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**From Process Signatures to Structural
Performance:
Mechanical Characterization of Additively
Manufactured Polymers and Architected
Structures**

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LIST OF ABBREVIATION

Abbreviation	Explanation
AM	Additive Manufacturing
MEX	Material Extrusion
FDM	Fused Deposition Modelling
FFF	Fused Filament Fabrication
IP	Infill Percentage
EM	Extrusion multiplier
DLP	Digital Light Processing
SLA	Stereolithography
MJF	Multijet Fusion
SLS	Selective Laser Sintering
BAAM	Big Area Additive Manufacturing
AWJ	Abrasive waterjet
ROI	Region of Interest
DfAM	Design for Additive Manufacturing
TPMS	Triply Periodic Minimal Surfaces
MJF	Multijet Fusion
FCC	Faced – Centered – Cubic
BCC	Body-Centered Cubic
ESMS	Energy storage multifunctional sandwich structures
DIA	Diamond
PLA	Polylactic Acid
PAEK	Polyarylether ketone
PEEK	Ppoly-ether-eter ketone
PEI	Polieterimide

PA6 CF	Carbon fibers reinforced Polyamide 6
TPU	Thermoplastic polyurethane
SEM	Scanning Electron Microscope
DCB	Double Cantilever Beam
BT	Beam Theory
MBT	Modified Beam Theory
NDT	Non Destructive Techniques
IRT	Infra – Red Termography
μCT	Micro – Computed Tomography
DSC	Differential Scanning Calorimetry
DMA	Dynamic Mechanical Analysis
SEM	Scanning Electron Microscope

LIST OF SYMBOLS

Symbol	Explanation
G_{IC}	Critical energy release rate [J/m^2]
P	Applied load [N]
ΔH	Specific Enatalpy [J/g]
T_g	Glass Transition Temperature [$^{\circ}C$]
$\chi(\%)$	Degree of crystallinity [-]
E	Young's Modulus [GPa]
σ	Engineering Stress [MPa]
σ_u	Tensile strength [MPa]
ε	Engineering Strain [-]
ε_f	Elongation strain at break [-]
t_l	Time required to complete a layer [s]
F_r	Flow rate [m^3/s]
M	Maxwell Number [-]
$\eta(\varepsilon)$	Absorption Efficiency [-]
ε_d	Densification Strain [-]
SEA	Specific Energy Absorption [J/g]
k	Spring constant [N/mm]

ABSTRACT

Additive Manufacturing (AM) of polymers is increasingly adopted for functional components and tooling in industrial sectors where lightweight design, geometric freedom and rapid customization are required. However, the mechanical reliability of additively manufactured polymers is strongly influenced by process-induced features such as anisotropy, interlayer bonding, thermal history and microstructural heterogeneity, which are not adequately captured by conventional material models.

This doctoral thesis investigates the process–structure–property relationships of polymer materials manufactured by Material Extrusion (FDM/HT-FDM) and Multi Jet Fusion (MJF), through an integrated experimental framework combining mechanical testing, thermal analysis and microstructural characterization. The study addresses both fundamental aspects of additive manufacturing and application-driven industrial scenarios, ranging from qualification of aerospace-grade polymers to the design of energy-absorbing lattice structures.

The work first establishes and validates advanced mechanical characterization methodologies using PLA as a reference material, demonstrating the dominant role of thermal conditions and interlayer adhesion in governing tensile performance and fractured behavior. These methodologies are then extended to high-performance PAEK materials, with a focus on PEEK processed by high-temperature FDM. Tensile, compressive and thermo-mechanical analyses are combined with DSC and DMA to quantify the influence of crystallinity, annealing and temperature-dependent behavior, with specific relevance to high-temperature tooling applications.

Finally, the compressive behavior of architected lattice structures manufactured by FDM (PA6-CF) and MJF (TPU) is systematically investigated. The results highlight topology-dependent deformation mechanisms, process-induced anisotropy and strain-rate effects, and are interpreted within an extended Gibson–Ashby framework. For TPU lattices, cyclic and rate-dependent tests enable the identification of optimized configurations for impact and energy absorption applications.

Overall, this thesis provides a comprehensive experimental basis for understanding and qualifying additively manufactured polymer materials, emphasizing the importance of process-aware mechanical characterization to support reliable industrial adoption of AM technologies.

THESIS OVERVIEW AND OBJECTIVES

The widespread adoption of Additive Manufacturing (AM) for polymer components has highlighted the need for advanced and process-aware characterization methodologies capable of capturing the intrinsic complexity of additively manufactured materials. Unlike conventionally processed polymers, AM components exhibit strong dependencies on deposition strategy, thermal history, build orientation and microstructural heterogeneity, leading to mechanical responses that cannot be reliably predicted using standard material data alone.

Within this context, the overarching objective of this doctoral thesis is to develop, validate and apply an integrated experimental framework for the mechanical and thermo-mechanical characterization of additively manufactured polymers, with direct relevance to industrial qualification and design.

The thesis is structured to progressively build from methodological foundations toward application-driven case studies.

1. Literature review provides a critical review of the state of the art in polymer Additive Manufacturing, with particular emphasis on Material Extrusion and powder-based technologies, focusing on interlayer adhesion, anisotropy, lattice mechanics and thermo-mechanical behavior.

2. Materials and Methods details the experimental methodologies adopted throughout the work, including additive manufacturing processes, mechanical testing protocols, microscopy techniques and thermal analyses, establishing a consistent and reproducible characterization framework.

3. Results chapter is organized into three main thematic blocks:

- **3.1 and 3.2** focus on PLA as a reference material to validate the experimental methodology. Tensile testing, thermal monitoring and microstructural analyses are combined to identify the dominant mechanisms governing interlayer bonding, fracture behavior and process-induced variability. This section serves as a methodological benchmark for subsequent studies.
- **3.3** extends the validated approach to aerospace-grade materials, using PAEK as a case study. Tensile behavior, microstructural features and thermal transitions are analyzed to support material qualification under industrial constraints. The influence of annealing and crystallinity is explicitly addressed to link processing conditions with mechanical performance.
- **3.4** is dedicated to the compressive characterization of architected lattice structures manufactured by FDM and MJF. PEEK and PA6-CF lattices are investigated to assess topology, density and anisotropy effects, while TPU lattices produced by MJF are analyzed for strain-rate sensitivity, cyclic behavior and energy absorption capability. These results support the selection and optimization of lattice designs for functional applications, including protective and impact-absorbing components.

4. Discussion chapter integrates the findings across materials, processes and length scales, highlighting common mechanisms and key differences between amorphous, semicrystalline and composite systems. Attention is devoted to the limitations of conventional modelling approaches and to the role of advanced experimental characterization in supporting numerical homogenization and multi-scale analyses.

Overall, this thesis aims to provide methodological tools and experimental evidence to support the reliable industrial adoption of polymer Additive Manufacturing, linking process conditions to material behavior and engineering design.

The investigation of multiple materials and additive manufacturing technologies reflects the diverse roles that AM currently plays across industrial sectors. In service-oriented companies, AM is widely used for rapid production and customer-specific components. In contrast, aerospace industries approach AM as a strategic solution for lightweight design, which requires stringent mechanical qualification of high-performance and costly polymers. Another emerging application concerns the use of AM for tooling, such as molds for composite curing, where the main objective is to reduce manufacturing costs, material waste, and energy consumption. These heterogeneous scenarios highlight the need for a rigorous and process-aware characterization framework, capable of supporting both material validation and design-driven industrial decision making.

1. LITERATURE REVIEW

Additive Manufacturing (AM) has progressed rapidly over the past three decades, evolving from a prototyping tool into a technologically and economically viable manufacturing route for functional components. This transition has been driven by fundamental advantages of AM, including geometric complexity, integrated functional design, and the capacity to produce customized or lightweight components without the constraints of subtractive or formative processes. Early analyses of AM adoption emphasized the paradigm shift from traditional manufacturing toward digitally driven, layer-wise material deposition [1]. Comprehensive overviews of AM technologies highlight similar trends, with polymer-based processes in particular experiencing widespread industrial uptake due to their accessibility and diverse material portfolio [2]. As AM continues to mature, its potential in end-use production, rapid tooling, and functional prototyping has begun to reshape engineering workflows and product development strategies across sectors spanning aerospace [3], healthcare [4], consumer products [5], and sports equipment [6].

Due to the broad material and technological diversity, AM now impacts a wide range of industries, as illustrated in Fig. I. 1.

Additive Manufacturing Usage by Industrial Sectors

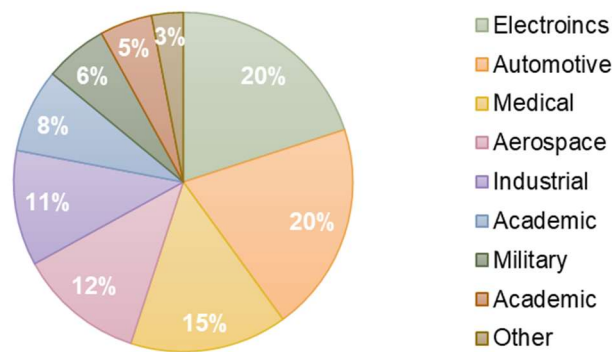


Fig. I. 1: Additive manufacturing usage by industrial sector [7]

Despite these developments, significant barriers remain in predicting and ensuring the mechanical performance of polymer AM components. Compared with conventionally manufactured polymers, additively manufactured materials exhibit distinct heterogeneities arising from the layer-by-layer deposition mechanism, thermal gradients, and incomplete fusion at interlayer boundaries.

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mechanism, thermal gradients, and incomplete fusion at interlayer boundaries. These challenges are strongly influenced by the type of AM technology used, as different polymer AM processes rely on radically different material consolidation mechanisms. For this reason, before focusing specifically on FDM, the central technology of this thesis it is necessary to introduce the main polymer AM families, their capabilities, and their limitations.

1.1 Polymers and Additive Manufacturing Technologies

Polymer AM encompasses multiple technological platforms, each with different processes, physics, material requirements, geometric constraints, and mechanical-performance implications. Among these, vat photopolymerization (SLA/DLP), powder-bed fusion (SLS&MJF), and material extrusion (FDM/FFF) are the most established and industrially relevant. Understanding their differences is essential for contextualizing why FDM is uniquely suited for printing engineering and high-performance thermoplastics such as PEI or PEEK.

1.1.1 Vat Photopolymerization: SLA and DLP

Vat photopolymerization technologies, including stereolithography (SLA) and digital light processing (DLP) rely on spatially controlled curing of liquid photopolymers using ultraviolet (UV) or visible light. SLA solidifies resin point-by-point through a scanning laser, while DLP cures entire layers simultaneously via projected light patterns as depicted in Fig. I. 2.

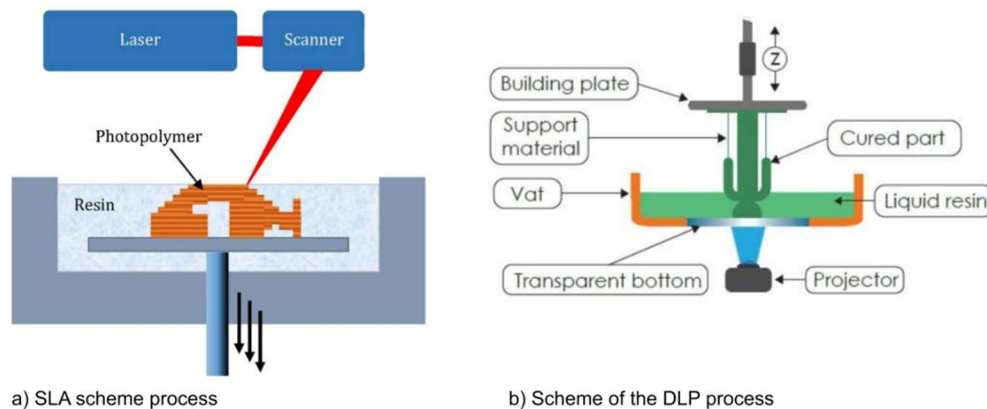


Fig. I. 2: Process schematic of vat photopolymerization technologies (SLA/DLP), illustrating: a) layer-wise curing of liquid photopolymers using laser scanning (SLA) [8]; b) projected light patterns (DLP) [9]

These methods offer exceptionally high resolution (25–100 μm) and smooth surfaces, enabling fabrication of intricate geometries, microfluidic structures, dental devices, and precision components. Typical resin families include:

- **Epoxy-based and acrylate photopolymers** constitute the backbone of most commercial SLA/DLP resins. Epoxy-based resins typically achieve higher stiffness, lower shrinkage, and improved dimensional stability, whereas acrylates exhibit faster polymerisation kinetics and lower viscosity, enabling high-speed DLP printing. Recent formulations demonstrate tensile strengths in the range

of 50–80 MPa, but they remain brittle (elongation < 10%) and have moderate thermal resistance ($T_g \approx 50\text{--}80\text{ }^\circ\text{C}$) [10,11]. Applications include microfluidic chips, housings, connectors, aesthetic parts, dental models, and hearing aids.

- **Toughened resins** incorporate urethane dimethacrylate (UDMA), rubber-modified domains, or interpenetrating networks to address the intrinsic brittleness of photopolymers. Mechanical performance improves significantly—tensile strain can reach 50–120%, with impact strength comparable to injection-moulded ABS—yet T_g values usually remain below 70–90 $^\circ\text{C}$. These materials are widely used for functional prototypes, low-load mechanical assemblies, and snap-fit components [12].
- **Elastomeric resins:** Elastomeric SLA resins such as polyurethane acrylates or thiol-ene systems achieve Shore A hardness from 30 to 90 and elastic strains >150%, although tear strength is typically lower than that of thermoplastic elastomers. Their thermal stability is limited (T_g near room temperature), making them unsuitable for sustained heating. Applications include soft robotics, grippers, shock absorbers, conformal padding, and biomimetic lattices [13,14].
- **High-temperature resins** can reach heat deflection temperatures (HDT) of 180–240 $^\circ\text{C}$ when fully post-cured, thanks to highly crosslinked aromatic monomers. However, despite good thermal resistance, they remain brittle and generally unsuitable for cyclic loading or impact. Their long post-curing cycles (UV + thermal) limit productivity [15,16]. Applications include moulds for low-pressure processes, test fixtures, fluidic connectors, and electronic housings.
- **Ceramic-filled resins** (e.g., silica, alumina, zirconia suspensions) allow production of dense ceramic parts through post-processing steps involving debinding and sintering. Mechanical properties after sintering can approach those of traditionally manufactured ceramics (e.g., compressive strength > 300 MPa), but shrinkage during sintering requires compensation and introduces defects. These materials exhibit excellent thermal stability and are used for investment casting, dental crowns, heat-resistant components. [17].
- **ISO-certified biocompatible resins (ISO 10993, USP Class VI):** these resins are engineered to meet cytotoxicity, irritation, and sensitisation requirements for medical use. Mechanical performance varies widely—dental splints require stiffness, whereas surgical guides require toughness and translucency. Thermal properties remain modest, with T_g typically 50–80 $^\circ\text{C}$. Applications include orthodontic models, surgical guides, dental splints, audiology devices, and patient-specific anatomical models [13,18].

Despite these options, photopolymers share inherent drawbacks due to their molecular structure: they undergo continued crosslinking, are susceptible to UV-induced degradation, show reduced fracture toughness, and possess poor long-term thermal stability. These issues limit their use in structural applications despite their micro-scale precision. Reviews consistently highlight these material limitations as the key barrier preventing SLA/DLP components from entering demanding industrial sectors such as aerospace or high-performance tooling [19].

Tab I. 1: Overview of the main SLA/DLP photopolymer resin families, summarizing their typical mechanical performance, thermal stability, curing or post-curing requirements, and representative application field

Resin Category	Mechanical Performance	Thermal Stability	Curing/Post-Cure Behavior	Typical Applications	
Epoxy/Acrylate systems	High stiffness, brittle; 50–80 MPa	T _g 50–80 °C	Fast curing; post-cure increases brittleness	Microfluidics, casings, aesthetic parts	[10,11]
Toughened resins	High elongation 50–120%	T _g 70–90 °C	Moderate curing time	Functional prototypes, snap fits	[12]
Elastomeric resins	Highly elastic; moderate tear strength	T _g ~RT	Rapid curing	Soft robotics, shock absorbers	[13,14]
High-temperature resins	Very stiff, brittle	HDT 180–240 °C	Requires thermal post-cure	Moulds, fixtures	[15,16]
Ceramic-filled resins	Ceramic-like after sintering	>500 °C	Multi-step curing/sintering	Dental ceramics, heat-resistant parts	[17]
Biocompatible resins	Application-specific	T _g 50–80 °C	UV + thermal curing	Dental, surgical tools	[13,18]

1.1.2 Powder-Bed Fusion (PBF) of Polymers: Selective Laser Sintering (SLS)

Selective Laser Sintering (SLS) is one of the most established polymer additive manufacturing technologies and remains the dominant industrial process for producing mechanically functional polymer parts. Unlike vat photopolymerization or material extrusion, SLS fabricates components by selectively fusing thermoplastic powders using a scanning infrared laser within a heated powder bed. A schematic of the process is shown in Fig. I. 3, illustrating the laser scanning, powder spreading operation, and the surrounding self-supporting powder environment.

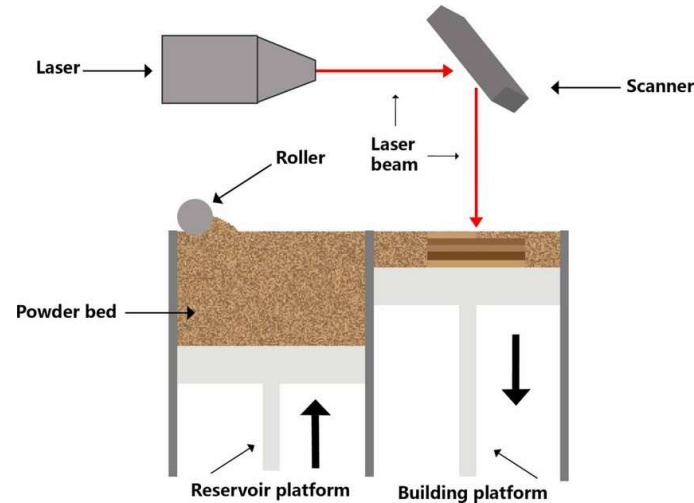


Fig. I. 3: : Schematic representation of the selective laser sintering (SLS) process, highlighting laser exposure, powder recoating, and self-support conditions [20]

SLS relies on heating the polymer powder bed to just below its melting temperature ($T_{bed} \approx 0.8\text{--}0.95 T_m$) so that only the laser provides the additional energy required to induce melting, coalescence, and inter-particle neck growth. This approach minimizes thermal gradients compared to FDM and allows the production of geometrically complex, support-free components. Typical laser sources include CO₂ lasers (10.6 μm) for polyamide powders and diode lasers for specific engineered polymer blends. The powder-bed environment enables uniform thermal exposure, leading to lower anisotropy than extrusion-based processes, although mechanical differences between build directions still exist due to layer-wise consolidation and partial sintering [21].

Numerical and experimental studies show that the primary microstructural drivers of SLS strength include particle size distribution, powder crystallinity, energy density, and recoating quality, all of which govern density and inter-particle bonding [22].

Unlike FDM, which can process a broad range of thermoplastics, SLS is constrained by the need for fine powders with suitable flowability, thermal stability, and predictable sintering behavior. As a result, its material portfolio is dominated by polyamide-based powders, though new developments are expanding this landscape.

PA12 offers an excellent balance of mechanical robustness, toughness, low moisture absorption, and high dimensional stability. Typical properties include tensile strength of 48–55 MPa, elongation at break of 10–20%, and usable operating temperatures around 90–110 °C. Its favorable sintering window ($T_m \approx 178$ °C) and stable crystallinity make it the most widely adopted commercial SLS polymer, used in aerospace ducting, housings, impact-resistant covers, gears, and medical-grade components [23].

PA11, derived from castor oil, offers higher ductility (elongation 30–50%), improved impact strength, and better performance under fatigue and dynamic loading. Its superior toughness makes it preferred for snap-fit components, orthotic devices, and flexible industrial parts. However, its narrower sintering window increases process sensitivity compared with PA12 [24].

TPU (Thermoplastic Polyurethane): SLS TPU powders enable production of elastomeric, energy-absorbing structures with Shore hardness ranging from 70A to 95A. These materials exhibit high resilience, excellent fatigue resistance, and strain levels >300%. They are widely used for sports padding, footwear midsoles, lattice structures, seals, and impact-absorbing components [25].

Carbon- or glass-filled versions of PA12 achieve increased stiffness (E up to 3–4 GPa) and improved dimensional precision due to reduced shrinkage. These are widely used for automotive brackets, robotic housings, and industrial jigs, though at the expense of reduced elongation at break [26].

Emerging High-Performance SLS Polymers (PEKK, PPS, PEEK Powders): Research efforts are underway to adapt high-performance polymers such as **PEKK, PPS, and PEEK** to powder-bed fusion, specifically for aerospace and high-temperature applications. However, these powders remain expensive, difficult to process due to narrow sintering windows, and susceptible to incomplete densification. Their mechanical properties do not yet match injection-molded equivalents without significant post-processing [27].

Tab I. 2: Comparison of Mechanical and Thermal Properties of typical SLS Materials

Material	Tensile Strength	Elongation	Thermal Performance	Application Domains
PA12	48–55 MPa	10–20%	~100 °C	Aerospace ducts, housings, machinery [23]
PA11	40–50 MPa	30–50%	~100 °C	Medical devices, flexible components [24]
TPU	5–10 MPa	>300%	~70–90 °C	Footwear, padding, lattice absorbers [25]
CF-PA12	70–90 MPa	2–5%	~100 °C	Structural brackets, stiff housings [26]
PEKK/PEEK	80–100+ MPa	3–10%	200–260 °C	High-temperature aerospace [27]

SLS excels when toughness, accuracy, internal lattice complexity, and support-free fabrication are required. However, it is intrinsically limited by:

- Residual porosity (5–10%), which reduces tensile strength
- Surface roughness ($R_a \approx 10$ –25 μm)

- Restricted material availability, largely polyamide-based
- Lower thermal and chemical resistance compared with PEEK/PEI via FDM

These limitations make SLS unsuitable for applications requiring high thermal stability, semi-crystalline control, or superior interlayer strength, which are central topics of your thesis. Indeed, SLS is widely used for environmental control system ducts, interior fittings, avionics housing, and custom brackets, where weight reduction and rapid manufacturability are essential. Furthermore, TPU SLS lattices are now standard in high-performance footwear, protective gear, and energy-absorbing structures due to their excellent resilience and form-fitting geometry [25]. Biocompatible PA11 SLS parts are used for orthoses, prosthetic sockets, dental devices, and custom implants, where toughness and geometric complexity are required. CF-PA12 and GF-PA12 components are used for under-the-hood brackets, robotic components, and jigs/fixtures, leveraging their stiffness and dimensional stability.

1.1.3 Powder-Bed Fusion of Polymers: Multi Jet Fusion (MJF)

Multi Jet Fusion (MJF) is a polymer powder-bed fusion technology developed to address productivity, scalability, and part uniformity limitations associated with laser-based sintering processes. In MJF, a thin layer of thermoplastic powder is selectively patterned using inkjet-deposited liquid agents, followed by uniform infrared energy exposure that induces localized melting and consolidation. A schematic representation of the MJF process is shown in Fig. I. 4, highlighting the deposition of fusing and detailing agents and the subsequent thermal consolidation step [28].

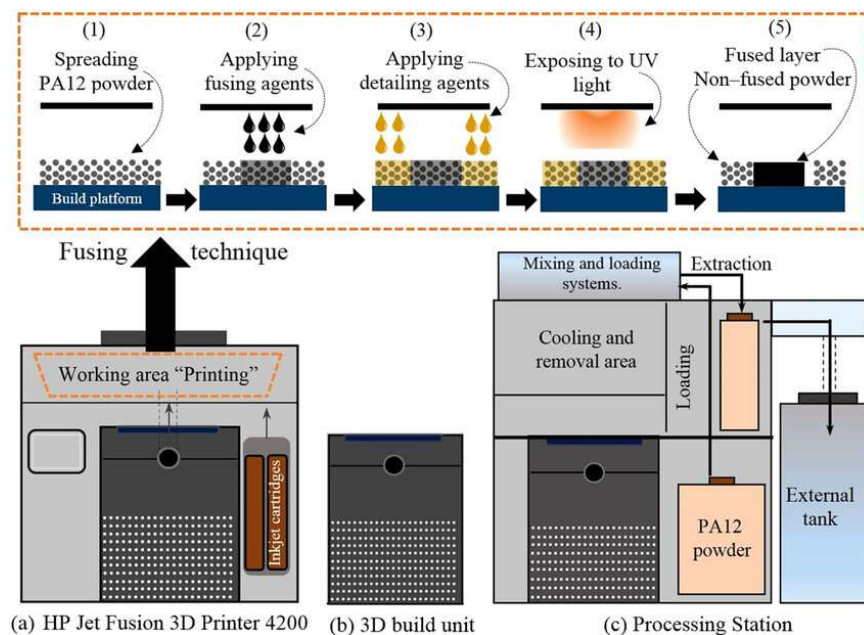


Fig. I. 4: Schematic overview of the Multi Jet Fusion (MJF) process and system architecture. (a) Layer-by-layer fabrication sequence showing powder spreading, selective deposition of fusing and detailing agents, infrared exposure, and formation of fused and unfused regions. (b) HP Jet Fusion 3D Printer 4200 build unit, illustrating the printing area and inkjet agent delivery system. (c) Processing station for powder mixing, cooling, depowdering, and material recycling [28].

Unlike SLS, where energy is delivered locally by a scanning laser, MJF employs area-wise thermal exposure, enabling faster layer processing and improved build rates. The surrounding powder bed provides self-support during fabrication, allowing the production of complex geometries without additional support structures. The consolidation mechanism in MJF results from the combined effect of agent distribution, infrared absorption, and thermal diffusion, which together govern part density and mechanical uniformity. The material portfolio of MJF is currently dominated by polyamide-based powders, particularly PA12, which exhibits favorable flowability, thermal stability, and reproducible melting behavior under MJF processing conditions. Typical mechanical properties of MJF PA12 parts include tensile strengths in the range of 45–55 MPa and elongation at break of 10–20%, comparable to SLS-produced PA12, with reports of reduced property anisotropy due to the more homogeneous thermal exposure [29].

Additional materials such as PA11, glass-filled PA12, and TPU have also been explored, though their industrial adoption remains more limited. From an application standpoint, MJF has gained significant traction in the production of functional polymer components for consumer goods, automotive interiors, housing, enclosures, and customized low- to medium-volume series production. The high build speed and repeatability of MJF make it particularly attractive for production-oriented workflows, including rapid spare-part manufacturing and mass customization scenarios. However, like SLS, the range of available polymers is largely restricted to polyamides and elastomers, and the process does not readily support high-temperature engineering thermoplastics.

As a result, while MJF represents an important and increasingly industrialized polymer PBF technology. However, its material limitations constrain its applicability for the investigation of high-performance thermoplastics. Consequently, MJF is not further explored experimentally in this thesis, which focuses instead on material extrusion processes capable of processing engineering and high-performance polymers such as PEI and PEEK.

1.1.4 Material Extrusion of Polymers: Fused Deposition Modelling

While polymer powder-bed fusion technologies such as SLS and MJF enable support-free fabrication and are widely applied to polyamide-based materials, their polymer portfolios remain comparatively constrained with respect to high-temperature engineering thermoplastics. In contrast, material extrusion technologies most notably Fused Deposition Modelling (FDM), also referred to as Material Extrusion or Fused Filament Fabrication (FFF) provide a direct route for processing a broad range of thermoplastic filaments, including engineering and high-performance polymers. For this reason, FDM constitutes the central polymer AM technology investigated in this thesis. FDM has become one of the most widely adopted polymer additive manufacturing technologies due to its accessibility, versatility, and steadily increasing performance. The process is based on extrusion of a thermoplastic filament through a heated nozzle, followed by deposition of the semi-molten polymer along prescribed tool paths to build a three-dimensional object in a layer-by-layer fashion. A schematic representation of the FDM process proposed by Vaes et al. is schematically illustrated in Fig. I. 5 [30].

Although initially employed primarily for rapid prototyping, the technological evolution of FDM over the past decade has substantially expanded its capabilities and positioned it as a viable method for manufacturing functional components. Improvements in hardware including actively heated build chambers, advanced nozzle designs, closed-loop motion control, and multi-material printheads have contributed to gains in dimensional accuracy, interlayer adhesion, and process repeatability.

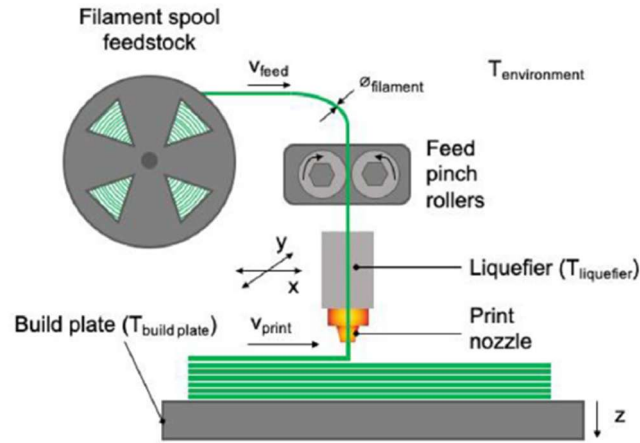


Fig. I. 5: Schematic representation of the Fused Deposition Modelling (FDM) process [30].

From a process-physics standpoint, studies have shown that precise control of deposition temperature, chamber heating, and raster strategy strongly influences filament bonding and final part strength, as interlayer consolidation depends on polymer diffusion and neck growth during and after deposition [31].

The selection of materials employed in Fused Deposition Modelling (FDM) spans a wide spectrum of thermoplastic polymers, ranging from low-cost commodity plastics to advanced engineering and high-performance materials. Their selection is governed by a combination of processing requirements, mechanical performance, thermal stability, chemical resistance, and cost–performance trade-offs. A useful framework for understanding the positioning of FDM materials is illustrated in Figure X, which classifies thermoplastics according to performance level, application domain, and permissible temperature of use, as depicted in Fig. I. 6 [32]. At the lower end of the performance spectrum, commodity polymers such as PLA, ABS, and PETG dominate low-cost and non-critical applications due to their ease of processing, low warpage tendency, and suitability for open-environment printing. These materials provide adequate stiffness and strength for conceptual prototyping, consumer products, and simple engineering geometries, though they typically exhibit limited thermal stability and reduced resistance to environmental degradation [33].

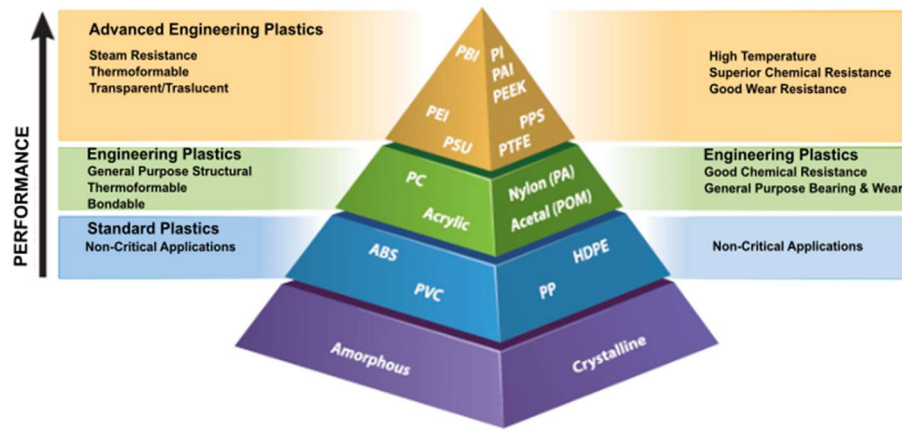


Fig. I. 6: Classification of thermoplastic materials by performance level, cost, and temperature of use [32]

As the technology matured, the materials palette expanded significantly toward engineering thermoplastics, including polycarbonate (PC), polyamide (PA), thermoplastic polyesters, and acetal (POM). These materials offer improved toughness, wear resistance, and thermal stability, enabling use in mechanical housings, fixtures, automotive components, and tribological applications. Multiple studies report that engineering polymers can achieve higher interlayer strength and better thermal-mechanical performance when processed under controlled temperature conditions, although they require enclosed heated chambers and tighter process control compared with commodity polymers [34]. Additionally, the emergence of fibre-reinforced filaments containing chopped or even continuous fibres—has expanded the performance window of this category. These composite materials exhibit enhanced stiffness-to-weight and strength-to-weight ratios, approaching or surpassing those of conventionally moulded engineering plastics [35].

The upper tier of the FDM materials landscape is composed of high-performance polymers, including the PAEK family (PEEK, PEKK), PSU, PPSU, PEI, and PBI. These thermoplastics exhibit outstanding thermal resistance, chemical inertness, fatigue strength, and dimensional stability, enabling use in aerospace, oil-and-gas, defense, and high-end biomedical applications. Their processing, however, remains challenging due to their high melt temperatures, narrow processing windows, and strong sensitivity to cooling rate and crystallization kinetics. Detailed reviews emphasize that high-performance polymers require highly controlled thermal environments—often involving chamber temperatures between 80–200 °C to ensure adequate interlayer fusion and to prevent excessive warpage [7]. These constraints also relate directly to the semi-crystalline nature of many high-performance polymers. For example, PEEK exhibits crystallization-driven changes in stiffness, fatigue behavior, and thermal response, making its final properties dependent on the exact thermal history during printing. Investigations into PEEK printed by material extrusion found that chamber temperature, nozzle temperature, and cooling profiles critically influence crystalline morphology and consequently mechanical performance [36]. The challenges associated with high-performance polymers extend beyond crystallization: environmental conditions during printing can strongly influence part density and mechanical reliability. Recent experimental evidence demonstrates that processing PEEK in vacuum or inert environments reduces oxidation and porosity, thereby improving tensile and interlaminar properties, particularly for long-term structural applications [37]. This reflects the broader trend that, as FDM moves upward along the performance pyramid, the sensitivity of polymer behavior to processing conditions

increases markedly. Taking together, the evolution of FDM materials demonstrates both the versatility of the process and the need for deeper mechanistic understanding, particularly as industrial users increasingly demand reliable, high-performance, application-specific thermoplastic components.

Polymer-based additive manufacturing has rapidly gained traction within the aerospace and aeronautics sectors due to its ability to reduce weight, shorten lead times, and enable on-demand production of customized or low-volume components. As illustrated in Fig. I. 7, airlines and aerospace OEMs increasingly employ AM for interior cabin components, environmental control system parts, and maintenance-replacement items. These applications leverage the geometric freedom of AM, allowing complex shapes, integrated features, and consolidation of multiple components into simplified monolithic parts [38]. Lightweight polymer ducts, air-conditioning fittings, and cabin ventilation grills printed from ULTEM 9085, or PEI have demonstrated significant reductions in production time while meeting flame, smoke, and toxicity (FST) requirements. Similarly, bracketry, cup holders, control-unit covers, and cosmetic interior elements can now be produced additively in small batches, reducing tooling costs and enabling efficient spare-part supply chains. The use of AM tooling—such as 3D-printed moulds for partition walls or composite lay-up—further highlights how high-performance thermoplastics and fibre-reinforced materials contribute to faster prototyping and structural repair operations. Across these applications, the key drivers remain weight reduction, certification-compliant materials, design flexibility, and rapid deployment, making polymer AM a strategic asset for commercial aviation, MRO (maintenance, repair, overhaul), and cabin customisation.

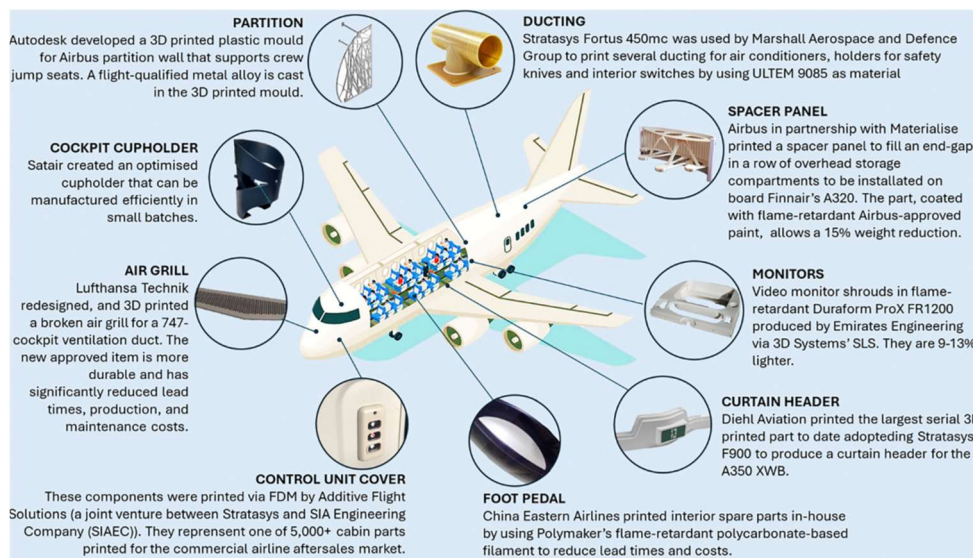


Fig. I. 7: AM-produced products used as components in aircraft[38]

1.1.5 Design for Additive Manufacturing and Topology Optimization

The increasing use of engineering and high-performance polymers in aerospace and industrial sectors has shifted additive manufacturing from a tool for prototyping toward a platform for fully optimized, application specific components. As materials such as PEI, PPSU, and PEEK demonstrate suitability for aircraft interior fittings, mechanical brackets, environmental ducting, and high-temperature tooling, the opportunity emerges not only to replicate existing geometries but to redesign them for superior performance. This shift requires

moving beyond conventional design practices and embracing methodologies tailored to AM's geometric freedom and process constraints. Within this context, Design for Additive Manufacturing (DfAM) has become a fundamental framework, enabling designers to exploit AM's capabilities in part consolidation, lightweighting, and functional integration while accounting for material behavior and process limitations [39]. Topology optimization is a central element of this approach, serving as the conceptual bridge between high-performance polymer applications and the geometric complexity achievable in AM. It also sets the stage for understanding how advanced designs interact with the defect mechanisms inherent to FDM, which are examined in the following sections. Topology optimization (TO) has become one of the most influential computational design tools in additive manufacturing, enabling engineers to identify optimal material layouts under structural, thermal, or multi-physics constraints. Unlike conventional design workflows, TO does not begin from a predefined geometry; instead, it determines the most efficient structural form within a prescribed design domain, often resulting in highly non-intuitive and organically shaped components. This makes TO particularly well aligned with AM, which can fabricate complex geometries without the restrictions of traditional subtractive or formative processes [40]. In polymer aerospace applications, TO has been successfully used to redesign brackets, seat-frame connectors, cabin interior supports, and electronic housings, achieving substantial weight reductions while preserving stiffness or improving load-carrying capacity. Several studies have demonstrated weight savings of 30–70% in AM-printed brackets and fittings when TO-derived geometries are combined with polymer AM techniques, particularly for non-critical or interior aerospace structures. The freedom to implement hollow regions, lattice-supported surfaces, and self-supporting shapes further enhances the structural efficiency achieved with polymers. This design paradigm is reinforced by DfAM methodologies, which emphasise exploiting AM's geometric freedom while managing constraints such as overhangs, anisotropy, and thermal-driven distortions [41]. Thus, effective use of TO requires iterative refinement of optimization outputs to satisfy AM manufacturability guidelines—adjusting overhang angles, thickening vulnerable regions, smoothing topology boundaries, or integrating support-friendly orientations. These considerations reflect broader industrial insights that highlight the need to integrate design, materials, and process constraints into a unified AM strategy [42].

As such, topology optimization represents not only a performance-enhancing design technique but also a context in which the limitations of FDM processes become most apparent. This interplay naturally leads to the discussion of defect formation mechanisms, process-induced anisotropy, and quality control strategies addressed in the following section. Fig. I. 8 presents representative applications of topology optimization, illustrating how computational material redistribution can achieve significant mass savings during the design stage (a). The figure also shows components manufactured from PEEK via FDM, highlighting its adoption in both aerospace structures (b) and patient-specific biomedical implants (c), where lightweight geometry and high-performance polymer properties are essential. The design evolution shown in Fig. I. 9 illustrates how topology optimization relies on iterative finite element analysis (FEA) to evaluate stress distributions and material efficiency within a predefined design space.

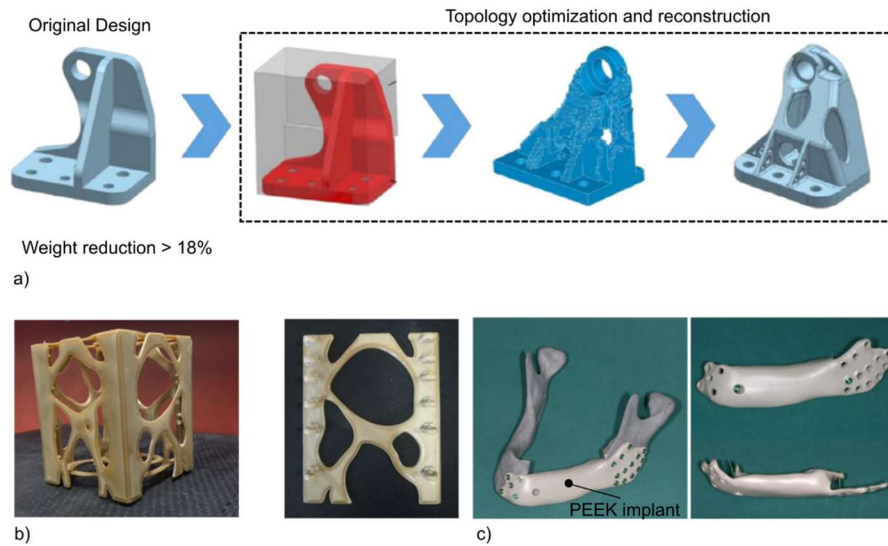


Fig. 1. 8: Examples of topology-optimized designs and reconstructed components (a) [41], alongside PEEK parts produced by FDM for aerospace applications (b) [43] and customized biomedical implants (c) [44]

In the GE aircraft engine bracket challenge [45], the optimization process repeatedly removes material from low-stress regions while reinforcing load paths identified through FEA, producing organic, weight-efficient geometries that remain structurally compliant under operational loading. The comparison between the initial topology-optimized forms (Fig. I.9 (c)) and the final printed brackets (Fig. I. 9 (e)) highlights how FEA-driven optimization enables substantial mass reduction while maintaining the required stiffness and strength.

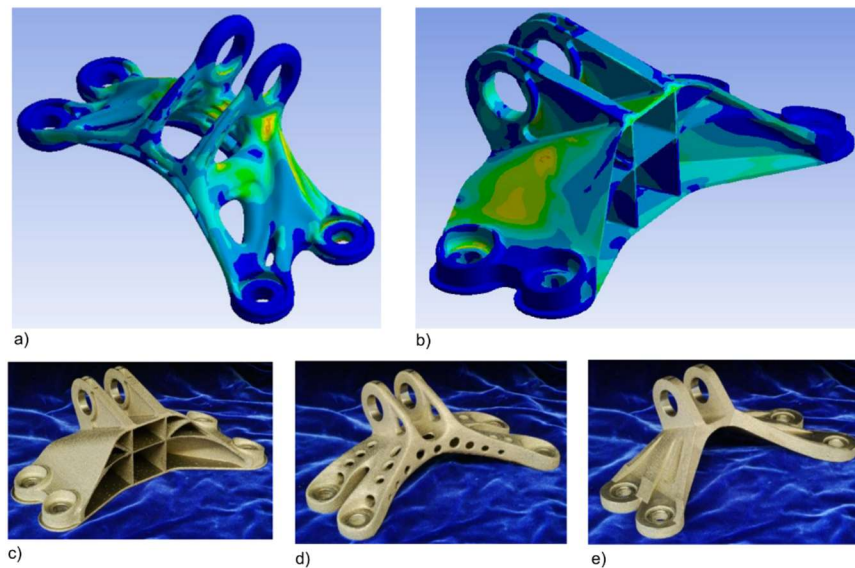


Fig. 1. 9: Examples of topology-optimized designs and corresponding manufactured components. Top row: results of topology optimization and finite element analysis (FEA), illustrating stress distribution and material redistribution within the design space (a). Bottom row: PEEK components produced by Fused Deposition Modelling (FDM), demonstrating the translation of optimized geometries into lightweight structural parts for aerospace applications(c,d,e) [45].

1.1.6 FDM: 3D printing of composite materials

Moreover, recent advances in reinforced filaments, including both short- and continuous-fiber composites, have significantly expanded the mechanical performance envelope of FDM. These developments enable stiffness and strength levels that can rival, or in some cases surpass, those of conventionally manufactured polymer components. A notable example is the work of Matsuzaki et al. [35], who demonstrated the feasibility of FDM using continuous carbon fiber–reinforced thermoplastic composites, achieving substantial improvements in load-bearing capability.

Building upon this concept, Fasel et al. employed continuous carbon fibers embedded within a PA12 matrix to fabricate a truss-based drone fuselage structure. Their work highlights the potential of continuous-fiber FDM for waste-free and cost-effective manufacturing of lightweight aerospace structures, particularly when combined with topology-optimized designs [46]. In such systems, FDM typically relies on a dual-nozzle configuration, where one nozzle extrudes the neat thermoplastic matrix while a second nozzle introduces the prepreg continuous fiber.

Further advancements were reported by Liu et al. [47], who developed a novel in-situ embedding approach in which continuous carbon fibers are directly impregnated within the molten polymer inside the nozzle. This method enables the fabrication of complex lattice and truss-core sandwich structures, such as CFR–PLA lattice cores, without the need for external support or post-processing steps, as illustrated in Fig. I. 10. These studies collectively demonstrate the strong synergy between continuous-fiber reinforcement, lattice-based design, and FDM, while also emphasizing the increased process complexity associated with fiber placement, bonding quality, and defect control.

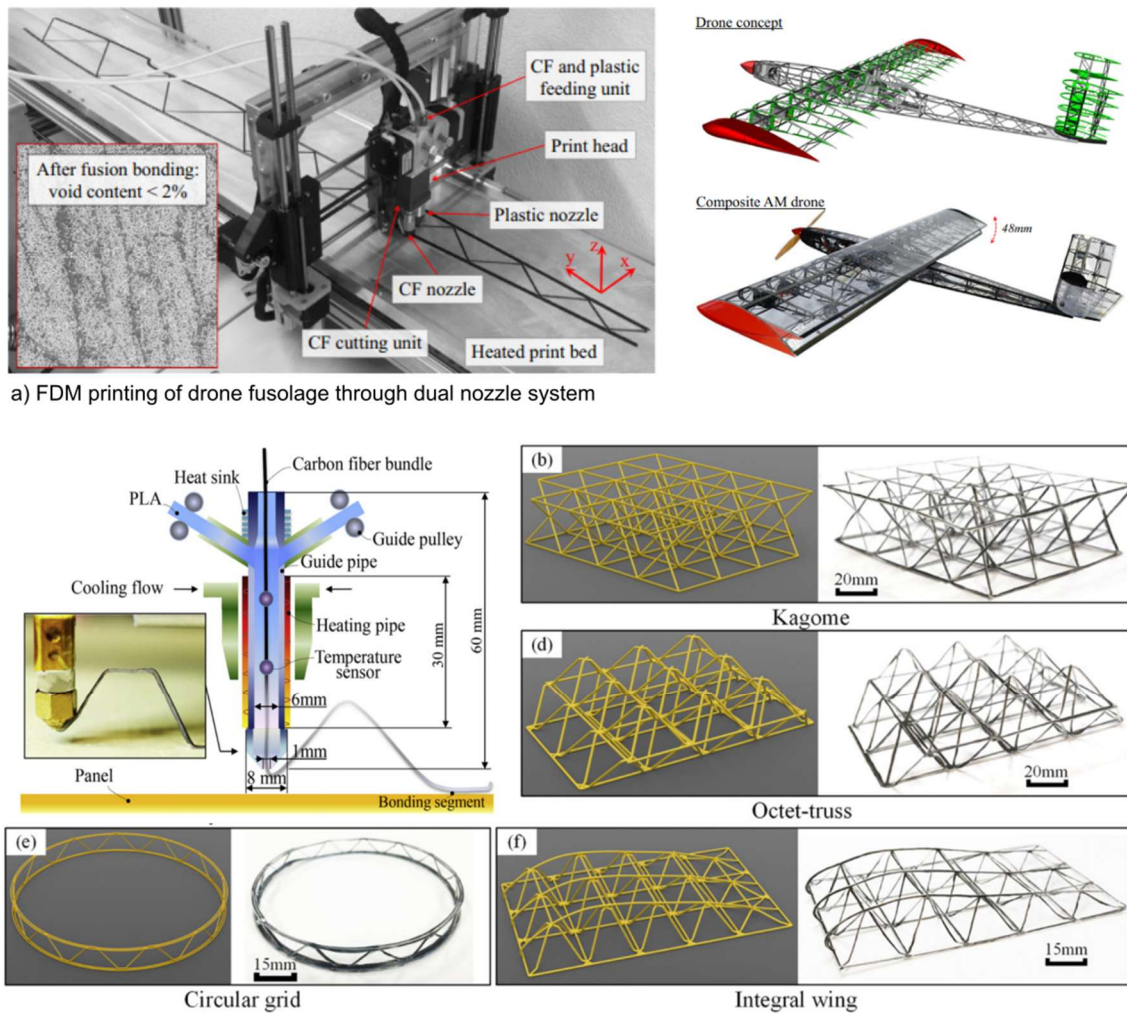


Fig. 1. 10: a) Examples of continuous carbon fiber–reinforced FDM systems and applications. Left: dual-nozzle FDM setup for in-situ deposition of thermoplastic filament and continuous carbon fiber. Right: topology-optimized truss-based drone structures fabricated via composite additive manufacturing [46].

b) Left: schematic of a nozzle system enabling in-nozzle impregnation and bonding of continuous carbon fiber within a molten polymer matrix. Right: examples of lattice and truss core geometries (Kagome, octet-truss, circular grid, and integral wing structures) manufactured using continuous fiber-reinforced FDM [47].

1.2 Defects in FDM technology

While these technological developments have transformed FDM into a robust and adaptable manufacturing platform, the process remains fundamentally limited by characteristic microstructural defects and heterogeneities. Among the most pervasive defects in FDM is the formation of interlayer voids, which arise from imperfect fusion between adjacent filaments or successive layers. The bond between extruded strands is governed by polymer chain interdiffusion and neck formation, both of which are highly sensitive to thermal history and deposition conditions. Insufficient heating or rapid cooling can inhibit diffusion and result in weak, partially fused interfaces that compromise mechanical properties, particularly in interlaminar tension or shear [48,49]. Microstructural analyses and CT imaging studies have shown that voids tend to align with the raster pattern, reinforcing directional anisotropy in stiffness and strength as first highlighted in foundational mechanical studies [50].

A second important defect category concerns geometric distortions and residual stress. As the extruded polymer cools, thermal contraction induces warpage, curling, and internal stresses, particularly in large or thin-walled components. Thermal gradients both within and between layers can produce cumulative distortions over the build, leading to dimensional inaccuracies and, in severe cases, interlayer delamination. Numerical and experimental analyses emphasize that many of these distortions originate from non-uniform temperature distributions and incomplete control of the cooling environment. Enhanced chamber heating and controlled cooling strategies can reduce these effects, yet they remain a fundamental challenge for high-precision applications.

Surface-related defects also represent a significant limitation of FDM. The stair-stepping effect, inherent to layer wise approximation of curved geometries, produces surface roughness that may influence mechanical performance, fluid flow, or aesthetic requirements depending on the application [51]. Additionally, inconsistencies in extrusion flow can create under-extrusion (leading to gaps and weak regions) or over-extrusion (leading to bead overlap and surface irregularities) [52]. These mesoscale defects are strongly linked to nozzle pressure, feed rate stability, filament diameter tolerance, and rheological behavior of the polymer melt, as detailed in reviews of FDM material behavior [53].

Several common microstructural defects introduced during material extrusion are illustrated in Fig. I. 11. Because many of these features are not visible to the naked eye, they are typically characterized using high-resolution SEM and μ CT imaging, as shown in Fig. I. 11(a–f). In Fig. I. 11 (a), voids located between adjacent rasters within the same layer and across consecutive layers are highlighted. Fig. INTR 2(b) illustrates under-extrusion, which results in discontinuities and insufficient material deposition, often associated with unstable flow conditions and nozzle pressure variations. Fig. I. 11(c) shows filament stirring, in which deposited material contributes to mass but does not effectively participate in load transfer, thereby reducing structural efficiency and motivating deeper understanding of process physics and defect formation mechanisms. Fig. I. 11(d) presents bubbles within the deposited filament, commonly associated with moisture in the feedstock material [54]; in this context, appropriate filament drying is typically required to ensure consistent deposition. Finally, Fig. I. 11(e) and (f) provide μ CT images of composite structures produced by FFF, revealing defects associated with fibre–matrix interactions. Large detachments between fibre and matrix

filaments can generate voids that propagate through the thickness, as shown in Fig. INTR 2(e) [55], while fibre waviness and misalignment during deposition—illustrated in Fig. INTR 2(f)—can significantly influence the mechanical response of printed composites [56,57].

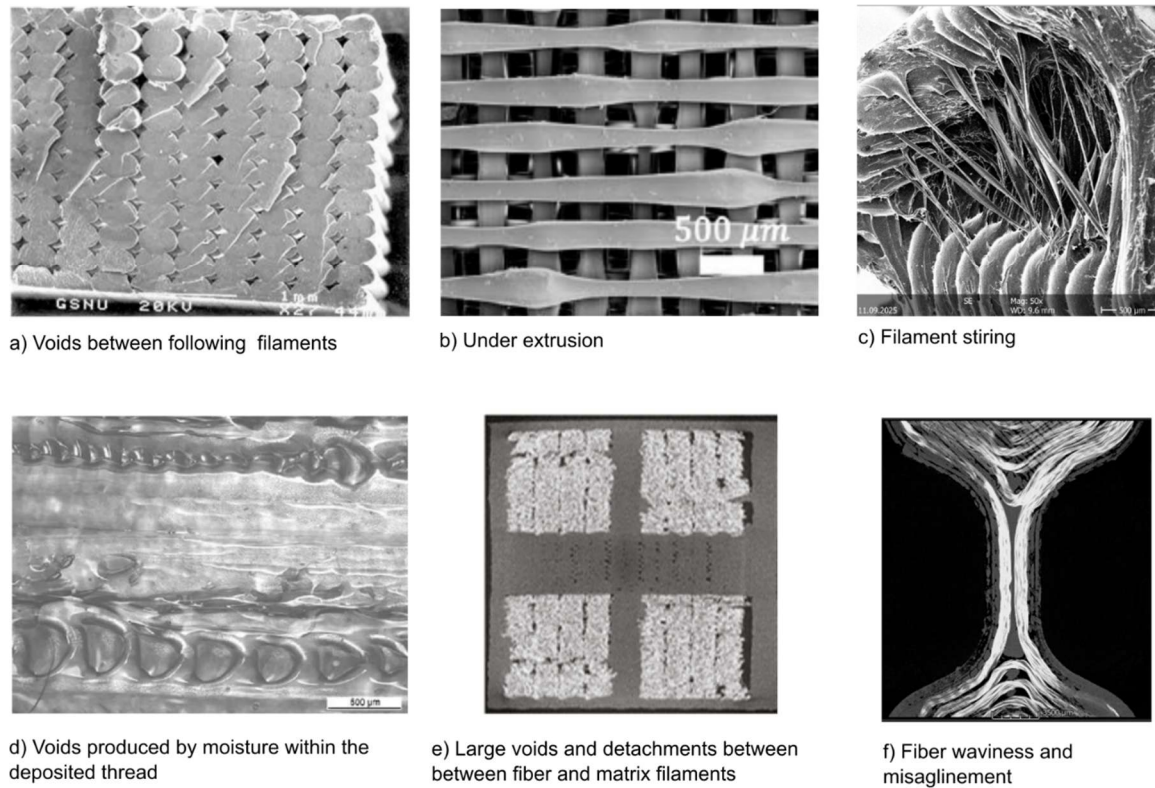


Fig. 11: Representative microstructural defects and heterogeneities in FDM components revealed by high-resolution SEM and μ CT imaging. (a) Inter- and intra-layer voids aligned with the raster pattern, arising from incomplete filament fusion [50]. (b) Under-extrusion leading to discontinuities and insufficient material deposition [52]. (c) Filament stirring, where deposited material contributes to mass but not effectively to load transfer. (d) Gas bubbles within extruded filaments, commonly associated with moisture in the feedstock material [54]. (e) μ CT image of a fibre-reinforced FFF structure showing fibre–matrix debonding and void propagation through the thickness [55]. (f) Fibre waviness and misalignment induced during deposition, which can significantly affect the mechanical response of printed composite components [56].

Beyond microstructural voids or bonding defects, FDM components frequently exhibit macroscopic, eye-visible defects that arise from the thermal and geometric characteristics of the process. One of the most common is warpage, where plate-like parts lift at the corners or edges due to non-uniform cooling and residual thermal stresses. Experimental and analytical studies show that cooling gradients between the hot inner core and colder outer layers induce bending moments that produce measurable curvature once the part is released from the build plate [58]. Warpage becomes more pronounced in large-format parts, where significant corner lifting and global distortion have been reported, especially when chamber and bed temperatures are insufficient to counteract shrinkage [59].

Another visible defect relates to edge quality, where contours appear wavy, thickened or rounded due to the discretization of edges into stepped layers, bead swelling, or inaccurate toolpath offsets. Detailed geometric analyses classify these defects, such as staircase effects, corner swelling, and support-induced artefacts—as

consequences of bead geometry, slicing resolution and local edge angles [60]. Reviews of dimensional accuracy confirm that warpage and edge deformation are among the dominant macroscopic imperfections in FDM, affecting both functional fit and aesthetic quality of printed parts [61]. Together, these visible defects underscore the impact of thermal management and toolpath strategy on the overall quality of FDM components.

To minimize both micro- and macro-scale defects in FDM parts, careful optimization of process parameters and printing strategy is essential. Voids and poor interlayer bonding can be mitigated by optimizing extrusion flow, bead geometry, infill strategy, and toolpath planning—approaches shown to reduce porosity and improve material continuity across layers [62]. Proper control of thermal conditions, especially build-plate (bed) temperature set slightly above the polymer's glass transition, significantly enhances adhesion of the first layers, reducing early-stage detachment, layer shifting, and warpage [63]. For larger or high-performance polymer parts (e.g., PEEK), warpage and distortion can be further reduced by optimising nozzle and platform temperature, layer thickness, and infill parameters, as demonstrated in recent multi-objective optimization studies [64]. In sum, these strategies tuning, thermal control, toolpath planning, and material specific optimization constitute a practical framework to reduce both visible and hidden defects in FDM components, thereby improving mechanical reliability and dimensional fidelity.

1.3 Mechanical Characterization and Structure–Property: Assessment of Polymer AM Components

The mechanical performance of additively manufactured polymer components is a central concern for their adoption in functional and load-bearing applications. Unlike conventionally processed polymers, additively manufactured materials exhibit pronounced anisotropy, process-induced heterogeneity, and architecture-dependent behavior, arising from the layer-wise deposition strategy, thermal gradients, and interfacial bonding mechanisms inherent to AM processes. These characteristics lead to strong dependencies of mechanical properties on build orientation, process parameters, and internal morphology, thereby necessitating careful and standardized mechanical characterization [65,66].

1.3.1 Standard Mechanical Characterization of AM Polymers

As illustrated in Fig. I. 12, mechanical characterization of AM polymers is typically performed using tensile, compressive, and flexural testing configurations, which are specifically selected to capture anisotropy, interlayer bonding quality, and geometry-dependent mechanical response. These established testing methodologies provide a consistent baseline for material comparison, process optimization, and design validation across different AM technologies and material classes [67,68].

Compression testing is particularly relevant for applications involving tooling, fixtures, and architected components, where instability phenomena, progressive plastic collapse, and densification mechanisms dominate the mechanical response. In these cases, compressive loading provides a more representative assessment of in-service behavior than tensile testing alone. Flexural testing further accentuates the sensitivity of AM components to interlayer bonding quality, as tensile and compressive stresses act simultaneously across layer interfaces, making this test especially effective for revealing weak interlaminar adhesion and premature failure modes [19].

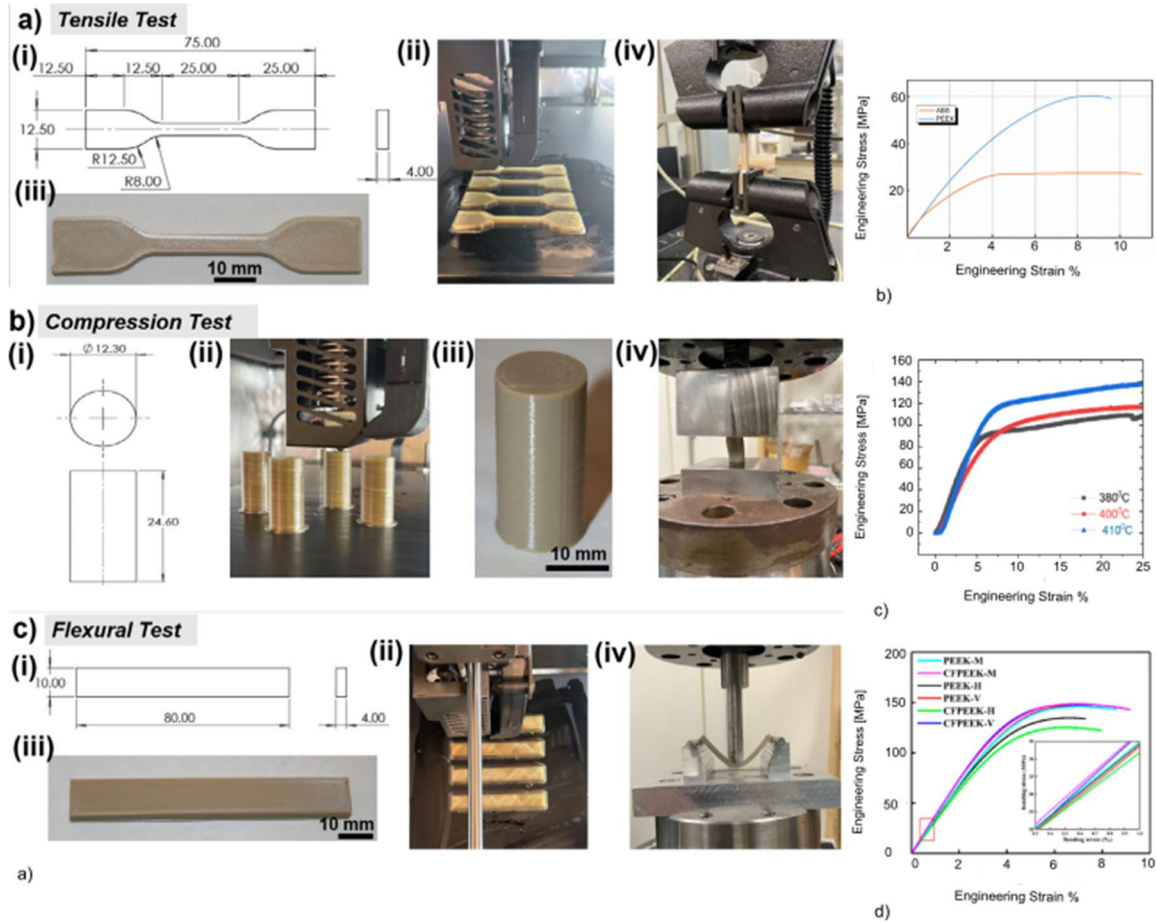


Fig. 1.12: Experimental configurations for the mechanical characterization of additively manufactured polymers: (a) tensile test, (b) compression test, and (c) flexural test. For each configuration, (i) specimen geometry, (ii–iii) additive manufacturing process and fabricated specimens, and (iv) test setup are shown. Representative mechanical responses are also reported. These tests are used to assess process-induced anisotropy, interlayer bonding quality, and geometry-dependent mechanical behavior [69].

1.3.2 Quasi-Static Testing: Fatigue, Creep, and Rate Effects

Beyond quasi-static loading conditions, increasing attention has been devoted to cyclic, fatigue, and rate-dependent mechanical behavior of polymer AM materials, reflecting the requirements of aerospace, tooling, and energy-absorbing applications. Fatigue studies consistently report reduced endurance limits compared with injection-molded counterparts, largely attributed to stress concentrations arising from internal voids, incomplete interlayer fusion, and surface roughness inherent to layer-wise fabrication processes [70]. Rate-dependent and viscoelastic effects also become increasingly important for thermoplastic AM materials, particularly under elevated temperatures or sustained loading. For high-performance polymers processed by FDM, such as PEEK and PEI, creep and stress relaxation are critical design considerations in tooling and structural applications where components are exposed to long-term thermo-mechanical loading [71].

1.3.3 Microstructural, Thermal and Non-Destructive Evaluation Approaches

Because the mechanical response of additively manufactured polymers is strongly governed by internal architecture and process-induced microstructural features, mechanical testing alone is insufficient to fully describe material behavior. Consequently, mechanical characterization is increasingly complemented by microstructural, thermal, and non-destructive techniques aimed at establishing reliable structure property relationships across multiple length scales.

Thermal analysis techniques, including differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), further contribute to understanding the mechanical behavior of AM polymers by quantifying crystalline evolution, glass-transition behavior, and viscoelastic response. This information is particularly relevant for semi-crystalline thermoplastics, such as PEEK and PEKK, whose stiffness, strength, and time-dependent behavior are strongly coupled to the thermal history experienced during layer deposition and cooling [36].

In parallel with ex-situ thermal analysis, non-destructive process-monitoring techniques (NDT) have gained increasing relevance for capturing the thermal history that governs interlayer bonding and phase development. Among these, infrared (IR) thermography has emerged as a powerful tool for monitoring the material extrusion process in real time. As shown in Fig. I. 13, IR imaging enables real-time mapping of surface temperature fields during deposition, capturing nozzle, part–bed interactions, cooling rates, and thermal gradients at the raster and layer scales. Such measurements provide indirect but critical insight into interlayer diffusion and healing conditions, which govern interfacial bonding strength in high-temperature polymers such as PEEK. Indeed, Fig. I. 13 a) shows the influence of temperature on the interaction between polymeric chains and its dependence with thermal history. Moreover, Fig. I. 13 b), and c) illustrates the temperature gradient during the MEX process and the time-dependent cooling behavior relevant to interfacial healing and bonding strength.

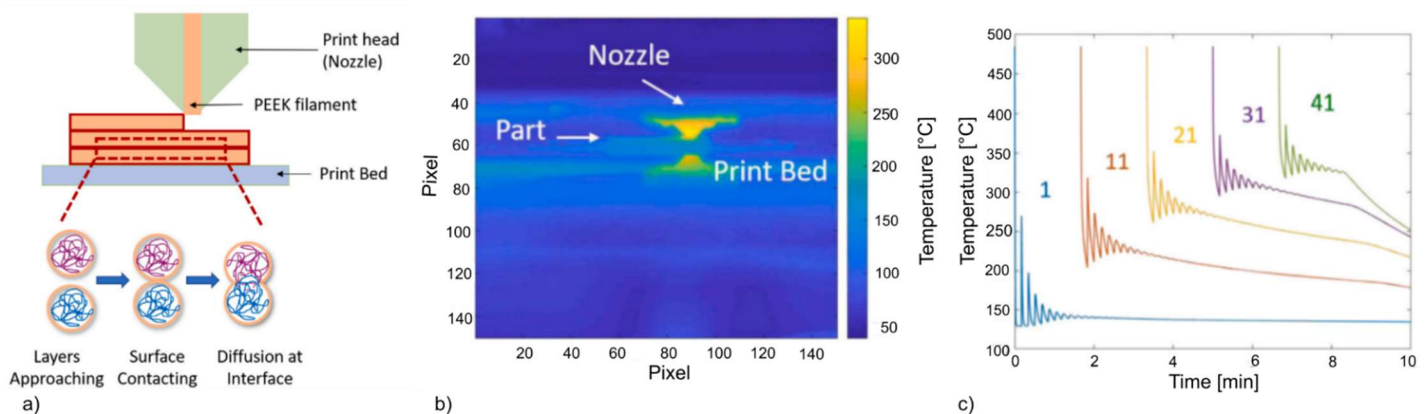


Fig. I. 13: Infrared (IR) thermography applied to the material extrusion (MEX/FDM) process for PEEK: (a) schematic illustration of interlayer contact, diffusion, and healing mechanisms as a function of thermal history during deposition; (b) IR thermal map showing surface temperature fields and nozzle–part–bed thermal interactions during printing; (c) representative time–temperature profiles highlighting layer-wise thermal cycling and cooling behavior. IR thermography

enables real-time, non-destructive monitoring of temperature gradients, cooling rates, and thermal histories that govern interlayer diffusion, healing conditions, and ultimately interfacial bonding strength in high-temperature semi-crystalline polymers [72].

While IR thermography primarily informs on process stability and thermal uniformity, volumetric defect assessment requires complementary techniques. In this context, X-ray computed tomography (μ CT) is widely regarded as the most comprehensive NDT method for polymer AM. μ CT enables three-dimensional quantification of porosity, void morphology, fiber orientation, and defect spatial distribution across multiple length scales, providing statistically robust descriptors of internal quality. Numerous studies report strong correlations between μ CT-derived defect metrics such as void volume fraction, shape anisotropy, and clustering and reductions in tensile strength, fatigue resistance, and interlaminar performance [42]. For example, X-ray CT has been used to quantify pore size, shape and spatial distribution in FDM PLA and to demonstrate a clear decrease in tensile stiffness with increasing porosity [73]. A representative example of this approach is shown in Fig. I. 14, where X-ray computed, tomography is used to visualize porosity size and spatial distribution in FDM specimens, and the resulting defect metrics are directly correlated with tensile stress–strain response under different raster configurations.

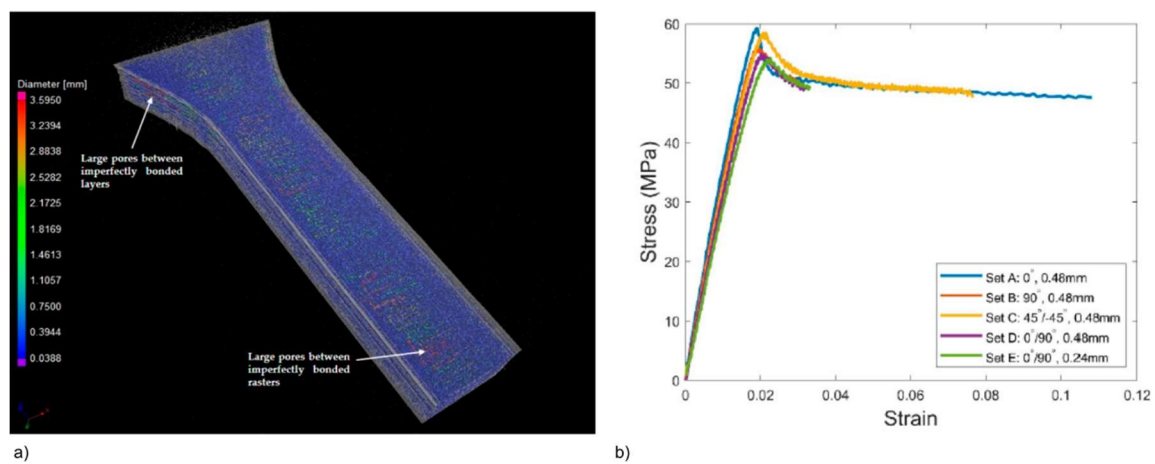


Fig. I. 14: X-ray computed tomography (μ CT)-based defect characterization of FDM polymers and its correlation with mechanical response: (a) three-dimensional μ CT reconstruction highlighting pore size, morphology, and spatial distribution, with large pores associated with imperfect interlayer and inter-raster bonding; (b) corresponding tensile stress–strain curves for specimens printed with different raster orientations and process parameters. μ CT enables quantitative assessment of internal porosity and defect architecture, providing direct links between processing-induced defects and reductions in stiffness, strength, and deformation behavior [73].

Taking together, these approaches highlight the necessity of an integrated, multi-scale characterization framework in polymer additive manufacturing, combining mechanical testing with thermal monitoring and defect analysis. This integrated methodology is particularly critical for FDM, where processing parameters directly control thermal history, interlayer bonding, crystallization behavior, and defect formation. Establishing robust links between processing conditions, internal structure, and mechanical response therefore represents a fundamental prerequisite for the reliable deployment of AM polymers in load bearing, high-temperature, and architected applications, and underpins the experimental investigations presented in this thesis.

1.4 Lattice Structures in Polymer Additive Manufacturing

Lattice structures represent one of the most distinctive design opportunities enabled by additive manufacturing. Unlike solid components, lattice architecture consists of periodic or aperiodic networks of struts or continuous surfaces forming interconnected porous structures. Their relevance in polymer additive manufacturing lies in the ability to decouple stiffness, strength, weight, and functional properties—such as permeability, compliance, and thermal exchange—within a single component. Such levels of structural tailoring are extremely difficult, if not impossible, to achieve using conventional subtractive or formative manufacturing processes. The increasing adoption of lattice structures is closely linked to the development of Design for Additive Manufacturing (DfAM) and topology optimization methodologies. While topology optimization identifies regions where material is structurally required, lattice architecture provides a practical and manufacturable strategy to replace bulk material with architected cellular networks. This approach enables lightweight designs while maintaining load-bearing capability and functional performance, particularly in polymer systems where weight reduction and process efficiency are critical [74,75].

1.4.1 Topology classification and mechanical response

A fundamental distinction among lattice architectures used in polymer additive manufacturing is illustrated in Fig. I. 15. Lattice structures can be broadly classified into three major families: strut-based lattices, surface-based lattices derived from triply periodic minimal surfaces (TPMS), and stochastic or foam-like architectures. Strut-based lattices consist of discrete beams connected at nodes and include widely used geometries such as cubic, BCC, and octet-truss cells. In contrast, TPMS lattices are defined by continuous surfaces that minimize stress concentrations and provide smoother load transfer. Stochastic lattices exhibit non-periodic architectures inspired by natural foams and trabecular bones. Each family exhibits distinct mechanical, manufacturing, and functional characteristics, which strongly influence their suitability for polymer AM and FDM-based applications [76].

From a mechanical standpoint, lattice structures are commonly classified according to their dominant deformation mechanisms, which strongly govern stiffness, strength, and energy absorption efficiency. Stretch-dominated lattices, such as octet-truss or optimized body-centered cubic (BCC) configurations, derive their load-bearing capacity primarily from axial deformation of their structural members. As a result, these architectures exhibit high stiffness-to-weight and strength-to-weight ratios and are often preferred in applications where structural efficiency is the primary objective [77]. In contrast, bending-dominated lattices, including simple cubic, Kelvin, or similar geometries, deform mainly through strut bending and rotation, leading to reduced stiffness but increased damage tolerance and deformation capacity [78].

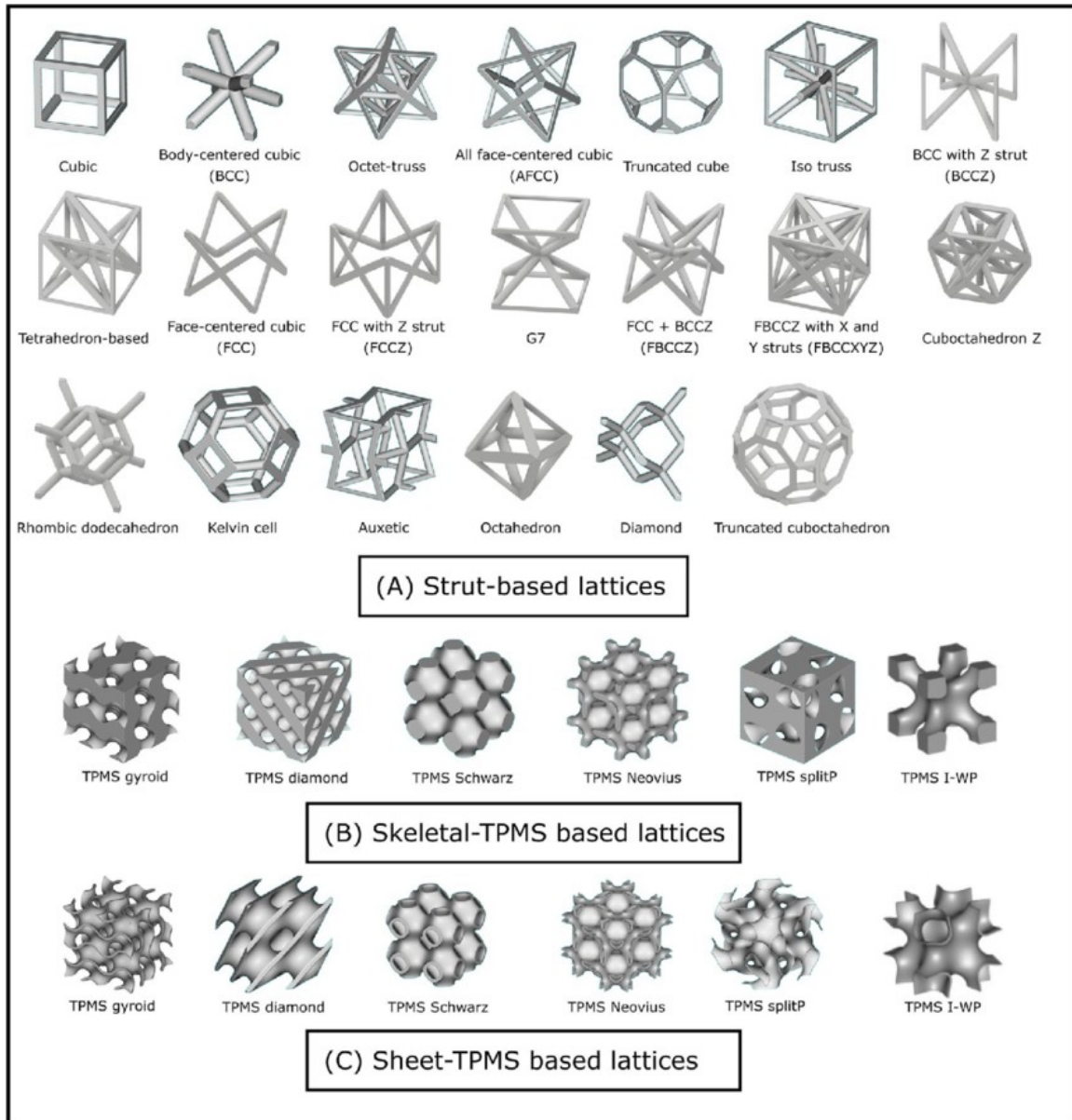


Fig. I. 15: Classification of lattice architectures used in polymer additive manufacturing: (a) strut-based lattices (cubic, BCC, octet-truss), (b) surface-based TPMS lattices (gyroid, diamond), and (c) stochastic foam-like architectures, highlighting fundamental geometric and mechanical differences [76].

This distinction is particularly relevant in the context of polymer additive manufacturing, where material ductility, interlayer bonding quality, and sensitivity to manufacturing defects strongly influence deformation and failure mechanisms. In polymer AM processes such as FDM, bending-dominated architectures often exhibit more stable post-yield behavior and reduced susceptibility to brittle failure, while stretch-dominated lattices can be more sensitive to imperfections at strut junctions and interlayer interfaces.

The compressive mechanical response of polymer lattice structures is schematically illustrated in Fig. I. 16 a). Regardless of topology, lattice materials typically exhibit a characteristic stress-strain response comprising an initial linear elastic regime, followed by a collapse or plateau region associated with progressive cell buckling or strut failure, and a final densification stage at high strain levels, where load rises

steeply as voids close and material compaction dominates [79]. This behavior under compression makes lattice architectures particularly attractive for applications involving energy absorption, load redistribution, and mechanical filtering. To quantitatively assess this performance, lattice structures are increasingly evaluated using the Specific Energy Absorption (SEA) metric, defined as the absorbed mechanical energy per unit mass up to the densification strain. SEA enables direct comparison between different lattice topologies and relative densities, providing a physically meaningful measure of efficiency in energy-absorbing applications. As shown in Fig. LAT-2(b), both topology and architectural grading strongly influence SEA, often more significantly than the base material itself [80].

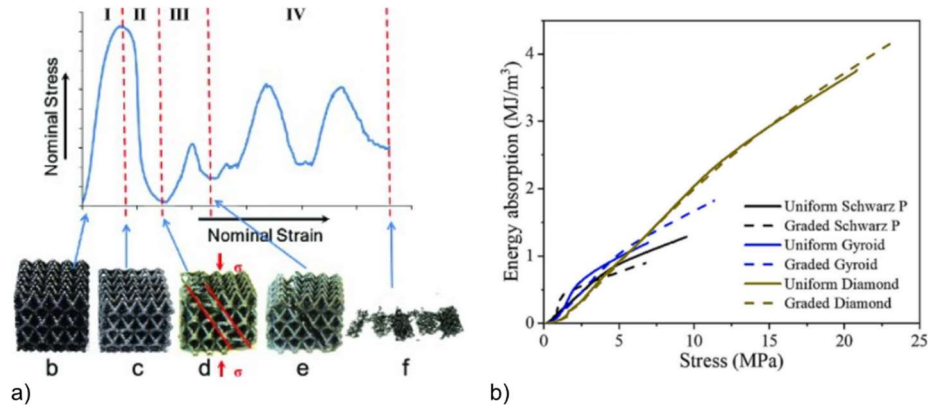


Fig. 1. 16: (a) Representative compressive stress–strain response of polymer lattice structures, highlighting elastic, collapse (plateau), and densification regimes [80]; (b) energy absorption behaviour and Specific Energy Absorption (SEA) comparison for uniform and graded lattice architectures, illustrating the influence of topology and grading strategy on mechanical efficiency [80].

Recent studies demonstrate that surface-based lattices, particularly triply periodic minimal surface (TPMS) architectures such as gyroid, diamond, and Schwarz-P, offer superior and more stable energy absorption compared with conventional strut-based lattices [75]. Furthermore, functionally graded lattices, in which relative density varies spatially, exhibit smoother stress redistribution, delayed localization of deformation, and enhanced cumulative energy absorption. Compression studies on polymer TPMS lattices manufactured by fused filament fabrication confirm that graded configurations achieve significantly higher SEA values across a wide stress range compared with uniform counterparts [80]. In polymer AM, the effectiveness of these deformation and energy absorption mechanisms remains closely tied to manufacturing-induced features such as filament bonding quality, layer orientation, geometric fidelity, and surface roughness. Consequently, lattice performance cannot be decoupled from process physics, reinforcing the importance of integrated design-for-additive-manufacturing strategies when exploiting lattice architectures for functional applications [80].

Beyond their mechanical performance, lattice structures offer additional functional advantages that are particularly relevant for polymer AM. Their inherent porosity enables controlled airflow, resin infiltration, and enhanced heat transfer, while their architecture can be locally graded to achieve spatially varying stiffness or compliance. These multifunctional characteristics have driven growing interest in lattice-based components for tooling, thermal management, and conformal moulding applications, especially when

combined with engineering and high-performance thermoplastics. Taken together, lattice structures represent a natural extension of topology optimization and DfAM principles, offering a powerful framework for tailoring both mechanical and functional performance in polymer additive manufacturing. Their compatibility with material extrusion processes, combined with the availability of engineering and high-performance thermoplastics, makes them particularly attractive for advanced tooling concepts. These aspects provide the foundation for the following section, which focuses on the application of techno polymer lattice structures as molds for the curing of thermosetting composites and resins.

1.4.2 Biomedical implants made by Lattice Structures through AM

In the biomedical field, additively manufactured lattice structures are most prominently employed in orthopedic, dental, and cranio-maxillofacial applications, where porous architectures enable stiffness reduction, weight savings, and improved interaction with surrounding tissues. Additive manufacturing has enabled the fabrication of patient-specific implants and assistive devices with complex internal architectures that would be infeasible using conventional manufacturing routes [81,82]. It is important to note, however, that metallic alloys remain the predominant materials for load-bearing biomedical implants. Titanium-based alloys (e.g., Ti-6Al-4V), cobalt–chromium alloys, and stainless steels are extensively used due to their high stiffness, fatigue resistance, corrosion resistance, and long-term clinical reliability. The integration of lattice and porous architectures into metallic implants enabled primarily by powder-bed fusion technologies has further enhanced osseointegration and reduced stress shielding, consolidating metals as the material of choice for structural biomedical applications [83,84]. In contrast, polymeric lattice structures occupy a complementary role, particularly in applications where full load-bearing capability is not required. High-performance polymers such as PEEK and PEKK are increasingly employed for cranial implants, spinal cages, surgical guides, and patient-specific orthoses, benefiting from their radiolucency, elastic modulus closer to cortical bone, reduced weight, and compatibility with medical imaging. In these contexts, lattice architectures are exploited to tailor compliance, promote ventilation or fluid transport, and enhance comfort rather than to maximize strength alone [85-87].

Representative examples of these biomedical applications are shown in Fig. I. 17, illustrating how lattice and porous architectures are exploited across polymeric and metallic additively manufactured implants and assistive devices.



Fig. I. 17: Examples of biomedical implants and assistive devices manufactured by additive manufacturing using lattice and porous architectures: (a) patient-specific PEEK cranial implant featuring a perforated lattice structure for stiffness reduction and tissue integration [87]; (b) customized PA12 forearm orthoses produced by AM, exploiting lattice designs for weight reduction, ventilation, and wearer comfort; (c) 3D-printed titanium sternum implant fabricated by metal additive manufacturing, incorporating lattice regions to enhance osseointegration and mechanical compatibility; (d) pelvic implant manufactured by powder bed fusion using Ti-6Al-4V ELI alloy, illustrating the use of porous structures to promote bone ingrowth and reduce stress shielding.

1.4.3 Industrial Applications of Lattice Structures in Polymer AM

In the automotive sector, lattice structures produced by polymer AM are increasingly used for lightweight interior components, functional housing, brackets, air-ducting systems, and energy-absorbing elements [88,89]. These components benefit from reduced mass, part consolidation, and improved functional integration compared with traditionally manufactured counterparts. Polymer lattices are particularly attractive for vibration damping, noise reduction, and impact mitigation, where controlled deformation and tailored compliance are more critical than absolute strength. Several studies report on the successful adoption of lattice-filled polymer components for interior structures, ventilation systems, and customized fixtures, especially in low- to medium-volume production and motorsport applications. In the context of electric vehicles, increasing emphasis is placed on lightweight, battery-integrated structural components for chassis and body panels. Recent research has explored the development of energy storage multifunctional sandwich structures (ESMS), in which conventional foam cores (e.g., PET) are replaced by additively manufactured continuous carbon fiber (CCF) lattice cores, enabling simultaneous load-bearing capability and structural energy storage functionality [90].

Similarly, in consumer and sports applications, polymer lattice structures have become a key enabling technology for footwear midsoles, protective gear, helmets, padding systems, and ergonomic products. In these cases, the primary performance drivers are energy absorption, comfort, and durability under cyclic loading. Lattice architectures allow precise control over stress–strain response, enabling designers to tune cushioning performance and resilience through geometry rather than material changes alone. The widespread adoption of lattice-based polymer components in footwear and protective equipment highlights the maturity of AM lattices for functional, production-grade applications [91].

Instead, in the field of techno-polymers, recent studies demonstrate that lattice-filled FDM components manufactured from PEEK or PEI can achieve sufficient stiffness and thermal stability for use in composite lay-up tooling, vacuum forming molds, and autoclave-compatible fixtures, while significantly reducing material consumption and machining time compared with conventional metal tooling. The Fig. I. 18 illustrates a structured comparison between additive manufacturing (AM) and traditional manufacturing (TM) approaches, highlighting their respective advantages and limitations in the context of polymeric mold and tooling development. The comparison is organized across several key aspects that are particularly relevant for manufacturing strategy selection.

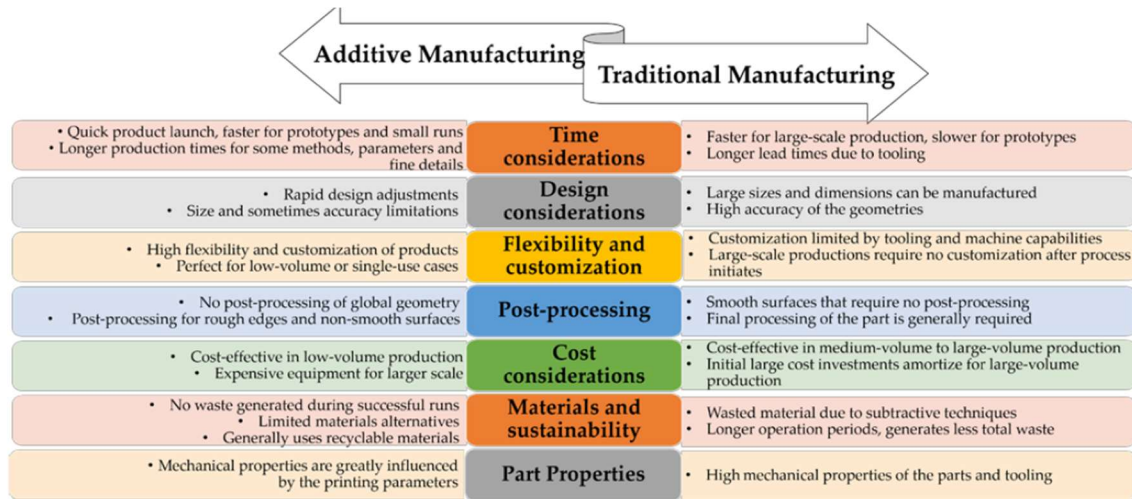


Fig. I. 18: Comparison between additive and traditional manufacturing [92]

- **Time:** AM is characterized by rapid product launch and reduced lead times, making it especially suitable for prototyping, design validation, and low-volume production. In contrast, traditional manufacturing is generally faster and more efficient for large-scale production but requires longer lead times due to tooling design and fabrication.

Design: AM enables rapid design modifications and supports complex geometries, albeit with potential limitations in size and dimensional accuracy. Traditional manufacturing allows the production of larger components with higher geometric accuracy, benefiting applications that demand tight tolerances and dimensional stability.

Flexibility and customization: AM offers a high degree of freedom, allowing tailored designs and mass customization without additional tooling costs. This makes it particularly advantageous for customized or single-use molds. Conversely, customization in traditional manufacturing is constrained by tooling and machine capabilities, and large-scale production typically relies on standardized designs.

Post-processing: AM can fabricate complex geometries without post-processing of the global shape, although additional finishing is often required to address surface roughness. Traditional manufacturing generally produces smoother surfaces but often requires final processing steps to achieve the desired part quality.

Cost perspective: AM is cost-effective for low-volume production, while traditional manufacturing becomes economically favorable at medium to high production volumes due to cost amortization.

Material usage and sustainability are highlighted, with AM generating minimal waste during successful builds, whereas traditional subtractive processes inherently produce material waste.

Fig. I. 18 also emphasizes that part properties in AM are strongly influenced by processing parameters, unlike the more predictable mechanical performance typical of traditionally manufactured tools.

1.4.4 AM Polymeric Tooling under Autoclave Curing Conditions

Traditional manufacturing of fiber-reinforced polymer matrix composites (FRP) relies on rigid molds capable of withstanding the combined effects of temperature and pressure during curing. In thermoset composite fabrication, processes such as vacuum-assisted resin transfer molding, resin transfer molding, and prepreg processing may be carried out either at room temperature under vacuum or at elevated temperature and pressure in autoclaves. Conventional metallic or specialized tooling materials provide reliable thermo-mechanical performance but involve high costs, long lead times, and substantial material waste.

Additive manufacturing (AM), particularly fused deposition modeling (FDM), has emerged as a promising alternative for composite tooling, offering significant reductions in cost and lead time while enabling design flexibility and rapid iteration [93]. However, the suitability of additively manufactured polymeric molds is strongly governed by their ability to maintain dimensional stability, stiffness, and surface integrity under simultaneous thermal softening and sustained pressure, which represent the most critical conditions during curing, particularly under autoclave conditions.

A screening of the open literature over the last decade was conducted to evaluate the extent to which additively manufactured polymeric tooling has been investigated for the curing of thermoset composites under autoclave conditions. The analysis considered only studies explicitly addressing the combined action of elevated temperature and sustained external pressure, which represents the most critical operating regime for composite molds. The number of relevant publications remains limited, with only a few contributions per year confirming that this application area is still at an early stage of development. Most published works focus on feasibility demonstrations, large-scale tooling concepts, surface finishing strategies, or dimensional stability at the tool level [94-96].

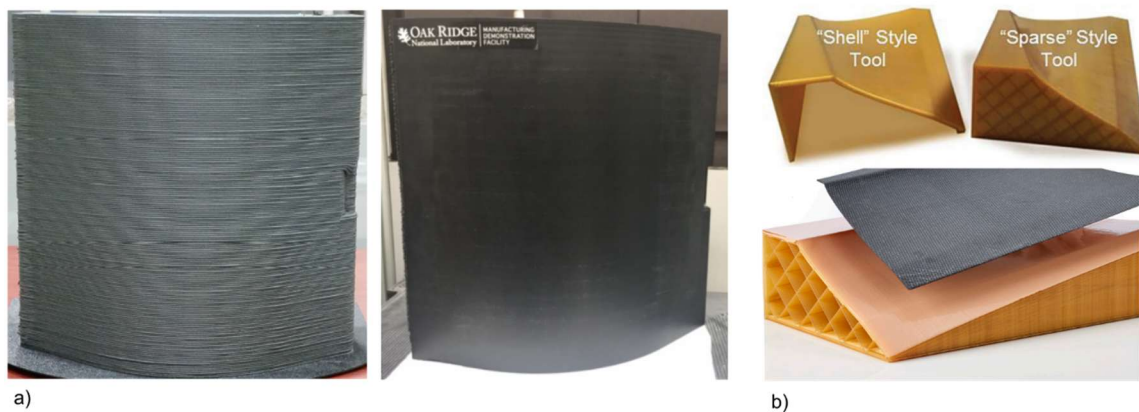


Fig. I. 19: Examples of additively manufactured polymer tooling for composite manufacturing: (a) large-format mold produced by extrusion-based additive manufacturing (BAAM-type process), characterized by pronounced layer-wise surface waviness [96]; (b) additively manufactured polymer mold after machining and surface finishing, suitable for composite lay-up and autoclave curing [93].

Fig. I. 19 a) illustrates a large-format tool produced via extrusion-based additive manufacturing, representative of BAAM-type processes. The intrinsic layer-wise deposition results in pronounced surface waviness and relatively high roughness, which become more severe as part size increases. For composite

curing applications, this condition is incompatible with direct lay-up requirements, as surface quality directly affects laminate finish and consolidation. Consequently, tooling geometries are often intentionally oversized during printing to allow subsequent subtractive machining or surface finishing, ensuring that the final mold geometry and surface quality meet composite manufacturing standards. This hybrid additive–subtractive approach is therefore not optional but essential, especially for large tools intended for autoclave curing as illustrated by Krunc et al. [95].

Material selection remains a key limitation. While SLA-printed molds can reduce cost and lead time, their durability under repeated high-temperature and high-pressure autoclave cycles is limited to one-part molds for vacuum bagging [97,98]. In contrast, high-performance thermoplastics such as polyetherimide-based ULTEM 1010 and carbon-fiber-reinforced thermoplastics have demonstrated the ability to withstand autoclave conditions above 180–200 °C with limited deformation under load, surviving multiple curing cycles [93,95]. Despite these advances, the widespread adoption of AM polymeric tooling for serial aerospace production remains constrained by material availability, certification, and long-term thermo-mechanical reliability under cyclic curing conditions.

To properly frame the thermo-mechanical characterization of additively manufactured PEEK lattice specimens, it is necessary to define the typical curing conditions encountered during autoclave processing of fiber-reinforced thermoset composites. Autoclave curing is widely recognized as one of the most demanding manufacturing environments for tooling materials due to the combined and sustained action of high temperature, external pressure, and long dwell time [92]. For aerospace-grade epoxy-based prepreg systems, curing temperatures typically range between 120–135 °C for medium-temperature resin systems and 175–185 °C for high-performance structural composites, with some advanced formulations requiring curing temperatures approaching 200 °C or higher [99]. The thermal cycle generally consists of controlled heating ramps, an isothermal dwell lasting from 1 to 4 hours, and a controlled cooling phase to limit residual stresses and distortion [100]. For high performance aerospace structures, vacuum bagging is commonly combined with external autoclave pressure to enhance fiber consolidation, reduce void content, and improve interlaminar properties. In such cases, autoclave curing typically involves total external pressures between 0.3 and 0.8 MPa, applied and maintained throughout the dwell period at elevated temperature. The applied pressure is generally held constant for extended durations, often ranging from 1 to 4 hours, while the resin viscosity decreases and crosslinking progresses. Under these conditions, the mold or tooling is subjected to a sustained compressive load at elevated temperature, which represents a critical scenario for polymeric tooling materials operating near their glass transition temperature [99].

From a tooling perspective, this pressure-temperature combination promotes time-dependent deformation mechanisms, such as creep and stress relaxation, which must be carefully accounted for when evaluating the suitability of additively manufactured polymeric molds for autoclave curing applications.

1.5 Concluding Remarks on the Literature Review

This literature review has examined polymer additive manufacturing technologies, design methodologies, and lattice architectures, highlighting the unique role of Fused Deposition Modelling (FDM) in processing engineering and high-performance thermoplastics such as PEI and PEEK. While lattice structures are now widely adopted in sectors such as automotive, consumer goods, and biomedical applications, their use in high-temperature polymer tooling, particularly under autoclave curing conditions, remains insufficiently explored.

The reviewed studies indicate that lattice architecture offers significant potential for weight reduction, energy absorption, and functional tailoring, but their mechanical and time-dependent behavior is strongly influenced by process-induced features, thermal history, and material properties. In the context of composite tooling, these factors become critical, as polymeric molds must maintain dimensional stability and stiffness under sustained temperature and pressure.

Consequently, a clear gap emerges in the systematic characterization of FDM-manufactured lattice structures made from techno-polymers for composite curing applications. The experimental work presented in this thesis addresses this gap by investigating the thermo-mechanical response of high-performance polymer lattice structures under conditions representative of autoclave processing.

2. MATERIALS AND METHODS

This chapter presents the experimental framework developed to investigate the mechanical behavior of additively manufactured polymer components, with particular emphasis on interlayer adhesion and process-induced anisotropy in fused deposition modelling (FDM). The methodology follows a deliberately bottom-up approach. The study begins with PLA processed on open-architecture printers, which enables a controlled investigation of deposition strategies, thermal history and interfacial bonding. This initial stage serves to establish and validate the experimental workflow under well-controlled and repeatable conditions.

Once the methodology is consolidated, the same characterization tools are extended to higher-performance polymers, including PEEK, PAEK and short carbon fiber reinforced PA6, manufactured under industrial FDM conditions. This transition allows the assessment of whether the process to structure property relationships identified for low-cost materials remain valid when applied to aerospace-grade polymers and industrial manufacturing environments. Attention is devoted to the persistence of anisotropic behavior across different materials and geometries, highlighting how process signatures influence not only bulk specimens but also components with increased geometric complexity.

In addition to solid specimens, the chapter introduces the mechanical characterization of architected lattice structures subjected to compressive loading. In these components, anisotropy emerges from the combined effects of printing direction, unit-cell topology and load orientation. These factors directly affect stiffness, strength, densification behavior and energy absorption, making lattice structures a critical test case for extending material-level observations to structural performance in lightweight and functional components.

A broad set of complementary characterization techniques supports this investigation:

- Infrared thermography is employed in situ to capture the transient temperature field during deposition under different printing orientations and component sizes, providing quantitative information on cooling rates, interlayer and interfilamentous times, and potential crystallization conditions.
- Optical stereomicroscopy and scanning electron microscopy are used to analyze cross-sections, porosity and fracture surfaces, allowing the distinction between interlayer and intralayer failure mechanisms.
- Thermal and thermo-mechanical analyses, including differential scanning calorimetry (DSC), density measurements and dynamic mechanical analysis(DMA), further support the interpretation of microstructural evolution and mechanical response, particularly for semicrystalline and high-performance polymers.

Instead, mechanical testing is organized into targeted experimental campaigns, each addressing a specific aspect of bonding quality and structural response:

- Mode I interlaminar fracture toughness tests directly quantify the energy required to initiate and propagate cracks along layer interfaces.

- Uniaxial tensile tests are used to evaluate the influence of build direction, interlayer time, extrusion conditions and component geometry on stiffness, strength and ductility.
- Compression tests on PEEK, PA6-CF and TPU lattice structures extend the analysis from bulk material behavior to architected systems, enabling the correlation of topology and relative density with energy absorption, collapse mechanisms and repeatability.

Moreover, temperature-dependent and time-dependent mechanical behavior of PEEK has been analyzed. Quasi-static compression tests performed at elevated temperatures, spanning the glass transition region and extending into the rubbery regime, are used to quantify the degradation of stiffness, strength and energy absorption under service-relevant thermal conditions. Complementary constant-load compression tests are conducted to investigate creep-driven deformation at high temperatures. In these tests, specimens are subjected to a prescribed compressive load while exposed to controlled thermal environments, and the resulting deformation is measured after thermal exposure. This approach provides insight into dimensional stability and long-term deformation mechanisms, which are critical for tooling applications such as additively manufactured molds for composite curing in autoclave environments.

Overall, this chapter outlines an integrated experimental methodology that combines mechanical testing, thermal analysis and microstructural characterization. The proposed framework enables the interpretation of fundamental process–structure–property relationships while simultaneously addressing the requirements of industrially relevant applications, which are discussed in detail in the subsequent chapters.

2.1 Experimental workflow and auxiliary techniques for interlayer adhesion

This section introduces the experimental methodologies and auxiliary techniques adopted throughout the thesis to support the mechanical characterization of interlayer adhesion in additively manufactured polymer components. Rather than focusing on a single test, it provides an overview of the preparatory and analytical steps that are repeatedly employed.

Fig. 2. 1 schematically illustrates the experimental workflow adopted for tensile tests, highlighting the main phases involved in specimen manufacturing, preparation and characterization.

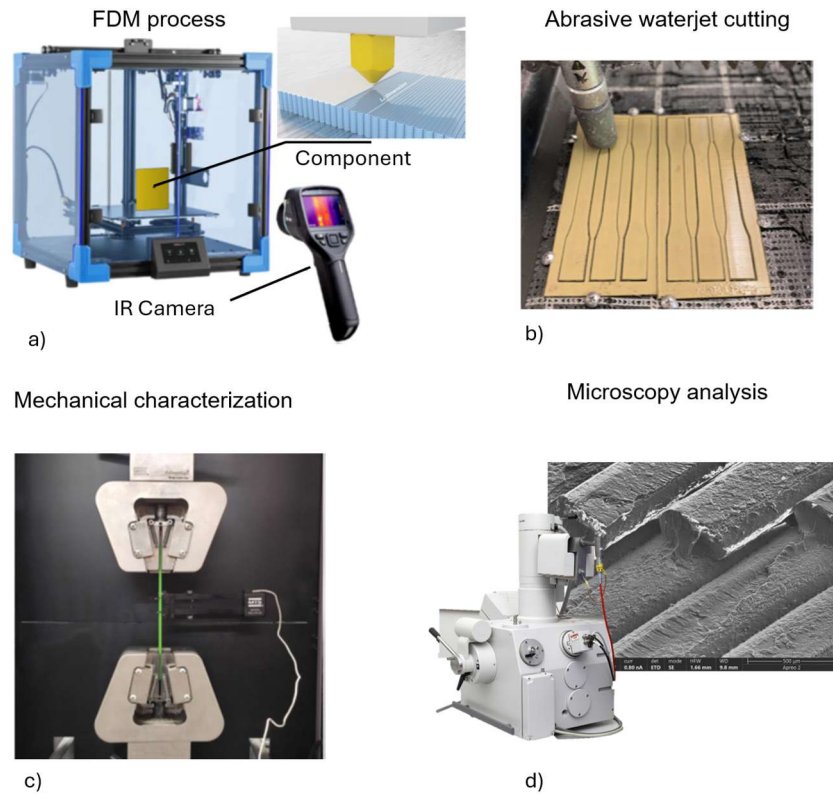


Fig. 2. 1: Overview of the experimental workflow: (a) FDM process with in situ infrared thermography; (b) specimen extraction by abrasive waterjet cutting; (c) mechanical testing setup; (d) microscopy analyses for microstructural and fracture characterization

Fig. 2. 1 a) shows the fused deposition modelling process and the integration of in situ infrared thermography. During printing, the infrared camera is used to monitor the transient temperature field of the deposited filaments and surrounding material, providing quantitative information on cooling rates, interlayer times and thermal gradients. These data are essential for correlating local thermal history with bonding quality and subsequent mechanical response.

After printing, specimens may be obtained through two alternative fabrication routes, depending on the objective of the analysis. As shown in Fig. 2. 1 b), abrasive waterjet machining is employed to extract test coupons from larger printed components. This cold cutting process avoids additional thermal input and

mechanical damage, preserving the original microstructure generated during printing. The use of AWJ enables the investigation of specimens representative of bulk regions of complex components, decoupling material behaviour from edge effects and local print kinematics.

Mechanical characterization is then carried out using standardized testing configurations, as illustrated in Fig. 2. 1 c) for uniaxial tensile tests. These experiments provide direct access to stiffness, strength, ductility and fracture behavior, and form the quantitative basis for assessing the influence of processing parameters, build orientation and specimen geometry.

Finally, microscopy analyses are performed to support the interpretation of mechanical results. Fig. 2. 1 d) illustrates the use of optical and scanning electron microscopy to examine fracture surfaces, interlayer interfaces and internal porosity. These observations allow the identification of dominant failure mechanisms, distinguishing interlayer debonding from intralayer fracture and providing direct evidence of process-induced defects and anisotropy.

2.1.1 Waterjet machining

Abrasive waterjet machining (AWJ) is a cold cutting process in which material removal is achieved by directing a high-velocity jet of water, entrained with abrasive particles, onto the workpiece surface. While pure waterjet cutting is typically employed for soft materials such as rubbers or foams, the addition of abrasive particles, most commonly garnet, enables the cutting of polymers, composites, metals, ceramics, and glass without inducing a heat-affected zone. This characteristic makes AWJ particularly suitable for additively manufactured polymers, where thermal damage, melting, or microstructural alteration must be avoided. In industrial AWJ systems, water is pressurized to values typically ranging between 40 and 60 kpsi (approximately 275–415 MPa), generating jet velocities close to Mach 3, corresponding to 700–900 m/s. The pressurized water passes through a jewel orifice, after which abrasive particles are entrained into the flow within a mixing chamber through a Venturi effect. The resulting water–abrasive mixture is then accelerated through a tungsten-carbide focusing tube, forming a coherent cutting jet. A schematic representation of a conventional AWJ system and the main nozzle components is shown in Fig. 2. 2 a) and Fig. 2. 2b), respectively [101].

The cutting performance of AWJ is governed by a combination of hydraulic, abrasive, mixing, and kinematic parameters. Hydraulic parameters include water pressure and orifice diameter, which control jet velocity, flow rate, and power density. Abrasive parameters comprise particle material, size, and mass flow rate. Mixing parameters are associated with the geometry of the mixing tube, namely its length and internal diameter, while kinematic parameters include traverse speed, jet angle, and standoff distance. Together, these variables determine kerf width, surface roughness, waviness, and achievable cutting depth [102].

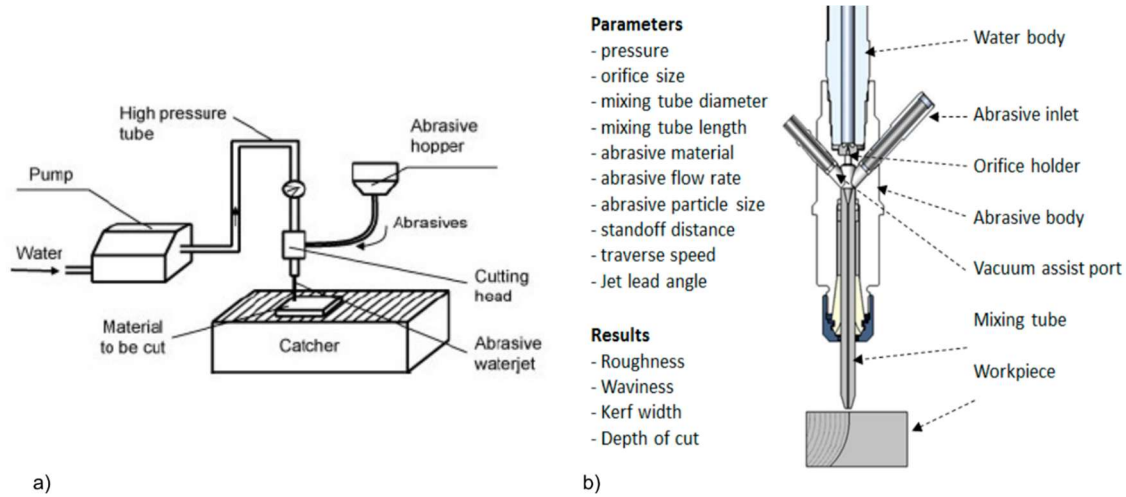


Fig. 2. 2 a) Schematic of an abrasive waterjet cutting system [101]; b) Nozzle scheme and AWJ parameters [102]

In this work, specimen extraction was performed using a WAZER Desktop abrasive waterjet system, a compact AWJ machine specifically designed for cold cutting of small components. The system provides a nominal working envelope of 305×457 mm, as illustrated in Fig. 2. 3 a). The hydraulic subsystem operates at a maximum pressure of approximately 32 MPa (≈ 317 bar), delivering a water flow rate of about $1.7 \text{ L} \cdot \text{min}^{-1}$. The jet is formed through a 0.48 mm jewel orifice, while abrasive particles are accelerated through a tungsten-carbide focusing tube with an internal diameter of approximately 1.1 mm, producing a kerf width on the order of 1.1 mm. Fig. 2. 3 b) shows a magnified view of the cutting head and nozzle assembly.

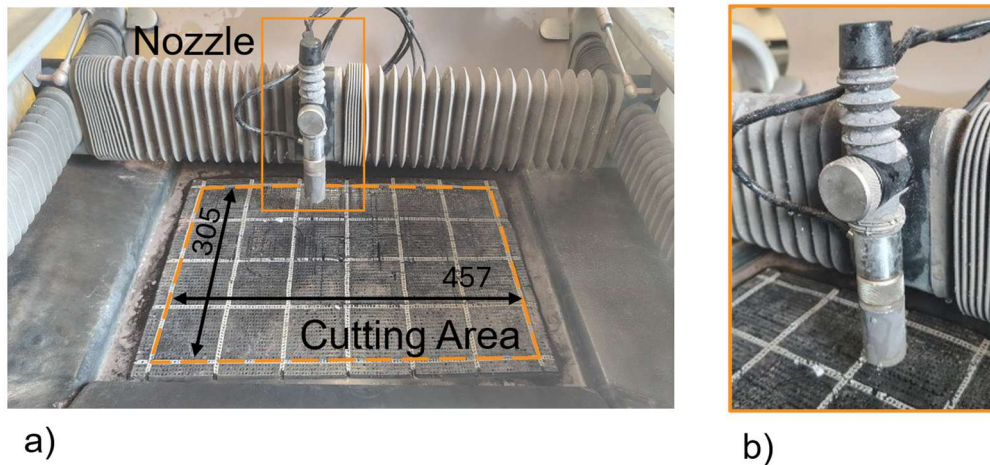


Fig. 2. 3: a) cutting area of the WAM WAZER awj; b) magnificated image of the cutting head

Cutting paths and process parameters were defined using the WAZERCAM software, an online open-source interface provided by the machine manufacturer. The software workflow, shown in Fig. 2. 4, begins with importing the two-dimensional specimen geometry in DXF format. The position and orientation of the cutting path within the working area are then defined, followed by the selection of material type and thickness. Although PLA is not included by default in the “Plastics and Rubbers” database, custom material entries can be created using the “Add/Edit Material” function, allowing the definition of appropriate cutting speeds as a

function of thickness. Once all parameters are set, the software generates the machine G-code, which contains the toolpath and local traverse speed for each segment.

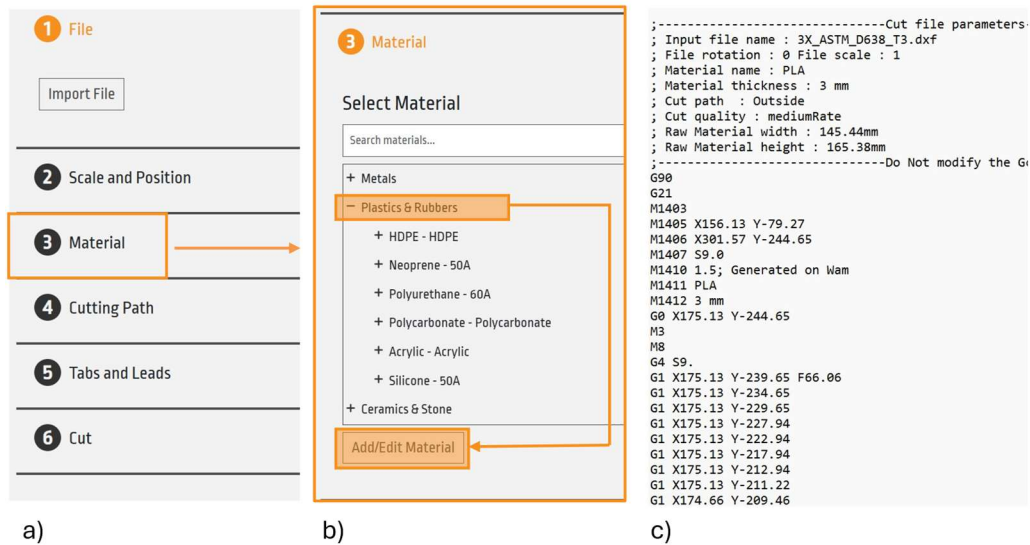


Fig. 2. 4: WAZERCAM software interface for abrasive waterjet toolpath generation: (a) workflow for importing the DXF geometry and defining scale and position; (b) material selection and customization via the Add/Edit Material function, used to define cutting parameters for PLA; (c) excerpt of the generated G-code, reporting the toolpath and local traverse (cutting) speed.

As highlighted in Fig. 2. 4 c), the generated G-code explicitly reports the cutting speed adopted during the operation. For polymer components with a thickness of 3 mm, a traverse speed of approximately 66 mm/min was employed. This relatively low cutting speed ensures complete penetration of the jet, minimizes kerf taper, and preserves the integrity of the additively manufactured microstructure, which is essential for subsequent mechanical and microstructural characterization.

2.1.2 Microscopy: Morphology studies

Morphological characterization was carried out using both optical microscopy and scanning electron microscopy (SEM) to investigate the internal structure of the specimens prior to testing and the fracture surfaces after mechanical failure. These analyses provide essential insight into process-induced features such as interfilamentous bonding, void distribution, porosity, and fracture mechanisms, which cannot be captured through mechanical testing alone. A LEICA M205 stereomicroscope was employed for low-magnification observations to quantify the actual cross-sectional dimensions of the specimens, assess surface morphology, and identify macroscopic fracture features. This instrument was particularly useful for evaluating geometric accuracy and detecting defects related to deposition strategy and raster arrangement. Higher-resolution optical microscopy was performed using a LEICA DM500 microscope to examine polished cross-sections and quantify internal interfilamentous voids. The two optical microscopy systems used in this work are illustrated in Fig. 2. 5: Fig. 2. 5 a) shows the LEICA M205 stereoscope, while Fig. 2. 5 b) reports the LEICA DM500 optical microscope. Scanning electron microscopy was employed to analyze fracture surfaces and to investigate the micromechanics governing interlayer and intralayer failure. SEM observations were carried out using a Philips XL40 microscope, shown in Fig. 2. 5 c). SEM analysis enabled the identification of ductile versus brittle fracture modes, filament pull-out, fiber–matrix interaction in reinforced polymers, and evidence of insufficient interfacial bonding.

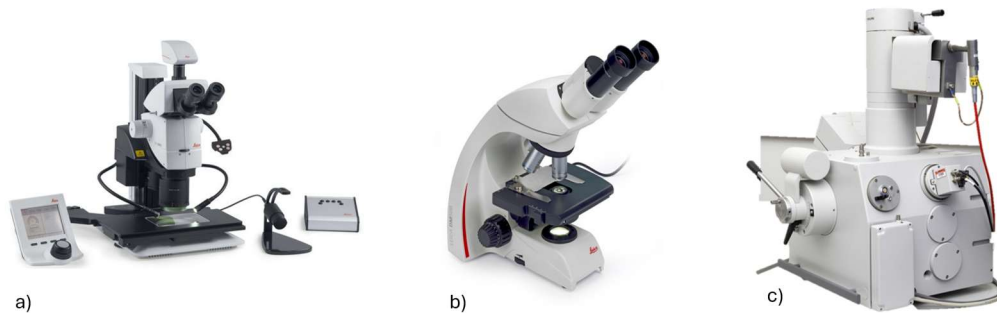


Fig. 2. 5: Microscopy equipment used for morphological characterization: (a) stereomicroscopy for geometric and surface inspection; (b) optical microscopy for internal morphology; (c) SEM for fracture and interfacial failure analysis.

To enable optical metallographic examination of internal morphology, selected specimens were embedded in epoxy resin prior to grinding and polishing. Metallographic embedding is a widely adopted procedure for the characterization of FDM-printed components, as it allows accurate inspection of internal process-induced defects such as interfilament voids, lack-of-fusion regions, and porosity. The embedding resin consisted of Epoxy 1000 Harz and Epoxy 1000 Härter, mixed at a resin-to-hardener ratio of 5:1 by weight. After pouring, specimens were kept fully submerged until complete polymerization. The resin was cured under ambient conditions for 6–12 hours, ensuring sufficient hardness for subsequent grinding.

After curing, up to four embedded specimens were mounted in the holders of a PRESI P.E.I.U. grinding and polishing machine. Grinding was performed using progressively finer abrasive papers (220, 400, 600, 1200, and 2500 grit) to obtain a flat and deformation-free surface. Water was used throughout as both lubricant and

coolant. During the final polishing step, a 0.2 μm alkaline fumed silica suspension was applied to enhance surface finish and achieve adequate reflectivity for high-resolution optical analysis.

Within the broader experimental framework of this thesis, microscopy plays a complementary role to mechanical and thermal characterization. Optical and SEM analyses support the interpretation of interlayer adhesion measured through fracture toughness and tensile tests, clarify the influence of thermal history on microstructural evolution, and assist in correlating processing parameters with mechanical anisotropy. Moreover, morphological observations are extended to lattice structures, where strut integrity, surface quality, and local defects strongly influence compressive response and energy absorption. In this way, microscopy provides a microstructural basis for understanding the process–structure–property relationships discussed throughout the subsequent sections.

2.2 Interlaminar fracture Toughness

The double cantilever beam (DCB) specimen of the present work, whose basic geometry was defined following ASTM D5528 standard [103]. Due to the similarities concerning the mechanical behavior between composite materials and additively generated components, testing standards for composite materials are used in most studies to assess the fracture toughness of additively manufactured parts. Additionally, the pre-crack was often accomplished by inserting a film in the midplane to fabricate the specimen [104-107]. The film only tenuously clings to the substrate, allowing further layers of deposition. This process required interrupting the deposition to install the film, which also calls for careful handling. As a result, the release film may cause some problems since the deposition process is interrupted, and a more extended period passes before moving on to the following layer. When installing the subsequent layer, this requires a faster cooling of the substrate. As a result, the substrate cools down more quickly than it would under normal deposition circumstances. A new sample configuration involving nested spacers was proposed in [108] to overcome such limitations. This sample configuration enabled the identification of the interlayer fracture toughness with great accuracy without coming across the abovementioned limitations. In this case, the influence of the deposition strategy on the fracture toughness of FDM samples was investigated using double cantilever beam (DCB) specimens as illustrated in Fig. 2. 6 a)

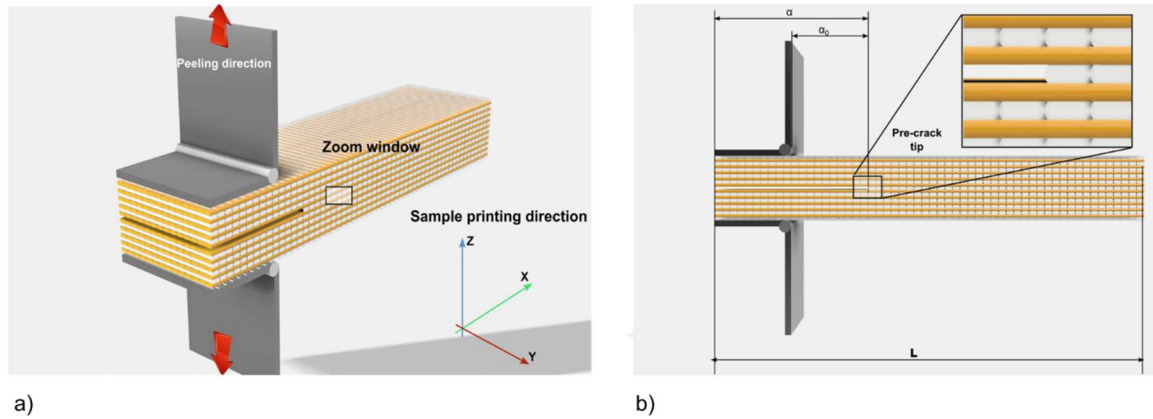


Fig. 2. 6: a) Relative direction between filaments and applied load; b) Main dimensions of the samples used in the experimentations.

A schematic view of the DCB specimen and the adopted printing strategy is reported in Fig. 2. 6 a), where successive deposited layers are highlighted using different colors to clarify the interfacial configuration. The overall specimen geometry was selected in compliance with ASTM D5528 recommendations and adapted to the mechanical properties of PLA. The key geometrical parameters include the specimen length L , width b , total thickness h , pre-crack length a , and the distance a_0 between the crack tip and the loading axis, as illustrated in Fig. 2. 6 b).

The dimensions of the specimens were verified against the geometrical constraints imposed by linear elastic fracture mechanics to ensure valid Mode I conditions. In particular, the following relations enumerated in were satisfied:

$$t \leq \frac{h}{4} + 0.01 \sqrt{\frac{0.04}{G_{Ic}} \frac{3+E_{11}}{a^2} + a^2} \quad \text{Eq. 1)}$$

$$h \geq 8.28 \sqrt[3]{\frac{G_{Ic} a_0^2}{E_{11}}} \quad \text{Eq. 2)}$$

$$\alpha_0 \leq 0.042 \sqrt{\frac{h^3 E_{11}}{G_{Ic}}} \quad \text{Eq. 3)}$$

where E_{11} is the Young's modulus along the printing direction (x-direction), G_{Ic} is the expected critical energy release rate, a is the pre-crack length, and a_0 is the distance from the load line to the initial crack tip.

The longitudinal Young's modulus E_{11} of PLA was determined experimentally through uniaxial tensile tests performed on specimens printed using the same filament and deposition conditions. Tensile tests were conducted using an MTS Criterion 43.50 universal testing machine, following ASTM D638, under ambient conditions and at a crosshead speed of 1 mm/min. Depending on filament orientation, the measured elastic modulus ranged between 2.8 and 3.3 GPa. These values were used to define the minimum specimen thickness and crack length required to satisfy the above criteria. Based on this analysis, the overall specimen thickness was selected in the range of 4.5–7.5 mm, depending on the initial crack length. The final geometrical dimensions adopted for the DCB specimens are summarized in Tab. II. 1.

Tab. II. 1: Characteristic dimensions of the specimens used for ASTM D5528 tests

Geometrical characteristics	Dimension [mm]
Length (L)	125
Width (b)	25
Pre-crack length (a)	55
Initial distance of the crack tip from the load axis (a_0)	30

2.2.1.1 Sample manufacturing methods

The experimental activity was organized into two distinct manufacturing campaigns, aimed at progressively refining the investigation of interlayer fracture toughness in FDM-printed PLA specimens. In the first campaign, a broad exploratory study was conducted to assess the influence of raster orientation on the mechanical response. A full factorial plan was defined to produce DCB specimens using the material extrusion (MEX) process, as schematically illustrated in Fig. 2. 7. Five different deposition strategies were adopted, characterized by distinct filament orientations within the printed layers. Three monodirectional linear raster patterns were employed, with filaments oriented at 0° , 45° , and 90° with respect to the loading direction, as shown in Fig. 2. 7 a-c). In addition, two alternating raster strategies were considered, in which the filament orientation alternates between successive layers. In the first alternating configuration, filaments were oriented at $0^\circ/90^\circ$, while in the second configuration a $\pm 45^\circ$ orientation was adopted. Representative schematics of these alternating strategies are reported in Fig. 2. 7 d-e).

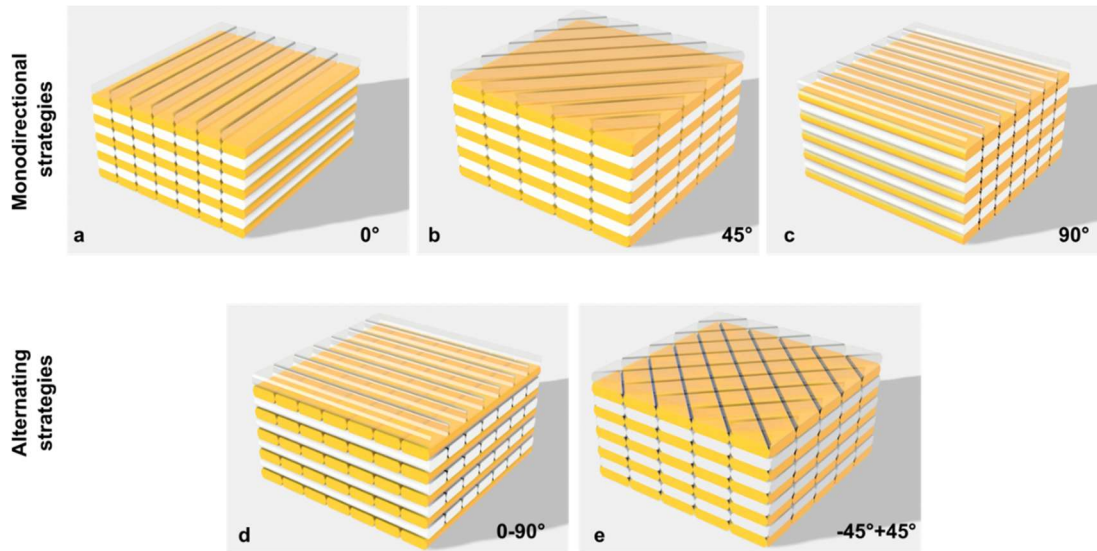


Fig. 2. 7: Schematic of the deposition strategy used to manufacture the specimens

Based on the results of this preliminary investigation, the second experimental campaign focused exclusively on the alternating deposition strategies, namely $0^\circ/90^\circ$ and $\pm 45^\circ$, which are more representative of practical infill patterns used in functional FDM components. In this phase, the influence of key process parameters on interlayer fracture toughness was systematically investigated.

The complete factorial design of the second campaign is summarized in Tab. II. 2 , which details the variation of three main process parameters during specimen fabrication:

1. Layer Deposition Strategy: The study encompassed two deposition strategies, $\pm 45^\circ$ and $0/90^\circ$, as elucidated in Section 2.1.
2. Extrusion Multiplier: Governing the volume of plastic extruded per unit distance through the print head's nozzle, this parameter was studied at three values: 0.96 (96%), 1.00 (100%), and 1.04 (104%).
3. Deposition Speed: The print head's movement speed was maintained at three levels: 1000, 2000, and 4000 mm/min for all deposition strategies.

Tab. II. 2: Test matrix with factors and assumed levels.

Level	Deposition strategy	Extrusion Multiplier [%]	Deposition speed [mm/min]
I	$\pm 45^\circ$	96	1000
II	$0/90^\circ$	100	2000
III		104	4000

For all specimens, the layer thickness was fixed at 0.2 mm, corresponding to half of the nozzle diameter, in order to ensure stable extrusion and consistent interlayer contact. Perimeter shells were deliberately omitted to eliminate the contribution of filaments not aligned with the infill raster direction and to isolate the effect of the selected deposition strategies. All remaining printing parameters were kept constant and are reported in Tab. II. 3.

Tab. II. 3: FDM process parameters to reproduce the DCB Mode I specimens

Printing Parameter	Value
Layer thickness [mm]	0.2
Nozzle Temperature [°C]	210
Printing Bed Temperature [°C]	60
Infill Percentage [%]	100
Internal Fill pattern	Grid

In addition, following the procedure described by [109], the pre-crack was produced by printing some thin spacers which can be easily cut, as schematized in fig. PLA2b.

The adoption of these five deposition strategies facilitates a comprehensive exploration of the influence of raster angle on specimen fracture toughness. This investigation aims to elucidate how different orientations affect mechanical behavior.

2.2.1.2 Set up of the specimens and mechanical test

The load is transmitted to the specimen through the top and bottom layer surface. In fact, the load line must be perpendicular to these surfaces, promoting the transverse opening of the crack. To ensure this load condition, two steel piano hinges have been applied to the specimen, following the protocol below:

Apply a neutralizing cleaning spray (WEICON cleaner) to improve the quality of the adhesion between the specimen surface and the hinges. This product is necessary to remove all the contaminant elements, like the powder on both the surfaces of the specimen and the hinges.

1. White paint was applied to the specimen's side surface. Tick marks were signed every 5 mm each of them, starting from the crack tip (signed with 0) to determine the position of the crack front during the test were signed on this surface. In the firsts 5 mm starting from the sign 0 the marks were drawn 1 mm apart. This is crucial in the first part of the test as it will be explained in detail afterwards as illustrated in Fig. 2. 8.

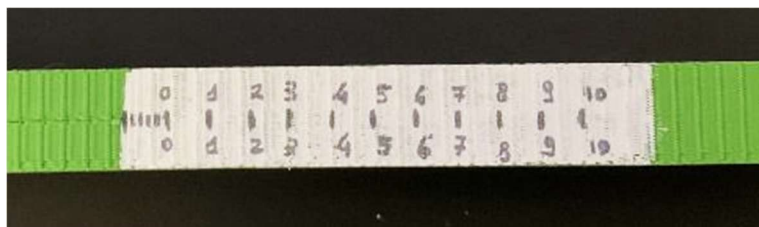


Fig. 2. 8: Macrograph of the painted specimen adopted for the analysis of crack front propagation

2. Subsequently, on the specimen's surfaces, at the hinge application area, is applied the Primer WEICON P400 chloric to prepare these surfaces before the application of the glue.

3. A two-component toughened methacrylate adhesive system Araldite $\text{\AA}$ 2022-1 was applied to both the surfaces in the hinge area through a constant dosage pistol with a mixing nozzle. This area is limited by the width of the specimens and a distance from the specimen's border (where the crack starts) of 30 mm. This adhesive is supplied by Huntsman Corporation (Texas, USA).
4. To provide a correct adhesion between the hinges and the specimen, it was necessary to wait at least 24 hours after the bonding at normal ambient condition of temperature as stated in the DIN EN 1465 standard.

The mechanical testing was executed using an MTS Universal Testing Machine, model C43.50, equipped with a 50 kN applied force capacity. All tests were conducted at a crosshead speed of 1 mm/min, which aligns with the lowest velocity recommended by the corresponding standard. To monitor the fracture tip, a Dino-Lite AM2111 traveling optical microscope with 200x magnification, mounted on a movable base mechanism, was employed. The configuration of the test specimen on the machine is illustrated in Fig. 2. 9.

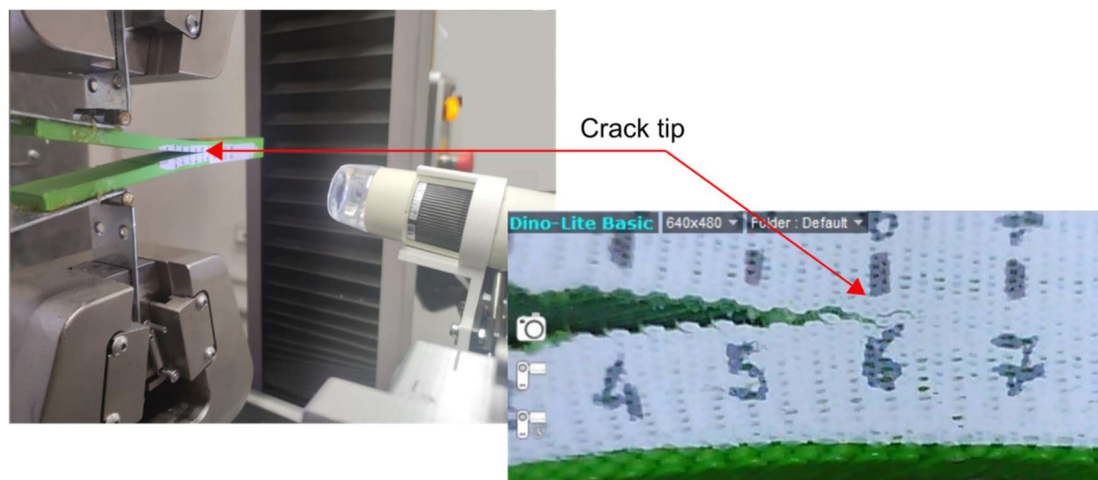


Fig. 2. 9: Picture of the adopted equipment during the execution of the mode I fracture toughness tests (testing machine, piano hinges, and the traveling microscope device).

The prescribed standard recommends splitting this mechanical test into two distinct phases. Specifically, the test was halted when the crack opening development, as recorded by the optical microscope, reached a value between 3 and 5 mm. As mentioned in the second step of the specimen setup, the distance between two consecutive marks in the initial five millimeters from the crack tip is 1 mm. Consequently, the specimen was completely unloaded at a crosshead speed of 8 mm/min. The objective of the first phase is to initiate the crack front and precisely determine the crack position. Additionally, it facilitates the elimination of any potential geometric imperfections introduced by the MEX process. In the second phase, the test was resumed at a constant crosshead displacement speed of 1 mm/min. Throughout this stage, the load applied by the machine on the specimen was recorded alongside the corresponding value of the crosshead displacement and the crack tip position. These three values were logged each time a crack front progression of 5 mm was observed through the optical microscope.

The critical energy release rate was determined using the VIS point technique. This method relies on visually identifying the initial crack motion through the microscope. Fig. 2. 10 a) illustrates two typical Load-Crosshead displacement diagrams for the two deposition strategies. The transparent plot represents the first

part of the test, while the sharper one corresponds to the crack propagation resulting from the movement of the mobile crosshead displacement. All the point-markers depicted on the propagation diagram signify a 5 mm increase in the crack length.

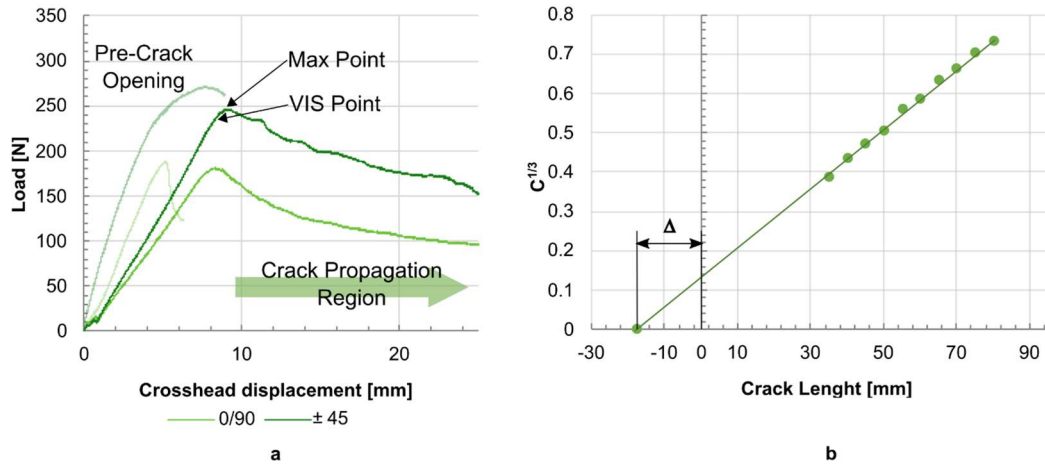


Fig. 2. 10: Typical load-crosshead displacement curve obtained by the execution of one of the tests (a) and defining the Δ factor for calculating the energy release rate using the modified beam theory (b)

The energy needed to spread a crack over a unit surface area is represented by the mode I strain energy release rate. The DCB test for an elastic linear construction is based on the change in compliance, C , which is determined by equation 4:

$$C = \frac{\delta}{P} \quad \text{Eq. 4)}$$

The loss of stored energy caused by the crack spreading from a value of a to $a + \Delta a$ causes a change in compliance. The beam theory assumes that the adherents are clamped at the crack tip and that there is little spinning in this area for calculating compliance. As a result, using the Irwin-Kies equation [28], which is given in eq. 5, it is possible to determine the strain energy release rate G_I :

$$G_I = \frac{P^2}{2b} \frac{\partial C}{\partial a} \quad \text{Eq. 5)}$$

where P stands for applied load, denotes machine crosshead displacement, b for specimen breadth, and the crack extension corresponding to applied load P and crosshead displacement. Thus, using eqs. 6 and the Euler-Bernoulli, it is possible to determine the compliance C , displacement, and moment of inertia I .

$$C = \frac{8a^3}{Ebh^3} \quad \delta = \frac{2Pa^3}{3EI} \quad I = \frac{bh^3}{12} \quad \text{Eq. 6)}$$

Thus, the strain release energy rate G_I is determined by equation 7:

$$G_I = \frac{3P}{2b} \quad \text{Eq. 7)}$$

The same reasoning suggests that the parameters of eq. 5 could be used to remedy any potential incorrect fracture front detection. According to eq. (8) the Modified Beam Theory approach was utilized to determine the energy release rate G_I :

$$G_I = \frac{3P\delta}{2b(\alpha + |\Delta|)} \quad \text{Eq. 8)}$$

the parameter Δ denotes the interception of the linear regression curve between the crack length and the cube root of C, as shown in Fig. 2. 10 c).

2.3 Tensile Tests

FDM parts are inherently anisotropic because filaments and layers create discrete interfaces whose quality governs the strength, durability, and failure mechanisms of the components [50,110]. When external loads tend to pull, bend, or open the part, cracks frequently nucleate and propagate along these interfaces. Raster orientation strongly affects this response: loading aligned with the deposited filaments (raster = 0°) mobilizes the bulk material, whereas loading transverse to the filaments (raster = 90°) stresses the bonded interfaces, leading to reduced strength, brittle fracture, and pronounced anisotropy, as depicted in Fig. 2. 11. Alternating raster orientations can mitigate in-plane anisotropy but cannot compensate for the weakness along the build (z) direction, making inter-layer adhesion the central focus of this study.

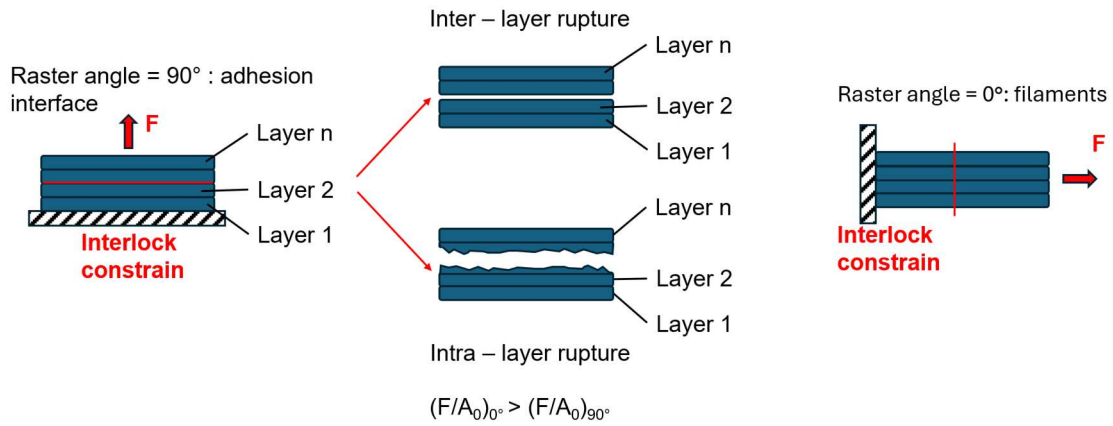


Fig. 2. 11: Schematic illustration of anisotropy and fracture mechanisms in FDM parts as a function of raster orientation under tensile loading. Loading aligned with the filaments (raster = 0°) promotes intra-layer failure governed by filament deformation, whereas loading transverse to the filaments (raster = 90°) concentrates stresses at inter-layer interfaces, leading to adhesion-controlled inter-layer rupture.

Moreover, the scheme depicted in Fig. 2. 11 allows a deeper understanding of the mechanism which overcome during a tensile test. In fact, it shows the differences between inter, and intra – layer fracture paths: inter – layer rupture is a common failure mode, especially when limited chain entanglement develops between consecutive layers. Fractography typically reveals a brittle mechanism. The crack runs along the bead – bead interface with minimal plastic deformation. Surfaces appear flat and glossy, often with mirror–mist–hackle regions. The path is relatively straight, with small step features and scarce evidence of filament necking or fibrillation. Ligament bridging is limited. These observations are consistent with low energy absorption at the interface. By contrast, intra – layer failures show rougher, matte surfaces and a more tortuous crack path through the filament. Evidence of plasticity is common: filament necking, craze whitening and fibrillation, shear bands, and pulled-out strands. Dimensional steps (“shear lips”) can appear near the specimen edges. Stretched polymer ligaments often bridge the crack wake before final separation. These features indicate higher local ductility and greater fracture energy within the filament material.

2.3.1 Material and sample preparation

The material, provided from RS PRO as a filament, boasting a starting diameter of $1.75 \text{ mm} \pm 0.2 \text{ mm}$. All the samples were produced through a Fused Deposition Modelling FDM 3D printer Creality Ender 6. Instead, to discretize the 3D models and realize the process g – code CAM slicer software as Simplify 3D and Prusa slicer. The present experimental campaign focuses on the assessment of layer interface adhesion during tensile tests. The study aims to investigate the influence of key process parameters on the mechanical behavior of printed components, with particular emphasis on the interlayer bonding quality, which plays a crucial role in determining the anisotropic mechanical properties of additively manufactured parts. Notably, another. All tensile specimens were designed in accordance with the ASTM D638 Type I [111] standard to ensure reproducibility and comparability of mechanical results. An image of the tensile test specimen employed for the characterization is depicted in Fig. 2. 12. The nominal thickness value of this specimen is 3.2 mm.

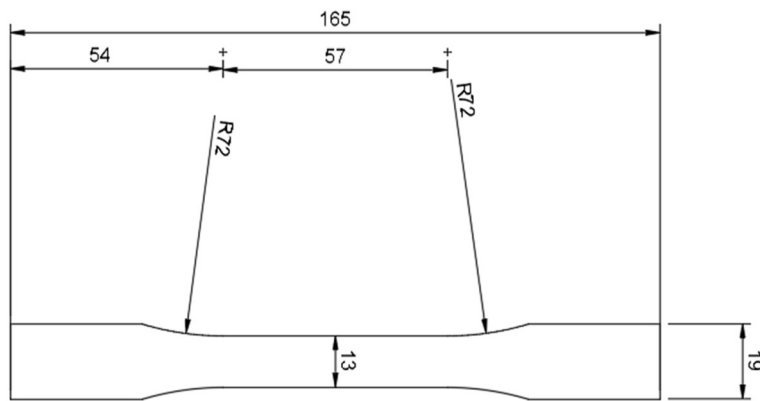


Fig. 2. 12: Tensile test geometry according to ASTM D638 Type I

The deposition parameters and characteristic temperatures used for PLA filaments in the following procedures are listed in Tab. II. 4:. The overall workflow, shown in Fig. 2. 13, encompasses the stages of specimen fabrication, process monitoring, sample preparation, and mechanical characterization, accompanied by multiple levels of microstructural and thermal analysis.

Tab. II. 4: Printing and deposition parameters adopted for PLA specimens manufactured by FDM, including thermal settings, infill configuration, and extrusion conditions.

Process Parameter	Value
Nozzle Temperature [°C]	210
Building Plate Temperature [°C]	60
Infill Percentage [%]	100
Infill Strategy	Rectilinear
Number of External Shell	0
Extrusion Multiplier [%]	100
Cooling Fan Speed [%]	100

Material: PLA
3D printer: Creality Ender 6

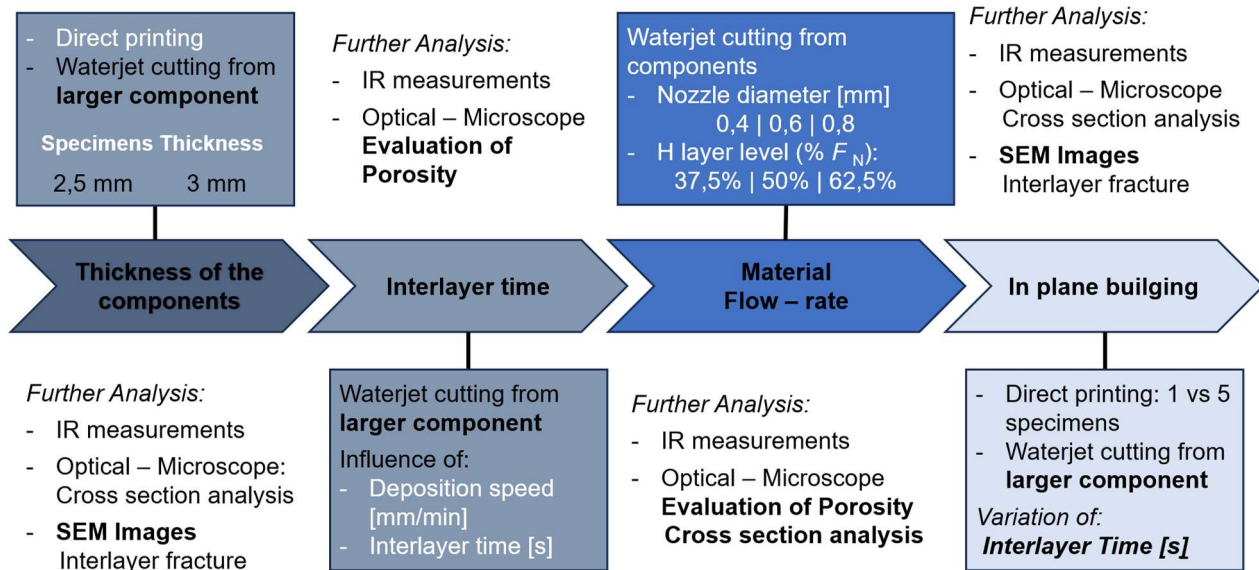


Fig. 2. 13: Schematic overview of the experimental workflow, process parameters, and characterization techniques used for PLA specimens fabricated by FDM

So far, the characterization of materials used for FDM has typically been carried out by printing the geometry of the test specimen only. While this methodology employs a dual aspect. In fact, the production of larger components with the simple geometry of plates or boxes avoids the side effects that are produced through direct printing of the samples. Moreover, thermal history and the cooling rates have a fundamental impact on the adhesion between the following layers, impacting on the mechanical performance of the component.

2.3.1.1 Different fabrication strategies, upright building:

- **Direct printing** of tensile specimens individually.
- **Sample extraction through waterjet cutting** of specimens from larger, previously printed plates. To this end, a box with side walls 4 mm of thickness was printed. Then, the samples were cut from the box. Waterjet cutting was used to cut the samples to avoid any material modification since no heating was produced during the cutting operation. This choice was supported by Alberdi [39], who demonstrated the advantages of abrasive waterjet cutting for polymer composites, highlighting its ability to produce high-quality cuts without thermal damage.

Furthermore, the nominal thickness of the specimens and of the massive components was varied. In this regard, two values were employed: respectively, 2.5 and 3.0 mm.

This dual approach enables the evaluation of potential differences in layer adhesion due to part geometry, cooling rates, and localized process variations during printing. This comparison highlights the effect of thermal history and build geometry on the final bonding strength. Besides, the strategy of production of the specimens and the thickness variation are enumerated in **Errore. L'origine riferimento non è stata trovata..**

A total number of 4 specimens were tested for each condition.

Tab. II. 5: Test matrix, summary of the experimental conditions for specimens or components realized with different thickness

Testing standard: ASTM D638 T1	
Sample manufacturing	Thickness [mm]
Direct sample printing	2.5
Waterjet cutting from printed component	3.0

Fig. 2. 14 illustrates the two different fabrication strategies adopted for the preparation of PLA tensile specimens used in the layer interface adhesion study.

Fig. 2. 14 a): Directly Printed Specimens

In the first configuration (Fig. 2. 14 a), tensile specimens were directly printed on the build platform. The models were oriented vertically, with the printing direction parallel to the tensile load axis, to promote interlayer bonding representative of typical structural printing conditions. Two nominal specimen thicknesses were investigated: 2.5 mm and 3.0 mm. This variation was introduced to assess the effect of component thickness on cooling dynamics and the subsequent bonding quality between adjacent layers. Directly printed specimens serve as a reference condition, where the layer deposition and consolidation occur without additional post-processing or mechanical extraction.

Fig. 2. 14 b): Rectangular Printed Box

This approach allowed a more controlled investigation of the interlayer time and thermal gradients occurring within bulk printed structures. The box dimensions were selected to ensure consistent thermal accumulation across layers, reproducing a representative in-situ environment for larger printed parts.

Fig. 2. 14 c): Extraction of Tensile Specimens

After fabrication, the tensile specimens were cut from the rectangular box using a waterjet cutting system, as shown in Fig. 2. 14 c).

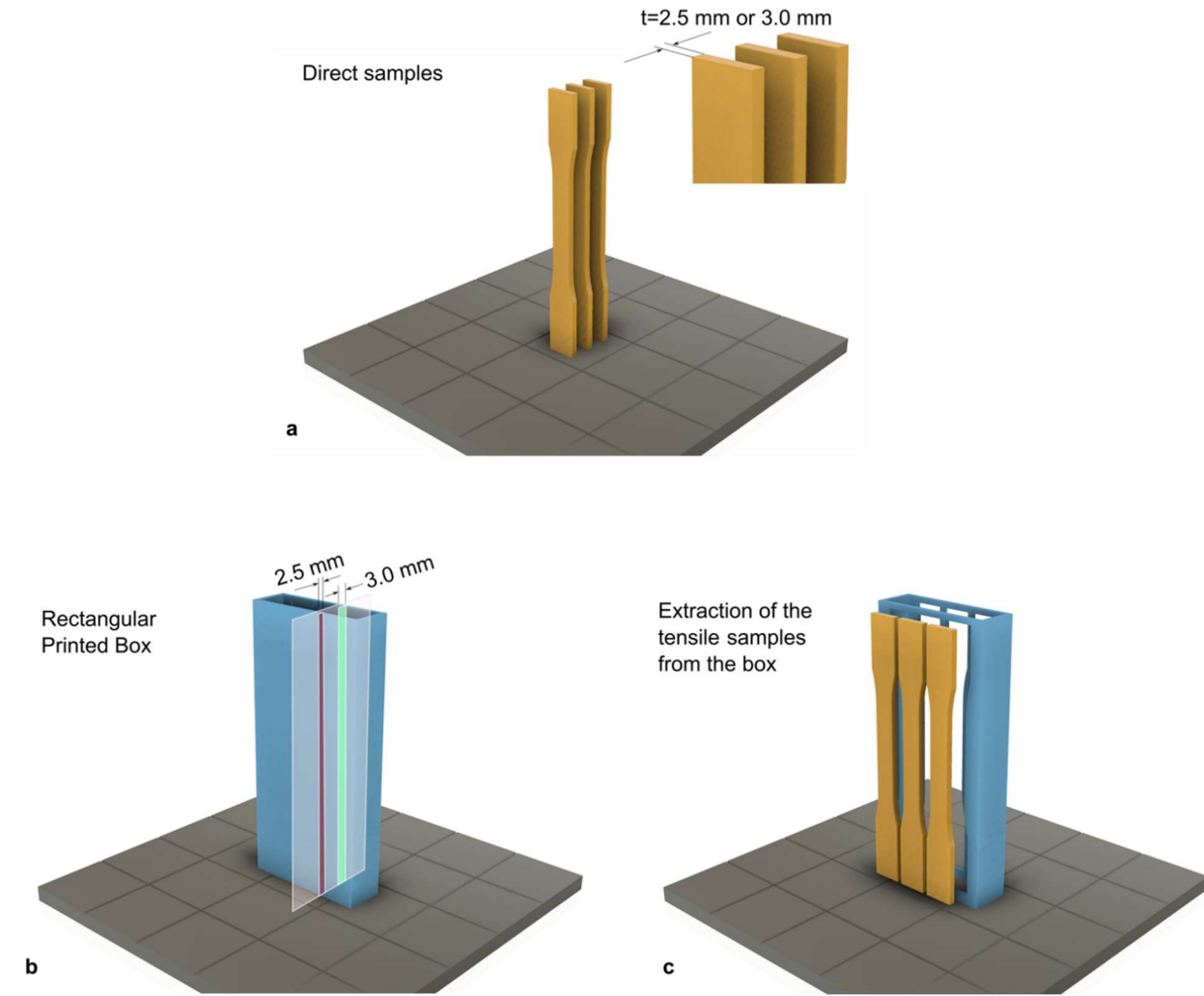


Fig. 2. 14: Schematic of the sample produced using different approaches: a) direct sample printing, b) rectangular box printing, and c) extraction of the specimens from a massive component

This non-thermal cutting technique was selected to prevent any alteration of the printed microstructure and to preserve the interlayer morphology. The extraction process enabled the preparation of specimens having the same geometry as the directly printed ones but with distinct thermal histories and interlayer consolidation characteristics due to their position within the larger printed component. The comparison between the directly printed and extracted samples thus provides valuable insight into the effects of printing strategy, cooling rate, and process continuity on the layer interface adhesion strength. The internal regions of the printed box correspond to the volume where the tensile samples were later isolated, ensuring identical printing parameters but different thermal boundary conditions compared to the directly printed specimens.

2.3.1.2 Influence of the interlayer time, upright building

Subsequently, controlled by adjusting deposition speed and printing sequence, the interlayer time defines the thermal exposure between successive layers. Samples were obtained by sectioning larger plates, allowing the analysis of different interlayer times without altering other parameters. In this regard, the deposition speed plays a key role in determining the thermal history. Moreover, the deposition speed significantly impacts interlayer bonding and, consequently, the overall quality of the printed material. The deposition strategy employed linear paths along the x-direction.

As illustrated in **Errore. L'origine riferimento non è stata trovata.** b), a full factorial experimental plan was conducted by varying the deposition speed and the pause time (through the adoption of G04 command) between consecutive layers. The pause time between the layers was involved as a process parameter to simulate the effect of printing of larger components as compared to smaller ones. Indeed, the time required to complete a layer (t_l) can be calculated as in Eq. 9):

$$t_l = \frac{A_l}{w \cdot v} \quad \text{Eq.9)}$$

where A_l is the area of the layer, w is the deposited filament width, and v is the deposition speed. This relationship, which neglects the influence of accelerations and decelerations, indicates that t_l is directly proportional to the layer dimension (A_l) and inversely proportional to the deposition speed (v). During MEX process, the adhesion behavior of consecutive layers is due to the entanglement of the polymeric chains. This is facilitated at higher temperatures in correspondence of which higher mobility of the polymeric chains is achieved. On the other hand, at higher temperatures, crystallization may occur owing to moderate cooling after melting and deposition. This also depends on the temperature of the substrate which influences the cooling rates after deposition. Thus, to investigate the effect of the thermal history on the mechanical behavior of MEX components, both the deposition speed and the interlayer time were varied. A full factorial experimental plan was carried out fixing the deposition speed and the interlayer time. To this end, some pauses between consecutive layers were opportunely designed with the aim of achieving the predefined interlayer time. The resulting experimental plan is summarized in Tab. II. 6.

Tab. II. 6: Summary of the experimental plan of the interlayer time employed to produce massive components

Condition	Deposition speed [mm/min]	Interlayer Time [s]
I	2000	21
II	2000	63
III	2000	105
IV	3000	21
V	3000	63
VI	3000	105
VII	4000	21
VIII	4000	63
IX	4000	105

Three deposition speed levels were chosen ranging from 2000 to 4000 mm/min, which are commonly utilized for PLA materials. Interlayer time selection involved setting 21 s as the lowest level, corresponding to the time required for layer production at the minimum speed. The highest level, 105 s, represents a component

five times larger, necessitating an interlayer time five times longer than the minimum. For each deposition condition, six samples were produced.

2.3.1.3 . INFLUENCE OF FLOWRATE DURING THE MEX PROCESS, UPRIGHT BUILDING

Furthermore, the characterization methodologies included the use of different nozzles and flow rate during the MEX process. The components produced are plates. The walls have a thickness of 3 mm, while the two major dimensions are 90 mm in width (along X axis) and 180 mm height (Z axis) respectively. From each plate, three samples were cut, as depicted in Fig. 2. 15 a), and six repetitions were performed for each process condition. The full experimental factorial plan is listed below in Tab. II. 7.

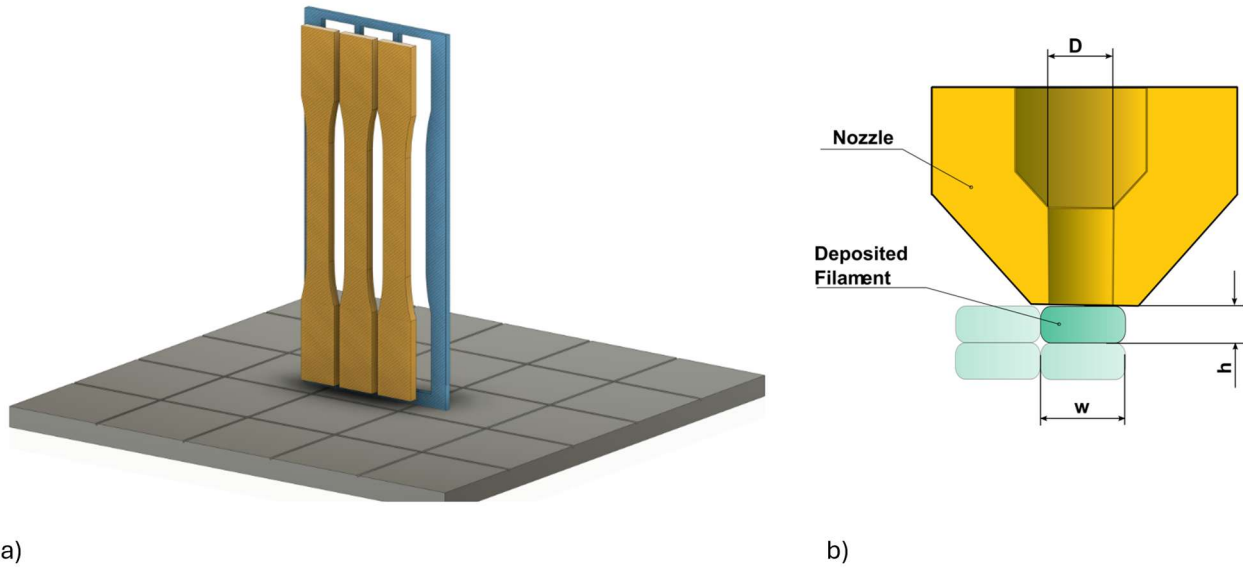


Fig. 2. 15: Influence of material flow rate during upright MEX fabrication: **(a)** schematic representation of the upright-printed plate and extraction of tensile specimens by waterjet cutting; **(b)** schematic of the deposited filament, highlighting the relationship between nozzle diameter (D), layer height (h), and bead width (w) used to define the volumetric flow rate.

Tab. II. 7: Deposition conditions adopted for the experimental investigation of material flow rate during upright MEX printing, including nozzle diameter, layer height (defined as $h = \alpha \cdot D$), percentage factor α , and deposition speed.

Percentage factor, α [% D]	37.5%	50%	62.5%
Nozzle Diameter, D [mm]	Layer height [mm]		
0.4	0.15	0.20	0.25
0.6	0.22	0.30	0.40
0.8	0.30	0.40	0.50
Deposition speed [mm/min]	2000		

As can be inferred, the layer height reported in Table 2 is calculated as the product of the nozzle diameter by the percentage factor α . A schematic representation of the deposited bead is shown in Fig. 2. 15 b).

Based on the deposition speed (v), the nozzle diameter (D) and the percentage factor (α) the material (volumetric) flow rate (F_r) can be calculated as:

$$F_r[mm^3/s] = \frac{v}{60} A = \frac{v}{60} (D \times 1.2 \times h) = \frac{v}{60} (D \times 1.2 \times \alpha \times D) = \frac{v}{50} D^2 \alpha \quad \text{Eq. 10}$$

where the cross section of the filament (A) can be calculated as the product of the width ($w=1.2 \times D$) by the layer height ($h= \alpha \times D$).

2.3.1.4 Influence of the component dimensions, in – plane building:

Test specimens were fabricated following ASTM D638 [111] Type IV geometry specifications. Three distinct manufacturing strategies were employed:

- 1) Type S - Single-specimen fabrication: direct printing of individual specimens (repeated five times);
- 2) Type M - Multi-specimen fabrication: simultaneous printing of five specimens;
- 3) Type C - Extracted specimens: waterjet cutting of test specimens from a larger printed plate

The manufactured MEX specimens' production is depicted in Fig. 2. 16. The experimental design was structured to investigate two critical temporal parameters in the MEX process:

1. The interlayer time (duration to complete a single layer) and the interfilamentous time (interval between adjacent filament depositions).
2. The interfilamentous time can be approximated, disregarding acceleration and deceleration effects, as the ratio between the layer's transverse dimension and the deposition speed.

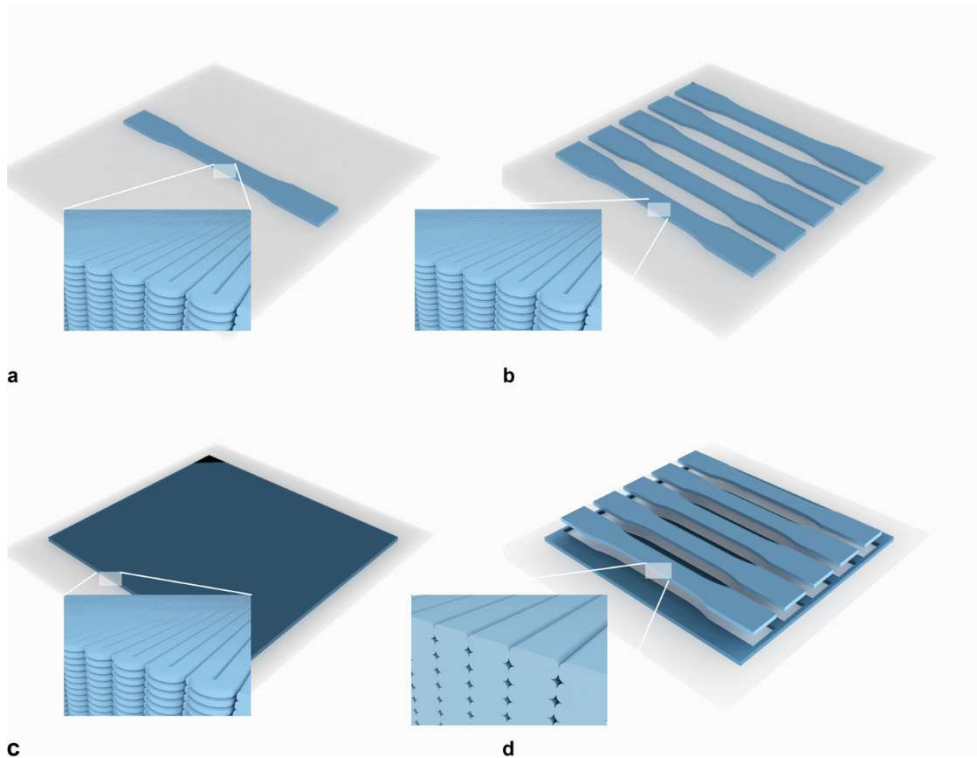


Fig. 2. 16: Schematic of the **flat** sample produced using different approaches: a) direct sample printing of single sample, b) direct sample printing of multiple samples; c) plate printing. c) extraction (through abrasive waterjet cutting) of tensile samples from the PLA.

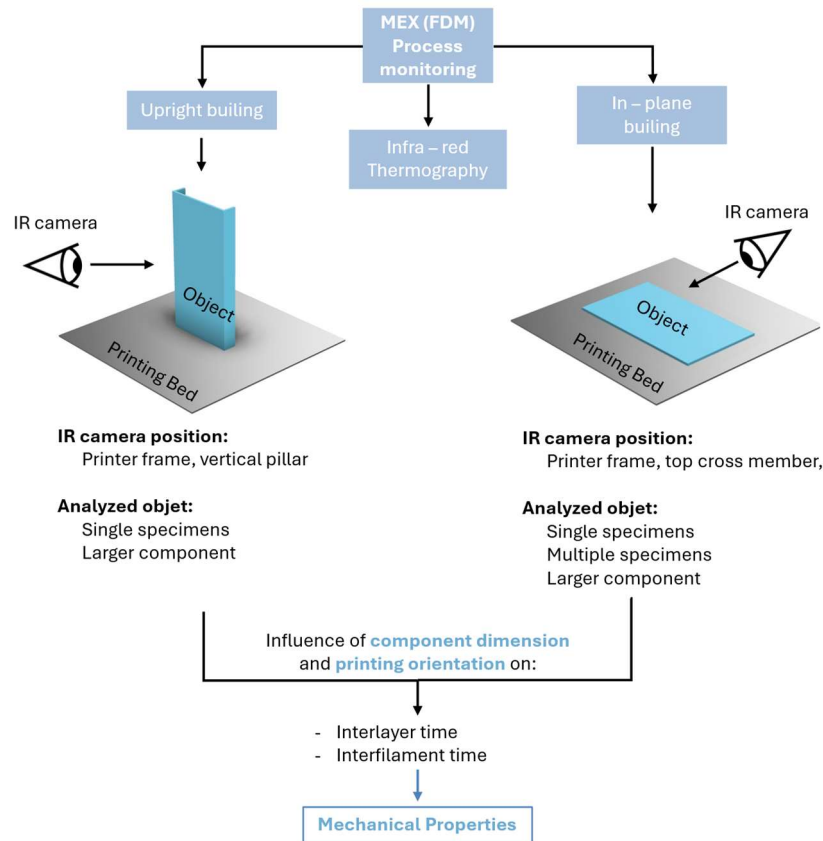
These manufacturing strategies created distinct thermal conditions:

- Strategies (S) and (M) maintained comparable interfilamentous times but exhibited significantly different interlayer times, with strategy (M) requiring approximately five times longer to complete each layer compared to strategy (S).
- Strategies (M) and (C) demonstrated similar interlayer times due to comparable layer areas, but markedly different interfilamentous times: 0.4 s for strategy (M) versus 3.6 s for strategy (C).

This systematic variation in temporal parameters enabled a comprehensive analysis of their influence on the final component properties. The distinct thermal histories resulting from these different time scales were expected to significantly impact polymer chain entanglement and, consequently, the mechanical properties of the printed components. The speed of the hot – end during the FDM process is 4000 mm/min.

2.3.2 Process Monitoring

Infrared thermography was employed to monitor the Material Extrusion (MEX/FDM) process in situ and to quantify the thermal history experienced by the deposited material under different printing conditions. Fig. 2. 17 schematically illustrates the two printing configurations investigated: upright building (vertical orientation) and in-plane building (horizontal orientation). In both cases, the infrared camera was rigidly mounted on the printer frame to enable non-contact, real-time acquisition of temperature fields during



deposition.

Fig. 2. 17: IR thermography setup for in-situ monitoring of the MEX/FDM process in upright and in-plane printing configurations.

For the upright configuration, the camera was positioned along the vertical pillar, monitoring single specimens and larger components. For the in-plane setup, the camera was mounted on the top cross member, observing single, multiple, or large specimens during deposition. This monitoring approach enables the assessment of the influence of component dimension and printing orientation on interlayer and interfilamentous times, which directly affect the mechanical properties of the printed PLA parts.

The mechanical properties of samples produced via the MEX process are intricately influenced by material viscosity (during the extrusion), polymer chain entanglement, and possible crystallization of the material. As mentioned earlier, while maintaining all the process parameters constant, the geometry and dimensions of the component (sample) significantly impact the temperature history. Consequently, thermal analysis was performed to uncover how sample dimensions affect the thermal history of different manufacturing strategies.

In fact, thermal analysis was conducted to elucidate how varying the deposition speed and the pause time affects the thermal history. Additionally, the potential development of edge effects during the printing process, which could impact sample characteristics, was investigated. Thus, infrared camera was involved in the MEX process for real-time temperature distribution monitoring. In this regard, two strategies of clamping of the camera have been employed: in Fig. 2. 18 the thermal camera model FLIR E40 was positioned adjacent to the specimen to record the thermal cycles occurring during printing. The thermal camera was securely mounted on the machine frame, enabling continuous temperature monitoring throughout the deposition process. This measurement configuration allows to understand the temperature field during the deposition. In fact, the R.O.I. (blue trigger) depicted in Fig. 2. 18 is centered on the extruded material level. It allows the measurement of the temperature and its cooling after the deposition during the entire interlayer time to identify possible material crystallization.

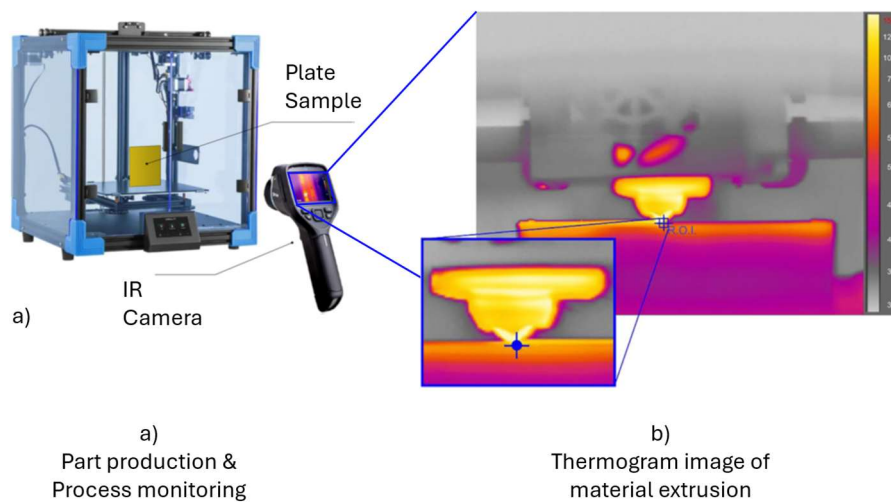


Fig. 2. 18: MEX process monitoring through Infra - red thermography; b) In - situ thermogram acquisition to the MEX process of a large part: upright building

In the upright configuration, the camera was positioned along the vertical pillar of the printer frame, allowing the monitoring of single specimens as well as larger components printed along the build direction. In the in-plane configuration, the camera was mounted on the top cross member of the printer, observing components printed flat on the build plate. This dual setup enables the investigation of how printing orientation and component dimensions influence interlayer time and interfilamentous time, which are key parameters governing interlayer bonding, polymer chain diffusion, and ultimately the mechanical performance of FDM parts.

The mechanical response of material-extruded polymers is strongly affected by the thermal state of the extruded filament during deposition. Material viscosity, polymer chain mobility and entanglement, and, in the case of semicrystalline polymers, the onset of crystallization are all controlled by the local temperature evolution. Even when nominal process parameters such as nozzle temperature, bed temperature and deposition speed are kept constant, the geometry and size of the printed component can significantly alter the thermal history due to differences in heat accumulation, cooling rate, and exposure time between successive layers.

For this reason, infrared thermography was used to systematically assess the influence of component dimensions, deposition strategy, and pause time on the temperature evolution of the deposited material. Particular attention was devoted to identifying edge effects and non-uniform cooling conditions, which may locally reduce interlayer adhesion and introduce variability in mechanical properties.

Fig. 2. 18 shows an example of in situ thermographic acquisition during material extrusion. The infrared camera (FLIR E40) was positioned adjacent to the specimen, and a region of interest (ROI) was defined at the height of the extruder. This ROI allows tracking the peak temperature immediately after filament deposition and its subsequent decay during the interlayer time. The temperature decay rate provides direct information on cooling kinetics and potential crystallization phenomena. The acquisition frequency was set to the maximum available value (30 Hz) to ensure sufficient temporal resolution. The recorded thermal data were post-processed using ResearchIR software.

In the second configuration, shown in Fig. 2. 19, a higher-performance infrared camera (FLIR A615) was mounted on the upper cross member of the printer frame to monitor in-plane printing. In this case, the acquisition frequency was increased to 100 Hz to capture rapid thermal transients during deposition. Temperature fields were analyzed using ResearchIR and further processed in MATLAB to extract quantitative indicators of interlayer and interfilamentous thermal conditions.

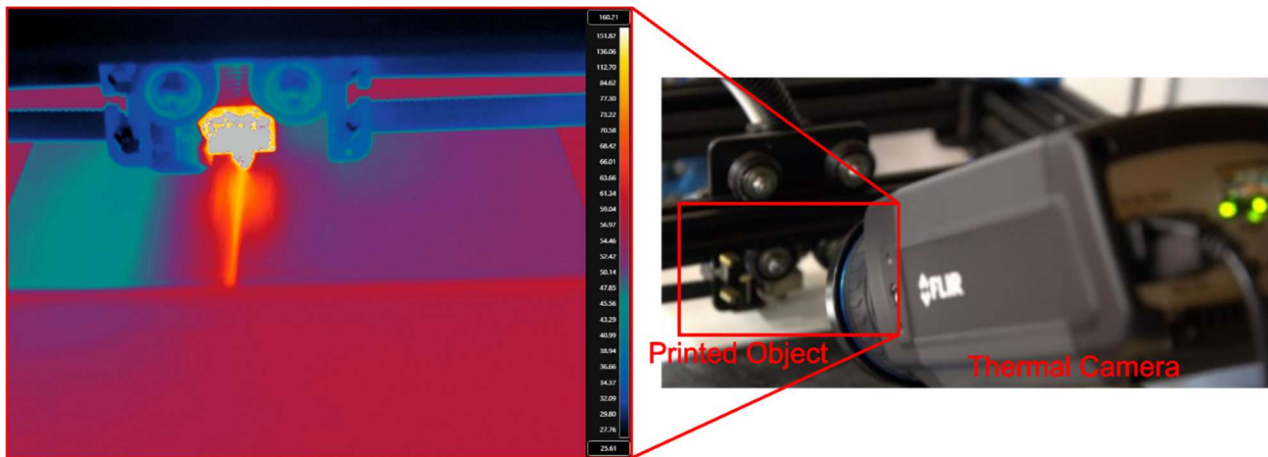


Fig. 2. 19: Picture of the IR thermal measurement during the MatExt process. Camera placed on the top – frame of the 3D printer to measure temperature during the flat in – plane deposition

In both configurations, thermal monitoring was performed on single specimens as well as on larger printed plates. These larger components were subsequently sectioned using abrasive waterjet machining to obtain tensile specimens. This approach enables a direct correlation between the thermal history measured during printing and the mechanical performance measured on specimens extracted from different regions of the same component.

Overall, the infrared thermography-based process monitoring provides a quantitative link between printing strategy, component geometry, and thermal history. This information is essential to interpret the observed variations in interlayer adhesion and fracture behavior, and to rationalize the mechanical response of FDM-printed polymers in terms of process-induced thermal signatures.

2.4 Tensile Tests: A Thales Alenia Space case of Study

This study delves into the manufacturing strategies employed for fabricating tensile samples utilized in the mechanical characterization of material extrusion (MEX) components constructed with polyaryletherketone (PAEK) for aerospace applications. As in the four previous works which use PLA, two distinct methods were investigated for obtaining tensile test samples: direct cutting and extraction from a box. These methods were examined under both as-printed and annealing conditions. Quasistatic tensile tests were conducted along the building direction to evaluate the impact of processing conditions on the adhesion of overlying layers. The results unveiled significant disparities in mechanical behavior and crystallinity between directly printed samples and those derived from the box. Polyaryletherketone (PAEK), provided by VICTREX, was utilized for the study, boasting an initial diameter of $1.75 \text{ mm} \pm 0.02 \text{ mm}$. The production of the samples was carried out using an industrial machine model Spectral 30 by 3ntr (from Novara, Italy). The critical deposition parameters, as listed in Tab. II. 8, influence the interlayer bonding behavior, surface roughness, and dimensional precision. Key factors, such as nozzle temperature, layer thickness, building plate temperature, extrusion multiplier, infill percentage, and nozzle speed, directly govern the thermal history, thereby influencing interlayer bonding and overall print quality. The samples were manufactured according to ASTM D638 [111] standards Type I.

Tab. II. 8: Deposition and processing parameters adopted for the fabrication of PAEK tensile specimens by material extrusion (MEX) using an industrial Spectral 30 printer, in accordance with ASTM D638 Type I geometry.

Process parameter	Value
Printer primary extruder temperature [°C]	398
Printer secondary extruder temperature [°C]	280
Printer plate temperature [°C]	120
Printer chamber temperature [°C]	120
Hotend speed [mm/min]	3600
Nozzle diameter [mm]	0.4
Extrusion width [mm]	0.5
Layer thickness [mm]	0.2
Infill percentage [%]	100
Infill strategy	Linear
Number of perimeter shells	4
Volumetric material flow [mm ³ /min]	360

Following the approach used in the previous works regarding, the specimens are produced according to the direct printing vs sample extraction through AWJ protocols. This approach allows to isolate the effects of the manufacturing strategy on the mechanical properties of PAEK components. In both cases, the entire thickness of the sample (of 4 mm) was filled by the external shells. Indeed, each shell was 0.5 mm wide; consequently, at least in the central region, the samples cut from the box and directly printed were produced with the same strategy. Of course, some differences existed in the side regions. Indeed, the cut samples were characterized

by filaments arranged along one direction, while directly printed samples showed a concentric shell strategy, as schematized in Fig. 2. 20.

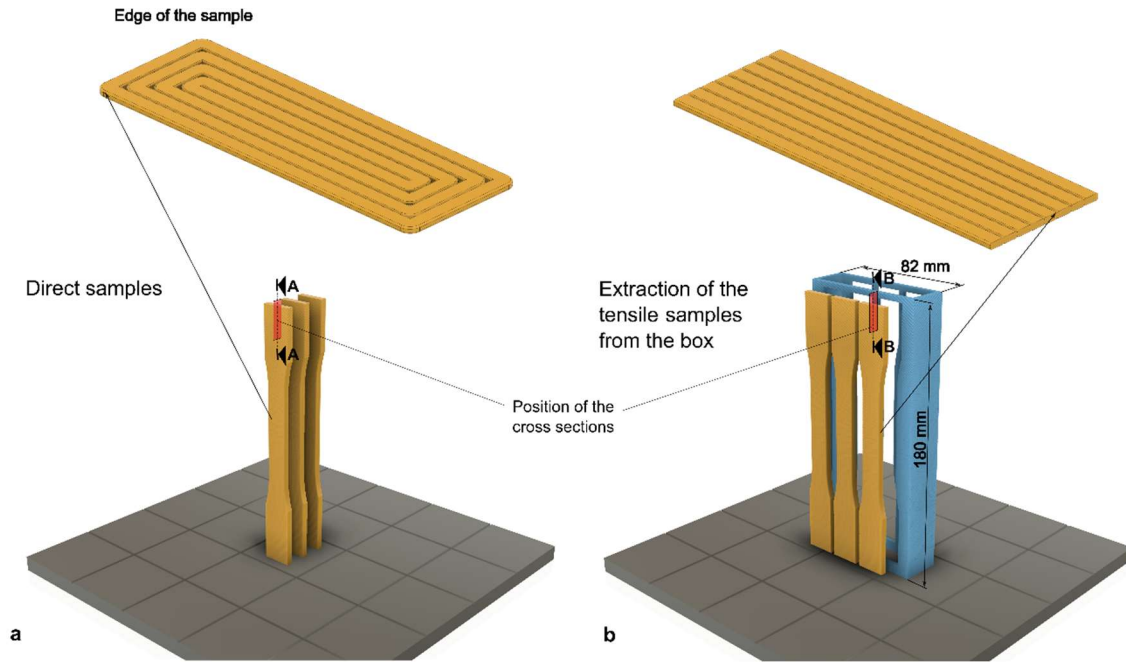


Fig. 2. 20: Manufacturing strategies adopted for PAEK tensile specimens: (a) directly printed specimens produced with a concentric shell deposition strategy; (b) specimens extracted by abrasive waterjet cutting (AWJ) from a printed box, characterized by unidirectional filament arrangement in the side regions. The figure also indicates the position of the analyzed cross sections. In both cases, the full specimen thickness is filled by external shells, ensuring comparable deposition strategies in the central region.

Additionally, some specimens were post-processed by annealing; Fig. 2. 21 shows the thermal cycle that the specimens were subjected during the annealing process. The test matrix of the experimental campaign is summarized in Tab. II. 9.

Tab. II. 9: Test matrix summarizing the experimental conditions used for the experimental investigation for the realization of tensile test PAEK specimens

Manufacturing	Post-processing
Direct print – AP	As printed
Direct print – A	Annealed
Cut from the box – AP	As printed
Cut from the box – A	Annealed

For each processing condition, five samples were produced. Thus, the mean value and standard deviation were computed. Optical microscopy served as a valuable tool in understanding the material's morphology both pre- and postmechanical tests. To this end, a stereoscope model M205 by LEICA was used to determine the real dimension of the cross section, the surface morphology, and the analysis of the fracture surface. Calorimetric analyses were conducted by differential scanning calorimetry (TA instrument DSC 8500) with 20 °C/ min heating/cooling rate. Heating was conducted from 25 to 350 °C.

Crystallinity ratio χ was determined for all the samples from the melting enthalpy ΔH_m according to Eq. 11.

$$\chi(\%) = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_{\infty}} \quad \text{Eq. 11}$$

where ΔH_{cc} is the cold crystallization enthalpy and ΔH_{∞} is the theoretical melting enthalpy of a 100% crystalline polymer. To this end, the ΔH_{∞} for PEEK was used which equals 130 J g^{-1} [40].

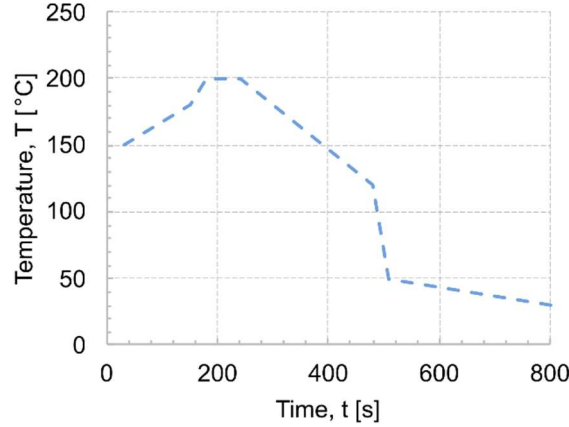


Fig. 2. 21: Annealing treatment for PAEK tensile specimens

2.4.1 Characterization processes

2.4.1.1 Mechanical Characterization

Before mechanical characterization, differential scanning calorimetry (DSC, TA Instruments, USA) was employed for calorimetric analyses, using a heating/cooling rate of $10 \text{ }^{\circ}\text{C/min}$. The procedure consisted of three stages: an initial heating phase from 25 to $190 \text{ }^{\circ}\text{C}$, followed by a 5-minute annealing period, then a cooling phase from $190 \text{ }^{\circ}\text{C}$ to $25 \text{ }^{\circ}\text{C}$, and finally a second heating cycle.

Furthermore, the samples were measured to ensure the proper dimension and tolerance.

Tensile testing was conducted using a universal testing machine (Model C43.50) equipped with a 50 kN load cell. Tests were performed at a constant crosshead speed of 2 mm/min until complete specimen failure. Strain measurements were acquired using an MTS extensometer (Model 632.24F-50) to determine the Young's modulus.

The tensile test configuration is illustrated in Fig. 2. 22.

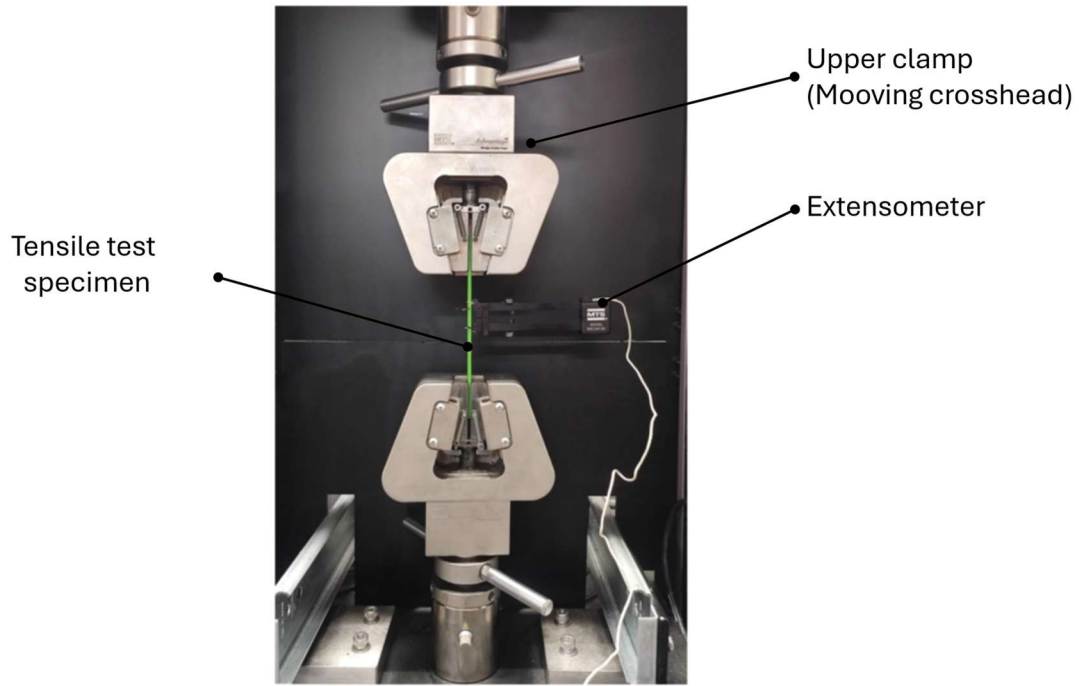


Fig. 2. 22: Mechanical tensile test through MTS Criterion model C43.50

$$\sigma = \frac{F}{A_0} \text{ [MPa]} \quad \text{Eq. 12}$$

The mechanical characteristic of **engineering stress** is calculated through Eq. 12. In fact, the measured force during the tensile test F is divided by the starting cross section value A_0 . Moreover, the **engineering strain** is calculated dividing the value of the extensometer displacement ($\Delta L - L_0$) by the starting length of the closed extensometer, which is 25 mm, as highlighted in Eq. 13.

$$\varepsilon = \frac{\Delta L - L_0}{L_0} \quad \text{Eq. 13}$$

The mechanical properties evaluated after the data analysis are:

- Elastic Modulus, measured in GPa: slope of the stress – strain diagram in the elastic region (first linear part of the curve).
- Tensile strength measured in MPa: maximum value of stress during the tensile test.
- Elongation at break (a – dimensional): strain value in correspondence of the final rupture of the specimen.

2.5 Compression characterization of Lattice Structures

2.5.1 FDM Printing of Lattice Structures

In this section, the compressive behavior of architected lattice structures manufactured by different additive manufacturing technologies was investigated. The aim is to assess their mechanical performance, anisotropy, and energy absorption capability. Two main additive manufacturing routes were considered: material extrusion-based Fused Deposition Modeling (FDM) and powder-bed fusion via Multi Jet Fusion (MJF).

For the FDM process, lattice structures were produced using two polymer systems with distinct mechanical and thermal characteristics: poly-ether-ether ketone (PEEK) and polyamide 6 reinforced with short carbon fibers (PA6-CF). These materials are representative of high-performance and structural-grade polymers commonly employed in industrial applications. In both cases, the influence of lattice topology and relative density on the compressive response was systematically investigated. For PEEK, a full factorial experimental plan was defined, combining three relative densities with three infill strategies, in order to explore the structure–property relationship across a wide design space.

PA6-CF was selected as a technologically relevant composite filament widely used for lightweight structural components and energy-absorbing elements, such as crush boxes. Similarly to PEEK, three lattice topologies and three relative densities were considered. In addition, compression tests were performed at different loading speeds to evaluate the strain-rate sensitivity of the material. Process-induced anisotropy was also quantified by testing cubic specimens loaded both along the build direction and perpendicular to it, highlighting the role of layer-wise deposition on mechanical performance.

In parallel, elastomeric lattice structures manufactured by Multi Jet Fusion were investigated using thermoplastic polyurethane (TPU). In this case, truss-based lattice architectures were selected to exploit the intrinsic compliance and high energy absorption capability of the material. Two initial lattice topologies were evaluated, and the most promising configuration was identified based on its ability to sustain large deformations and absorb energy prior to densification. For this selected topology, the influence of loading speed was analyzed, and cyclic compression tests were performed to assess repeatability and mechanical stability under multiple loading cycles.

Finally, the results obtained from the MJF TPU lattice characterization were transferred to an industrially relevant application, namely the design and fabrication of a shock absorbing sport device. This case study demonstrates how systematic compression testing of lattice structures can directly inform design choices for functional components, bridging experimental characterization and real-world applications.

2.5.1.1 PEEK & PA6CF: FDM Process

INTAMSYS provides the PEEK raw material utilized. The initial diameter of the filament is $1.75 \text{ mm} \pm 0.02 \text{ mm}$. The specimens were fabricated via fused deposition modeling (FDM), using an INTAMSYS FUNMATH 410 PRO machine. Instead, PA6CF lattice structure were produced using Bambulab X1E FDM printer. An image of the two 3D printers is illustrated in Fig. 2. 23 a) represents an image of the Bambulab X1E printer with a cubic specimen printed through PA6-CF. While, Fig. 2. 23 b) shows a picture of the PEEK specimens inside the INTAMSYS 3D printer before the annealing treatment.

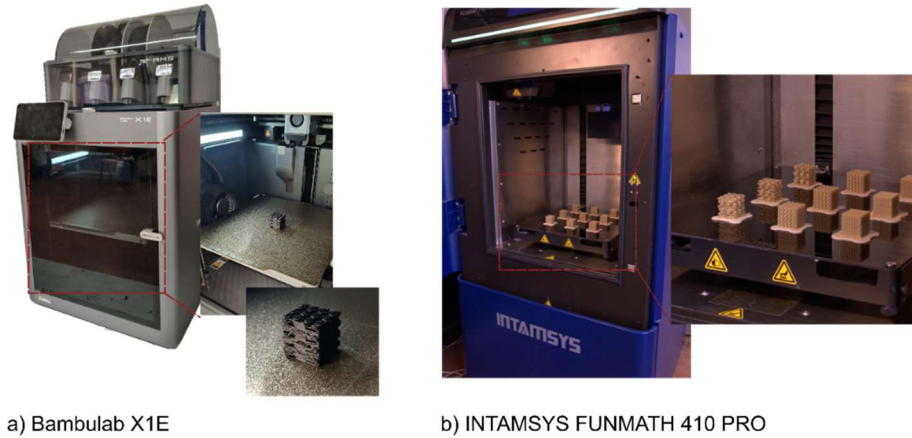


Fig. 2. 23: FDM processing of PA6–CF and PEEK compression lattice specimens. (a) Bambu Lab X1E FDM printer used for the fabrication of PA6–CF lattice structures, with a representative cubic specimen shown on the build plate. (b) INTAMSYS FUNMAT PRO 410 FDM system employed for PEEK processing, showing the printed specimens inside the heated build chamber before annealing.

Deposition parameters are enumerated in Tab. II. 10 for the FDM deposition process for the two materials.

Tab. II. 10: FDM process parameters adopted for the fabrication of PEEK and PA6–CF specimens, including extrusion temperatures, build plate and chamber temperatures, deposition speed, geometric settings, and build plate adhesion strategies.

	PEEK	PA6 CF
Process parameter	Value	Value
Left nozzle temperature [°C]	440	275
Right nozzle temperature [°C]	300	/
Building plate temperature [°C]	130	100
Building chamber temperature [°C]	90	60
Hot-end speed [mm/min]	3600	16200
Nozzle diameter [mm]	0.40	0.4
Extrusion width [mm]	0.40	0.42
Layer thickness [mm]	0.15	0.12
Number of external and perimeter shells	0	0
Extrusion multiplier	100%	100%
Build plate adhesion strategy	Raft	None

These parameters were determined based on the material manufacturer's recommendations and preliminary optimization trials. To mitigate warping and enhance interlayer adhesion, the build plate was maintained at 130°C and 100 °C, and the enclosed build chamber was temperature-controlled at 90°C and 60 °C to minimize thermal gradients and reduce residual stress. A layer thickness of 0.15 and 0.12 mm was chosen respectively for PEEK and PA6 CF to balance print resolution with manufacturing efficiency. For both materials a drying treatment is required before the printing itself. Especially PA6 CF is an hygroscopic material. For this reason the PA6 CF filament rowing was dried within a Sunflu Filadrier S2 at the temperature of 70 °C for 8 hours as recommended by the supplier. PEEK filament, on the other side, requires a drying treatment at the temperature of 150 °C for 12 hours using an industrial oven Memert UF160PLUS.

Three distinct infill densities (10%, 20%, and 30%) were tested to determine its influence on the compressive performance. These densities were achieved by varying the dimension of the unit cell, while keeping constant the thickness of the wall. Such characteristics are strictly connected to the adoption of FDM technology which does not enable (with the adopted nozzle diameter of 0.4 mm) wall thickness (extrusion width) smaller than 0.40 mm. Additionally, three infill patterns were employed to investigate their influence on the mechanical behavior under compressive loading for the two materials. This approach followed a factorial experimental design to isolate the effects of infill density and strategy. To ensure adhesion and minimize warping, a raft structure was printed using SP5000, as a support material, via the printer's dual-nozzle system. This strategy eliminated mechanical removal stresses that could damage the PEEK specimen. Conversely PA6CF does not require a support material. Indeed, 2 layers of raft with an offset of 2.5 mm is enough to guarantee the correct adhesion of the part with the printing bed.

The cubic samples (25 mm edge length) were manufactured in accordance with ISO 604:2002 [112] ("Determination of Compressive Properties"). A schematic of the three types of structures fabricated with PEEK is depicted in Fig. 2. 24.

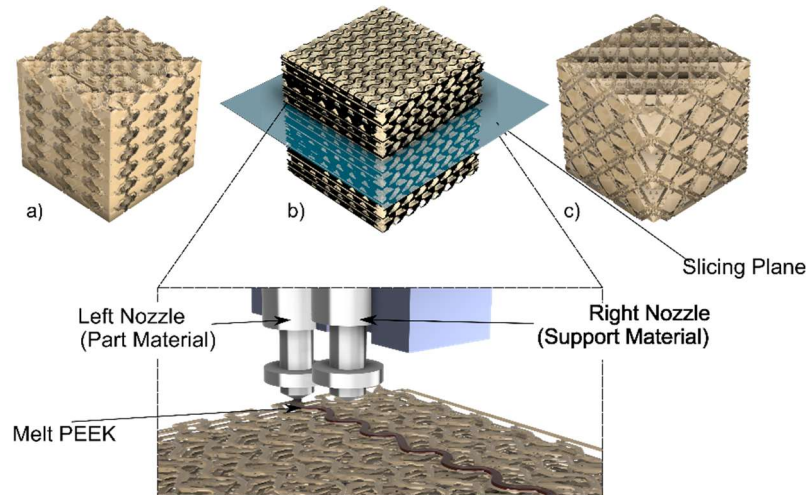


Fig. 2. 24: Schematic representation of the lattice structures and dual-nozzle FDM printing strategy adopted: a) Cross 3D; b) Gyroid with schematic illustration of filament path deposition. The magnified image shows the dual-nozzle configuration: the left nozzle extrudes the part material and the right nozzle deposits the SP5000 support material used to fabricate a raft for PEEK specimens; c) Octect topology.

Cross 3D, Gyroid and Octed are three architectures employed to study the mechanical behavior of PEEK lattice structures produced through FDM. Instead, for PA6CF the three-topology used are Crosshatch, gyroid, and 3D honeycomb. The complete printing factorial plan for the two materials is listed in the Tab. II. 11 below. Five specimens for each condition were printed for the PEEK lattice structure, while for PA6CF three specimens were used for the characterization, due to the extension of the full-factorial plan.

Tab. II. 11: Factorial experimental plan adopted for the compressive characterization of PEEK and PA6–CF lattice structures, including lattice architecture, relative density levels, applied test speeds, and anisotropy assessment.

MATERIAL: PEEK			
Architecture	Relative Density	Test speed [mm/min]	Anisotropy YES/NO
Cross 3D	10 – 20 - 30	2	NO
Gyroid	10 – 20 - 30	2	NO
Octect	10 – 20 - 30	2	NO
MATERIAL: PA6CF			
	Relative Density	Test speed [mm/min]	Anisotropy YES/NO
Cross-hatch	10 – 20 - 30	10 – 100 – 600	YES (10 mm/min)
Gyroid	10 – 20 - 30	10 – 100 – 600	YES (10 mm/min)
3D Honeycomb	10 – 20 - 30	10 – 100 – 600	YES (10 mm/min)

2.5.1.2 Peek thermal treatment: annealing

After printing the samples were thermally treated using an instrumented furnace model UF160PLUS by Memmert. The thermal cycle for annealing is schematized in a). This cycle was adopted according to the material manufacturer datasheet. An image of the as printed and the annealed structure obtained after the thermal cycle is depicted and magnified in a) and b).

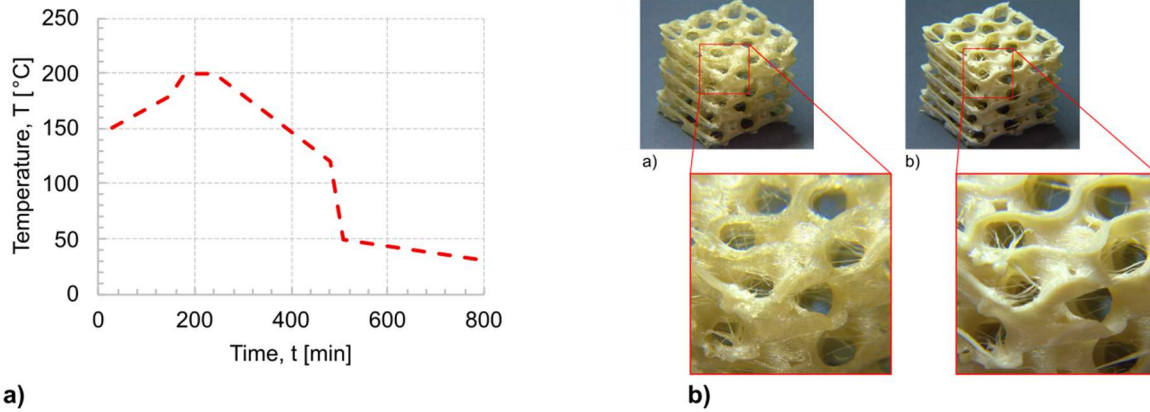


Fig. 2. 25: Effect of thermal annealing on FDM-printed lattice structures. (a) Temperature–time profile adopted for the annealing treatment. (b) Comparison between the as-printed sample, characterized by a predominantly amorphous morphology, and the annealed structure, where increased crystallization leads to a more defined microstructural appearance.

2.5.1.3 Physical characterization: DSC&DMA

The physical and thermo-mechanical characterization of the materials was performed by combining density measurements, Differential Scanning Calorimetry (DSC), and Dynamic Mechanical Analysis (DMA). These techniques were employed to quantify the degree of crystallinity, thermal transitions, and temperature-dependent viscoelastic behavior, which are essential for interpreting the mechanical response of both bulk specimens and lattice structures. Sample density was determined by dividing the measured mass by the nominal volume. Mass measurements were performed using an analytical balance with a resolution of ± 0.0001 g.

Additional DSC analyses were conducted on specimens in their as-printed condition and after annealing to evaluate the influence of thermal treatment on crystallinity. DSC measurements were performed using a TA Instruments DSC25 system. For each test, the sample mass was kept below 5 mg. Sample preparation followed a standardized procedure consisting of:

- (i) Cutting a small portion of material from the printed specimen
- (ii) Weighing the sample using the analytical balance
- (iii) Sealing the sample in an aluminum crucible using the press shown in Fig. 2. 26 (b,c)
- (iv) Positioning the sealed crucible inside the DSC furnace alongside an empty reference crucible, as illustrated in Fig. DSC(d).

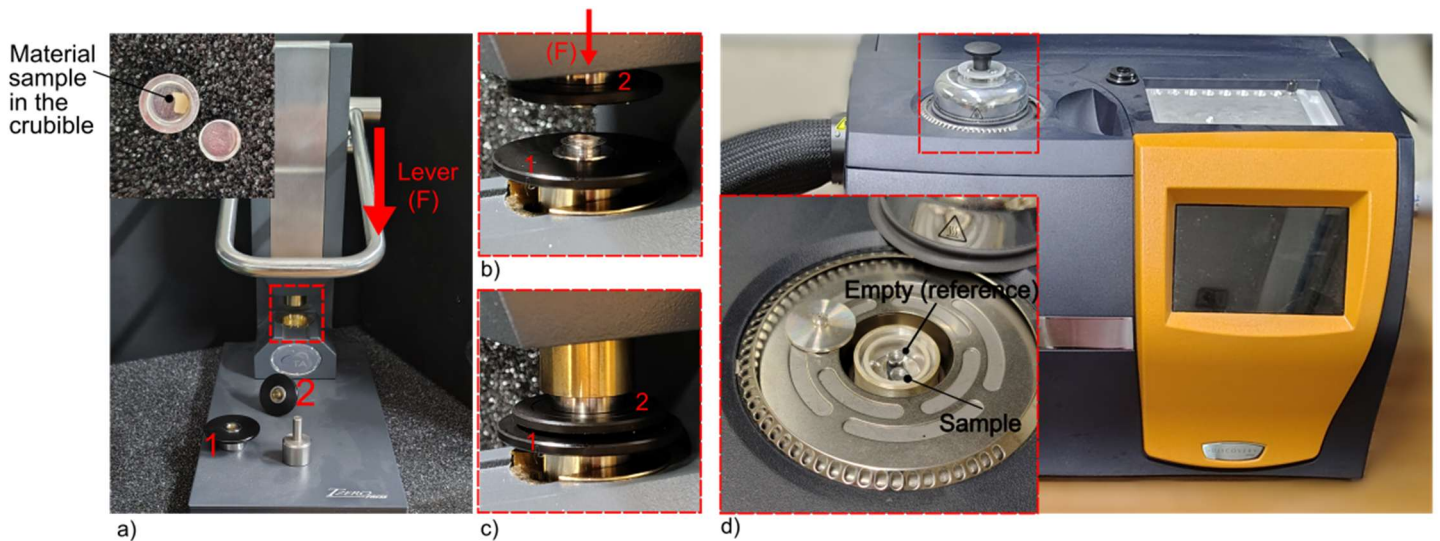


Fig. 2. 26: Experimental setup for Differential Scanning Calorimetry (DSC) measurements. (a) Press used to hermetically seal the aluminum crucibles containing the polymer samples with the applied force and lever mechanism used during crucible sealing are highlighted (b);(c). DSC instrument showing the furnace chamber with reference (empty) and sample crucibles positioned on the sensor platform (d).

Prior to testing, the DSC flange was cooled to -90 °C by continuously supplying nitrogen gas to the system. The DSC thermal programs adopted for PEEK and PA6–CF specimens are summarized below in Tab. 2.12 and consisted of an equilibration step, isothermal holds, controlled heating and cooling ramps, and a second heating cycle to assess thermal history effects.

Tab. 2.12: DSC procedures for PEEK and PA6 - CF

	PEEK	PA6 – CF
Equilibrate temperature	40 °C	30 °C
Isothermal	40 °C, 10 minutes	30 °C, 10 minutes
Ramp (first heating)	To 400 °C at 10°C/min	To 300 °C at 10 °C/min
Isothermal	400 °C, 10 minutes	300 °C, 10 minutes
Ramp (cooling)	To 40 °C, 10°C/min	30 °C, 10 °C/min
Isothermal	40 °C, 10 minutes	30 °C, 10 minutes
Ramp (second heating)	To 400 °C, 10 °C/min	To 300 °C at 10 °C/min

PEEK samples were extracted from extruded material both before and after the annealing treatment, enabling direct evaluation of the influence of annealing on the thermal and mechanical behavior of the polymer. The degree of crystallinity, χ_c , was calculated with Eq. 14 from the melting enthalpy (ΔH_m) and cold crystallization enthalpy (ΔH_{cc}) obtained during the first heating cycle, following the method described in [113]:

$$\chi_c = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_m^{100\%}} \quad \text{Eq. 14}$$

where $\Delta H_m^{(100\%)} = 130 \text{ J/g}$ represents the melting enthalpy of a fully crystalline PEEK material. For annealed samples, in which cold crystallization was not observed, the degree of crystallinity was calculated as:

$$\chi_c = \frac{\Delta H_m}{\Delta H_m^{100\%}}$$

The PEEK sample was obtained from the extruded material previous and after annealing process. Thus, it is possible to address the influence of the annealing process on the material thermic and mechanical behavior.

Furthermore, the temperature-dependent mechanical response of the materials was investigated by means of Dynamic Mechanical Analysis (DMA). The tests were performed using a double cantilever beam (DCB) configuration, which enables the evaluation of viscoelastic properties under flexural loading conditions. Rectangular specimens were manufactured according to the dimensional requirements for DMA testing, with nominal dimensions of 12.7 mm in width, 3.2 mm in thickness, and 60 mm in length. Each specimen was rigidly fixed within the three-clamp DCB fixture using a torque wrench, applying a tightening torque of 0.8 Nm to ensure reproducible boundary conditions throughout the test.

Temperature sweep tests were carried out starting from an initial temperature of 30 °C, with a constant heating rate of 5 °C/min. The upper temperature limit was set to 200 °C for PA6–CF specimens and to 300 °C for PEEK specimens, in order to fully capture the relevant thermo-mechanical transitions of each material.

DMA measurements were performed on specimens in both the as-printed condition and after the annealing treatment, allowing direct assessment of the effect of post-processing on the viscoelastic response. For both

PEEK and PA6-CF, DMA specimens were printed using full-density infill. The raster orientation was selected such that deposited filaments were perpendicular to the applied loading direction, as shown in the magnified view in Fig. 2. 27, to minimize geometric effects and highlight intrinsic material behavior.



Fig. 2. 27: Dynamic Mechanical Analysis (DMA) experimental setup: DMA instrument equipped with an integrated furnace for temperature-controlled testing. In the magnified images of the internal of the furnace the double cantilever beam (DCB) fixture with the specimen clamped between three supports is presented, highlighting the loading configuration adopted for flexural DMA measurements.

2.5.2 MJF of truss – based lattice structures

This study examines the mechanical behavior and manufacturing repeatability of Face-Centered Cubic (FCC) and Diamond lattice architectures produced through binder jetting technology of thermoplastic polyurethane (TPU) across four density levels, with comprehensive characterization of energy absorption performance and failure mechanisms. Uniaxial compression testing was conducted, with detailed analysis of deformation behavior, densification characteristics, and manufacturing consistency. The deformation evolution of the analyzed structures was captured by a digital camera and analyzed through Digital Image Correlation. The study compares Face-Centered Cubic and Diamond lattice topologies across four density levels. It provides comprehensive characterization of deformation mechanisms, failure modes, and energy absorption performance under standardized conditions. Furthermore, the research quantifies manufacturing repeatability metrics—essential for enabling mass customization applications—and establishes design frameworks that translate patient-specific or application-specific energy requirements into manufacturable lattice specifications with predictable performance. These outcomes enable systematic implementation of customized protective devices across automotive, aerospace, and medical device sectors, bridging the current divide between design intent, manufacturing reality, and personalized functional requirements. Additionally, different speeds were used for the diamond structure which exhibited the best properties as energy absorber. Moreover, cyclical tests have been carried out to understand the repeatability of these structures under multiple load conditions.

2.5.2.1 Manufacturing strategies

2.5.2.1.1 Experimental Design and Specimen Configuration

Two lattice architectures were selected for systematic comparison based on their mechanical characteristics and established manufacturing compatibility with binder jetting processes. Face-Centered Cubic (FCC) truss-structures feature orthogonal strut arrangements with nodes positioned at cube face centers, creating stretching-dominated deformation mechanisms under compressive loading. Diamond lattice structures exhibit tetrahedral connectivity with nodes arranged in a diamond cubic crystal structure, characterized by bending-dominated deformation with enhanced load distribution capabilities. A rendering representation of the two truss-based lattice employed is depicted in Fig. 2. 28

The mechanical behavior of lattice structures can be described using the Maxwell criterion, originally formulated by Maxwell and subsequently extended by Calladine. The Maxwell number (M) is determined according to Equation 16:

$$M = s - 3n + 6 \quad \text{Eq. 16}$$

where s represents the number of struts and n denotes the number of nodes within the unit cell.

The Maxwell number describes the mechanical response of lattice structures by determining whether external loads are accommodated through flexural or axial deformation. Negative Maxwell numbers induce bending-dominated behavior, wherein insufficient strut density necessitates flexural compliance at nodal junctions. This response is advantageous for energy absorption and vibration isolation applications. Conversely, positive Maxwell numbers yield stretch-dominated behavior, wherein adequate strut redundancy enables predominantly axial load transmission, resulting in superior stiffness-to-weight ratios [114].

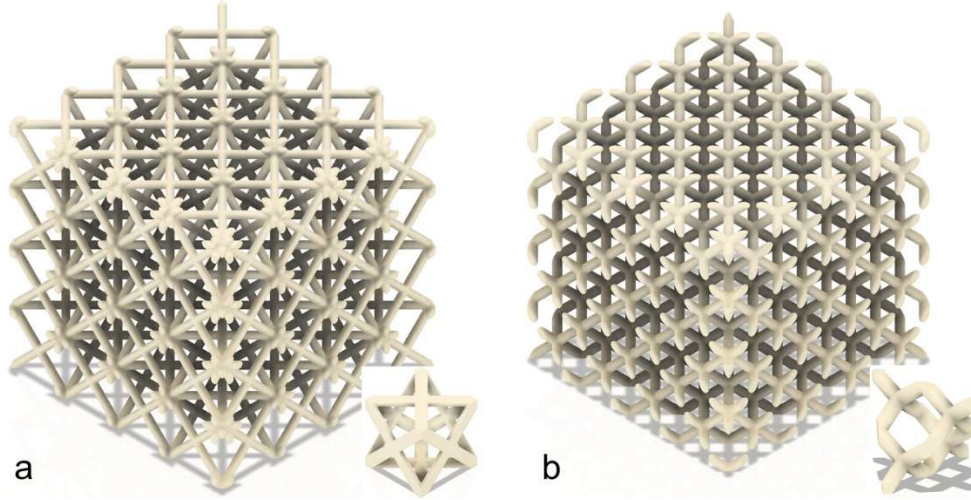


Fig. 2. 28: Lattice architectures selected for mechanical comparison. (a) Face-Centered Cubic (FCC) truss structure, characterized by orthogonal strut arrangements and stretching-dominated deformation under compressive loading. (b) Diamond lattice structure, featuring tetrahedral connectivity and a bending-dominated deformation mechanism with enhanced load distribution.

Based on the unit-cell topology illustrated in Fig. 2. 28, the mechanical behavior of the FCC and Diamond lattice structures was quantitatively assessed using the Maxwell criterion. The corresponding number of nodes and struts for each architecture, together with the resulting Maxwell number and the expected deformation regime, are summarized in Tab. II. 12.

Tab. II. 12: Maxwell number calculation for the FCC and Diamond lattice unit cells, including the number of nodes and struts and the resulting expected deformation behavior under mechanical loading.

Lattice Type	Nodes (n)	Struts	Maxwell number	Expected Behavior
FCC	4	12	$12-3(4)+6=6$	Stretch-Dominated
Diamond	8	16	$16-3(8)+6=-2$	Bending dominated

A systematic experimental matrix was developed to comprehensively explore the design space relevant for customization applications. All specimens were designed with cubic geometry ($25 \times 25 \times 25 \text{ mm}^3$). The experimental matrix comprised 8 distinct specimen configurations with 5 replicate specimens manufactured for each condition, resulting in 40 total specimens for mechanical characterization. This replication strategy captured both intra-build and inter-build variability essential for assessing manufacturing consistency. The related test matrix is listed in Tab. II. 12.

Tab. II. 13: Experimental matrix for FCC and Diamond lattice specimens, reporting architecture type, density level, specimen code, target relative density, unit cell size, and corresponding strut diameter. All specimens feature a cubic geometry ($25 \times 25 \times 25 \text{ mm}^3$) and were designed by varying strut diameter at constant unit cell size.

Architecture	Density Level	Specimen Code	Target Relative Density (%)	Unit Cell Size [mm]	Strut Diameter [mm]
Face-Centered Cubic	1	FCC -1	10	6×6×6	$0.73 \pm 0.1 \text{ mm}$
Face-Centered Cubic	2	FCC -2	12	6×6×6	$0.79 \pm 0.1 \text{ mm}$
Face-Centered Cubic	3	FCC -3	14	6×6×6	$0.87 \pm 0.1 \text{ mm}$
Face-Centered Cubic	4	FCC -4	16	6×6×6	$0.96 \pm 0.1 \text{ mm}$
Diamond	1	DIA-1	10	6×6×6	$0.9 \pm 0.1 \text{ mm}$
Diamond	2	DIA-2	12	6×6×6	$0.96 \pm 0.1 \text{ mm}$
Diamond	3	DIA-3	14	6×6×6	$1.08 \pm 0.1 \text{ mm}$
Diamond	4	DIA-4	16	6×6×6	$1.22 \pm 0.1 \text{ mm}$

Unit cells were designed using parametric n-Topology software with control over strut diameter, unit cell dimensions, and connectivity patterns. Relative density values were varied through modification of strut diameter while maintaining constant unit cell size, enabling isolation of density effects from geometric scaling influences.

2.5.2.2 Sample Manufacturing And Materials

The powder material employed was BASF Ultrasint-TPU01 (TPU 88/90 Shore A) being characterized by a nominal density of 1.1 g/cm^3 . Particle morphology was assessed via scanning electron microscopy to verify sphericity and identify potential agglomeration. The binder system comprised HP 3D700 Fusing Agent. Lattice specimens were fabricated using a HP Multi Jet Fusion model 5600 binder jetting machine. Tab. II. 14 summarizes the main printing conditions.

Tab. II. 14: Main printing parameters adopted for the fabrication of TPU lattice specimens using the HP Multi Jet Fusion 5600 system

Parameter	Value	Units
Layer thickness	100	μm
Powder spreading speed	254	mm/s
Print head traverse speed	254	mm/s
Drying time per layer	3.5	s
Powder bed temperature	104	$^{\circ}\text{C}$
Percentage of recycled powder from previous prints	80	%

Process parameters were optimized through preliminary experimentation to achieve complete lattice structure fabrication while maintaining dimensional accuracy and minimizing manufacturing defects.

Following completion of printing, specimens underwent a standardized post-processing sequence: controlled depowering extracting the samples from the build after cooling followed by sandblasting.

2.5.3 Compression Mechanical characterization & data analysis

This section describes the procedures adopted to characterize the compressive response of PEEK, PA6-CF, and TPU lattice structures. The methodology covers quasi-static tests at room temperature, high-temperature tests for PEEK, and the post-processing analysis used to extract the main mechanical parameters. All tests were performed under displacement control and followed the recommendations of ISO 604:2002.

2.5.3.1 Environmental compression characterization

Compression tests were performed on an MTS C43.50 machine (50 kN load cell). Testing continued until reaching 20 mm displacement, corresponding to a nominal strain of $\varepsilon = 0.8$, to fully characterize the complete material response.

PEEK cube specimens were loaded under displacement control of 2 mm/min. Indeed, for PA6 CF the experimental plan includes the influence of speed and anisotropy. For each deposition conditions five replicates were performed. The full factorial plans, which includes the structures topologies and the relative densities is listed below in Tab. II. 15.

Tab. II. 15: Full factorial experimental plan adopted for compression testing of lattice specimens manufactured via FDM and MJF, reporting lattice topology, relative density levels, test speeds, and anisotropy conditions for PEEK, PA6-CF, and TPU materials.

PEEK (FDM)	Cross 3D	Gyroid	Octet	
Relative Density	10%	20%	30%	
PA6 CF (FDM)	Crosshatch	Gyroid	3D Honeycomb	
Relative Density	10%	20%	30%	
Test speed [mm/min]	10	100	600	
Anisotropy	x	x	x	
TPU (MJF)	FCC	DIA		
Relative Density	10%	12%	14%	16%

The compression tests performed on PA6-CF specimens were designed to investigate the influence of both loading rate and material anisotropy on the compressive response. In particular, the experimental plan included nine different lattice configurations, obtained by combining three lattice topologies with three relative density levels. Each configuration was tested at displacement rates of 10, 100, and 600 mm/min to assess strain-rate sensitivity.

Material anisotropy was also systematically evaluated by varying the orientation of the cubic specimens with respect to the build direction during compression testing. Specimens were loaded both parallel and

perpendicular to the build direction in order to capture the effect of layer-by-layer deposition on the mechanical response. When the applied load is perpendicular to the build direction, interlayer interfaces are predominantly subjected to shear loading, making the compressive behavior particularly sensitive to interlayer cohesion and bonding quality.

2.5.3.2 High temperature compression tests

To evaluate the temperature-dependent compressive behavior of PEEK lattices, compression tests were additionally performed at 25 °C, 140 °C, 160 °C and 200 °C, covering a range that spans the glass transition temperature of PEEK ($T_g \approx 143\text{--}150\text{ °C}$). This allows quantifying the degradation of stiffness, plateau stress and energy absorption capabilities as the polymer transitions from a glassy to a rubbery state. All high-temperature compression tests were carried out on an MTS C43.50 universal testing machine (50 kN load cell) equipped with a temperature-controlled environmental chamber. The cubic lattice specimens ($25 \times 25 \times 25\text{ mm}^3$), manufactured with the same process parameters described in Section 2.5.1, were placed between lubricated compression platens to minimize friction.

Before loading, each specimen was thermally equilibrated for 15 minutes at the target temperature to ensure a uniform thermal field throughout the structure. A picture of the set – up of the test with the use of the climatic chamber is illustrated in Fig. 2. 29.

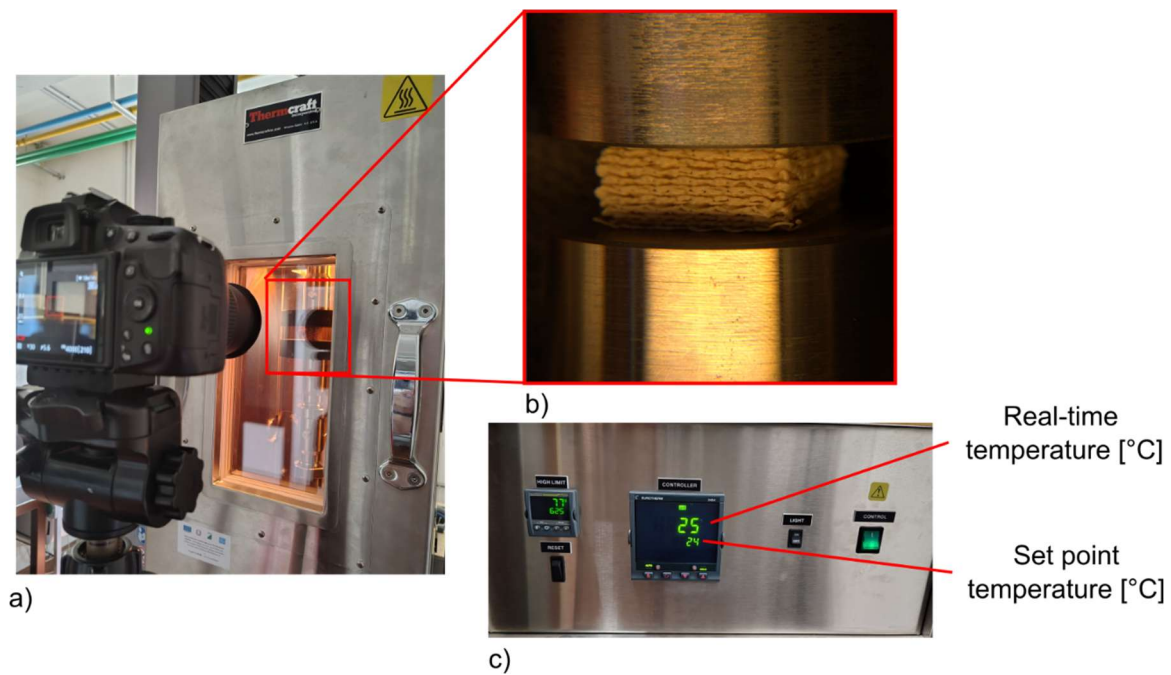


Fig. 2. 29: Experimental setup for high-temperature compression testing of PEEK lattice specimens. (a) MTS C43.50 universal testing machine equipped with a temperature-controlled environmental chamber. (b) Magnified view of a PEEK lattice specimen compressed between heated platens inside the chamber. (c) Environmental chamber control panel displaying the stabilized test temperature.

Tests were performed under displacement control, adopting the same nominal speed used in the room-temperature characterization (2 mm/min). These thermal-compression tests provide the necessary data to quantify:

1. knock-down factors $E(T)/E(25\text{ °C})$ and $\sigma_y(T)/\sigma_y(25\text{ °C})$
2. Softening behavior around T_g and its consistency with DMA trends
3. High-temperature efficiency of PEEK cellular materials for aerospace applications.

The full factorial plan of the architecture lattice tested under different temperatures are listed below in Tab. II. 16:

Tab. II. 16: full factorial plan of the architecture lattice tested under different temperatures

Topology: Gyroid			
Relative Density [%]	10	20	30
Test Temperature [°C]	140	160	200

2.5.3.3 Compressive data analysis

After the tests, the following parameters were determined: Young's modulus, compressive strength, densification strain, energy and energy to densification. To precisely determine the densification strain, the procedure reported in [20,21], was adopted. Particularly, the absorption efficiency was determined through Eq. 16 and Eq. 17.

$$\eta(\varepsilon) = \frac{1}{\sigma(\varepsilon)} \int_0^\varepsilon \sigma(\varepsilon) d\varepsilon; \quad \text{Eq. 16}$$

$$\varepsilon_d = \left. \frac{d\eta(\varepsilon)}{d\varepsilon} \right|_{\varepsilon=\varepsilon_d} = 0 \quad \text{Eq. 17}$$

Then, the strain to densification is represented by strain corresponding to the peak of the absorption efficiency. Then, the energy to densification was calculated as the integral of the energy over the strain upon densification strain. In Fig. 2. 30 is represented a picture of a PA6 – CF specimen within the compressive plate. Furthermore, the three characteristic phases of a lattice compressive test and the mechanical response are illustrated.

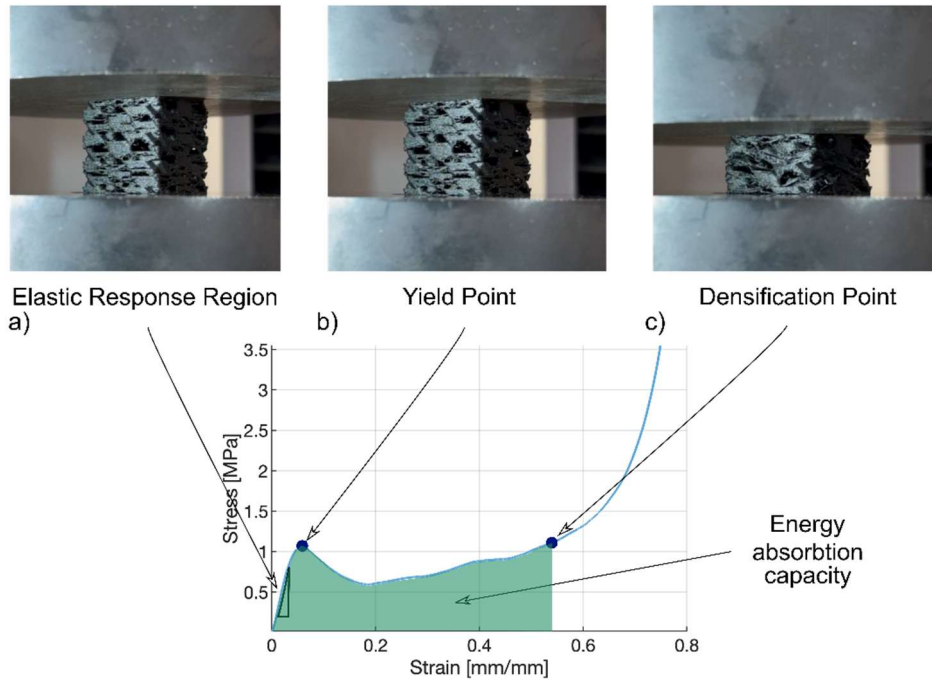


Fig. 2. 30: Characteristic deformation stages and mechanical response of a lattice structure under compressive loading. (a) Elastic response region, characterized by linear stress–strain behavior. (b) Yield point, marking the onset of cell collapse and stress plateau formation. (c) Densification point, where progressive cell collapse leads to a rapid increase in stress. (d) Representative stress–strain curve highlighting the elastic region, yield stress, densification strain, and the absorbed energy up to densification.

To compare mechanical properties with solid materials, a theoretical model relating density, topology, and behavior is required. To this end, the widely used model proposed by Gibson and Ashby theory [78] was adopted. According to this theory, the mechanical response of lattice structures mainly depends on the dominant deformation mechanism: bending, which refers to the flexural deformation of the structure, and stretching, i.e., the axial tension or compression of the structure. These mechanisms influence the relationship between relative density and mechanical properties, described by the following equations:

$$\frac{E}{E_s} = C_1 \left(\frac{\rho}{\rho_s} \right)^{n_1} \quad \text{Eq. 18}$$

$$\frac{\sigma}{\sigma_s} = C_2 \left(\frac{\rho}{\rho_s} \right)^{n_2} \quad \text{Eq. 19}$$

where C_1 and C_2 , n_1 and n_2 are constants identified by fitting the model to the experimental data. C_1 and C_2 dependent on geometry; the exponents n_1 and n_2 typically range between 1 and 2 depending on whether stretching or bending is the dominant deformation mechanism, respectively. E and σ are the elastic modulus and compressive yield strength of the lattice structure, respectively, while E_s and σ_s refer to those of the base solid material; ρ and ρ_s represent the density of the lattice structure and the solid material, respectively.

2.5.3.4 High-temperature constant-load compression fixture

To evaluate the creep-like compressive behavior of PEEK lattice structures under conditions representative of autoclave curing of composite laminates, a dedicated spring-loaded compression fixture was designed and manufactured. The apparatus consists of a three-plate assembly connected through threaded rods and pre-loaded compression springs, enabling the application of sustained compressive loading during thermal exposure. A CAD representation of the system is shown in Fig. 2. 31 a), while Fig. 2. 31 b) reports the manufactured device.

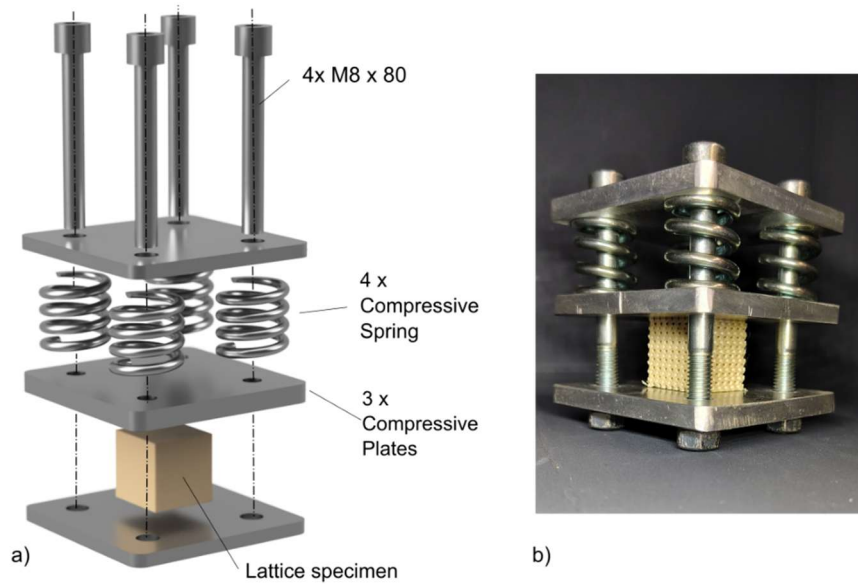


Fig. 2. 31: High-temperature constant-load compression fixture: (a) CAD model of the three-plate spring-loaded fixture used to apply a constant compressive load on PEEK lattice specimens. (b) Photograph of the manufactured device, designed to operate inside a furnace for creep-like compression tests at elevated temperature.

The adoption of a spring-loaded compression fixture was motivated by practical considerations related to load application at elevated temperature. At the temperatures relevant for autoclave curing cycles, the use of conventional hydraulic or pneumatic loading systems is inherently challenging due to thermal stability and compatibility issues of working fluids and actuators. For this reason, a purely mechanical loading system based on elastic elements was selected, allowing reliable and reproducible application of sustained compressive loading inside a furnace environment with minimal system complexity.

The fixture comprises:

- a **lower support plate**, onto which the cubic lattice specimen ($25 \times 25 \times 25 \text{ mm}^3$) is positioned.
- an **intermediate floating plate**, guided by four threaded screw-columns, which transmits the load to the specimen.
- an **upper loading plate**, connected to compression springs arranged symmetrically around the specimen area.
- four **high-temperature steel rods** with nuts that allow controlling the spring compression.

By tightening the upper nuts, the springs are compressed by a prescribed amount, generating a known axial force on the intermediate plate and therefore on the specimen.

After pre-loading, the entire assembly is placed inside a Memert UF160PLUS, where it is held at the target temperature. Following thermal exposure, the apparatus is removed from the furnace, and the final specimen height is measured using a digital micrometer (resolution 1 μm). This test configuration provides a creep-like compressive assessment of PEEK lattice architectures, enabling the quantification of their dimensional stability under sustained load at elevated temperature. Since autoclave curing cycles typically involve temperatures in the range of 160–190 $^{\circ}\text{C}$ combined with external pressure, this method effectively replicates the thermo-mechanical environment experienced by lightweight lattice molds.

2.5.3.4.1 Mechanical formulation of the spring–specimen system

From a mechanical standpoint, the compression fixture can be idealized as a one-dimensional spring–specimen system arranged in series, subjected to uniaxial compression along the build direction of the lattice, as depicted in Fig. 2. 32.

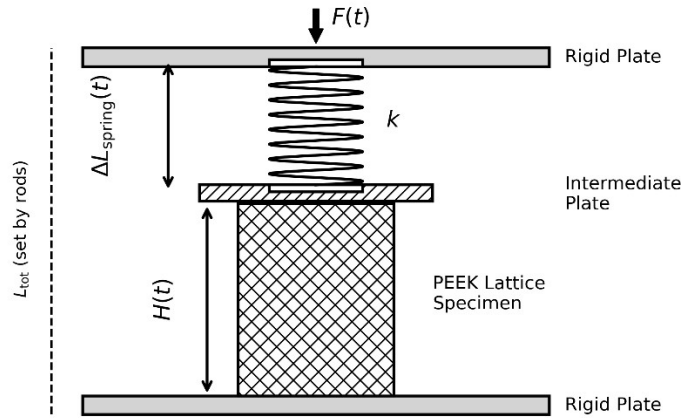


Fig. 2. 32: Mechanical schematic and free-body diagram of the spring–specimen system: Simplified one-dimensional representation of the compression fixture, modeled as an elastic spring assembly with stiffness k connected in series with a deformable lattice specimen of height $H(t)$. Time-dependent specimen deformation results in spring extension and evolution of the applied compressive load $F(t)$.

In this representation, the upper and lower plates are treated as rigid bodies, while the threaded rods serve solely as guides ensuring alignment of the loading direction. The spring assembly is characterized by an equivalent stiffness k , whereas the lattice specimen is represented as a deformable element whose height evolves under sustained compressive loading at elevated temperature.

The applied compressive force $F(t)$ acts uniformly along the loading axis and is governed by the elastic response of the springs. Time-dependent deformation of the specimen results in spring extension and progressive load relaxation, according to the mechanical relations described in the previous section.

In this configuration, the applied load is not controlled directly but is generated by imposing a prescribed displacement to the elastic elements of the system.

At room temperature, tightening of the threaded nuts imposes an initial compression ΔL_0 to the spring assembly, thereby generating an initial compressive force:

$$F_0 = k \Delta L_0$$

where k is the equivalent stiffness of the spring system. This force is transmitted through the intermediate plate to the lattice specimen, defining the initial mechanical state of the system prior to thermal exposure. The compression fixture was designed to apply a nominal compressive stress of 1 MPa to the lattice specimens, representative of the pressure levels typically experienced by lightweight tooling components during autoclave curing cycles. Therefore, the required nominal compressive force to apply is:

$$\sigma_{comp} A = 1 \frac{N}{mm^2} \cdot 25 \cdot 25 mm^2 = 625 N$$

To achieve this target condition, the four steel compression springs, are characterized by a nominal stiffness of $k = 50 N/mm$ each, arranged symmetrically around the specimen loading area. The total stiffness of the spring assembly is therefore given by:

$$k_{eq} = 4k = 200 N/mm$$

Thus, the spring displacement to achieve this load condition is $\Delta L_0 \approx 3.1 mm$

In practice, a nominal pre-compression of $\Delta L_0 = 3.0 \pm 0.2 mm$ was applied to each spring.

When the pre-loaded assembly is placed inside the furnace and exposed to elevated temperature, the lattice specimen is free to undergo time-dependent compressive deformation under sustained loading. Owing to the **series configuration of the spring-specimen system**, any **incremental reduction in specimen height occurring during deformation at high temperature** is instantaneously accommodated by a corresponding extension of the spring assembly, ensuring kinematic compatibility of the system under load.

This kinematic condition can be expressed at the differential level as:

$$\delta H(t) = \delta L_{spring}(t)$$

where the above relation holds **instantaneously during deformation under load at elevated temperature** and does not apply to the final configuration measured after unloading and cooling.

As the specimen deforms over time, the applied compressive load evolves according to the elastic response of the springs and can be expressed as:

$$F(t) = k \Delta L_{spring}(t)$$

Consequently, the applied load gradually decreases as deformation progresses. The resulting mechanical boundary conditions therefore correspond to a **quasi-constant load condition governed by elastic feedback**, rather than a strictly constant stress condition. This configuration allows the specimen to deform under sustained compression while avoiding geometric over constraint and is representative of compliant tooling conditions encountered during autoclave curing processes.

Following thermal exposure and subsequent cooling to room temperature, elastic recovery of both the spring assembly and the lattice specimen occurs. As a result, the final spring compression measured after cooling does not directly correspond to the total compressive deformation accumulated by the specimen at high temperature, but instead reflects the residual load supported by the lattice structure at the end of the test. Conversely, the final specimen height measured in the unloaded, cooled state quantifies the irreversible

component of deformation induced by the combined action of sustained compressive loading and elevated temperature.

2.5.3.4.2 Deformation stages during the compression test

The mechanical response of the spring-loaded compression system can be further clarified by considering the three distinct deformation stages experienced by the fixture during the test, which are documented in Fig. HT-2 through photographic images of the experimental setup. The figure highlights both the specimen height and the spring compression measured at each stage.

Initial pre-loading stage (Fig. 2. 33 a): In the initial configuration at room temperature, the lattice specimen is positioned between the lower and intermediate plates, and the threaded nuts are tightened to impose a prescribed compression on the spring assembly. At this stage, the specimen height H_0 and the corresponding spring length L_0 are measured. This configuration defines the reference mechanical state of the system prior to the application of the nominal compressive load.

Pre-loaded configuration prior to thermal exposure (Fig. 2. 33b): Further tightening of the threaded nuts imposes a controlled additional compression of the springs, corresponding to the target pre-loading condition. In this state, the specimen height H_{pre} and the spring length L_{pre} are measured at room temperature. This configuration represents the initial loaded condition of the test, from which the nominal compressive pressure is defined before thermal exposure.

Final cooled configuration (Fig. 2. 33 c): After high-temperature exposure and subsequent cooling to room temperature, the fixture reaches a final configuration characterized by a different specimen height H_f and spring length L_f . Elastic recovery of both the lattice specimen and the spring assembly occurs during cooling. Measurements performed in this state provide information on the irreversible component of compressive deformation accumulated by the specimen and on the residual compressive load supported by the lattice structure.

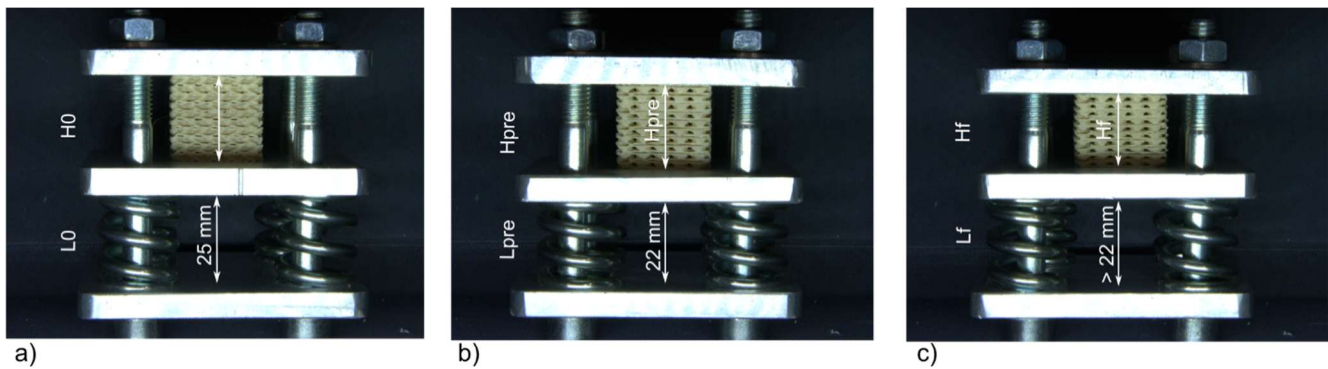


Fig. 2. 33: Deformation stages of the spring-loaded compression fixture: (a) Initial configuration after spring pre-loading at room temperature. (b) Configuration during high-temperature exposure under sustained compressive loading. (c) Final configuration after cooling to room temperature, showing residual spring compression and permanent specimen deformation.

Based on the test configuration described, the following geometrical and mechanical quantities were measured or derived and are reported in the Results section:

H_0 : specimen height measured at room temperature after spring pre-loading and prior to thermal exposure.

H_f : specimen height measured at room temperature after thermal exposure and cooling

ΔL_0 : initial spring compression imposed during pre-loading

ΔL_f : residual spring compression measured after cooling

$F_0 = k \Delta L_0$: initial compressive load applied to the specimen

$F_f = k \Delta L_f$: residual compressive load supported by the specimen after thermal exposure.

The permanent compressive strain is defined as:

$$\varepsilon_{\text{perm}} = \frac{H_0 - H_f}{H_0}$$

This quantity represents the irreversible component of deformation accumulated during the test and is used as a metric to assess the high-temperature dimensional stability of the lattice structures. The residual load ratio F_f/F_0 is additionally used to quantify the ability of the lattice architecture to sustain compressive loading after prolonged exposure to elevated temperature.

To complete the description of the experimental procedure, the thermal cycles applied during the spring-loaded compression tests are summarized in Fig. 2. 34, while the corresponding test matrix is reported in Tab. II. 17. As shown in Fig. 2. 34, three different thermal treatments were applied, corresponding to target temperatures of 140 °C, 160 °C, and 200 °C.

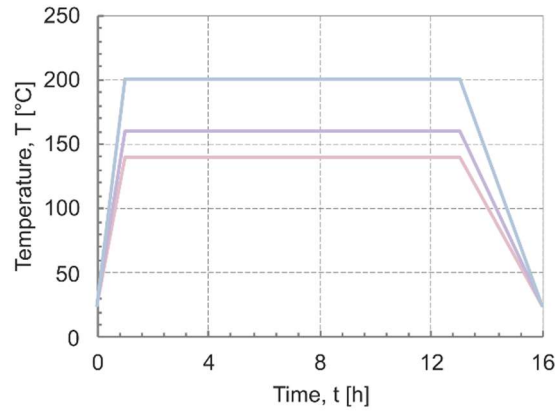


Fig. 2. 34: Temperature–time profiles corresponding to the three thermal treatments investigated in this study (140 °C, 160 °C, and 200 °C). In all cases, specimens were held at the target temperature under sustained compressive loading for 12 h before cooling to room temperature.

In all cases, the pre-loaded compression fixture was heated to the target temperature, held under isothermal conditions for 12 h, and subsequently cooled to room temperature. The heating and cooling ramps were kept comparable across all tests to ensure consistent thermal histories.

The combination of lattice relative density and test temperature investigated in this study is summarized in Tab. II. 17.

Tab. II. 17: Combination of lattice relative density and test temperature investigated for gyroid lattice specimens in the spring-loaded compression tests.

Topology: Gyroid	Relative Density [%]		
Temperature [°C]	10	20	30
140	X	V	V
160	X	V	V
200	X	V	V

All tests were performed on gyroid lattice specimens with relative densities of 10%, 20%, and 30%, allowing a systematic evaluation of the influence of both temperature and lattice density on the thermo-mechanical response under sustained compressive loading.

3. RESULTS

The present chapter reports the experimental results obtained by applying the methodology developed in the previous sections. The discussion is structured in a progressive manner, beginning with PLA, which is used as a reference material to validate the overall workflow and to quantify the sensitivity of inter-layer adhesion to thermal history, deposition strategy, and process parameters. Once the methodology has been consolidated on PLA, it is transferred to high-performance thermoplastics, with specific focus on PAEK within the framework of a collaboration between the University of L'Aquila and Thales Alenia Space. This collaboration, established in the context of the OCEANOS project (**Next-generation Satellite Technologies**, lead partner: **Thales Alenia Space**), addresses the characterization and metallization of polymeric components for aircraft lightweighting, as well as the assessment of additive manufacturing integration in the aerospace supply chain for components operating under demanding thermal conditions in orbital environments. Within this framework, the aim is to perform a quality assessment of PAEK components produced via FDM, as the company is evaluating the adoption of MEX-based technologies for the fabrication of non-structural polymer parts to reduce overall aircraft mass.

Subsequently, the chapter extends the analysis to the compressive behavior of architected materials manufactured by FDM (PEEK, PA6-CF) and MJF (TPU). PEEK and PA6-CF are representative technical polymers with potential application in aerospace, biomedical, and automotive sectors, particularly where weight reduction and high specific performance are required. Their mechanical response under compressive loading is investigated in terms of elastic modulus, yield strength, and deformation mechanisms. In addition, their suitability for energy-absorbing applications is examined through the evaluation of densification strain and specific energy absorption (SEA), which are key indicators for the design of impact-mitigation and crashworthy structures.

Finally, TPU lattice structures produced via MJF are analyzed to enable a multi-scale interpretation of deformation and failure mechanisms, spanning from bulk material behavior to cellular architectures. These results are exploited to support the development and validation of a functional shock-absorbing component for sport-biomedical applications.

Across all sections, the results are presented through mechanical response curves, microstructural observations, and quantitative metrics. The discussion is consistently framed within a unifying perspective that links process-induced thermal history, resulting morphology, and the mechanical performance of the additively manufactured components.

PLA is employed as the baseline material to establish the experimental methodology and to isolate the role of deposition strategy, thermal history, and process parameters on inter-layer adhesion.

Mechanical Mode I and tensile tests are used to understand the mechanical response of layer adhesion.

The validation consists of three complementary steps:

- (i) The first Mode I fracture study focused exclusively on the effect of raster orientation,
- (ii) a second investigation where thermal history is directly measured via infrared thermography to quantify the influence of deposition parameters on adhesion, and
- (iii) tensile testing under controlled manufacturing strategies to confirm the same correlations under a different loading mode.

Microstructural analyses (optical and SEM) are used throughout to link fracture paths and interface morphology with changes in process temperature and mechanical response.

3.1 PLA, Mode I: Across Deposition Strategies and Process Parameters

The Mode I fracture tests performed on PLA provide a comprehensive view of how deposition strategy and process parameters govern inter-layer adhesion. The results obtained across the two complementary studies converge toward a unified interpretation, in which the mechanical response is dictated by three interacting factors: raster orientation, thermal history, and the resulting interfacial morphology. The first campaign isolates the role of deposition geometry, while the second introduces controlled thermal variations through changes in extrusion multiplier and print speed, supported by infrared thermography and microscopic analyses. Together, these investigations establish the physical principles underlying crack initiation and propagation in MEX-printed PLA.

3.1.1 Effect of deposition strategy: geometric contribution

In the first set of experiments, fracture toughness was evaluated by varying the raster orientation while keeping the process parameters constant. This configuration allows the intrinsic effect of filament alignment on crack propagation to be quantified. The load–displacement for the five raster orientation are shown in Fig. 3. 1 a). The curves show clear differences among deposition strategies. The blur curves represent the load-crosshead displacement during the first phase of the execution of the test.

Specimens printed with 0° rasters exhibit a sharp load drop immediately after crack onset, consistent with brittle interfacial separation. Conversely, $\pm 45^\circ$ specimens reach the highest peak loads and show a smoother post-peak softening, indicating enhanced energy absorption and improved interlayer cohesion. Intermediate behaviors are observed for 45°, 90°, and 0/90° patterns, each characterized by varying degrees of micro-instability during crack growth.

The evolution of crack length during the test, reported in Fig. 3. 1(b) is described by the R-curves. The R-curves are clustered into the five group that refer to the printing adopted strategies. In the graph, the dashed

line represents the initial pre-crack length while the various specimen categories are grouped using a basic color and point shape for each one of them. Individually, the various specimen categories showed different energy dissipation behavior.

highlights the relationship between mechanical response and crack progression. 0° specimens exhibit a nearly linear and monotonic increase in crack length after initiation, consistent with a weak and planar interface. 90° and $0/90^\circ$ rasters show a stepped crack-growth pattern, confirming the influence of raster transitions on local crack deflection. The $\pm 45^\circ$ configuration shows smooth but slower crack advance, indicative of higher crack-growth resistance.

The energy release rate G_I was computed using the equations of the simple and modified beam theories described in Section 2.2 through the Eq. 8:

$$G_I = \frac{3P\delta}{2b(\alpha+|\Delta|)} \quad \text{Eq. 8}$$

Fig. 3. 1 c) shows the average values of the critical energy release rate and the crack propagation for the deposited strategies. The results illustrate in Fig. 3. 1 c) reveal strong influence of the deposition strategy on the G_{Ic} and G_{Iprop} .

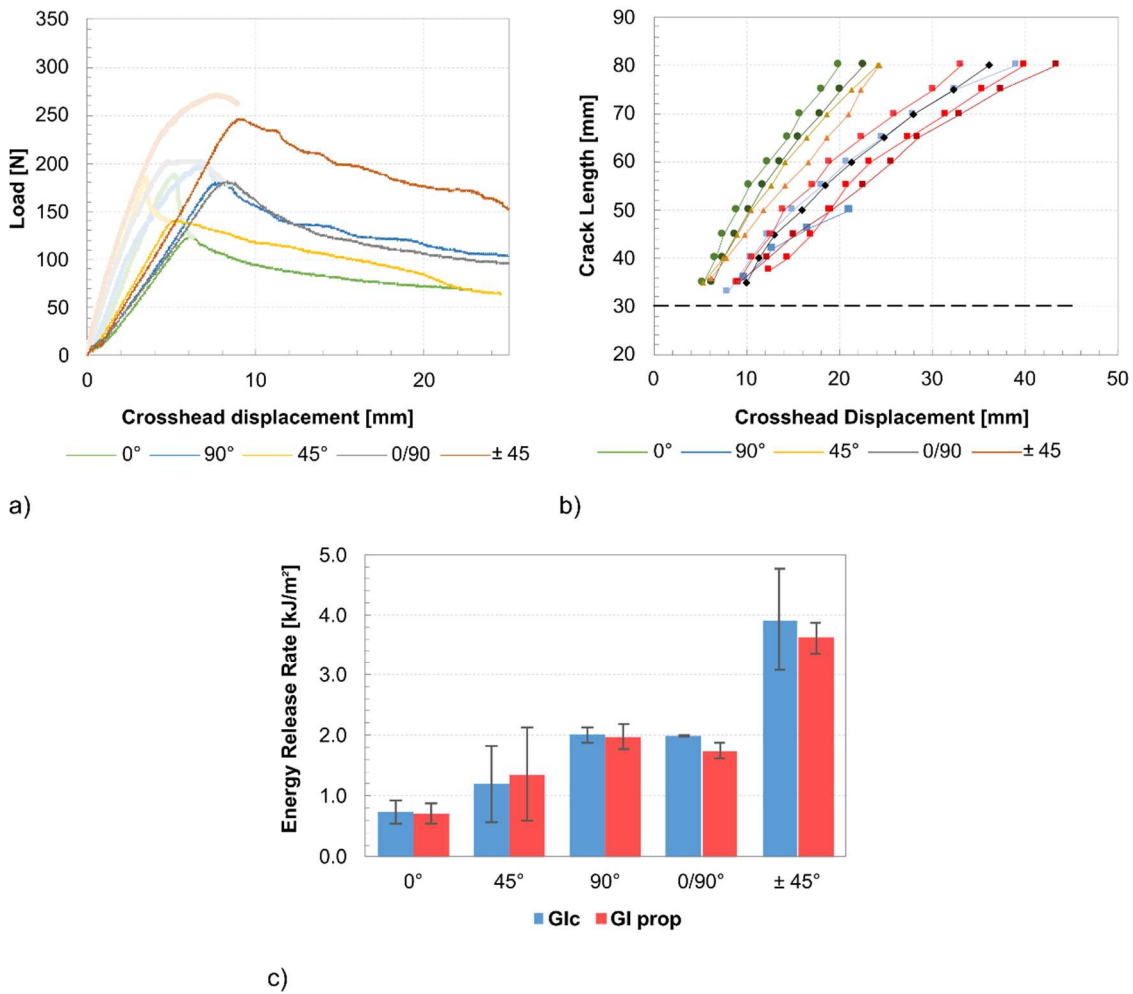


Fig. 3. 1: a) Load–displacement responses for 0° , 45° , 90° , $0/90^\circ$, and $\pm 45^\circ$ deposition strategies. (b) crack-length evolution for the different deposition strategies; c) initiation (G_{Ic}) and propagation (G_{Iprop}) energy release rates

The results reveal that 0° specimens exhibit the lowest interlaminar fracture toughness ($G_{Ic} \approx 0.74 \text{ kJ/m}^2$), accompanied by stable and planar crack propagation along the layer interface. Conversely, the $\pm 45^\circ$ configuration results in the highest toughness ($G_{Ic} \approx 3.9 \text{ kJ/m}^2$), characterized by an initial region of enhanced plasticity followed by a stable propagation regime. This value is almost 4.5 times calculated for 0° samples. Intermediate behaviour is observed in 90° and $0/90^\circ$ samples, where the crack repeatedly jumps between adjacent filaments, producing recurrent load drops. The 45° orientation displays higher variability, often associated with local regions of insufficient inter-layer bonding (“kissing bonds”).

The optical micrographs of the propagation region, shown in Fig. 3. 2 (a–e), provide direct microstructural evidence for the mechanisms captured by the mechanical response and R-curves. Key observations include:

- a) 0° : smooth, planar fracture with negligible bridging $\rightarrow$ correlates with lowest G_{Ic} and G_{Iprop}
- b) 45° : diagonal crack trajectory with partial bridging $\rightarrow$ matches oscillatory R-curve.
- c) 90° : stepped fracture advancing along filament rows $\rightarrow$ reflected in staircase GI trend.
- d) $0/90^\circ$: heterogeneous crack path with mixed bridging events $\rightarrow$ produces wider GI variability.
- e) $\pm 45^\circ$: highly tortuous path with pronounced ligament formation $\rightarrow$ agrees with high and stable G_{Iprop} plateau.

These images collectively demonstrate that the morphology of the propagation region directly encodes the energy dissipation mechanisms active during fracture.

To rationalize the observed fracture behaviour, the representative unit cell (RUC) geometry is used to quantify the effective bonding area (A_a) between adjacent rasters. Enlarged views of the local filament architecture are shown in Fig. B (RUC insets). The RUC analysis highlights three geometric descriptors:

- w_v : vertical projected bonding width
- w_a : axial effective contact width
- A_a : total effective adhesion area, dependent on raster orientation

As illustrated in Fig. 22 of the article and reproduced schematically in Fig. B, the effective bonding area increases from 0° to $\pm 45^\circ$, following:

$$A_a, 0^\circ < A_a, 90^\circ \approx A_a, 0/90^\circ < A_a, 45^\circ < A_a, \pm 45^\circ$$

This geometric hierarchy corresponds exactly to the trends in G_{Ic} and G_{Iprop} illustrated in Fig. 3. 1(c) and the R-curve plateau levels represented in Fig. 3. 2(f).

The $\pm 45^\circ$ deposition provides the greatest interfacial contact and mechanical interlocking, which explains the highest crack-growth resistance. Conversely, the 0° configuration aligns beads parallel to the crack plane, minimizing contact area and promoting brittle propagation.

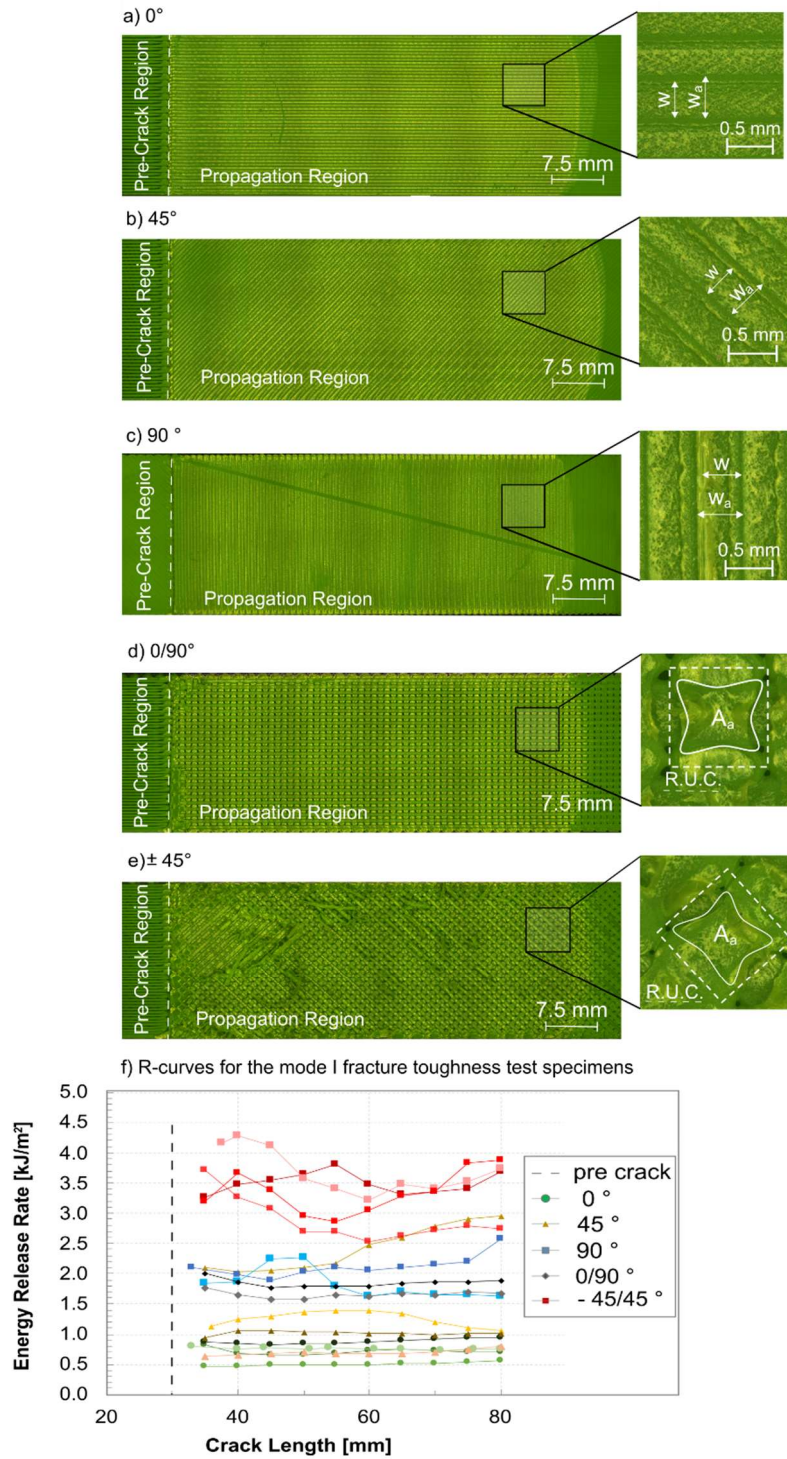


Fig. 3. 2: Fracture surface morphology and GI evolution during crack propagation for different deposition strategies: a-e) Optical macrographs of the propagation region for raster orientations 0° , 45° , 90° , $0/90^\circ$, and $\pm 45^\circ$. f) Corresponding Mode I energy release rate GI as a function of crack length.

The evolution of the strain energy release rate during crack propagation provides further insight into the interplay between raster orientation and interfacial failure mechanisms. As shown in Fig. 3. 2 f), the GI -C curves exhibit markedly different trends depending on the deposition strategy. Specimens printed with a 0° raster orientation display the lowest and most stable GI values along the propagation path, consistent with

the planar and featureless fracture surfaces previously observed. The 45° and 90° configurations show oscillatory behavior, with repeated increases and drops in GI, reflecting the intermittent crack deflections caused by the filament geometry and the transitions between adjacent beads. The $0/90^\circ$ pattern exhibits a similar stepped response, with peaks corresponding to local bridging events followed by sudden reductions once the crack bypasses these microstructural obstacles. In contrast, the $\pm 45^\circ$ specimens maintain the highest GI values throughout propagation, with a broad plateau that indicates sustained energy dissipation and a stable toughening mechanism. These trends, taken together, confirm that both the local filament architecture and the associated stress redistribution govern not only crack initiation but also the kinetics of crack growth along the interlayer interface. The combined representation in Fig. 3. 2, where the fracture surfaces for each deposition strategy are aligned above the corresponding GI-a curves, clearly visualizes how the morphology of the crack path directly translates into the mechanical response. Smooth, planar interfaces yield low and steady GI values, while tortuous, fiber-bridged fracture surfaces correspond to elevated and more stable propagation resistance.

Optical microscopy corroborates these observations: planar crack fronts in 0° , segmented propagation in 90° , partial debonding in 45° , and extensive filament pull-out in $\pm 45^\circ$. These results confirm that raster orientation defines the baseline fracture behavior through its influence on the effective interfacial contact area and the mechanical continuity across layers.

3.1.2 Process, Thermal, and Mechanical Correlation

The second experimental campaign extends the Mode I characterization by integrating infrared thermography (IRT) to directly monitor the thermal history experienced by the previously deposited layer during printing. This approach provides a process–thermal–mechanical framework that is not limited to fracture testing and can be later transferred to tensile specimens and to any MEX-printed geometry. The objective is to quantify how extrusion multiplier, printing speed, and filament deposition conditions influence the interlayer temperature field, and subsequently affect G_{Ic} , G_{Iprop} , and fracture morphology.

This methodology represents a generalizable tool for the broader characterization protocol developed in this thesis.

3.1.3 Mechanical Response Under Varying Process Parameters

Mode I fracture tests were performed on samples printed using two reference raster configurations ($0/90^\circ$ and $\pm 45^\circ$), each manufactured at three extrusion multipliers (EM = 96%, 100%, 104%) and three print speeds, namely 1000, 2000, and 4000 mm/min. The mechanical responses demonstrate two distinct behaviors primarily distinguished by the presence or absence of the crack propagation path. Only the $0/90^\circ$ specimens produced under specific processing conditions exhibited stable crack propagation. Most samples manufactured with this raster strategy showed immediate, catastrophic delamination, yielding a null GIc, as no stable propagation phase could be established as illustrated in Fig. 3. 3, with some displaying out-of-plane crack propagation relative to the nominal direction introduced during the MEX process. All specimens printed with EMP = 104% displayed crack propagation, independent of deposition speed. Conversely, for EMP = 96% and EMP = 100%, propagation occurred only at the intermediate deposition speed (2000 mm/min). At both 1000 mm/min and 4000 mm/min, these samples failed in a brittle manner without measurable propagation. This confirms that, for $0/90^\circ$, propagation is strongly conditioned by the combined effect of extrusion multiplier and deposition speed, with only a narrow processing window enabling stable crack growth. Conversely, all samples produced with the $\pm 45^\circ$ strategy exhibited a crack propagation path, except for the condition 1000 mm/min and 96% value of EMP. It highlights a significant sensitivity of fracture toughness to the loading path. In these specimens no filament was perpendicular to the crack propagation direction, enabling a more uniform load distribution along the filaments and preventing the stepped decay observed in the $0/90^\circ$ samples. Additionally, the $\pm 45^\circ$ deposition strategy induced increased plasticity during the initial crack displacement, likely owing to the path orientation between consecutive layers.

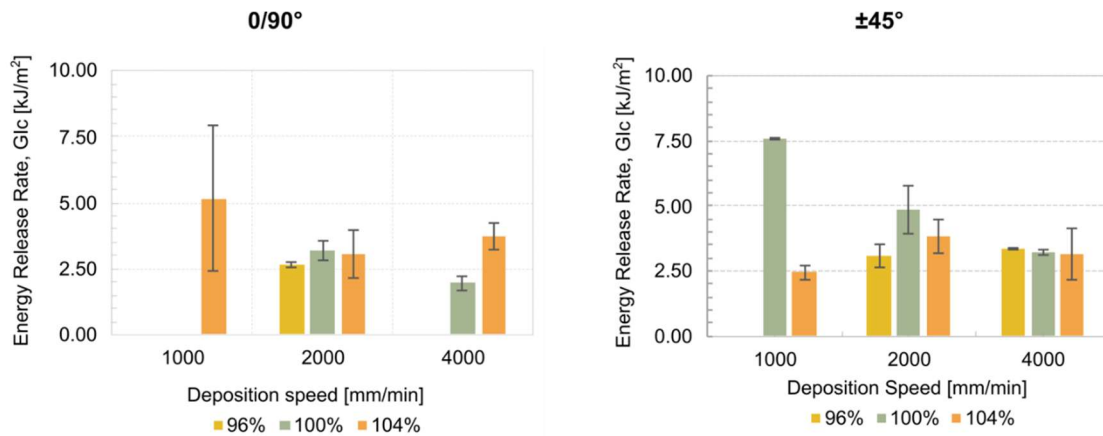


Fig. 3. 3: Influence of the process parameters (extrusion multiplier and deposition speed) on GIc using different deposition strategies: (a) $0/90^\circ$, (b) $\pm 45^\circ$.

Based on the analysis of force-displacement curves and observations of crack propagation, the computation of the energy release rate was conducted utilizing the modified beam theory equation (Eq. 8: $G_I = \frac{3P\delta}{2b(\alpha + |\Delta|)}$), following the methodology outlined in Section 2.

The results of the GIc are highlighted in Fig. 3. 3 and in listed in Tab III. 1.

Tab III. 1: Experimental Results from Mode I tests: Influence of deposition speed (1000 – 2000 – 4000 mm/min) and material flow rate (e.m.p.) during FDM process.

Deposition Strategy	Printing Speed [mm/min]	EMP [%]	Average Glc [kJ/m ²]	σ Glc [kJ/m ²]
0/90	1000	96	-	-
		100	-	-
		104	5.16	2.74
	2000	96	2.66	0.07
		100	3.19	0.36
		104	3.04	0.91
	4000	96	-	-
		100	3.22	0.08
		104	3.73	0.51
$\pm 45^\circ$	1000	96	-	-
		100	7.58	0.04
		104	2.44	0.25
	2000	96	3.10	0.45
		100	4.85	0.94
		104	3.85	0.65
	4000	96	3.36	0.05
		100	3.22	0.08
		104	3.16	0.96

3.1.3.1 Thermal History: Infrared Thermography

The integration of infrared thermography enables a direct measurement of the thermal field during deposition and provides quantitative insight into how the extrusion multiplier influences the temperature experienced by the underlying layer. The infrared image acquired during printing is shown in Fig. 3. 4 a), highlighting the distinct thermal regions corresponding to the hot-end, the extruded filament, and the printed sample. To monitor and compare temperature variations between samples printed under different processing conditions, a measurement point was selected on the upper surface of the specimen. The corresponding temperature–time histories, reported in Fig. 3. 4 b), compare two depositions at the same printing speed (1000 mm/min) and identical raster orientation ($\pm 45^\circ$), produced with extrusion multipliers of EMP = 100% and EMP = 104%. Indeed, these printing conditions exhibit respectively the highest and lowest value for the Glc. Both conditions exhibit temperature peaks of approximately 210 °C as the hot end passes over the measurement point, followed by a rapid cooling. While the peak temperature is essentially identical for both extrusion multipliers, significant differences emerge in the substrate temperature (the temperature of the previously deposited layer). For EMP = 100%, the mean substrate temperature is ~59 °C, whereas for EMP = 104%, it increases to nearly 70 °C.

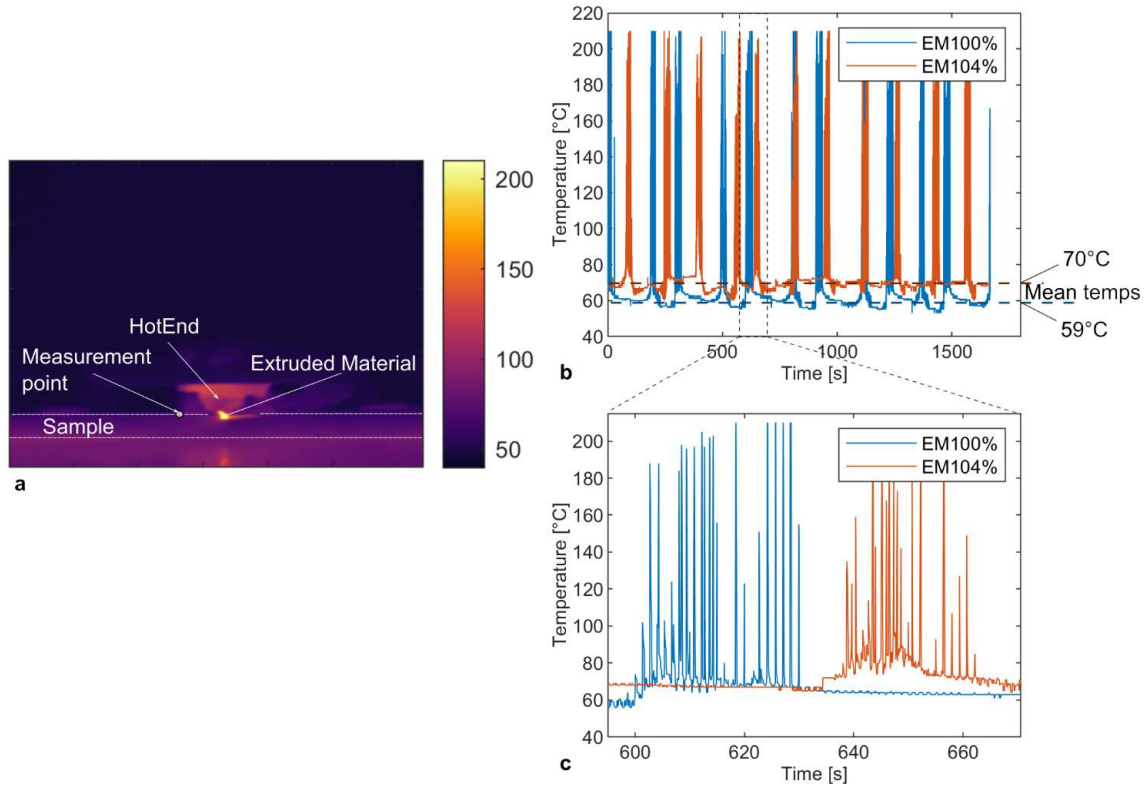


Fig. 3. 4: Infrared thermography during deposition: (a) thermal image with ROI definition; (b) substrate temperature evolution for $EM = 100\%$ and $EM = 104\%$; (c) temperature profile during layer deposition.

A more detailed view of the temperature evolution during deposition is shown in Fig. 3. 4 c). The higher extrusion multiplier ($EMP = 104\%$) produces a slower cooling rate and a higher average temperature throughout the deposition stage when compared to $EMP = 100\%$. This behavior is attributed to the increased flow rate associated with higher EMP values, which introduces additional molten material onto the previously deposited layer, increasing thermal inertia and reducing the cooling rate. As a result, the underlying layer remains at a higher temperature for a longer period. However, the study also notes that excessive extrusion may lead to incomplete melting, indicating a potential trade-off between thermal inertia and proper fusion of the extruded filament. These observations demonstrate that thermal energy available at the interface, rather than nominal material flow alone, is the dominant factor governing interlayer toughness. These thermal observations explain the trends observed in fracture toughness (Fig. 3. 3), demonstrating that both too low ($EMP = 96\%$) and too high ($EMP = 104\%$) material flow can negatively affect interlayer adhesion: insufficient flow results in reduced heating, whereas excessive flow increases thermal inertia but may limit complete melting, leading to suboptimal bonding.

3.1.3.2 Thermo-Mechanical Interpretation (DSC & DMA)

To complement the mechanical and thermal analyses presented earlier, specimens printed with EMP = 100% and EMP = 104% were further investigated through dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC). These techniques provide insight into the viscoelastic response and crystallinity of the printed PLA and clarify how process-induced thermal history affects the polymer state.

Dynamic mechanical analysis was used to evaluate the evolution of storage modulus (E') and damping factor ($\tan \delta$) as a function of temperature. The results are reported in Fig. 3. 5, which compares the behavior of samples produced with the two extrusion multipliers. T (°C)

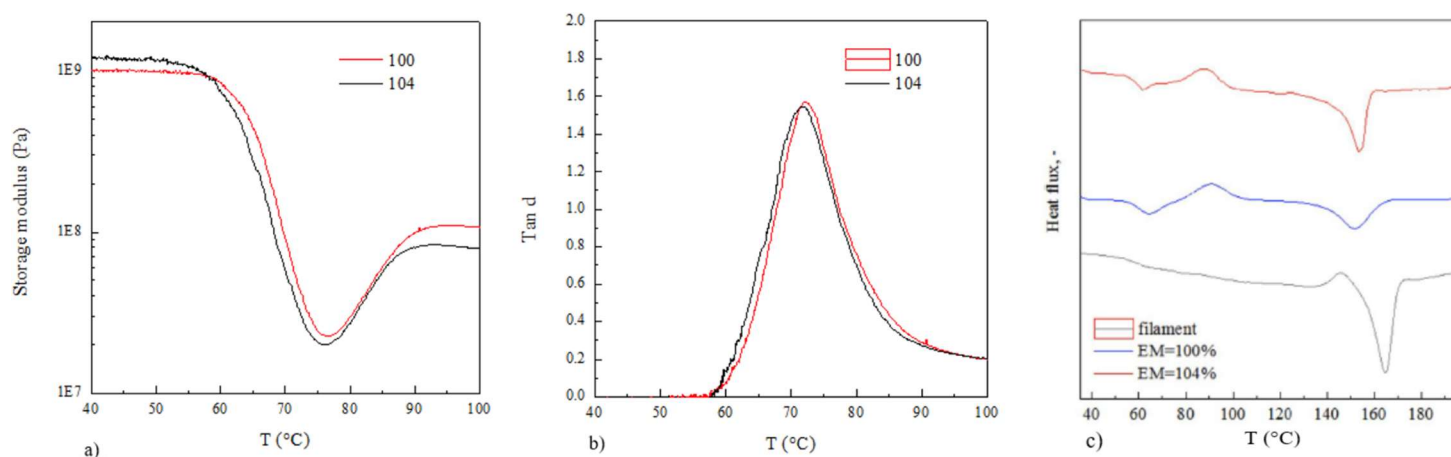


Fig. 3. 5: Thermo-mechanical and thermal characterization of PLA specimens printed with different extrusion multipliers (EMP = 100% and EMP = 104%). (a) Storage modulus (E') as a function of temperature. (b) Damping factor ($\tan \delta$) as a function of temperature, highlighting the glass transition region. (c) DSC thermograms of the raw filament and printed specimens, showing cold crystallization and melting behavior.

The specimen printed with EMP = 100% exhibits a slightly lower storage modulus at the lowest temperatures but shows a higher glass transition temperature, as indicated by the position of the $\tan \delta$ peak, compared with the sample obtained with EMP = 104%. As temperature approaches T_g , the storage modulus decreases for both materials. After reaching the minimum, E' increases again due to crystallization. The specimen obtained with EMP = 100% shows a more pronounced increase in storage modulus after the minimum, suggesting a lower crystalline degree achieved during deposition. Conversely, the sample obtained with EMP = 104% exhibits a smaller modulus recovery, which is consistent with a higher crystallinity degree already present in the as-printed condition.

DSC analysis of the filament reveals a high crystalline degree. For both EMP conditions, DSC curves of the 3D printed parts exhibit exothermic peaks associated with cold crystallization, confirming that the rapid cooling during the MEX process leads to the formation of predominantly amorphous structures. The melting peak of the printed samples shifts towards lower temperatures compared to the filament, which is attributed to the formation of a different crystalline phase during processing. Overall, DSC confirms that the part produced with EMP = 104% has a higher crystalline degree than the one printed with EMP = 100%. Despite

this difference in sampling depth, the analyses are consistent and confirm that $EMP = 104\%$ leads to a slightly higher crystallinity than $EMP = 100\%$.

These trends are in agreement with the thermal histories derived from infrared thermography, which indicate that higher extrusion multipliers promote slower cooling and facilitate crystallization during printing.

3.1.3.3 Microstructural Analysis: SEM of Delaminated Surfaces

Microstructural characterization by scanning electron microscopy (SEM) was carried out to link the thermo-mechanical findings to the actual interlayer morphology and fracture mechanisms. SEM observations focused on the delaminated surfaces obtained after Mode I fracture tests and are reported in Fig. 3. 6.

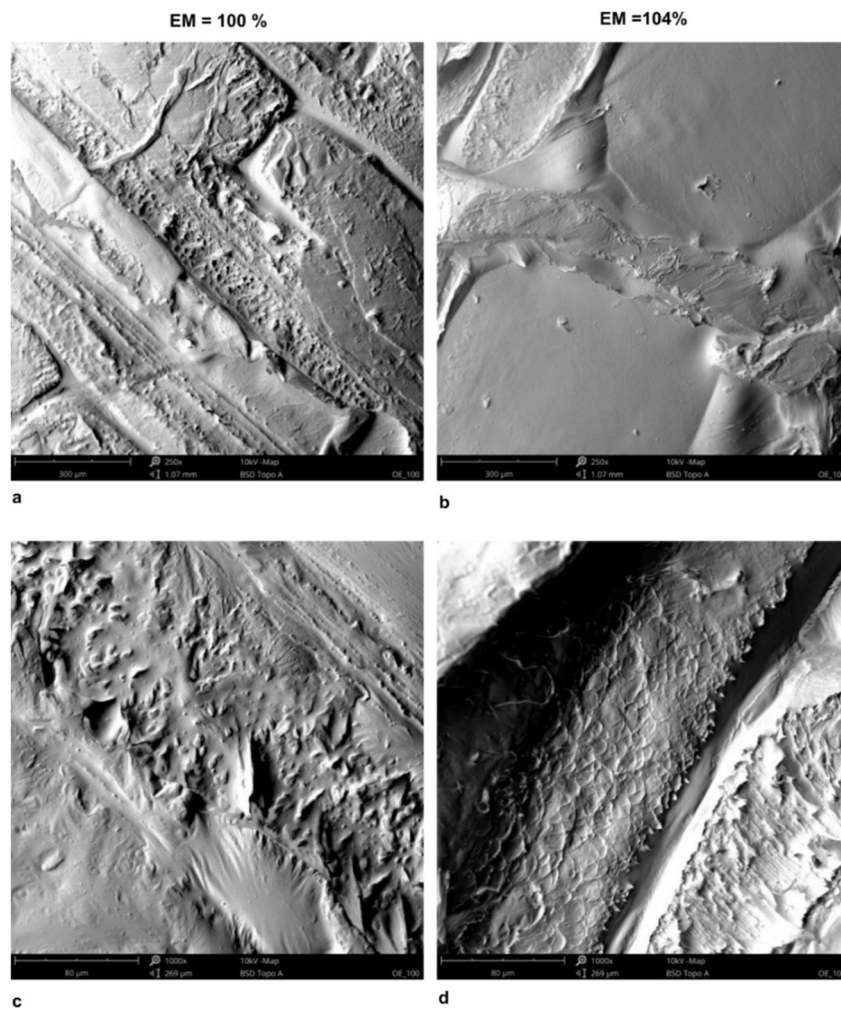


Fig. 3. 6: SEM micrographs obtained with two magnifications, $250\times$ (a, b) and $1000\times$ (c, d), for parts realized with $EMP = 100\%$ and $EMP = 104\%$ after the delamination test

SEM micrographs clearly reveal that fracture occurs primarily through detachment of adjacent layers, and that the extension and texture of the detached regions depend strongly on the extrusion multiplier. For $EMP = 100\%$, a larger detached area is observed, with a rougher surface morphology. This roughness suggests more extensive mechanical interlocking and significant polymer-chain diffusion at the interface. In contrast, the detached surface of the part obtained with $EMP = 104\%$ appears smoother and more uniform, indicating weaker adhesion and a more brittle delamination. At higher magnification (Fig. 3. 6 d)), ordered

microstructures, likely associated with spherulitic crystalline domains, are visible on the surface of the deposited layers. This observation is consistent with the higher crystallinity degree inferred for EMP = 104% from DMA and DSC analyses. By crossing the information provided by thermal, mechanical, and microstructural investigations, it can be hypothesized that, in parts printed with EMP = 104%, crystallization occurs too early, i.e. before polymer chains can adequately diffuse across the interface between adjacent layers, thereby hindering additional diffusion and limiting adhesion. In contrast, for EMP = 100%, crystallization is sufficiently delayed allowing significant chain diffusion at the interface, resulting in stronger interlayer bonding.

The higher crystallinity observed at EMP = 104% can be ascribed both to the slower cooling rate during deposition and to the presence of small crystalline nuclei already present at the exit of the liquefier, which accelerate crystallization and restrict the rearrangement of polymer chains at the interface.

3.2 PLA Tensile Behavior: Integrated Analysis of Thermal History, Manufacturing Strategy, and Microstructural Evolution

The tensile behavior of PLA components manufactured via Material Extrusion (MEX) was investigated through a unified analysis that consolidates the results of four complementary experimental studies. Although each study examined different process parameters—interlayer time, deposition speed, nozzle diameter, layer height, build orientation, and specimen preparation method, the combined findings converge toward a single governing framework: the thermal history imposed during deposition dictates polymer chain mobility, interlayer diffusion, void morphology, and ultimately the mechanical performance of printed parts.

This section integrates all results into a coherent interpretation of how thermal, mechanical, and microstructural factors interact to govern tensile behavior in MEX-fabricated PLA.

3.2.1 Thermal Analysis of the Deposition Process

Understanding the thermal history experienced by the extruded filament is fundamental to rationalizing interlayer adhesion in Material Extrusion (MEX) processes. Across the different experimental campaigns, infrared thermography was systematically employed to quantify the dynamic temperature evolution at the deposition interface, with all measurements acquired from a point Region of Interest (ROI) placed on the top surface of the growing component. Although the test cases differed in printing strategy, geometry, flow rate, or nozzle configuration, the thermal results reveal a consistent physical picture: interlayer time, flow rate, and toolpath length determine the thermal environment in which polymer chains diffused across interfaces, thereby dictating the mechanical response of the printed structures.

3.2.1.1 Effect of Sample Thickness and Manufacturing Strategy

The thermal behavior associated with the fabrication of tensile specimens was first examined by comparing two manufacturing approaches:

- (i) direct printing of the tensile specimen, and
- (ii) printing of a larger plate followed by waterjet extraction.

The corresponding infrared (IR) maps are reported in Fig. 3. 7. During printing, IR thermography reveals clear differences in the spatial distribution of heat for the two fabrication strategies. The directly printed specimen (Fig. 3. 7 a) displays a narrow, intense hot zone, localized around the extrusion path, with steep thermal gradients extending over only a few millimeters. In contrast, the plate fabrication case (Fig. 3. 7 b)) shows a broader thermal footprint, with heat spreading over a wider area and more gradual spatial gradients due to the larger deposited surface.

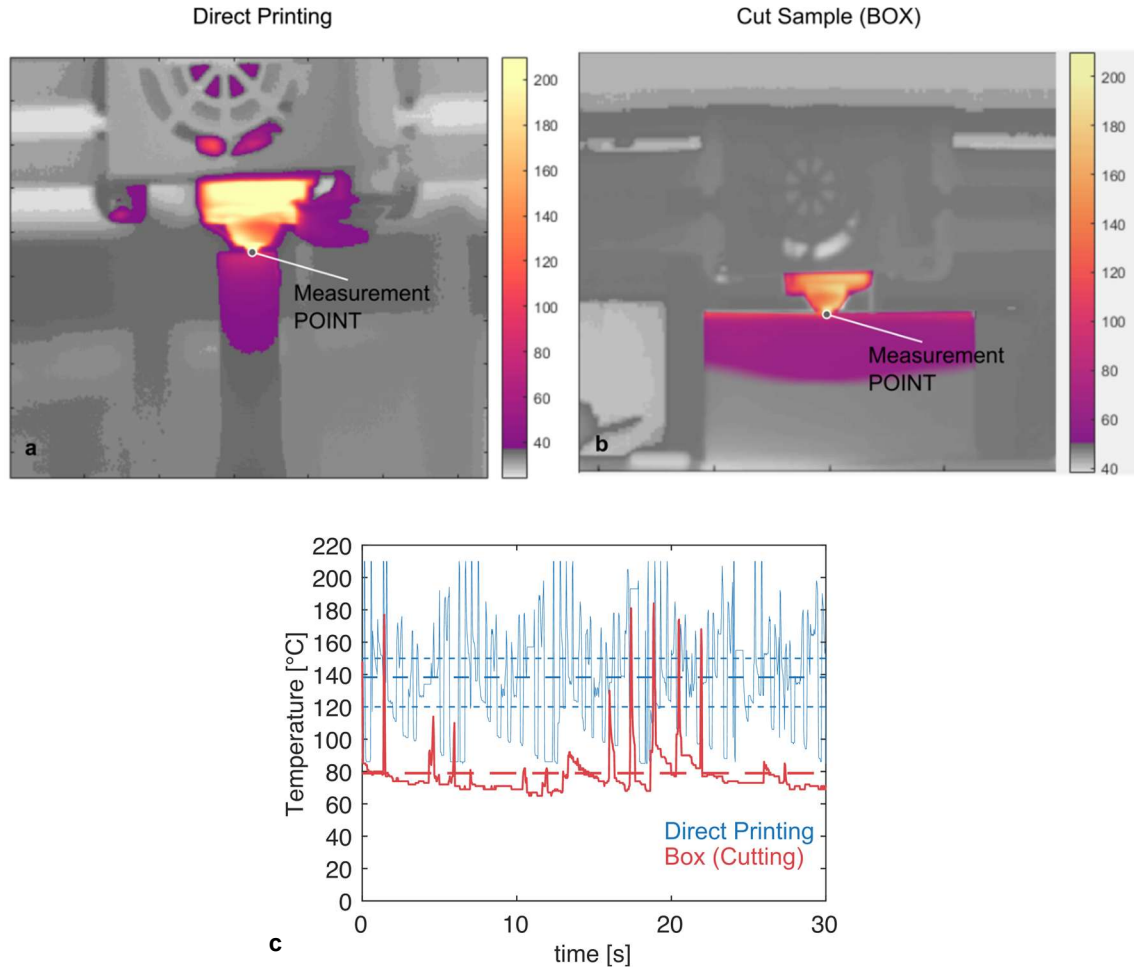


Fig. 3. 7: Infrared thermography analysis of two manufacturing strategies for tensile specimen fabrication. (a) IR map acquired during direct printing of the tensile specimen, showing a localized high-temperature zone near the extrusion path. (b) IR map recorded during printing of a larger plate subsequently cut into tensile specimens, characterized by a broader thermal footprint. (c) Temperature–time histories measured at the indicated points, comparing direct printing and plate fabrication (box cutting) and highlighting differences in interlayer thermal evolution.

The thermographic data illustrated in Fig. 3. 7 c) clearly demonstrates that part geometry and toolpath length directly govern the thermal regime experienced during layer deposition. Specifically:

Direct printing keeps the interlayer interfaces at higher temperatures for longer, enhancing interlayer adhesion. In fact, the substrate temperature maintains a higher minimum value between successive passes (≈ 122 °C), while the interlayer time is of 0.4 s. This condition limits the cooling between following layers. Indeed, large plates with long inter-pass distances, experience more severe cooling. Notably, a longer interlayer time has been observed (≈ 3.6 s) reducing the substrate temperature to a value of ≈ 72 °C. Thus, a lower chain mobility at the interface leads to weaker mechanical performance (as will be shown in the mechanical section).

3.2.1.2 Influence of Interlayer Time and Deposition Speed

In the first set of experiments, the thermal response was analyzed as a function of deposition speed (2000, 3000, and 4000 mm/min) and interlayer time (21 s, 63 s, and 105 s). The interlayer time was deliberately selected to reproduce increasingly larger components:

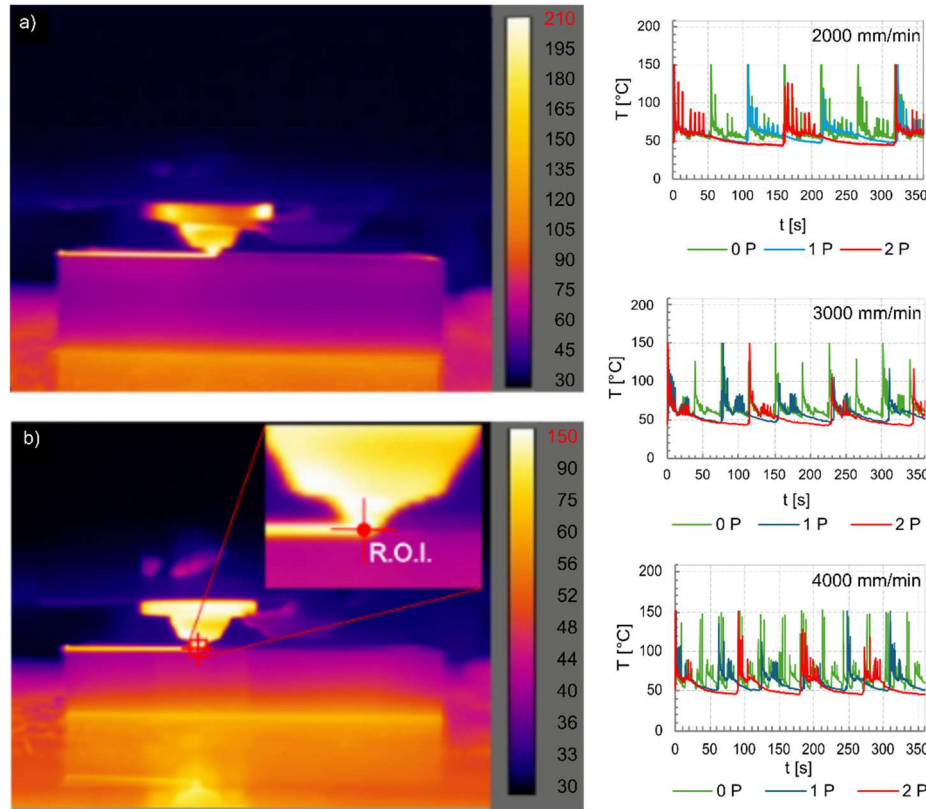


Fig. 3. 8: Infrared thermography analysis of the thermal response as a function of deposition speed and interlayer time. (a) Representative IR image during deposition, highlighting the thermal field around the extrusion zone. (b) Magnified view indicating the region of interest (ROI) used for temperature tracking. Right: temperature–time histories recorded at the ROI for deposition speeds of 2000, 3000, and 4000 mm/min, showing the effect of increasing interlayer time on substrate cooling.

- 21 s corresponds to the minimum time required to complete a layer at the lowest deposition speed.
- 63 s represents an intermediate condition.
- 105 s reproduces a component approximately five times larger, requiring proportionally longer layer times.

Representative IR images and the corresponding thermal histories recorded at the ROI for all deposition speeds are shown in Fig. 3. 8. The temperature signal displays a characteristic periodic pattern, where each cycle reflects a single nozzle pass above the ROI. After each peak, associated with the arrival of the hot-end, the temperature rapidly decreases until the next layer is deposited.

A critical observation is that peak temperatures remain relatively similar across interlayer times (≈ 150 – 160 °C), since extrusion parameters were unchanged. The key distinction arises in the temperature of the substrate immediately before the arrival of the subsequent layer. Longer interlayer times provide more time for cooling,

reducing the substrate temperature. Thus, when the interlayer time increased from 21 s $\rightarrow$ 63 s $\rightarrow$ 105 s, the substrate temperature progressively decreased (e.g., from $\approx 52^\circ\text{C}$ to $\approx 42^\circ\text{C}$), reducing polymer-chain mobility at the interface and leading to inferior mechanical performance. This first dataset therefore establishes a fundamental correlation: interlayer time, not deposition speed, is the dominant factor controlling the substrate temperature and hence interlayer diffusion.

3.2.1.3 Nozzle Diameter and Layer Height: Influence of Flow Rate

The second investigation replicated the same thermographic methodology but focused on how flow rate (Fr)—modified via combinations of nozzle diameter and layer height—affects the thermal environment. Since the spatial resolution of the IR camera does not allow fully resolving the melt pool, the ROI was again placed on the upper surface of the part to quantify the thermal signature of the underlying layer. A composite of the thermal profile and cooling-time analysis is shown in Fig. 3. 9. The cooling time is defined as the interval required for the substrate to cool from its local peak temperature down to the glass transition temperature of PLA ($\approx 59.5^\circ\text{C}$). This interval corresponds to the window where polymer chains retain enough mobility to interdiffusion across the interlayer interface. The results show that:

1. Small nozzles ($D = 0.4\text{ mm}$) lead to short cooling times ($\approx 1\text{--}1.5\text{ s}$). The filament has low thermal inertia and rapidly cools below T_g .
2. Medium nozzle ($D = 0.6\text{ mm}$) $\rightarrow$ longest cooling times ($\approx 4\text{--}5\text{ s}$). Higher thermal inertia leads to slower cooling and extended diffusion time.
3. Large nozzle ($D = 0.8\text{ mm}$) $\rightarrow$ cooling time surprisingly shorter than $D = 0.6\text{ mm}$.

This inversion is explained by:

- lower specific energy supplied to the filament (lower melt temp)
- steeper thermal gradients between core and surface.

Thus, the cooling time is controlled simultaneously by flow rate, thermal inertia, and the actual temperature of the extruded filament, yielding a non-monotonic relationship. This second dataset reinforces the key idea that the thermal window available for chain interdiffusion is highly sensitive to process-induced thermal inertia, not merely to peak temperatures.

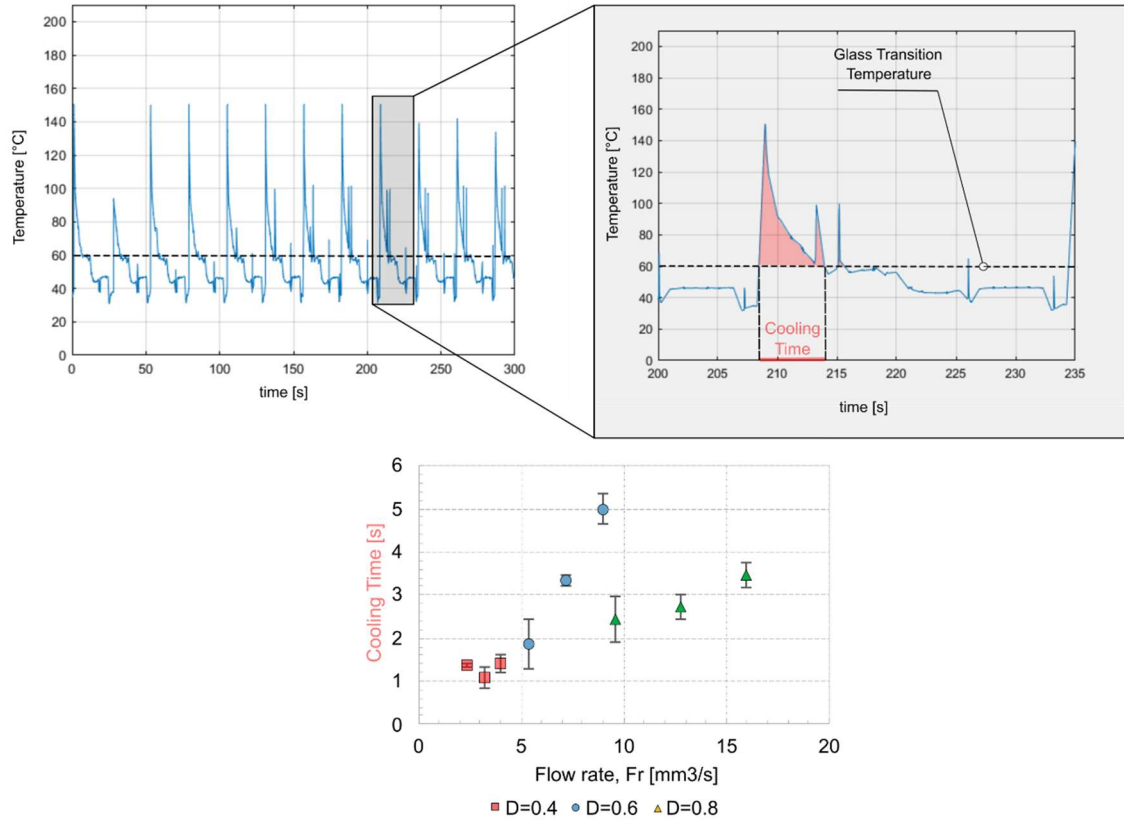


Fig. 3. 9: Thermographic analysis of cooling behavior as a function of flow rate. Top left: temperature–time history measured at the region of interest (ROI) during deposition. Top right: magnified view highlighting the cooling interval from the local temperature peak to the glass transition temperature of PLA. Bottom: cooling time as a function of flow rate for different nozzle diameters ($D = 0.4, 0.6$, and 0.8 mm).

3.2.1.4 Flat Orientation: Single specimen VS Large Palte

This step explored thermal histories for two extreme different manufacturing strategies:

1. Direct printing of a singular tensile specimen
2. Printing a large plate followed by specimens extraction.

The IR maps and associated thermal histories are shown in Fig. 3. 10. Additionally, linear R.O.I is considered to measure temperature alongside the length during the deposition.

In plate fabrication, the longer toolpath between adjacent filaments (along the Y direction) produces an extended cycle period of about 3.6 s, with temperatures dropping to ≈ 72 °C between passes. Peak temperatures also progressively decrease from 154 °C to 138 °C, evidencing cumulative heat loss.

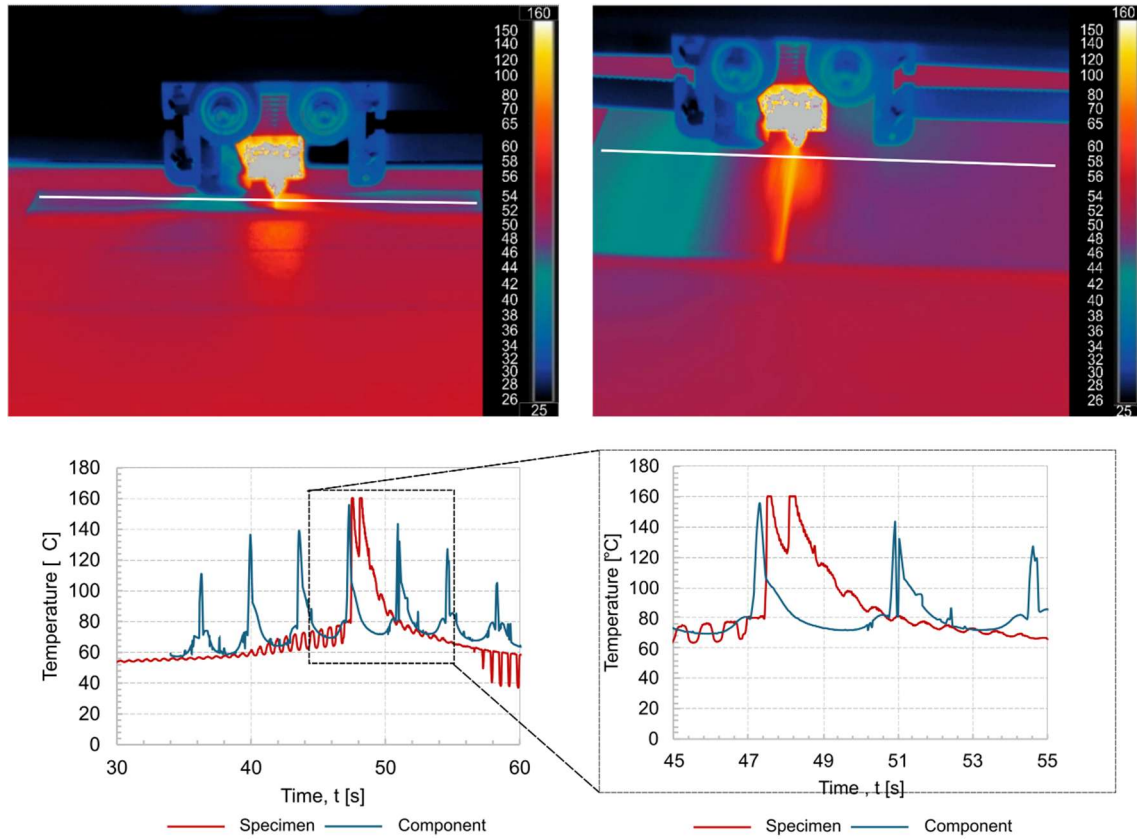


Fig. 3. 10: Infrared thermography comparison of two manufacturing strategies for tensile specimens. Top row: IR maps acquired during (a) direct printing of a single tensile specimen and (b) printing of a large plate for subsequent specimen extraction, with the linear region of interest (ROI) indicated. Bottom row: corresponding temperature–time histories measured at the ROI for the specimen and surrounding component, including a magnified view highlighting differences in thermal cycling between the two strategies.

Direct printing, by contrast, is characterized by highly compressed cycles of 0.4 s. The short inter-pass interval keeps the interface at higher minimum temperatures (≈ 122 °C) and generates peak temperatures above 160 °C. As a result, the material spends longer above the glass-transition region within each layer, promoting enhanced intermolecular diffusion and entanglement between filaments. These thermal signatures correlate directly with the observed mechanical behavior: the extended high-temperature exposure in direct printing leads to superior interlayer adhesion and higher tensile performance, whereas the more severe thermal cycling in plate fabrication reduces interlayer bonding strength.

Overall, the thermographic evidence confirms that interlayer adhesion in PLA is primarily controlled by the substrate temperature and the time the interface remains above T_g . Regardless of whether interlayer time, toolpath length, nozzle diameter, layer thickness, or specimen preparation method is modified, the governing mechanism is the same: effective bonding occurs only when the thermal cycle allows sufficient chain interpenetration before vitrification. This thermal framework underpins the interpretation of all subsequent mechanical results (fracture, tensile response, and delamination) within a unified and predictive perspective.

3.2.2 Mechanical Properties of PLA Tensile Specimens

This section consolidates the mechanical characterization of PLA tensile specimens manufactured under a diverse range of process conditions and fabrication strategies. Despite the variety of printing parameters, such as deposition speed, interlayer time, nozzle diameter, layer height, flow rate, and toolpath geometry, all results were assessed through the same tensile testing protocol. This procedure allows a unified interpretation of the underlying mechanisms governing mechanical performance.

3.2.2.1 Stress – strain response

The representative stress–strain curves for all manufacturing conditions are reported in Fig. 3. 11.

Across all datasets, the tensile response exhibits three dominant regimes:

- Linear elastic region, reflecting the stiffness of the filament network.
- Nonlinear hardening, leading to a clearly identifiable peak stress.
- Abrupt load drop, signifying brittle fracture with minimal post-peak deformation.

Despite this common shape, the magnitude of the mechanical properties varied significantly across specimen categories, revealing the sensitivity of interlayer adhesion to the thermal and morphological history.

- ***Directly printed specimens***, either with 2.5 mm or 3.0 mm thickness, exhibit the highest stress levels and the most extended nonlinear region before failure. The curves rise rapidly after yielding and maintain elevated stress up to strains of ~2–2.5%, indicating strong interlayer bonding and effective chain interdiffusion as illustrated in Fig. 3. 11 a). This behavior aligns with the short inter-pass times and elevated substrate temperatures characteristic of direct printing, which preserve the interface near or above the glass transition temperature long enough to promote molecular entanglement. ***Specimens extracted from larger width plates*** also display relatively high stiffness but lower peak stress and reduced ductility. Their stress–strain curves plateau earlier and fail at lower strains, reflecting the longer interlayer times and more severe thermal cycling associated with plate fabrication. These conditions cause the substrate temperature to drop below T_g before the subsequent layer arrives, limiting polymer mobility and weakening the interfacial weld.

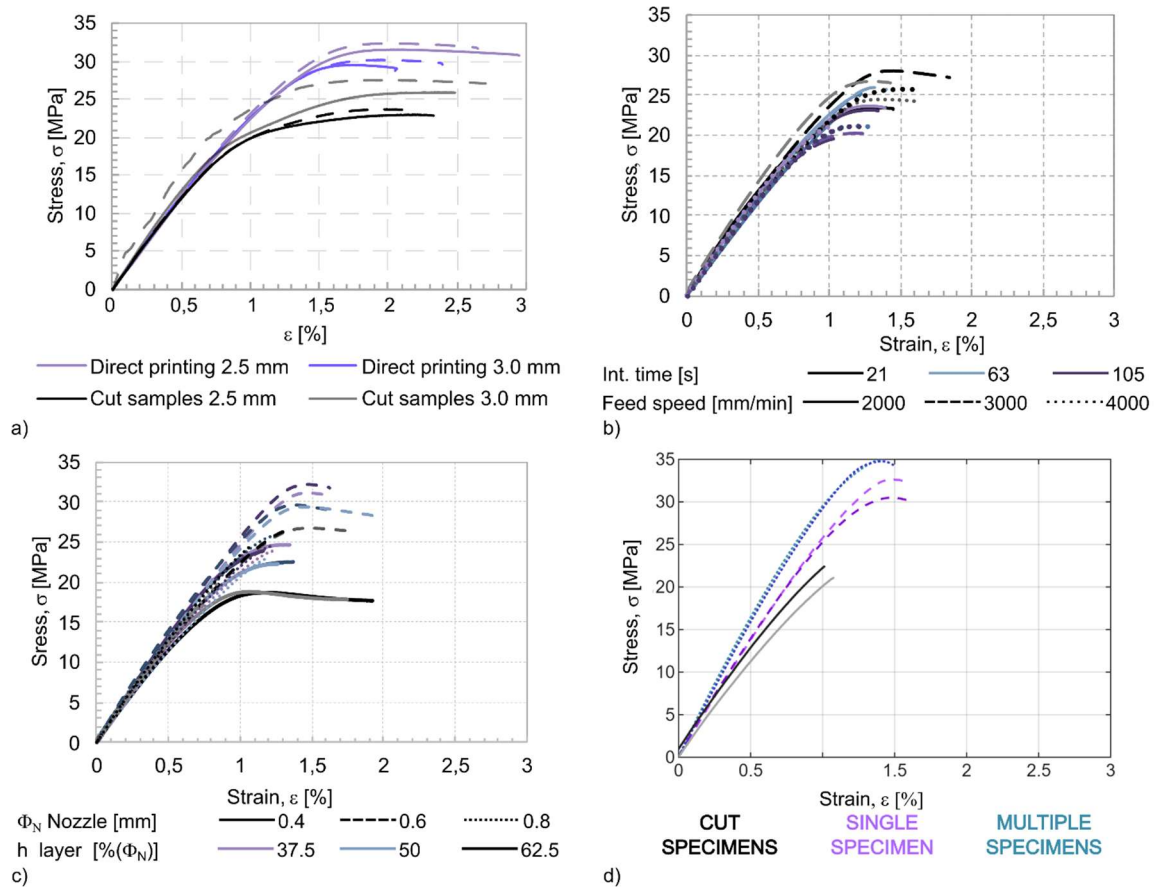


Fig. 3. 11: Representative tensile stress–strain curves of PLA specimens manufactured under different process conditions and fabrication strategies. The plots compare the influence of specimen preparation method (direct printing vs plate cutting (a)), interlayer time and deposition speed, (b), nozzle diameter and layer height, (c), and toolpath strategy (single-production vs multi-production vs extraction from flat-printed plate), (d), highlighting the strong dependence of tensile performance on interlayer bonding conditions.

- The influence of process timing is clearly visible in the curves corresponding to different interlayer times and deposition speeds. Short interlayer time (21 s) consistently produces higher peak stresses and more gradual post-yield softening, whereas long interlayer time (105 s) leads to a marked decrease in both strength and elongation at break. Similarly, the best-performing combination 3000 mm/min with 21 s shows an extended hardening region, while the worst-performing cases 3000 – 4000 mm/min combined with 105 s exhibit an abrupt post peak drop and brittle behavior. These observations corroborate the strong link between substrate temperature and interlayer adhesion: the longer the cooling interval, the lower the interlayer bond strength.
- Variations in nozzle diameter and flow rate produce another distinct family of stress–strain responses. Thin layers deposited with a small nozzle ($D = 0.4$ mm) result in higher stresses and a wider plastic regime due to reduced void size and higher specific energy input, which slow down cooling and strengthen interlayer bonding. Intermediate nozzle diameter ($D = 0.6$ mm), particularly when combined with a layer height equal to 37.5% of the nozzle diameter, yields the best overall behavior, with high peak stress and moderate ductility. In contrast, samples printed with a large nozzle ($D = 0.8$ mm) show a significantly steeper post-yield drop and fail almost immediately after

the elastic region. This brittle failure mode is attributed to the combination of larger inter-filament voids and insufficient polymer diffusion due to fast cooling.

- Finally, the comparison between single-pass, multi-pass, and plate-cut specimens confirms the positive role of toolpath-induced reheating. Multi-pass printing generates recurrent temperature recovery within the same layer, producing the highest stress levels and the most ductile behavior among the three. Conversely, plate-cut specimens suffer from weaker bonding and early delamination, consistently showing the lowest strength and the most limited strain capacity.

Overall, the aggregated curves reveal a coherent trend: higher and more sustained temperatures at the interlayer interface—achieved through shorter inter-pass times, smaller nozzle diameters, thinner layers, or reheating associated with specific toolpaths—lead to stronger interlayer adhesion and superior mechanical performance.

This thermal dependence manifests directly in the progression of the stress–strain curves, which serve as a mechanical fingerprint of the interlayer bonding quality established during deposition.

3.2.2.2 Mechanical Behavior: Tensile Properties of PLA

For each curve, the **Young's modulus**, the **tensile strength** (the highest value reached by the stress during the tensile test), and the **elongation at rupture** were determined. Thus, the average and the standard deviation were calculated to identify the influence of each deposition condition. The trends for the elastic modulus are summarized in Fig. 3. 12. Indeed, the tensile strength and the elongation at rupture are shown respectively in Fig. 3. 13 and Fig. 3. 14. Across all datasets investigated, the elastic region of the curves was highly consistent, confirming that the stiffness of MEX-printed PLA is only marginally influenced by processing parameters. In fact, Young's Modulus values result more influenced by the amount of deposited material and the amount of internal porosity. By contrast, both the peak strength (σ_u) and the elongation at break (ϵ_f) exhibited substantial variability across fabrication strategies, reflecting the sensitivity of interlayer consolidation to the thermal and morphological history established during deposition. To facilitate interpretation, the experimental conditions were grouped into four families corresponding to the different printing strategies considered in the four studies.

3.2.2.2.1 Young's Modulus

Fig. 3. 12 summarizes the Young's modulus values obtained across all manufacturing strategies investigated. Despite the wide range of process parameters: specimen thickness, fabrication strategy (direct printing vs. plate extraction), nozzle diameter, layer height, interlayer time, and toolpath geometry, the elastic response of PLA remains remarkably consistent, with all values lying between 2.40 and 2.80 GPa. This narrow variation band indicates that stiffness is the least sensitive mechanical property to changes in processing conditions, as commonly observed in semicrystalline polymers. The elastic stiffness of MEX-printed PLA is robust against process variations and primarily controlled by geometric features and void morphology, rather than interlayer adhesion phenomena.

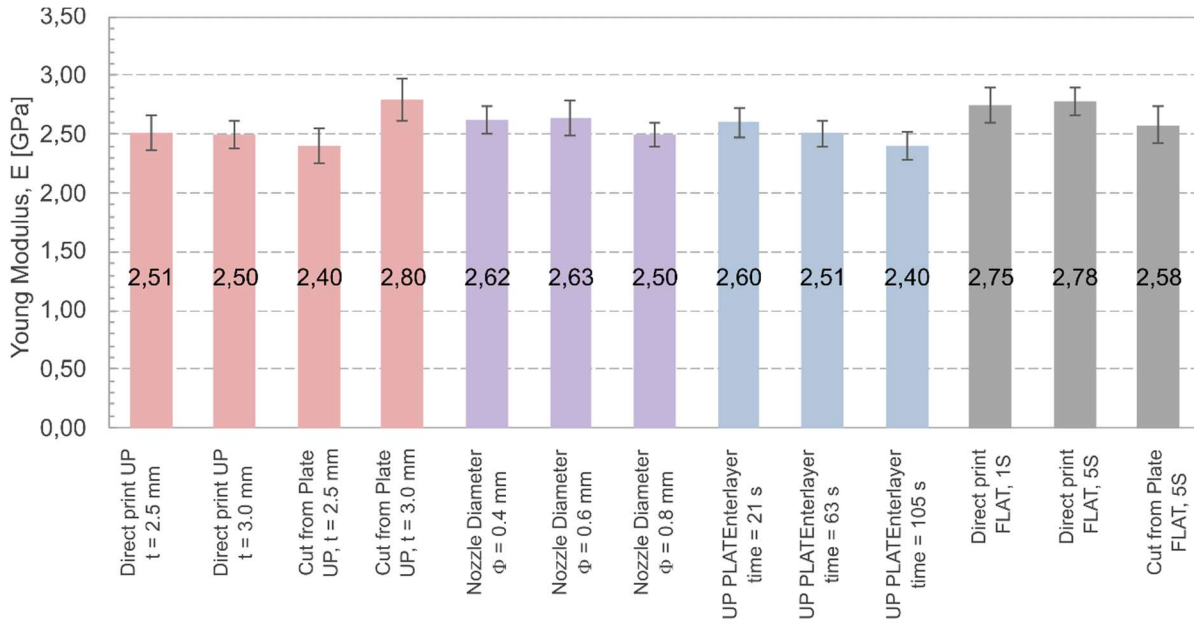


Fig. 3. 12: Young's modulus of PLA tensile specimens manufactured under different printing strategies and process conditions. Error bars represent the standard deviation obtained from replicate tests.

By contrast, the tensile strength (σ_u) and elongation at break (ϵ_f) of the PLA specimens exhibited substantial variability across manufacturing strategies and process conditions. Although all samples shared the same overall stress–strain shape, the magnitude of the peak stress and ductility was governed by the thermal and morphological characteristics established during deposition. For clarity, the results are discussed by grouping the printing conditions into four primary families.

3.2.2.2.2 Ultimate Tensile Strength

The consolidated results of ultimate tensile strength (σ_u) for all manufacturing strategies are summarized in Fig. 3. 13(a–d). Despite the diversity of printing parameters investigated across the four studies, clear and consistent trends emerge, reinforcing the dominant role of thermal history and interlayer bonding quality on the mechanical performance of PLA components.

Fig. 3. 13(a) compares directly printed specimens with those extracted from plates, for two different thicknesses (2.5 mm and 3.0 mm). Directly printed specimens systematically exhibit higher tensile strength, with $\sigma_u \approx 31$ – 32 MPa, whereas plate-extracted samples range from 21 to 28 MPa. In addition, the effect of thickness is opposite for the two fabrication strategies. For direct printing, thinner specimens show slightly higher strength owing to shorter interlayer times and hotter substrates. Conversely, in plate-cut specimens, the thicker walls exhibit higher σ_u , reflecting the greater thermal inertia and slower cooling that favor interlayer adhesion. These observations correlate well with the infrared measurements discussed previously: during direct printing, the substrate remains significantly hotter, while the plate cools rapidly and non-uniformly, reducing the available time for interdiffusion.

Fig. 3. 13(b) isolates the effect of interlayer time (21, 63, and 105 s) and deposition speed (2000–4000 mm/min), grouped through the corresponding substrate temperature T_{sub} . A clear monotonic behavior emerges: short interlayer time (21 s) results in the highest substrate temperature (≈ 52 – 53 °C) and yields $\sigma_u \approx$

25–26 MPa; intermediate interlayer time (63 s) lowers T_{sub} to $\approx 45\text{--}46\text{ }^{\circ}\text{C}$ and strength to $\approx 24\text{--}25\text{ MPa}$; finally, long interlayer time (105 s) produces the coldest substrate ($\approx 42\text{--}44\text{ }^{\circ}\text{C}$) and the lowest $\sigma_u \approx 21\text{ MPa}$. Deposition speed does not introduce a systematic trend, confirming that the principal controlling parameter for strength is the interlayer time—and the associated cooling behavior—rather than the feed rate. The nearly linear correlation between σ_u and T_{sub} mirrors the thermal analyses presented in Section 3.2.2.1.

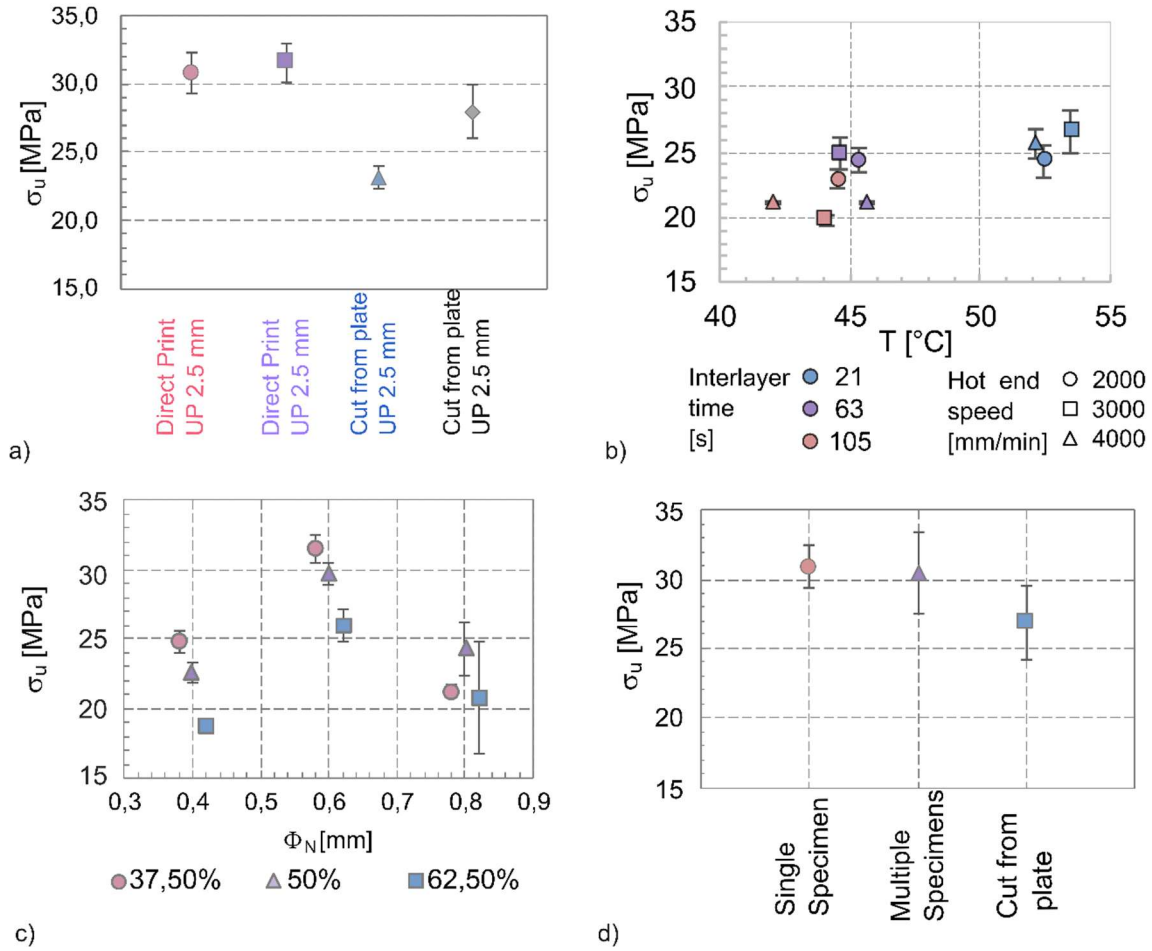


Fig. 3. 13: Tensile strength from PLA ASTM D638 T1 specimens from different printing conditions: a) Influence of specimens thickness and component dimensions; b) Evaluation of interlayer time due to the different printing speed; c) Flow rate dependence (nozzle diameter and layer height); d) Flat orientation: printing of single, multiple specimens, and flat larger plate (specimens obtained by AWJ machining).

Fig. 3. 13(c) presents the influence of nozzle diameter (0.4, 0.6, 0.8 mm) and of layer height expressed as a percentage of the nozzle diameter. Three main features emerge. First, the 0.6 mm nozzle provides the most favorable balance between deposited volume, thermal inertia, and interfacial pressure, consistently yielding the highest strength ($\approx 30\text{--}32\text{ MPa}$). Second, for the 0.4 mm nozzle, σ_u decreases as layer height increases, consistent with reduced filament compaction and shorter cooling times. Third, the 0.8 mm nozzle systematically results in lower σ_u ($\approx 22\text{--}24\text{ MPa}$) with greater scatter; these samples display brittle behavior, reflecting the presence of larger voids and limited interfacial entanglement.

Finally, Fig. 3. 13(d) compares single-specimen direct printing, multiple-specimen direct printing, and extraction from a larger plate. Multiple-pass direct printing shows the highest σ_u (≈ 32 MPa), attributable to increased heat accumulation along overlapping toolpaths and slower cooling. Single-pass printed specimens exhibit slightly lower strength (≈ 30 MPa), while plate-extracted samples remain the weakest (≈ 26 – 27 MPa), due to steep thermal gradients and extended inter-pass distances. This comparison demonstrates directly that toolpath geometry alone can modify tensile strength by more than 20 %, even under nominally identical printing parameters.

3.2.2.2.3 Elongation at Break

The elongation at break (ϵ_f) displayed the greatest sensitivity to processing conditions among all mechanical properties, confirming ductility as the most direct and discriminating indicator of interlayer bonding quality in MEX-printed PLA. The consolidated results for all manufacturing strategies are summarized in Fig. 3. 14(a–d).

In Fig. 3. 14(a), directly printed specimens systematically exhibit the highest ductility, with ϵ_f generally between 1.8% and 2.6%, whereas specimens extracted from plates show markedly lower values, often close to or below 1.2%. As observed for tensile strength, the influence of specimen thickness differs between the two fabrication routes. Thinner directly printed specimens (2.5 mm) retain slightly higher ductility due to shorter interlayer times and elevated substrate temperatures, which promote enhanced interfacial mobility. Conversely, thicker plate-cut specimens benefit from the higher thermal inertia of the surrounding material, exhibiting slightly improved ϵ_f relative to thinner plate-cut samples, though still significantly lower than directly printed ones. These differences align with the documented thermal histories, confirming the detrimental effect of steep, rapid cooling when specimens are extracted from large components.

The influence of interlayer time and deposition speed is reported in Fig. 3. 14(b), through the substrate temperature T_{sub} measured just before deposition. The trend mirrors that observed for tensile strength but is more pronounced. Short interlayer times (21 s), associated with the highest substrate temperatures (~ 52 – 53°C), produce the highest ductility, with ϵ_f approaching or exceeding 1.6%. Increasing the interlayer time to 63 s reduces T_{sub} to approximately 45°C and lowers ϵ_f to ~ 1.2 – 1.4% . Under the longest interlayer time (105 s), the substrate temperature falls near 42°C , and the ductility declines to ~ 0.9 – 1.1% . Deposition speed does not introduce a systematic influence, further confirming that thermal exposure above T_g —not feed rate—is the dominant variable governing interfacial ductility. The nearly linear relationship between ϵ_f and T_{sub} emphasizes the central role of the substrate temperature in determining the extent of polymer chain interdiffusion.

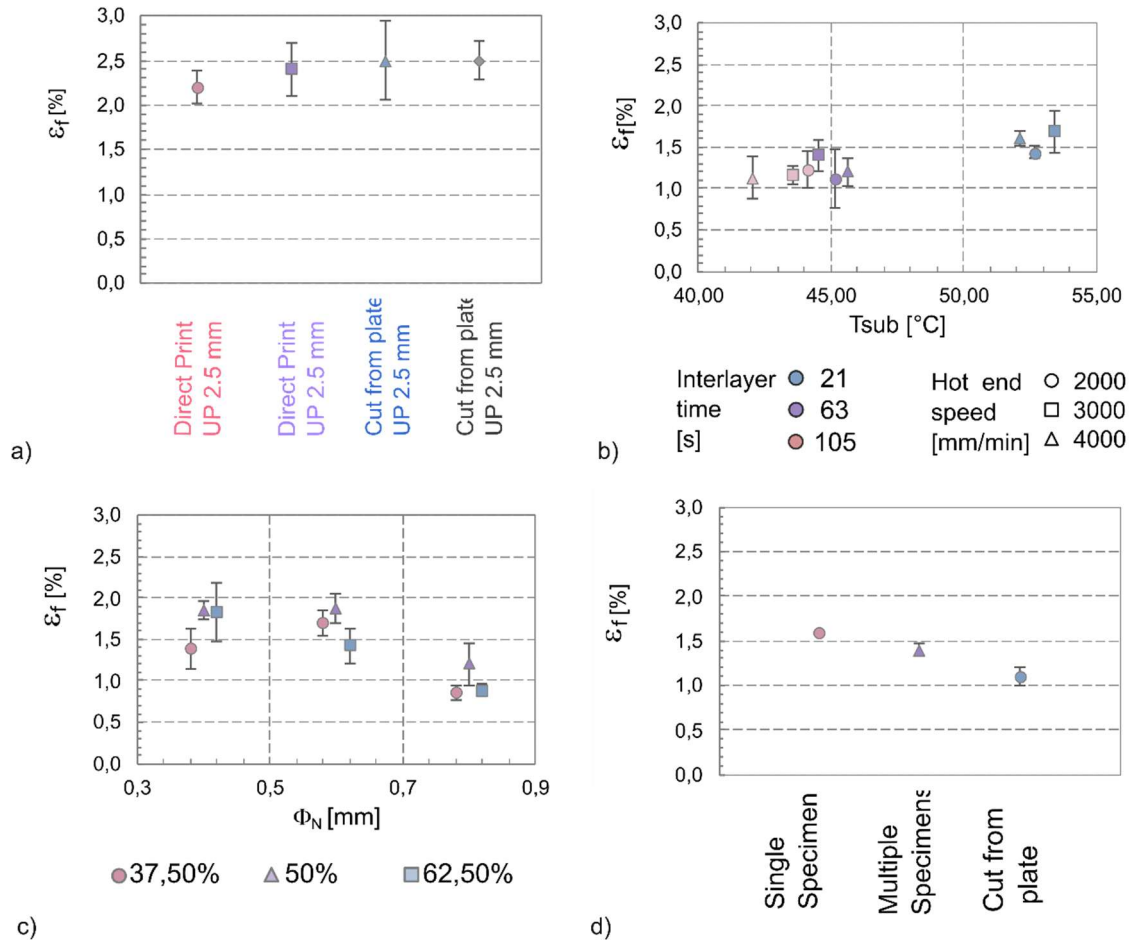


Fig. 3. 14: Elongation at break (ϵ_f) for all manufacturing strategies investigated: (a) Direct printing vs. plate-cut specimens; (b) Effect of interlayer time and substrate temperature; (c) Influence of nozzle diameter and layer height; (d) Effect of printing strategy (single, multiple, plate-cut).

To further assess whether the reduction in tensile strength and ductility at longer interlayer times is associated with changes in bulk porosity, cross-sections were examined for specimens printed at a deposition speed of 3000 mm/min with interlayer times of 21 s and 105 s. As shown in Fig. T_X, both conditions exhibit a broadly similar distribution of interfilament voids, with no appreciable increase in pore area or characteristic void size at 105 s. This evidence supports the conclusion that the degradation of σ_u and ϵ_f with increasing interlayer time is not primarily driven by a loss of apparent density, but rather by a deterioration of interlayer adhesion caused by lower substrate temperatures and reduced polymer-chain interdiffusion.

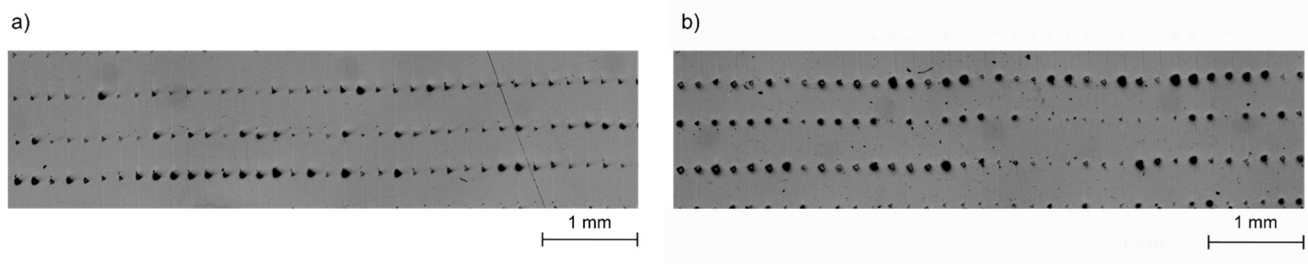


Fig. 3. 15: cross-sections of specimens printed at $v = 3000$ mm/min with interlayer times of 21 s and 105 s

The effects of nozzle diameter and layer height, shown in Fig. 3. 14(c), follow the same physical logic. The 0.6 mm nozzle yields the highest elongation for the tested layer heights, with ϵ_f values around 1.8–2.2% for the 37.5% and 50% layer-height conditions. This confirms that the balance of flow rate, interlayer pressure, and thermal inertia offered by the 0.6 mm nozzle is optimal for ductility. For $\Phi_n = 0.4$ mm, ductility decreases as layer height increases, consistent with reduced contact area and reduced time above T_g . The poorest performance is consistently observed for $\Phi_n = 0.8$ mm, where samples fail in a brittle manner with ϵ_f values as low as $\sim 0.9\%$. This behavior correlates with the presence of large voids and insufficient thermal stabilization during deposition, as observed in the morphological analyses of cross-sections.

Finally, Fig. 3. 14(d) compares single-specimen direct printing, multiple-specimen direct printing, and plate-cut specimens under flat orientation. Multiple-specimen direct printing leads to the highest ductility, confirming the beneficial effect of cumulative heating and reduced cooling rates when adjacent specimens are deposited sequentially. Single-printed specimens retain moderately high ϵ_f values, though slightly lower than the multi-printed ones due to reduced heat accumulation. Plate-extracted specimens again exhibit the lowest ductility ($\sim 1.1\%$), consistent with their steep thermal gradients and limited interlayer molecular mobility.

Overall, the elongation at break consolidates the conclusions drawn from the tensile strength analysis. While the strength varied by roughly 20–30% across printing strategies, ductility varied by nearly a factor of two, making ϵ_f the most sensitive mechanical signature of the underlying thermal history. These results confirm a unified mechanistic picture: processing conditions that sustain the interlayer region above the glass transition temperature for a longer period invariably produce superior ductility, enhanced interfacial bonding, and mechanical reliability in PLA.

3.2.3 Microstructural Analysis

Microstructural observations were performed to elucidate how fractured mechanisms, filament morphology, and internal porosity govern the tensile behavior of PLA fabricated under the different processing strategies examined in this study.

Reflected-light microscopy of fracture surfaces reveals two dominant failure modes: intralayer fracture and interlayer delamination. Directly printed specimens predominantly fail within individual filaments, generating tortuous, rough surfaces with clear evidence of plastic deformation, filament tearing and local fibrillation. By contrast, specimens extracted from larger components exhibit extended regions in which the fracture propagates along bead–bead interfaces, producing flatter and smoother surfaces indicative of reduced interlayer adhesion. These characteristic features—including layer start/stop regions and edge-related overfill—are illustrated in Fig. 3. 16 for both fabrication routes.

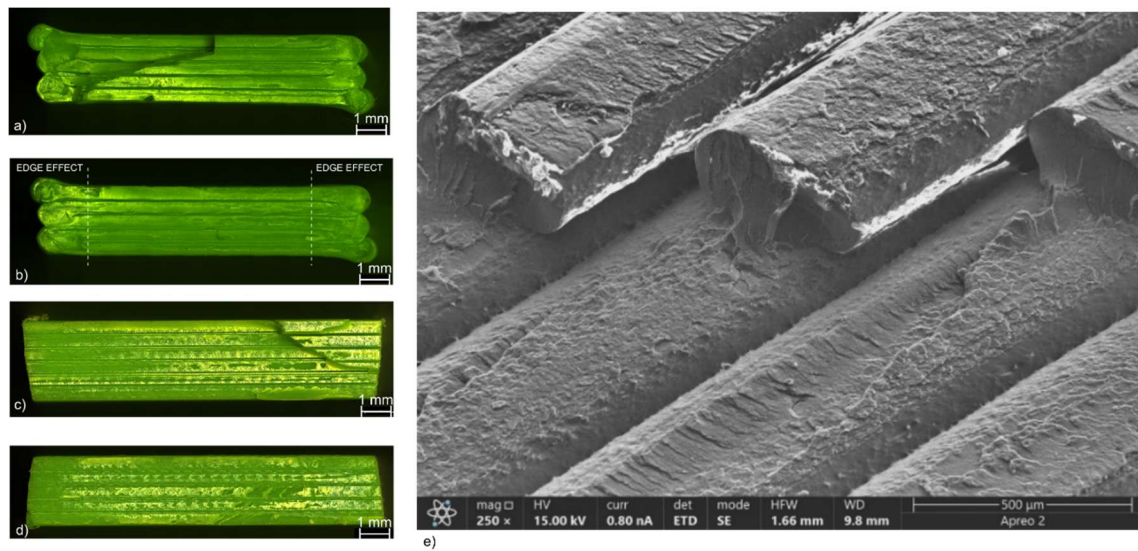


Fig. 3. 16: Optical fracture surfaces of PLA tensile specimens: (a–b) directly printed samples with intralayer fracture and visible edge effects; (c–d) plate-cut specimens showing prevalent interlayer delamination.

Scanning electron microscopy provides higher-resolution confirmation of these trends. Directly printed samples show widespread filament necking and fibrillation, with rough, ductile microfeatures consistent with strong interfilamentous bonding. Conversely, plate-cut specimens display smoother planar regions, limited filament rupture, and mixed cohesive–adhesive failure, consistent with their lower tensile strength and elongation at break. Representative SEM images at increasing magnification are reported in Fig. 3. 17.

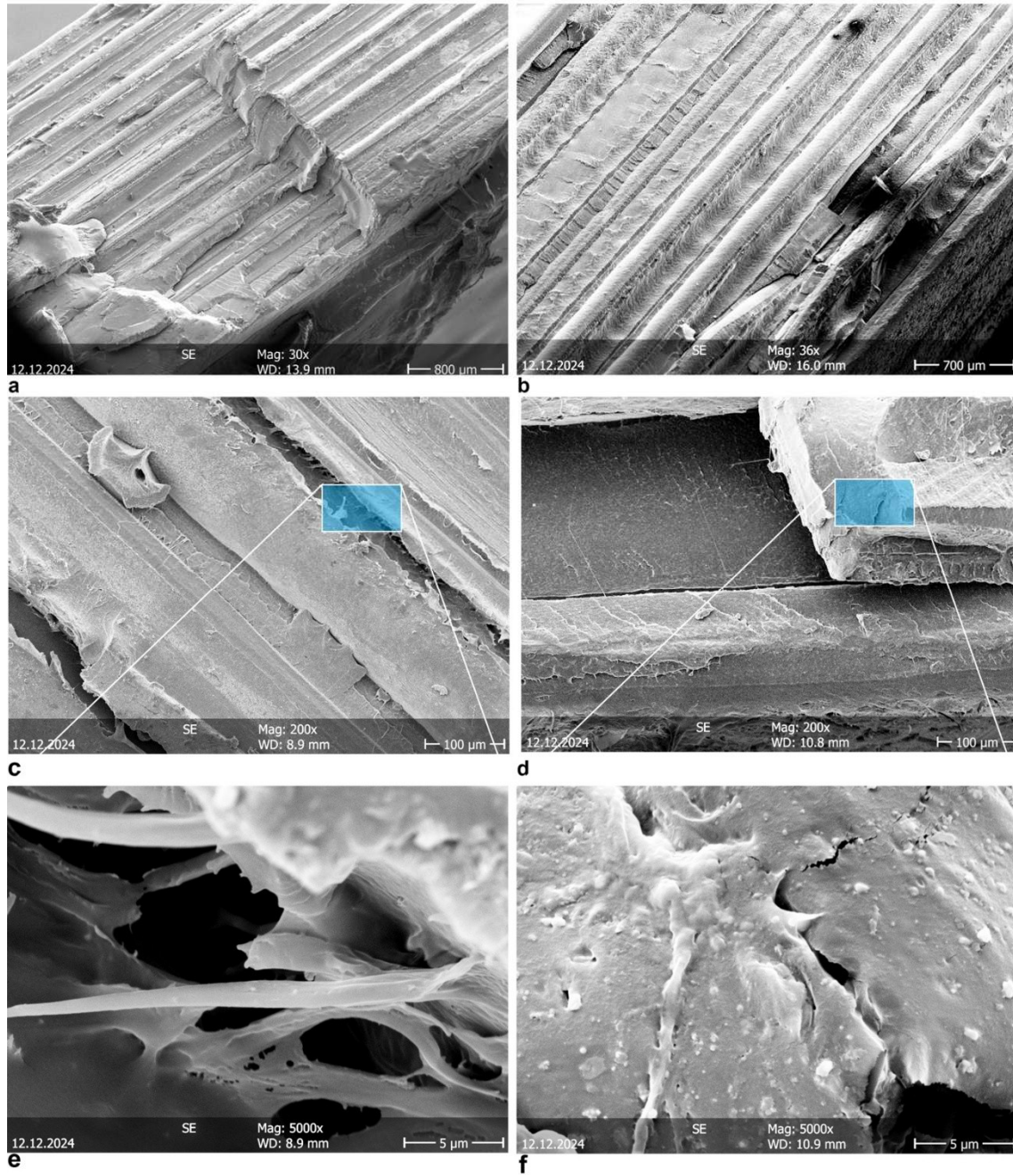


Fig. 3. 17: SEM micrographs of PLA fracture surfaces: (a,c,e) directly printed samples showing intralayer rupture and extensive fibrillation; (b,d,f) plate-cut specimens with pronounced delamination and smoother interfaces.

To quantify the internal structure, metallographic cross-sections were prepared for directly printed and plate-extracted specimens manufactured under identical nominal printing conditions. As shown in Fig. 3. 18, both sets of samples contain interfilamentous voids, but with distinct morphological distributions: directly printed specimens contain fewer but slightly larger voids, while plate-cut specimens show a denser population of smaller pores. These trends are quantitatively confirmed by the corresponding histograms of void area and major-axis length reported in Fig. 3. 18(c,d). These results indicate that the small variations in Young's

modulus observed between fabrication strategies cannot be attributed solely to porosity fraction; rather, pore morphology and the quality of filament coalescence play a stronger role in mechanical performance.

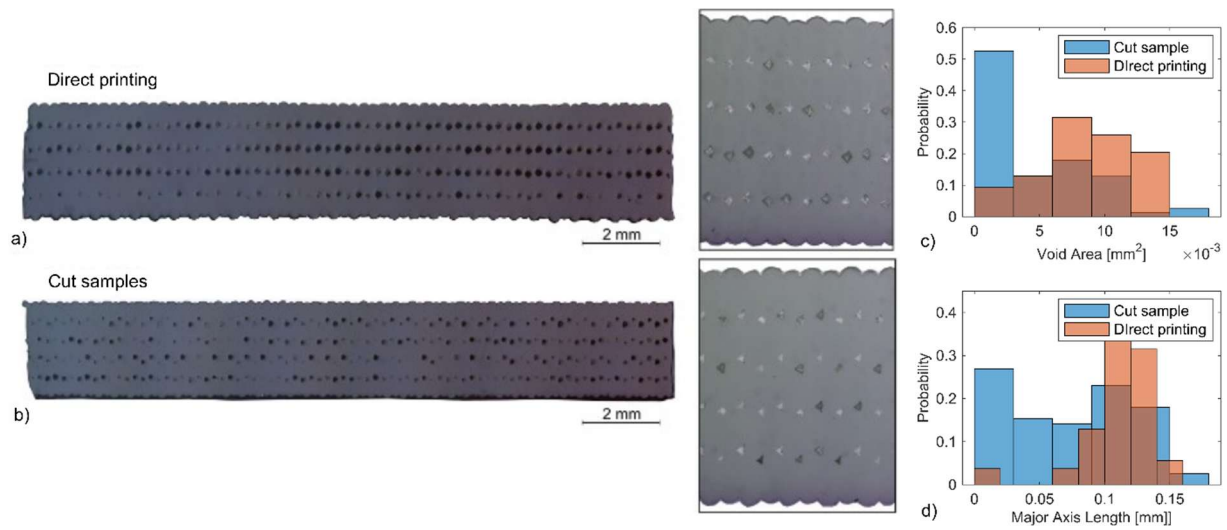


Fig. 3. 18: Metallographic analysis of cross-sectional porosity in PLA tensile specimens manufactured by direct printing and plate extraction. (a) Directly printed specimen and (b) plate-cut specimen under identical nominal printing conditions. (c) Probability distribution of void area and (d) distribution of void major-axis length, highlighting differences in pore morphology between the two fabrication strategies.

The combined influence of nozzle diameter and layer height on porosity was further examined by selecting three representative deposition conditions: $\Phi = 0.4 \text{ mm} - h = 0.20 \text{ mm}$, $\Phi = 0.6 \text{ mm} - h = 0.23 \text{ mm}$, and $\Phi = 0.8 \text{ mm} - h = 0.50 \text{ mm}$, as shown in Fig. M4. The metallographic cross-sections reveal how the amount of material extruded per filament strongly affects both the size and morphology of interfilamentous voids.

Specimens printed with the 0.4 mm nozzle exhibit a dense distribution of very small, uniform voids. This morphology results from the rapid solidification of the thin filaments and the relatively low compaction pressure achieved during deposition. In contrast, the 0.8 mm nozzle, coupled with a much larger layer height, generates significantly larger voids (up to 0.8–1.0 mm), with irregular shapes and broader dispersion. Such porosity is consistent with the brittle mechanical response observed for these printing conditions.

The intermediate configuration (0.6 mm – moderate layer height) provides an optimal condition, with small, uniformly distributed voids and no localized accumulations. This reflects a favorable balance between extruded volume, cooling time, and interfilamentous contact pressure, which aligns with the superior mechanical performance recorded in these samples.

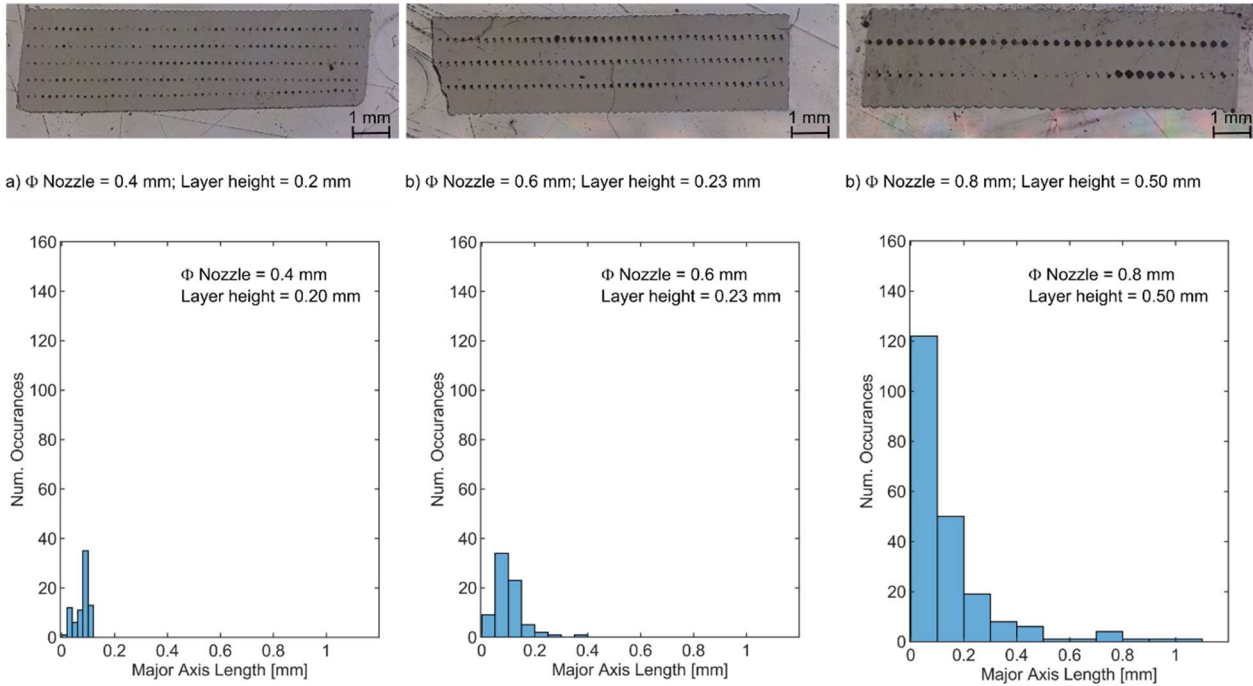


Fig. 3.19: Influence of nozzle diameter Φ_n and layer height (expressed as % of Φ_n) on internal porosity: metallographic cross-sections and histograms of void major-axis length for three representative conditions (0.4 mm–50%, 0.6 mm–37.5%, 0.8 mm–62.5%). Larger nozzles combined with thick layers promote the formation of larger and more elongated voids, in agreement with the observed reduction in tensile strength and ductility.

Overall, microstructural evidence demonstrates that porosity and fracture morphology are governed not by a single processing variable but by the coupled interplay of filament geometry, thermal dissipation, interlayer time and deposition kinematics. These factors determine local viscosity, spreading behavior, and the extent of polymer chain interdiffusion during consolidation, ultimately dictating the mechanical integrity of the printed PLA components.

3.3 Mechanical characterization of PAEK Components: tensile behavior

After the detailed analysis carried out on PLA, the same experimental framework was applied to aerospace-grade PAEK to validate the methodology on a high-performance semicrystalline polymer. Tensile properties were evaluated for two manufacturing strategies:

- I. Dog-bone specimens directly printed as isolated samples, and
- II. Dog-bone specimens extracted from full components produced under industrial MEX conditions.

This comparison is crucial because qualification in aerospace requires that mechanical data represent the behavior of real parts rather than idealized laboratory coupons. All the tensile test specimens are manufactured according to ASTM D638 T1. During tensile testing, most fractures occurred within the gauge length, although some directly printed specimens exhibited failure slightly outside the mid-section. This behavior is consistent with the presence of local geometric irregularities inherent to single-specimen deposition. In contrast, all plate-cut specimens fractured exactly at mid-gauge, demonstrating higher geometric uniformity.

Macroscopically, the samples also displayed different visual and surface characteristics. Directly printed specimens showed a light opaque beige color and slightly irregular edges, typical of the MEX process, indicating the onset of crystallization during deposition. Plate-cut samples, instead, appeared translucent brown in the as-printed condition, consistent with a predominantly amorphous microstructure, and became uniformly opaque only after annealing.

These preliminary visual differences already suggest that manufacturing strategy influences both microstructure and deformation behavior. A detailed analysis of fracture morphology, internal porosity and surface features is provided in Section 3.3.4 (Optical analysis), where microscopic evidence is directly correlated with mechanical performance.

3.3.1 Stress–strain response

The stress–strain curves collected for all configurations are shown in Figure Fig. 3. 20. All samples exhibit an initial linear elastic region, followed by a clear peak load and a subsequent plateau corresponding to plastic deformation. However, the extent of the plastic region strongly depends on the manufacturing strategy. Directly printed specimens experience an abrupt stress drop immediately after peak load, failing in a brittle manner with very limited plasticity. In contrast, samples extracted from the plat both as printed and annealed show extended plastic deformation before fracture, indicating a fundamentally different failure mechanism.

This divergence is the first major evidence that directly printed tensile coupons do not replicate the consolidation mechanisms of full components and therefore provide a non-conservative estimate of mechanical performance.

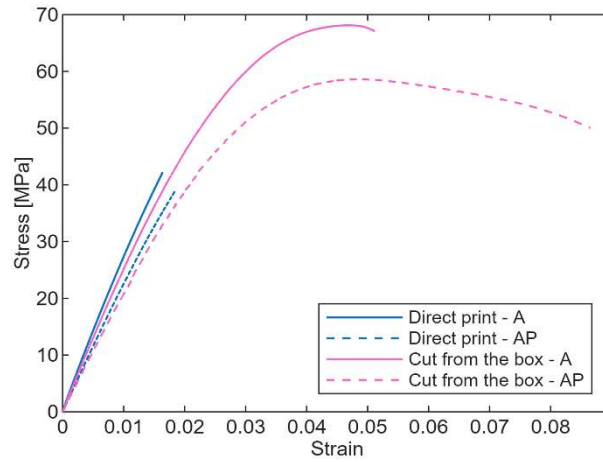


Fig. 3. 20: Representative tensile stress–strain curves of PAEK specimens manufactured using different fabrication strategies and post-processing conditions. The curves compare directly printed tensile specimens and samples extracted from a printed plate, both in the as-printed (A) and annealed (AP) conditions. Mechanical properties: E , σ_{max} , ϵ_r :

The tensile properties obtained from the elaboration of stress–strain curves are shown in Fig. 3. 21.

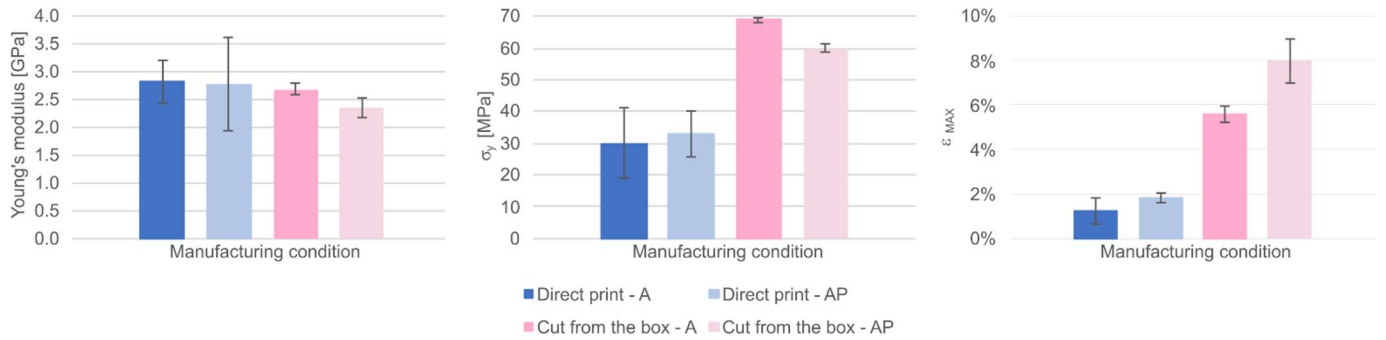


Fig. 3. 21: Mechanical properties of PAEK tensile specimens manufactured using different fabrication strategies and post-processing conditions. From left to right: Young's modulus (E), ultimate tensile strength (σ_{max}), and elongation at rupture (ϵ_r) for directly printed specimens and specimens extracted from a printed plate, both in the as-printed (A) and annealed (AP) conditions.

Despite the diversity in manufacturing strategies, the Young's modulus remains remarkably consistent across all conditions, averaging approximately 2.66 GPa. Neither annealing nor the printing strategy significantly affects the stiffness, confirming that the elastic response is primarily governed by overall density and bead geometry—parameters that do not differ substantially between the specimen types.

In contrast, peak stress and elongation at rupture show substantial variation. Directly printed specimens reach only ~31 MPa, roughly half the strength of cut samples (60 MPa as printed, 69 MPa annealed). Ductility follows the same trend: direct printing results in very limited elongation (~2%), whereas as-printed cut samples reach ~8%, and annealed cut samples ~6%. This reinforces the central observation that the mechanical integrity of PAEK is dominated by interlayer adhesion quality and thermal history.

3.3.2 Microscopical Analysis: Optical and SEM

Optical and electron microscopy were employed to clarify the failure mechanisms associated with the different manufacturing strategies and to relate them to the tensile response.

Fracture surfaces observed under reflected-light microscopy reveal two main failure modes: interlayer delamination and intralayer fracture. In directly printed specimens, the crack path predominantly follows the interfaces between adjacent layers, producing extended, relatively smooth and planar regions that are characteristic of delamination. By contrast, specimens cut from the larger component, especially after annealing, more frequently exhibit intralayer fracture, with cracks intersecting and breaking individual filaments rather than propagating along the interfaces. These differences between annealed and “as-printed” cut specimens are clearly visible in the optical images reported in Fig. 3. 22. In fact, Fig. 3. 22 a) illustrates a plate-cut annealed fracture surface, which displays rougher, more tortuous surfaces associated with stronger interlayer bonding. Moreover, this image shows a fracture that involves three following layers. This fracture mechanism is indicative of a much stronger adhesion between the layers achieved through the annealing post process. Whereas as-printed cut specimens show smoother, interface-controlled fracture regions, as depicted in Fig. 3. 22 b).

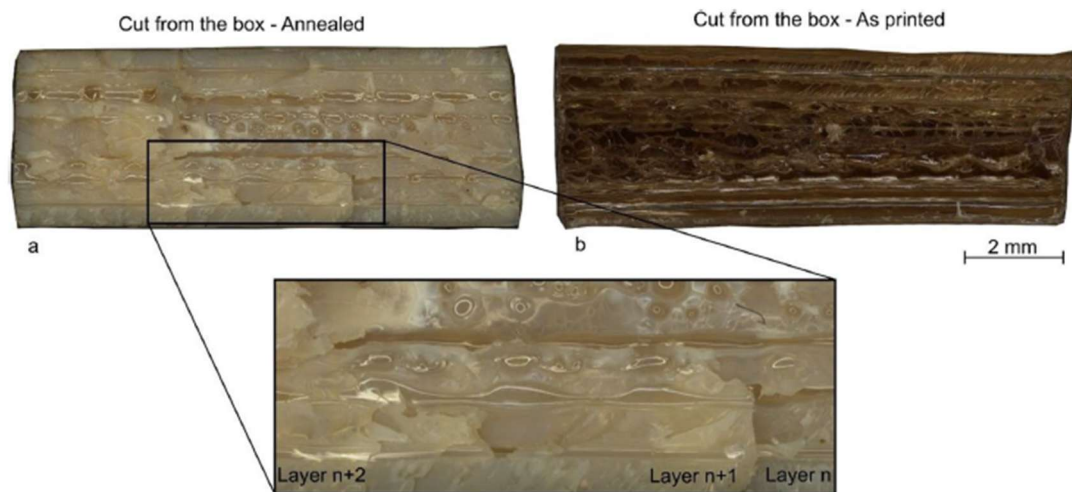


Fig. 3. 22: Optical microscopy images of fracture surfaces of PAEK tensile specimens extracted from a printed plate. (a) Annealed specimen, showing a rough and tortuous fracture surface with crack propagation across multiple consecutive layers (layers n , $n+1$, and $n+2$), indicative of strong interlayer adhesion and intralayer fracture mechanisms. (b) As-printed specimen, characterized by smoother and more planar fracture surfaces dominated by interlayer delamination

These macroscopic fracture features are confirmed and refined by SEM observations. A representative comparison between the two failure modes at increasing magnifications is shown in Fig. 3. 23. At low magnification, directly printed samples present wide delaminated areas with limited filament rupture, in agreement with their lower tensile strength and reduced elongation at break. At higher magnification, as illustrated in Fig. 3. 23 a) the fracture surface remains relatively smooth, with only modest fibrillation and scarce evidence of extensive plastic deformation, indicating that failure is governed by interlayer decohesion. In contrast, SEM micrographs of specimens cut from is presented in Fig. 3. 23 b): the plate after annealing

reveal highly fragmented fracture surfaces, with marked filament tearing, rough morphology and localized plastic flow. This intralayer-dominated failure mode is consistent with the higher strength and ductility measured in these samples and reflects a significant improvement in interlayer cohesion.

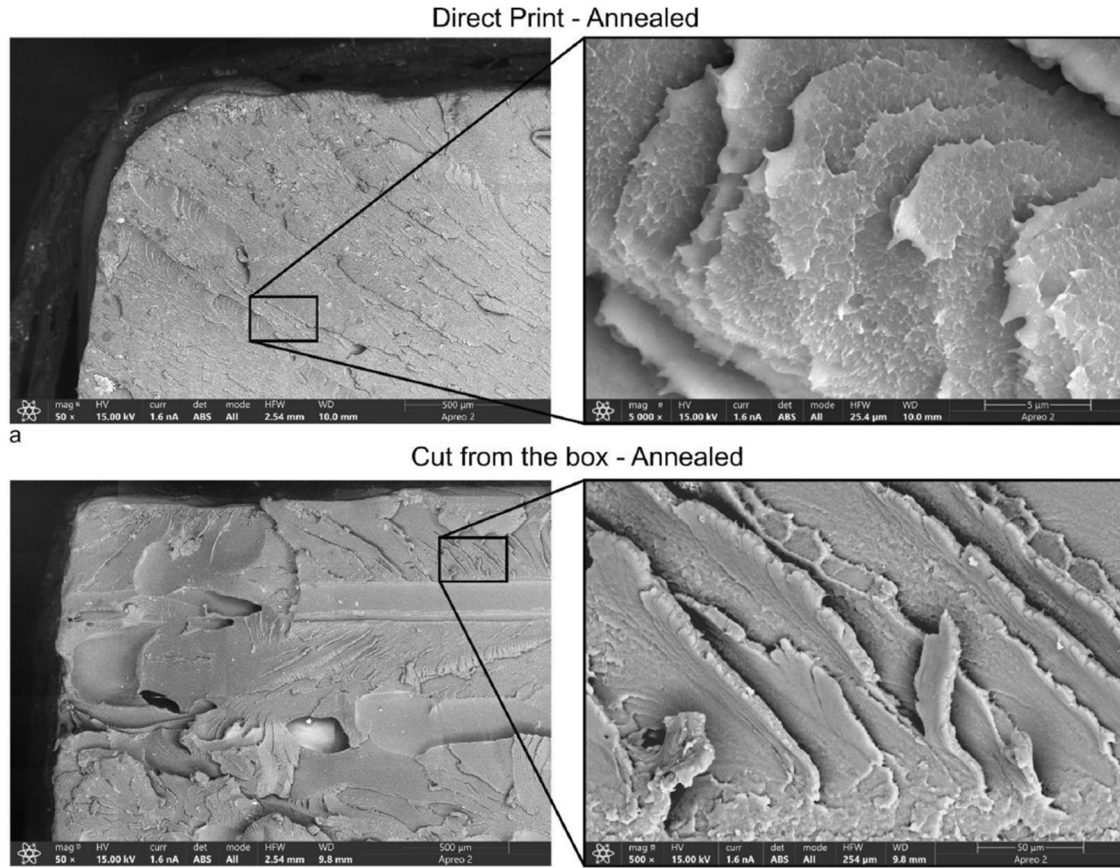


Fig. 3. 23: SEM images of fracture surfaces of annealed PAEK tensile specimens manufactured by different strategies. (a) Directly printed specimen, showing extended interlayer delamination with relatively smooth fracture surfaces and limited filament tearing, indicative of interlayer-controlled failure. (b) Specimen cut from a printed plate, exhibiting rough and highly fragmented fracture surfaces with pronounced filament rupture and plastic deformation, consistent with intralayer-dominated failure and enhanced interlayer cohesion

Additional insight is provided by surface topography analyses of the gauge region depicted in Fig. 3. 24. Directly printed specimens exhibit wavy edges and localized grooves associated with acceleration/deceleration phases of the printhead and with changes in toolpath direction. These geometrical artifacts act as stress concentrators and help explain why some directly printed samples could fail outside the exact mid-gauge section and show a larger scatter in tensile properties. Conversely, specimens cut from the plate exhibit a much more regular and planar surface, with shallower grooves and higher geometric uniformity, which supports the improved repeatability and higher average mechanical performance. This contrast in surface morphology is illustrated in Fig. 3. 24. The main cause of these geometric artifacts could be addressed to the acceleration/deceleration that occur at the direct printed samples' edges. Indeed, at the edge, the extruder change in direction leads to an acceleration up to the selected printing speed from zero. Similarly, as the extruder approaches the edges, it decelerates from the set speed to zero. Within acceleration and deceleration ramp regions, uneven material flow is produced since the difficulty (for the MEX machine)

to precisely control the material feeding under such speed gradients. This is also due the low viscosity of the material and pressure variations within and underlying the nozzle, which makes very complex the material flow control under unsteady deposition conditions.

Overall, the optical and SEM observations converge on a coherent picture: directly printed PAEK specimens are limited by weaker interlayer adhesion and local geometric defects, leading to premature delamination and brittle failure, whereas plate-cut specimens, particularly after annealing, benefit from stronger interlayer bonding and smoother, more homogeneous gauge surfaces, which shift failure towards intralayer fracture and enable higher tensile strength and elongation at break.

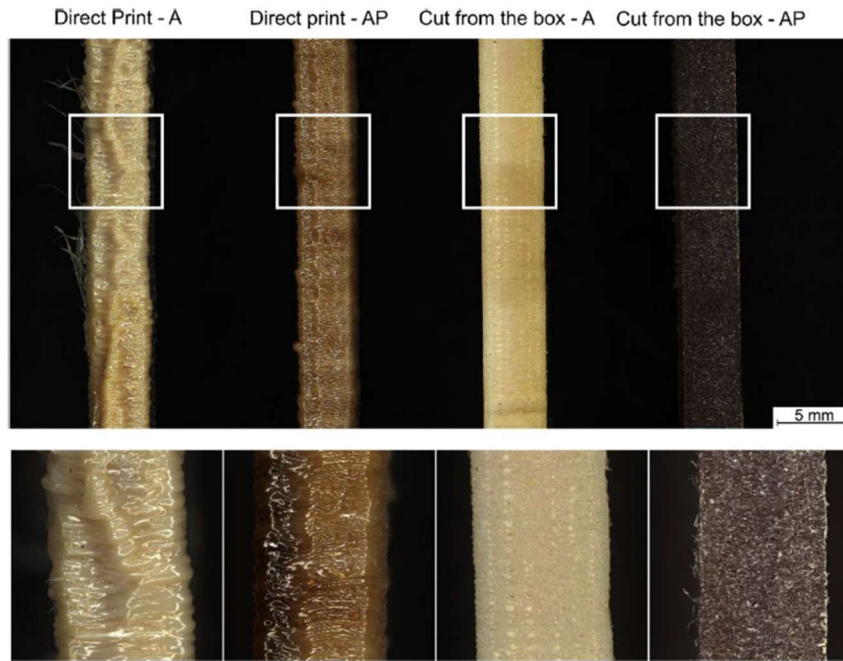


Fig. 3. 24: Optical microscopy images of the gauge region of PAEK tensile specimens manufactured using different fabrication strategies and post-processing conditions: directly printed as-printed (Direct print – A), directly printed annealed (Direct print – AP), plate-cut as-printed (Cut from the box – A), and plate-cut annealed (Cut from the box – AP). Top row: full gauge-length views with highlighted regions of interest; bottom row: magnified views of the selected areas. Directly printed specimens exhibit pronounced surface waviness, grooves, and geometric irregularities associated with printhead acceleration/deceleration and toolpath changes. In contrast, plate-cut specimens show smoother, more planar, and geometrically uniform surfaces, supporting improved repeatability and enhanced mechanical performance

3.3.3 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry was performed to quantify the degree of crystallinity associated with the different manufacturing strategies and thermal states of the PAEK specimens. The results allow correlating crystallization kinetics with the tensile behavior discussed in the previous sections.

DSC heating curves for all conditions: directly printed samples, plate-cut samples in the as-printed state, and plate-cut samples after annealing are shown in Fig. 3. 25. Each thermogram exhibits a distinct cold-crystallization peak followed by the melting endotherm, from which crystallization percentages were calculated.

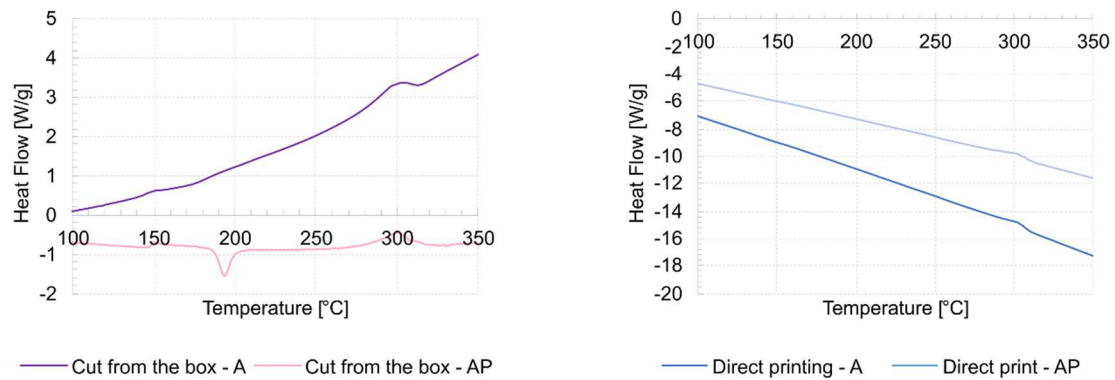


Fig. 3. 25: DSC curves of the samples produced through different deposition and post-process conditions

The melting enthalpies and the cold crystallization enthalpies were calculated for each condition to determine the crystallization percentages, which are summarized in Tab III. 2.

Tab III. 2: Differential scanning calorimetry (DSC) results for PAEK specimens manufactured using different fabrication strategies and post-processing conditions.

Manufacturing condition	ΔH_{Cryst} [J/g]	ΔH_{Melt} [J/g]	χ [%]
Direct print — as printed	—	15.01	11.55
Direct print — annealed	—	15.11	11.62
Direct print — annealed	— 16.66	20.91	3.27
Cut from the box — annealed	—	15.31	11.77

As can be inferred, the percentage of crystallinity of the samples cut from the box under “as-printed” condition was 3.27%; such value was much smaller than that achieved after the annealing post-treatment (11.77%). These values confirm the almost amorphous nature of the as-printed samples cut from the box and the increase in the crystallization after the annealing process. On the other hand, the directly printed samples under “as-printed” condition showed a much higher percentage of crystallinity (12.18%). This confirms the onset of an incipient crystallization achieved during the deposition process owing to the smaller layer dimension which led to shorter interlayer time and higher substrate temperature. This promoted the crystallization observed on the directly printed samples under “as-printed” conditions.

3.4 Compressive Characterization of Architected Lattice Structures

Additive manufacturing of lattice structures has emerged as one of the most effective strategies for designing lightweight components with tailored stiffness, controlled energy absorption, and progressive failure under compression. Compared with their solid counterparts, cellular architectures provide an exceptional strength-to-weight ratio and can be engineered to exhibit either bending-dominated or stretching-dominated behavior depending on geometry, relative density, and deformation mode — as widely discussed in the foundational framework of Gibson and Ashby[74].

Material Extrusion (MEX/FDM) and powder-bed fusion technologies such as Multi Jet Fusion (MJF) have made it possible to fabricate these architectures using both high-performance semicrystalline polymers (e.g., PEEK, PA6-CF) and elastomeric materials (e.g., TPU). Each material and process combination exhibits distinct thermal, rheological, and morphological characteristics that directly affect strut quality, interlayer bonding, and ultimately the compressive response of the resulting lattice. As demonstrated in[115], the interplay between material properties and cell topology is central in determining stiffness scaling, collapse modes, and densification mechanisms. In this context, understanding how architecture, relative density, strain-rate sensitivity, and anisotropy influence the compressive performance of polymeric lattices is essential for applications in aerospace, biomedical impact protection, and energy-absorbing devices. Several studies have highlighted the importance of correlating process parameters with structural behavior [75]

Building upon these insights, the present section extends the methodological framework previously developed for the tensile characterization of PLA and PAEK by applying it to the compressive testing of three classes of lattice structures:

- i. PEEK lattices fabricated via high-temperature FDM, where crystallinity, layer adhesion, and filament geometry strongly influence collapse mechanisms.
- ii. PA6-CF composite lattices, in which strain-rate sensitivity and printing-direction anisotropy introduce additional complexity in the mechanical response.
- iii. TPU truss-based lattices produced via MJF, representative of elastomeric metamaterials used in impact-mitigation and shock-absorbing applications.

This section presents a systematic comparison of the mechanical behavior across these materials and architectures. Particular attention is given to the stress–strain evolution, plateau response, densification strain, and energy absorption capability — parameters widely recognized as critical descriptors for lattice design.

The objective is twofold:

- i. Validate the generality and robustness of the experimental methodology across different AM processes and material classes
- ii. Provide quantitative guidelines for designing polymeric lattice structures with predictable compressive behavior, bridging the gap between manufacturing constraints and functional performance.

3.4.1 Compressive behaviour of PEEK lattice structures (HT-FDM)

PEEK lattice structures exhibited a characteristic compressive response consistent with the behavior of cellular solids produced by high-temperature FDM. Representative specimens for each topology and relative density are shown in Fig. PEEK-1, highlighting the geometric accuracy obtained after printing and annealing. All specimens maintained dimensional stability during manufacturing, confirming the effectiveness of the thermal conditioning implemented during the HT-FDM process.

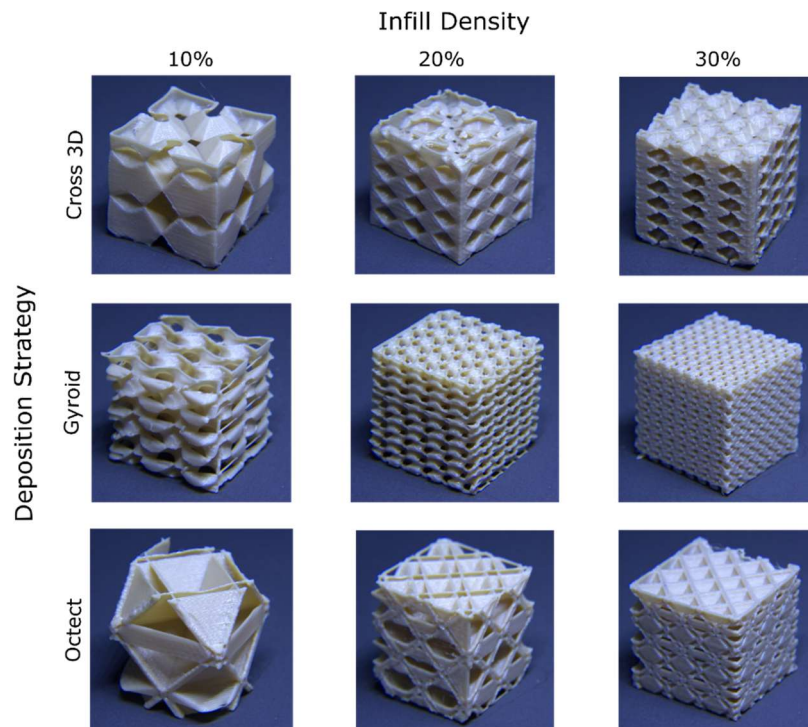


Fig. 3. 26: Photos of PEEK lattices for the three architectures and densities

The compressive stress–strain curves for the three investigated topologies (Cross-3D, Gyroid, Octet) are illustrated in Fig. PEEK-2. All configurations display the three classical regions of cellular solids:

- (1) an initial linear elastic regime,
- (2) a plateau governed by progressive cell wall collapse, and
- (3) densification at large strains.

Despite being manufactured with the same base material and printing parameters, the three lattices show markedly different behaviors.

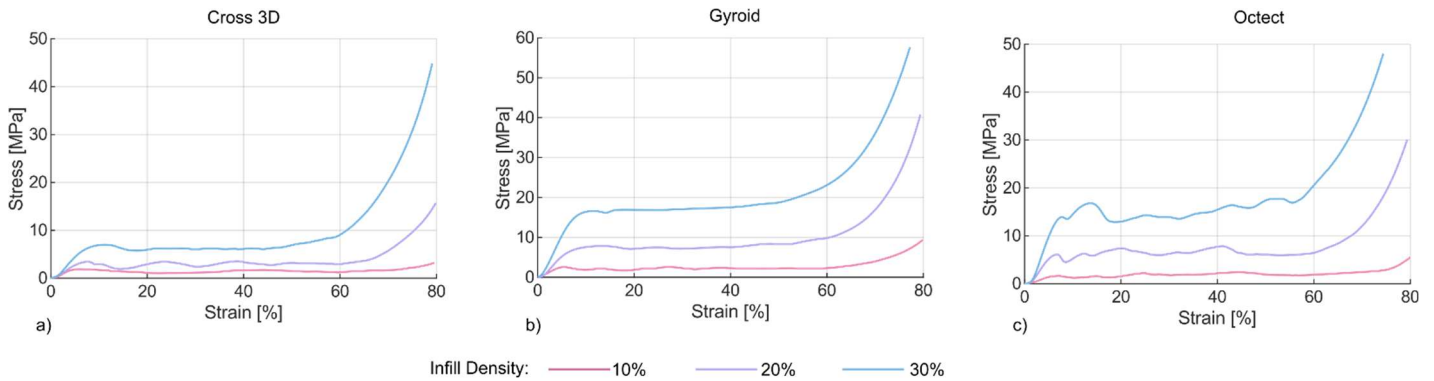


Fig. 3. 27: Stress–strain curves obtained from compression tests of lattice structure grouped by architecture and density

Cross-3D: the structure shows a relatively steep rise in stress following the elastic region, with pronounced stress fluctuations during the collapse phase. This behaviour is indicative of local, layer-by-layer buckling of vertical struts, reflecting a deformation mechanism dominated by bending.

Gyroid: specimens exhibit the smoothest and most stable plateau. Collapse progresses through distributed micro-buckling of the curved surfaces, reducing stress localisation and making this topology the most stable in terms of energy absorption.

Octet: the response is significantly stiffer in the elastic regime and reaches higher plateau stresses, especially at 20% and 30% relative density. Deformation occurs through axial stretching and symmetric node collapse, consistent with a stretch-dominated architecture.

These collapse mechanisms are clearly observed in the online test images shown in Fig. 3. 28: , where Gyroid specimens, that show progressive deformation with high uniformity are illustrated. The different phases of the compression test, namely until the yield point, during the phase of cells collapse, and the reaching of the densification point are depicted alongside the three infill densities employed. Indeed, 10, 20, and 30% infill densities are shown in the three rows of Fig. 3. 28: .

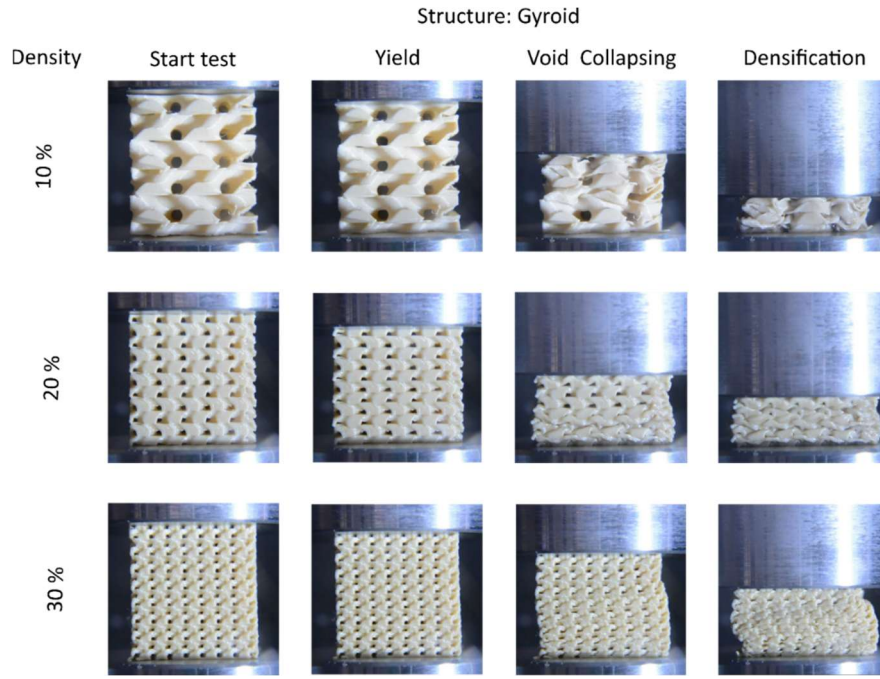


Fig. 3. 28: Progressive images acquired during compression tests of PEEK gyroid lattice specimens, illustrating the progressive deformation and collapse mechanisms at different relative densities. Each row corresponds to a different infill density (10%, 20%, and 30%), while the columns depict successive stages of deformation: elastic loading up to the yield point, progressive cell collapse within the plateau region, and final densification.

Fig. 3. 29 a) and Fig. 3. 29 b) depicts the trends of the Young's modulus and the Yield strength, respectively. As expected, increasing the infill density promoted stiffer structures with higher yield strength. However, linear increase of the Young's modulus was observed for Cross and Octet structure, while the stiffness of Gyroid structures increased more than linearly with the infill density. Similarly, the yield strength of the cross structure increased linearly with the infill percentage while those of gyroid and Octet structure raised more than linearly with the infill density. Substantial differences existed when comparing the absolute values of the compressive characteristics of the structures; actually, the cross structure showed the lowest values of both the Young's modulus and the yield strength.

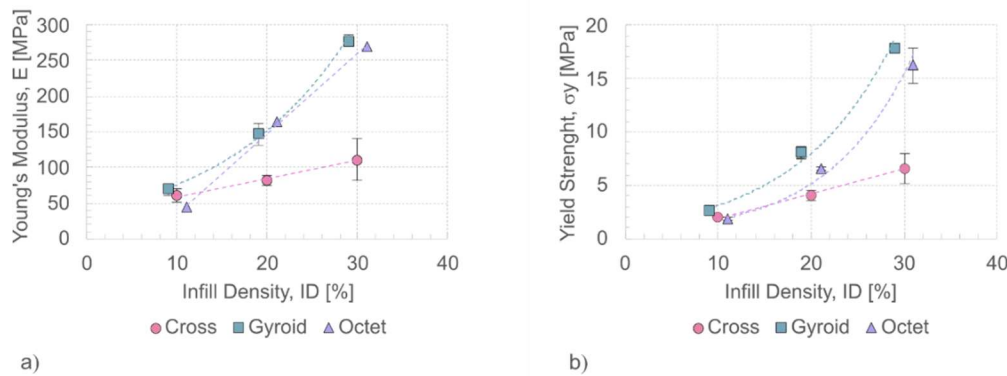


Fig. 3. 29: Effect of infill density on the compressive properties of PEEK lattice structures. (a) Young's modulus and (b) yield strength as a function of infill density for Cross, Gyroid, and Octet architectures.

As an example, for an infill density of 30% the Young's modulus and the yield strength of gyroid and octet structures were almost 2.54 (Young's modulus) times higher and 2.61 (yield strength) of a cross structure. Minor differences existed between Gyroid and Octet structures in terms of stiffness, while gyroid exhibited higher yield strength (18 MPa) as compared to Octet (16 MPa) corresponding to a reduction of 12%.

3.4.1.1 Energy absorption and densification strain

So far, the early compressive characteristics of the analyzed lattice structures were examined. However, energy absorbance also represents a key aspect while using such structures as shock absorbers. Energy absorption characteristics of the lattice structures were determined, as reported in Fig. 3. 30. To this end, the densification strain was determined for all conditions using Eq. 17 $\left(\varepsilon_d = \frac{d\eta(\varepsilon)}{d\varepsilon} \Big|_{\varepsilon=\varepsilon_d} = 0 \right)$ in the Materials and Methods chapter. As expected, the densification strain decreased with increasing the infill density. Indeed, in denser samples (with higher infill density) smaller voids existed; this promoted reaching the densification under lower strain values. Under the adopted building conditions, the densification strain was between 0.48 and 0.65. The densification strain was employed to determine the specific energy to densification (Fig. 3. 30 b)); which represents the energy absorbed by the lattice structure per unit volume. As can be inferred, the specific energy of all the analyzed structures increased with the infill density. However, gyroid structures were characterized by the highest specific energy to densification. Compared to Cross structures, gyroid and Octet structures achieved the highest Soecific energy to densification since their higher yield strength. On the other hand, gyroid structures performed even better to the Octet since the absence of localized collapses (which can be observed in Fig. 3. 27 b)) during the void collapsing phase. To better compare the different structures, the energy to densification strain was normalized per unit weight (Fig. 3. 30 c)); this enabled to better compare the structures made with different infill densities. In the Gyroid and Octet structures the energy to densification per unit weight reached 22.8 J/g and 17.8 J/g respectively.

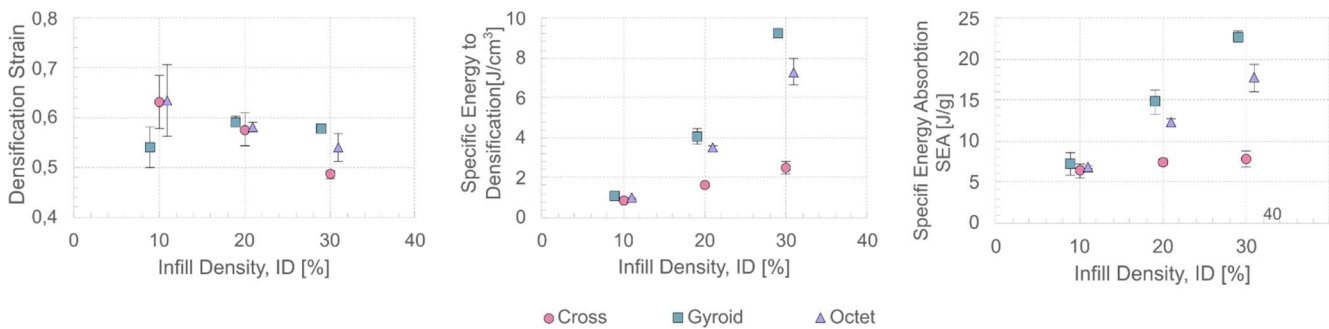


Fig. 3. 30: Energy absorption behavior of PEEK lattice structures as a function of infill density: (a) densification strain, (b) specific energy absorbed up to densification per unit volume, and (c) specific energy absorbed up to densification normalized by specimen mass for Cross, Gyroid, and Octet architectures.

This further confirms the better performances of Gyroid structures for energy absorbance. On the other hand, Cross structures showed a negligible improvement in terms of energy absorption capability and lower values of the maximum normalized energy to densification (7.8 J/g).

3.4.1.2 Scaling behavior: Gibson–Ashby fitting

The experimental data were fitted using the Gibson–Ashby power-law relationships described respectively with Eq. 18 and Eq.19 listed below.

$$\frac{E}{E_s} = C_1 \left(\frac{\rho}{\rho_s} \right)^{n_1} \quad \text{Eq. 18}$$

$$\frac{\sigma}{\sigma_s} = C_2 \left(\frac{\rho}{\rho_s} \right)^{n_2} \quad \text{Eq. 19}$$

Where $E_s = 3600$ MPa, $\sigma_s = 120$ MPa and ρ_s for PEEK are respectively the Young's Modulus, the yield strength, and the density of the bulk material as determined by [116]. The coefficients C_1 and C_2 and exponents n_1 and n_2 of the Gibson and Ashby's model were determined by fitting equations 3 and 4; they are summarized in Tab III. 3. The exponent values were analyzed according to the classification framework proposed by Viswanath et al.[117], who investigated hierarchical cubic lattice structures exhibiting both bending and stretching-dominated behaviors for enhanced buckling resistance.

The Cross structure, characterized by $n_1=0.68$ and $n_2=1.35$, exhibited stretching-dominated behavior, as indicated by efficient load transfer and a pronounced increase in elastic modulus and strength with increasing relative density. However, the low coefficient values ($C_1=0.08$, $C_2=0.37$) result in comparatively lower absolute stiffness and strength. The Gyroid structure, with $n_1=1.37$, $n_2=1.89$, and coefficients $C_1=0.37$, $C_2=1.32$, and the Octet structure, characterized by $n_1=1.80$, $n_2=2.15$, $C_1=0.65$, $C_2=1.58$, were bending-dominated.

Although the mechanical response curves of the Gyroid and Octet structures partially overlap within the examined range of relative density, the distinction between them remains relevant: the gyroid structures show similar Young's modulus ratio to the Octet while slightly higher yield strength ratio. Larger scaling exponents and coefficients, indicative of a more pronounced bending-dominated behavior and superior critical load capacity.

These findings confirm that while the exponents are fundamental for classifying the mechanical behavior of lattice structures, the absolute values of the coefficients critically influence the actual experimental performance. Notably, bending-dominated lattices exhibited comparable or even superior critical load capacities compared to stretching-dominated lattices.

Tab III. 3: Gibson and Ashby's coefficients and exponents of analyzed structures

Structure	n_1	C_1	n_2	C_2
Cross	0.68	0.08	1.35	0.37
Gyroid	1.37	0.37	1.89	1.32
Octet	1.80	0.65	2.15	1.58

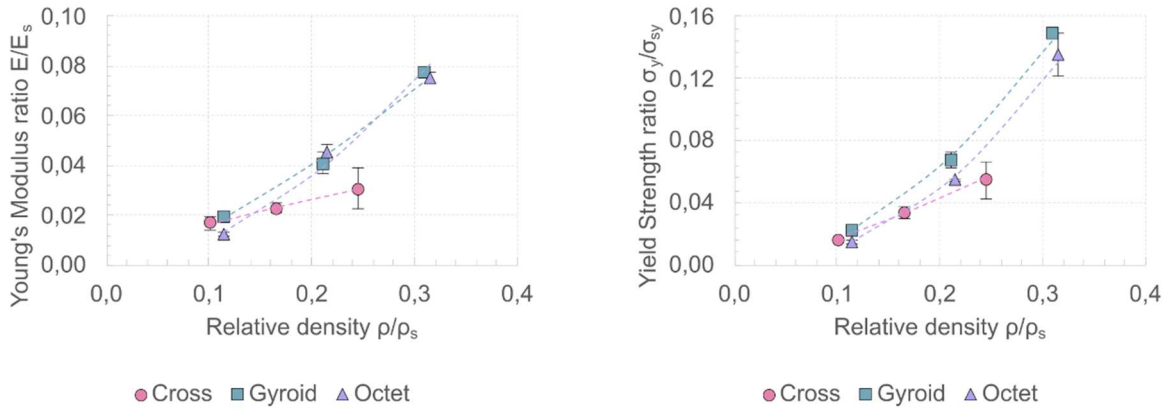


Fig. 3. 31: Normalized mechanical properties of PEEK lattice structures as a function of relative density. (a) Normalized Young's modulus (E/E_s) and (b) normalized yield strength (σ_y/σ_s) for Cross, Gyroid, and Octet architectures. Experimental data are fitted using Gibson–Ashby power-law relationships.

3.4.1.3 PEEK Thermo-mechanical assessment: DSC & DMA analysis

Differential Scanning Calorimetry (DSC) was performed on PEEK samples in the as-printed condition and after the annealing cycle, in order to quantify the effect of thermal treatment on crystallinity and to relate these findings to the mechanical behavior of the printed lattice structures.

The heat-flow curves obtained during the first heating, cooling and second heating cycles are shown in Fig. 3. 32, highlighting clear differences between the two material states. During the first heating of the as-printed specimen, the DSC trace exhibits a pronounced exothermic peak prior to melting, corresponding to cold crystallization.

This indicates that a significant fraction of the material remained amorphous after printing and crystallized only upon heating. In contrast, the annealed sample shows no cold-crystallization peak, confirming that the thermal treatment successfully induced crystallization of amorphous regions, eliminating the need for further ordering during DSC heating.

Melting occurs in both samples at comparable temperatures (~ 340 °C), but with different melting enthalpies. The as-printed specimen displays a higher melting enthalpy ($\Delta H_m = 44.0$ J/g) than the annealed sample ($\Delta H_m = 41.1$ J/g). This apparent inversion, despite the higher overall crystallinity of the annealed sample, is attributed to differences in crystal morphology and thermal history.

The rapid solidification during FDM produces imperfect, less stable crystals that melt with higher apparent enthalpy, while annealing promotes slower reorganisation into more uniform and thermodynamically stable crystals, which do not contribute to cold crystallization and therefore show a lower ΔH_m during the first heating.

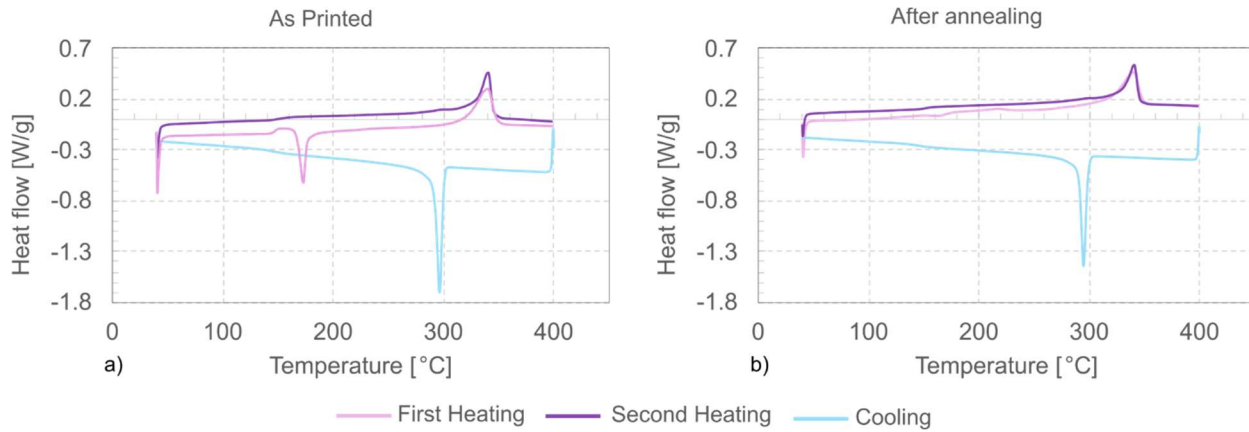


Fig. 3. 32: Differential Scanning Calorimetry (DSC) thermograms of PEEK samples in the as-printed condition and after annealing: first heating, cooling, and second heating cycles. The as-printed sample exhibits a pronounced cold-crystallization peak during the first heating, indicating residual amorphous content after printing, while the annealed sample shows no cold crystallization, confirming increased crystallinity induced by thermal treatment.

The degree of crystallinity χ_c , calculated from the enthalpy values using Eq. (5–6), increases substantially after annealing, rising from 12.5% in the as-printed state to 31.7% as listed in Tab III. 4. This value approaches the upper limit reported in literature for PEEK ($\approx 40\%$ according to Tardif et al.[118]), confirming the effectiveness of the applied thermal cycle while indicating the presence of some residual amorphous phase. A summary of the DSC results is provided in Table PEEK-DSC, which reports the melting enthalpy, cold-crystallization enthalpy and resulting crystallinity for the two material states.

Tab III. 4: Enthalpy values and crystallinity degree of as printed and after annealing PEEK samples

Sample	ΔH_m [J/g]	ΔH_{cc} [J/g]	χ_c [%]
As printed	44.0	27.7	12.5
After annealing	41.1	-	31.7

Dynamic Mechanical Analysis (DMA) was performed to assess the temperature-dependent viscoelastic response of PEEK in the as-printed and annealed states. The evolution of the storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$) as a function of temperature is reported in Fig. PEEK-DMA.

DMA results, shown in Fig. 3. 33, are fully consistent with the DSC observations.

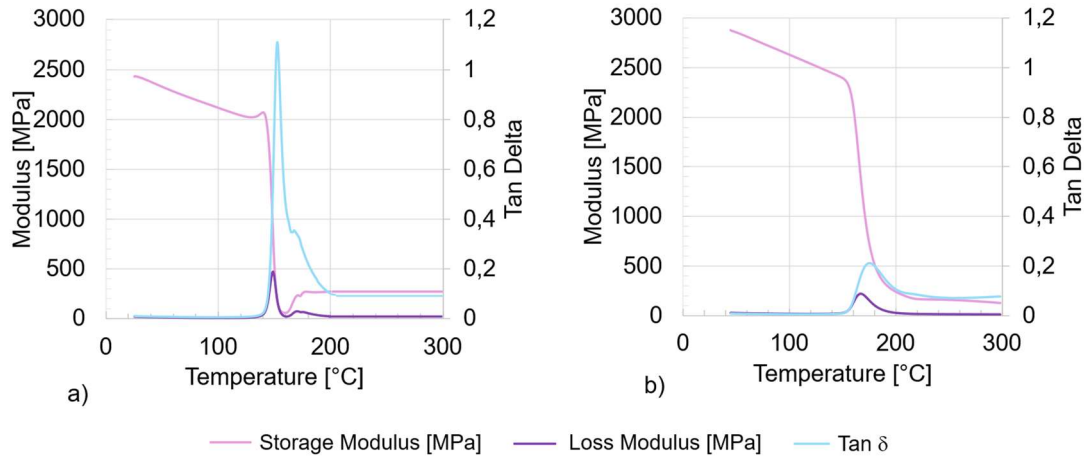


Fig. 3.33: Dynamic Mechanical Analysis (DMA) of PEEK in the as-printed and annealed conditions: (a) heat-flow curves obtained during first heating, cooling, and second heating cycles from DSC; (b) temperature dependence of storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$) from DMA. Annealing increases the storage modulus over the entire sub- T_g region, suppresses cold crystallization, reduces viscoelastic losses, and shifts the $\tan \delta$ peak to higher temperature, reflecting increased crystallinity and stronger constraint of the amorphous phase.

In the as-printed condition, the storage modulus E' is about 2.4 GPa at room temperature and decreases gradually up to ≈ 140 – 145 °C, where a sharp drop marks the onset of the glass transition. The loss modulus E'' and $\tan \delta$ exhibit pronounced peaks in the same temperature range, with a maximum E'' of ≈ 475 MPa and a relatively sharp $\tan \delta$ peak. After annealing, the room-temperature storage modulus increases to about 2.8 GPa and remains higher over the entire sub- T_g range, with an improvement of 16,7%. Just before the transition, E' is still ≈ 2.3 GPa for the annealed material, compared to ≈ 2.0 GPa for the as-printed state. At the same time, the loss modulus peak decreases to ≈ 170 MPa and the $\tan \delta$ peak becomes broader and less intense, while its maximum shifts to higher temperature, around 160 – 162 °C. These combined trends with higher E' , lower E'' peak and reduced $\tan \delta$ amplitude are typical of semicrystalline polymers after crystallization or annealing treatments, as the amorphous phase becomes more constrained by the surrounding crystalline lamellae. Similar behavior has been reported for PEEK grades subjected to controlled annealing or post-curing, where an increase in crystallinity leads to higher storage modulus and a reduction in damping over the entire glassy region, as explained by TA instruments reports [TA033_PEEK_storage_crystallinity]. Hence, the material is more likely to fail under the sudden stress of impact.

The apparent shift of T_g from ≈ 145 °C (as-printed, DMA) to ≈ 160 °C (annealed, DMA) can be rationalized by considering both the change in morphology and the different nature of DSC and DMA measurements. DSC detects T_g through a step in heat capacity, and in this case yields a midpoint T_g of about 150 °C for both conditions. DMA, instead, probes the viscoelastic response under oscillatory loading: the loss-modulus peak is often close to the DSC T_g , while the $\tan \delta$ peak tends to appear at slightly higher temperature and is very sensitive to chain mobility, crystalline constraint and testing frequency, as observed by Z. Wei et al [119].

In summary, DSC and DMA provide a coherent picture of the printing process leaves PEEK in a partially crystalline state, with significant amorphous content that cold-crystallizes on first heating; furthermore, the annealing drives the crystalline phase towards 30%, eliminates cold crystallization, stiffens the material

across the glassy region, and reduces mechanical damping. Moreover, the glass transition measured by DMA shifts to higher temperature after annealing because the amorphous phase is more constrained by the crystalline network, while DSC T_g remains around 150 °C, as expected from its different sensitivity to morphology.

Having established the thermo-mechanical behavior of PEEK and the effects of annealing on its viscoelastic response, it is now possible to interpret the mechanical performance of the lattice structures under elevated-temperature compression. The following section presents the stress–strain response of Gyroid lattices at 25–200 °C and discusses these trends in light of the DSC and DMA observations.

3.4.1.4 Thermal-dependent compressive behavior of PEEK lattice structures.

The compressive response of PEEK lattice structures was further investigated under elevated temperature conditions, with tests carried out at 25, 140, 160 and 200 °C for the three examined relative densities (10, 20 and 30%). Representative stress–strain curves for the Gyroid architecture at the three density levels are reported in Fig. 3. 34, while the evolution of the Young’s modulus and yield strength as a function of temperature is summarized in Fig. 3. 35.

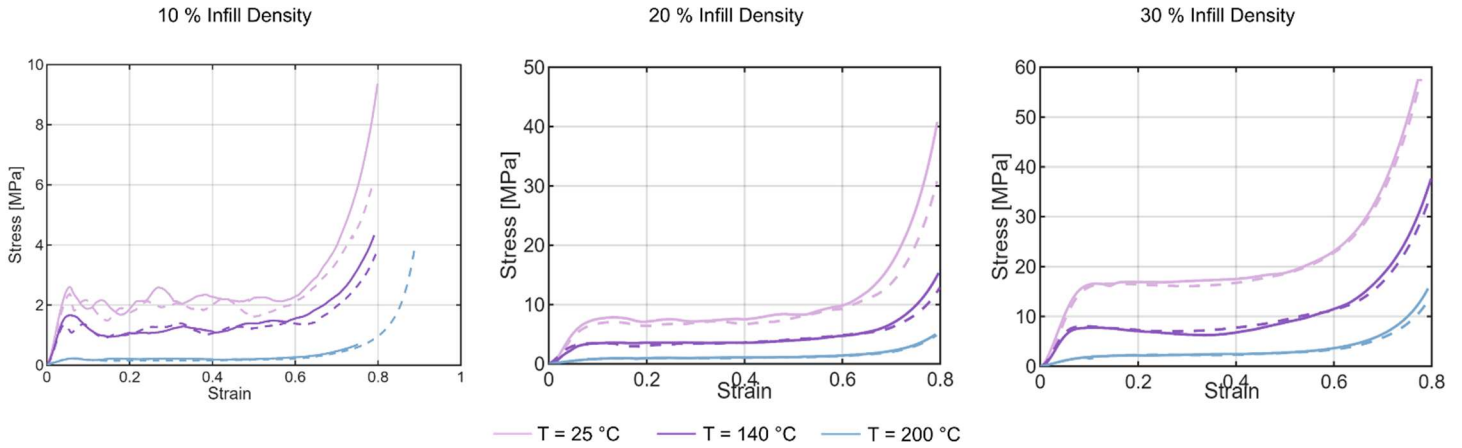


Fig. 3. 34: Compressive stress–strain response of Gyroid PEEK lattice structures tested at elevated temperatures for different relative densities. Representative curves are reported for (a) 10%, (b) 20% and (c) 30% infill densities, tested at 25, 140, 160 and 200 °C. Increasing the test temperature results in a progressive reduction of the elastic stiffness and yield stress, accompanied by a smoother and less stable plateau region. Higher relative densities preserve larger load-bearing capacity over the entire strain range, despite the pronounced thermal softening observed at elevated temperatures.

All structures exhibited the characteristic three-stage compressive behaviour observed at room temperature, namely an initial elastic regime, a plateau associated with progressive cell collapse, and a final densification stage. Increasing the test temperature led to a progressive reduction of both elastic stiffness and yield strength, accompanied by a smoother plateau region. This degradation was strongly dependent on the relative density, with higher-density lattices retaining greater load-bearing capacity over the entire temperature range.

At 25 °C, all densities displayed highly reproducible stress–strain responses, in agreement with the results discussed in Sect. 3.1. When the test temperature was increased to 140 °C, a moderate reduction in the initial slope and yield strength was observed, while the overall deformation mechanisms remained essentially unchanged. At 200 °C, close to the onset of pronounced softening of the amorphous phase, the plateau region became markedly smoother and collapse occurred at substantially lower stress levels, indicating a significant loss of stiffness and structural resistance.

The temperature dependence of the Young’s modulus E and yield strength σ_y , extracted from the compression curves, is reported in Fig. 3. 35 a) and Fig. 3. 35 b) respectively.

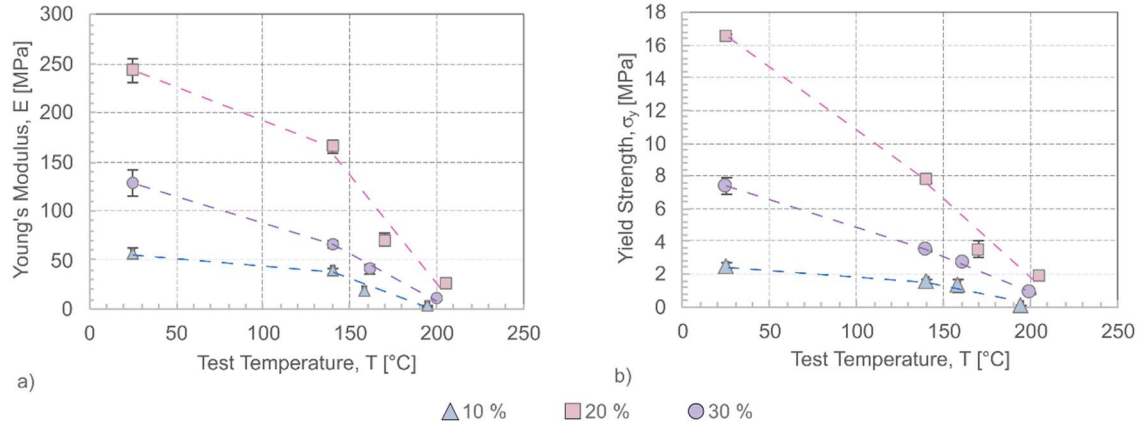


Fig. 3.35: Effect of test temperature on the compressive properties of Gyroid PEEK lattices at different relative densities. (a) Young's modulus as a function of temperature; (b) yield strength as a function of temperature. Dashed lines represent bilinear fits used to describe the temperature-dependent degradation below and above the glass transition region.

Both properties decrease non-linearly with temperature, with a pronounced reduction occurring between 140 and 200 °C. This behaviour reflects the increasing mobility of the amorphous phase and the progressive viscoelastic softening of PEEK as the temperature approaches and exceeds the glass transition region.

To provide a simplified constitutive description of the observed trends, the experimental data were fitted using bilinear temperature-dependent relationships for both $E(T)$ and $\sigma_y(T)$, with separate linear regimes identified below and above the glass transition temperature. The resulting expressions, determined for each relative density, are reported in Tab III. 5 and enable a compact representation of the stiffness and strength degradation of the lattice structures over the investigated temperature range.

Tab III. 5: Bilinear temperature-dependent laws for the Young's modulus and yield strength of Gyroid PEEK lattice structures at different relative densities. The table reports the linear relationships describing the evolution of the Young's modulus E and the yield strength σ_y as a function of test temperature in the sub-Tg and above-Tg regimes, for relative densities of 10%, 20% and 30%. The transition between the two linear regimes occurs in the temperature range associated with the glass transition of PEEK, as identified by DSC and DMA analyses

Relative Density %				
	10 %	20 %	30 %	
Young's Modulus				
$E(T)$	$\begin{cases} -0.16T + 61.10 \\ -0.60T + 117.86 \end{cases}$	$\begin{cases} -0.55T + 142.7 \\ -0.89T + 188.32 \end{cases}$	$\begin{cases} -0.97T + 260.0 \\ -2.2T + 452.1 \end{cases}$	$\begin{cases} T < 140\text{ }^{\circ}\text{C} \\ T > 140\text{ }^{\circ}\text{C} \end{cases}$
Yield strength				
$\sigma_y(T)$	$\begin{cases} -0.01T + 2.70 \\ -0.3T + 5.51 \end{cases}$	$\begin{cases} -0.03T + 8.23 \\ -0.05T + 9.81 \end{cases}$	$\begin{cases} -0.08T + 18.40 \\ -0.09T + 19.90 \end{cases}$	$\begin{cases} T < 140\text{ }^{\circ}\text{C} \\ T > 140\text{ }^{\circ}\text{C} \end{cases}$

The identified bilinear laws capture the progressive degradation of compressive stiffness and strength with increasing temperature and highlight a clear change in slope between the low- and high-temperature regimes. Across all densities, the slopes associated with the high-temperature regime are significantly larger in

magnitude than those observed at lower temperatures, indicating an increased sensitivity of the compressive properties to temperature above the transition region.

Qualitative comparison between compression modulus and DMA response

To further contextualize the temperature-dependent degradation of the compressive stiffness, a normalized comparison between the Young's modulus obtained from compression tests and the storage modulus measured by DMA was performed. Fig. 3. 36 reports the ratio $E(T)/E(25\text{ °C})$ for Gyroid lattices at different relative densities, together with the corresponding normalized DMA storage modulus $E'(T)/E'(25\text{ °C})$ measured on annealed bulk specimens.

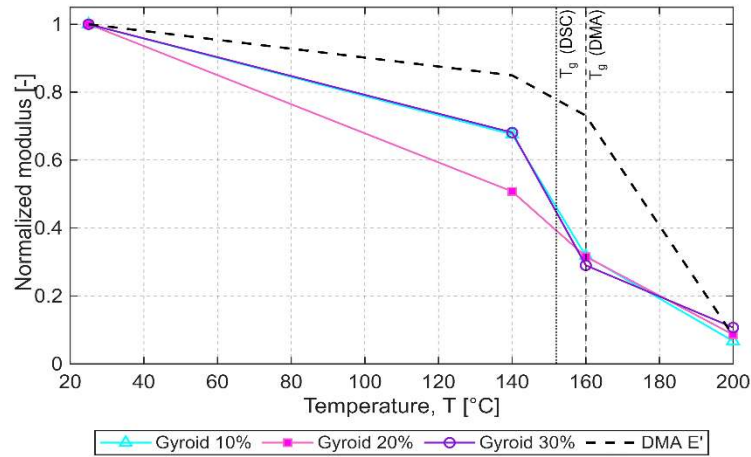


Fig. 3. 36: Normalized Young's modulus $E(T)/E(25\text{ °C})$ obtained from compression tests on Gyroid PEEK lattice structures with relative densities of 10%, 20% and 30% is compared with the normalized DMA storage modulus $E'(T)/E'(25\text{ °C})$ measured on annealed bulk PEEK specimens. Vertical dashed lines indicate the glass transition temperatures identified by DSC and DMA analyses

For all relative densities, the normalized compressive modulus exhibits a progressive reduction with increasing temperature, with a marked drop occurring in the temperature range between 140 and 200 °C. A similar qualitative trend is observed for the DMA storage modulus, which remains relatively stable up to the glass transition region and then decreases sharply at higher temperatures.

The comparison highlights that, despite the different nature of the tests and length scales involved, both approaches capture a comparable transition in thermo-mechanical behavior. In particular, the steep reduction in normalized stiffness observed above the glass transition temperature is consistently reflected in both compression-derived moduli and DMA measurements.

3.4.2 High-temperature compressive response of PEEK lattice structures

The experimental results obtained from the spring-loaded compression tests are presented in this section. The response of the lattice structures is characterized in terms of permanent compressive deformation and residual nominal compressive stress measured after thermal exposure and cooling. The influence of temperature and lattice relative density is examined, and the relationship between irreversible deformation and residual load-bearing capability is subsequently highlighted.

3.4.2.1 Permanent compressive deformation

Fig. 3. 37 reports the permanent compressive strain measured after cooling for lattice specimens with relative densities of 20% and 30% following 12 h of exposure at elevated temperature under nominal compressive loading.

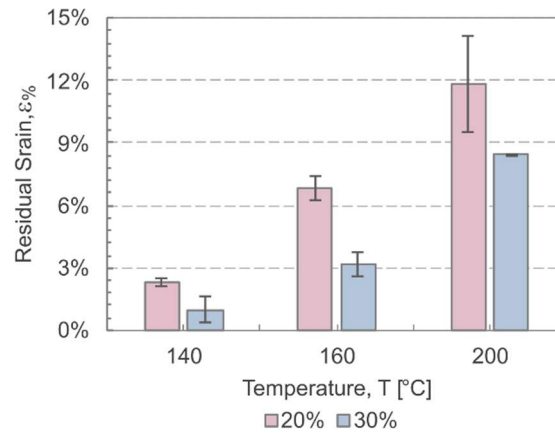


Fig. 3. 37: Permanent compressive strain measured after cooling as a function of temperature for lattice structures with relative densities of 20% and 30%.

For both lattice densities, the permanent strain increases with increasing temperature. At 140 °C, the measured deformation remains limited for both configurations. A clear increase in permanent strain is observed at 160 °C, followed by a further and more pronounced increase at 200 °C. At all the temperatures investigated, lattice structures with a relative density of 30% exhibit lower permanent strain than those with a relative density of 20%. The difference between the two densities becomes more pronounced at higher temperatures.

3.4.2.2 Residual nominal compressive pressure

The residual nominal compressive pressure retained after thermal exposure and cooling is reported in Fig. 3. 38. The values represent the compressive pressure sustained by the lattice specimens after unloading and cooling to room temperature, referenced to the nominal pressure initially applied through spring pre-loading.

At 140 °C, both lattice densities retain a relatively high residual compressive pressure, close to the initially applied nominal value. A reduction in residual pressure is observed at 160 °C, with a more pronounced

decrease for the 20% infill configuration. At 200 °C, the residual compressive pressure further decreases for both densities, reaching the lowest values among the investigated temperature conditions.

For all temperatures, the 30% infill lattice consistently maintains a higher residual compressive pressure compared to the 20% configuration, indicating a greater ability to sustain compressive loading after thermal exposure. The difference between the two densities becomes increasingly evident as temperature increases.

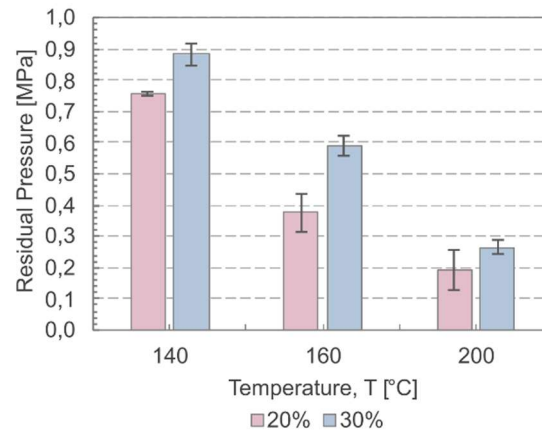


Fig. 3. 38: Residual nominal compressive pressure measured after cooling as a function of temperature for lattice structures with relative densities of 20% and 30%. Error bars represent the standard deviation of the measurements. The initial nominal compressive pressure applied during pre-loading was approximately 1 MPa.

The relationship between permanent compressive strain and residual nominal compressive pressure is presented in Fig. 3.39. Each data point corresponds to a specific temperature condition (140, 160, and 200 °C) and lattice relative density (20% or 30%).

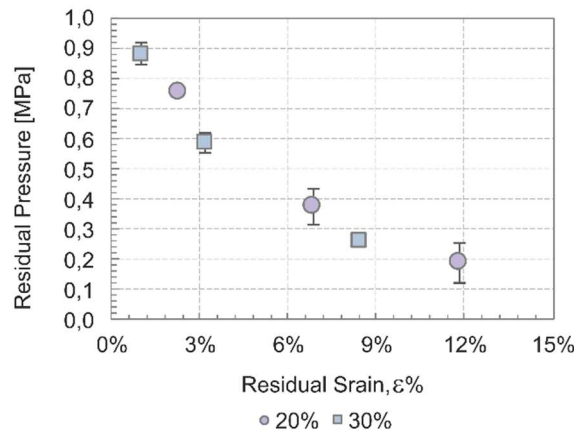


Fig. 3. 39: Residual nominal compressive pressure plotted against permanent compressive strain for lattice structures with relative densities of 20% and 30%. Marker color indicates the test temperature, while marker shape distinguishes lattice density.

The results show that the residual compressive pressure decreases as the permanent strain increases; however, the relationship is non-linear. Conditions characterized by moderate permanent deformation may exhibit similar or even lower residual pressure compared to conditions associated with larger irreversible strains.

For a given level of permanent strain, the 30% infill lattice consistently exhibits higher residual compressive pressure than the 20% configuration. This trend indicates that lattice relative density influences not only the magnitude of permanent deformation, but also the ability of the structure to retain compressive pressure after thermal exposure.

3.4.3 Compressive of PA6-CF Lattice Structures

Before discussing the compressive behavior of the PA6-CF lattice structures, a complete characterization of the feedstock material was carried out to establish the thermal transitions, fiber content, and morphology of the short-fiber reinforcement. These analyses provide essential context for interpreting the mechanical response of the printed lattices, particularly about stiffness, anisotropy, and deformation mechanisms.

3.4.3.1 Thermomechanical and morphological characterization of PA6-CF

Fig. 3. 40 shows the fracture surface of the as-received PA6-CF filament at two magnifications. At low magnification, the section exhibits a heterogeneous microstructure composed of a PA6 matrix embedding a dense population of short carbon fibres. At higher magnification, Fig. 3. 40 b) highlights the geometry of the fibres, which appear uniformly dispersed with diameters of $\sim 6\text{--}8\text{ }\mu\text{m}$ and lengths in the order of tens of micrometres. This morphology is consistent with commercial short-fibre PA6 compounds, where fibres are intentionally discontinuous to preserve printability in extrusion-based AM. The random fibre orientation in the filament cross-section results in an intrinsically quasi-isotropic behaviour for the feedstock itself; however, during printing, shear-induced alignment and extensional flow within the nozzle are expected to orient fibres preferentially along the deposition direction—a well-known mechanism responsible for anisotropy in printed fibre-reinforced polymers.

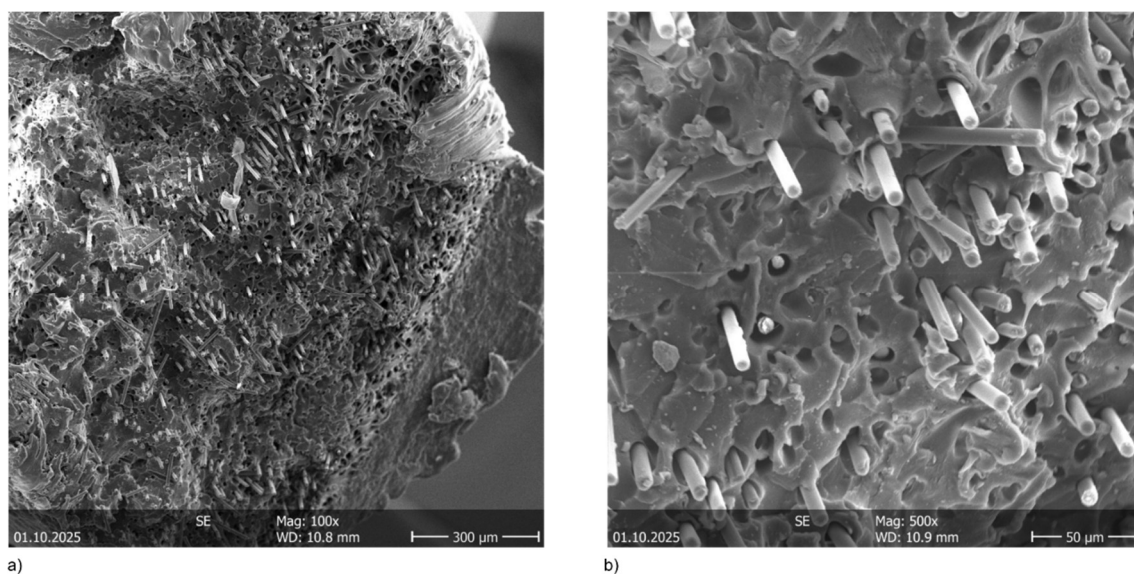


Fig. 3. 40: SEM micrographs of the PA6-CF filament fracture surface: a) 100 $\times$ overview; b) 500 $\times$ detail showing fibre dispersion.

DSC and TGA Analysis: evaluation of degree of crystallinity

Furthermore, the feedstock filament was characterized through different techniques, including Differential Scanning Calorimetry (DSC). Thermogravimetric Analysis (TGA), and Dynamic Mechanical Analysis (DMA). To calculate the degree of crystallinity the Eq. 14 is employed:

$$\chi_c = \frac{\Delta H_m - \Delta H_{CC}}{\Delta H_m^{100\%}} \quad \text{Eq. 14}$$

The value of $\Delta H_m^{100\%}$, related to the neat PA6 found in literature is 230 J/g according to [120,121]. This value must be corrected considering the amount of fiber volume fraction present in the filament described by Mossinger et al [122]. Consequently, the value of $\Delta H_m^{100\%}$ was corrected with the value of ΔH_{comp} as shown in the following Eq. 20:

$$\Delta H_{comp} = \Delta H_m^{100\%}(1 - w_f) \quad \text{Eq. 20}$$

Indeed, fibers do not contribute to the melting heat in a composite structure. The amount of fiber fraction was estimated through thermogravimetric analysis (TGA). A sample of material with a weigh of 40.2 mg was tested in static air environment from room temperature to 1000 °C at a ramp rate of 10°C/min, to identify degradation phenomena occurring during heating. Then the test was replicated until 480°C which was identified during the previous test as the temperature of total degradation of the PA matrix. This enabled to determine the weight of the remaining material (carbon fibers). The TGA diagram obtained is illustrated in Fig. 3. 41.

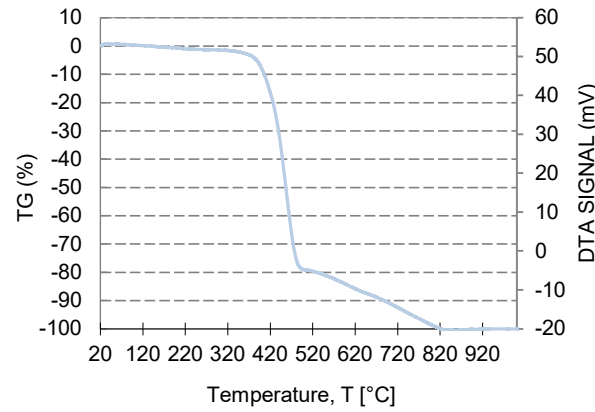


Fig. 3. 41: TGA curve of PA6-CF filament showing polymer decomposition onset at 390 °C, complete matrix degradation by 500 °C, and ~20 wt% carbon-fibre residue.

A first weight loss of 5%—commonly used as an indicator of thermal degradation onset—was detected at ≈ 390 °C, marking the beginning of PA6 matrix decomposition. The polymer underwent complete volatilization by ≈ 500 °C, where the DTA signal stabilized, and no further mass loss associated with organic content was recorded. The remaining mass fraction, corresponding to non-combustible carbon fibers, was approximately 20 wt%. This value is fully consistent with typical short-fiber PA6 composites and matches the trends reported by A. Das et al. [123], who observed similar decomposition behavior and fiber residue levels for PA6-based carbon-fiber filaments.

The degree of crystallinity, χ_c , was determined from the melting enthalpy ΔH_m and the cold-crystallization enthalpy ΔH_{cc} obtained during the first heating scan. Thus, the DSC thermogram is depicted below Fig. 3. 42.

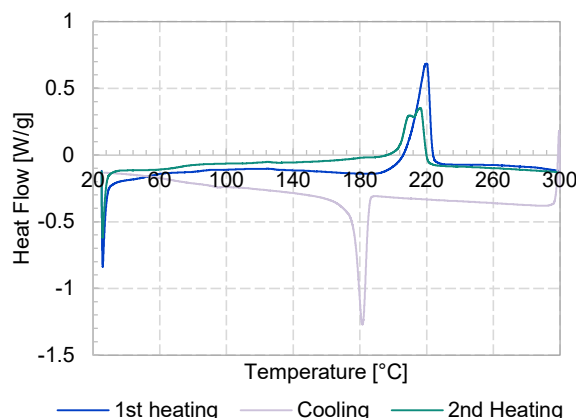


Fig. 3. 42: DSC thermogram of Bambu PA6CF filament showing first heating, cooling, and second heating scans. The first heating exhibits a single dominant endothermic melting peak with no cold-crystallization exotherm, indicating that the as-received filament is already partially crystalline. The second heating reveals a double melting peak, associated with the coexistence of PA6 crystalline populations with different lamellar thicknesses (γ - and α -phases).

The first heating shows a single dominant endothermic melting peak without any detectable cold-crystallization exotherm, indicating that the as-received filament is already partially crystallized and does not undergo further crystallization upon heating. This behavior is typical of PA6 processed by melt extrusion, where rapid cooling during filament manufacturing results in a mixed population of crystalline lamellae. During the second heating, a distinct double endothermic peak is observed. This feature is commonly associated with the melting of two crystalline populations with different lamellar thicknesses. The lower-temperature shoulder is typically attributed to the melting–recrystallisation–melting sequence of the γ -phase (thin lamellae) of PA6, whereas the main peak located at approximately 216 °C corresponds to the melting of the more stable α -phase (thicker lamellae). This interpretation is consistent with the behavior reported by Millot et al. [124], who observed a similar dual endothermic signature in PA6 systems, indicating the coexistence of lamellae with different stability and crystallization histories.

The value of the melting normalized enthalpy ΔH_m , calculated during the first heating is 53 J/g. Therefore, the degree of crystallinity of the PA6 matrix was found from the Eq. 14 together with Eq. 18. The DSC measurement does not show significant cold crystallization peak during the first heating. Hence, the value obtained of the degree of crystallinity is $\chi_{c\%} = 28.80 \%$.

DMA Analysis

The DMA curves in Fig. 3. 43 show the typical viscoelastic behavior of a short-carbon-fiber-reinforced PA6. At low temperature, the material exhibits a high storage modulus ($E' \approx 13\text{--}15$ GPa), which reflects the stiff, glassy response of the polyamide matrix combined with the reinforcing action of the carbon fibers. Similar high-modulus values are reported for PA6-CF composites, where the fibers carry a significant share of the load and suppress molecular mobility in the polymer phase. In fact, as reported by Karsli et al. [125] the Storage Modulus exhibits an improvement with the amount content of carbon fiber reinforced polyamide 6, while the T_g is not affected by the fraction of carbon fiber.

As temperature increases, the storage modulus E' decreases steadily and shows a marked drop between 70 °C and 110 °C. This drop corresponds to the α -relaxation of polyamide, associated with segmental motion of the amide chains. The $\tan \delta$ peak appears in the same temperature interval, and its maximum at 76 °C is taken as the glass-transition temperature (T_g) according to ASTM D4065. The loss modulus (E'') also reaches its maximum in this region, further confirming the position of the primary relaxation. Accordingly to the results obtained from [125] the T_g obtained from the DMA analysis is few degree higher compared to the one found through the DSC. In fact, this is probably due to the frequency effect in the DMA that freezes the movements. Molecular relaxation of the polymer chain can only occur at higher temperatures compared to DSC.

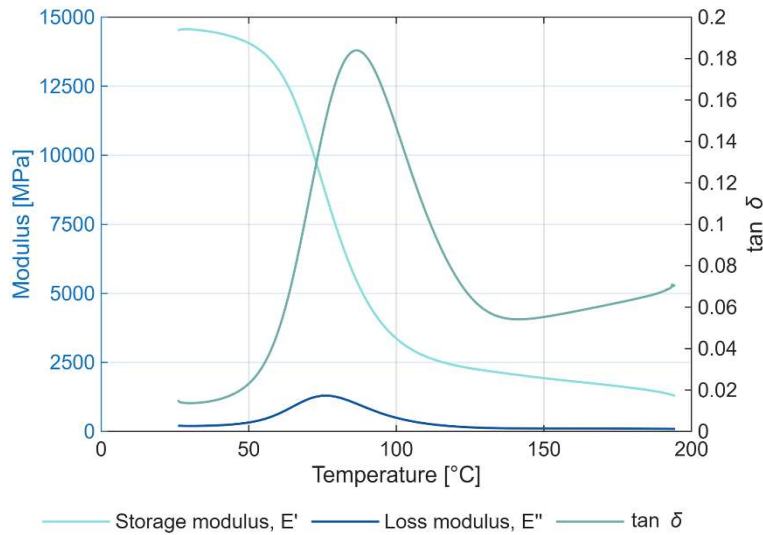


Fig. 3. 43: Dynamic Mechanical Analysis (DMA) curves of PA6CF showing the temperature dependence of the storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$).

3.4.3.2 Mechanical characterization of PA6-CF lattice structures fabricated by FDM

Three lattice topologies, namely Cross-hatch, Gyroid, and 3D Honeycomb were manufactured at 10%, 20% and 30% relative density, and tested under compression. For each condition, cubic specimens were tested at three speeds (10, 100, 600 mm/min). As shown in the following sections, both the base material and the lattice structures exhibits minimal strain-rate sensitivity. Instead, the mechanical compressive response is strongly governed by topology and deposition orientation, with anisotropy emerging as one of the most relevant design constraints.

Compression tests on full density samples

Stress-strain curves calculated from data from compression tests performed on cylindrical samples made with 100% relative densities ($\varnothing 12 \times h 24$ mm) at different strain rates are reported in Fig. 3. 44. The comparison among different curves and from Young's modulus, Yield strength and peak strength indicate negligible strain rates effect on the full-density samples.

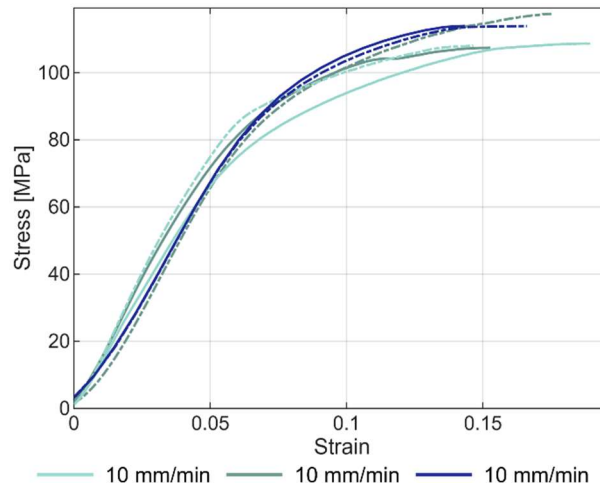


Fig. 3. 44: Stress-strain curves of full density samples during compression tests under different testing speeds.

Tab III. 6: Analysis of compression mechanical properties of cylindrical specimens

Test speed [mm/min]	10	100	600
E [GPa]	1.46 ± 0.14	1.38 ± 0.16	1.32 ± 0.01
σ_y [MPa]	71.5 ± 5.2	68.0 ± 5.3	$85,6 \pm 1.3$
σ_f [MPa]	108.4 ± 0.5	112.5 ± 7.1	113.9 ± 0.1

These results demonstrate that PA6-CF shows negligible strain-rate sensitivity within the quasi-static regime explored. Elastic modulus, yield stress, and peak strength vary only slightly (within experimental scatter), confirming that the polymer–fiber microstructure does not exhibit significant rate-dependent stiffening or softening at these speeds. Consistent with the behavior of the bulk material, the lattices also exhibited minimal variations with strain rate. Differences observed among curves at 100 and 600 mm/min remain within the intrinsic dispersion associated with FDM processing (geometric variability, fiber reorientation, local defects). For this reason and to maintain clarity and avoid redundant plots only the curves at 10 mm/min are shown, and all subsequent analyses (Gibson–Ashby scaling, anisotropy) are based on the reference speed.

Strain rate sensitivity of the PA6CF lattice structures

Representative stress–strain curves for the three investigated lattice topologies: Cross-hatch, Gyroid, and 3D Honeycomb. The stress – strain charts obtained from the test conducted at 10, 100, and 600 mm/min are shown in Fig. 3. 45. Despite sharing the same manufacturing parameters and base material, the three architectures show markedly different deformation modes:

- Cross-hatch demonstrates early local buckling of the vertical members and pronounced stress oscillations during collapse, indicating a primarily bending-dominated mechanism.
- Gyroid shows the smoothest plateau and the most stable collapse sequence, attributable to its continuous surface-based geometry, which distributes stresses without concentrated hinges.
- 3D Honeycomb exhibits intermediate behaviour, with higher initial stiffness than Cross-hatch but more localization than Gyroid, consistent with a mixed bending/stretching character.

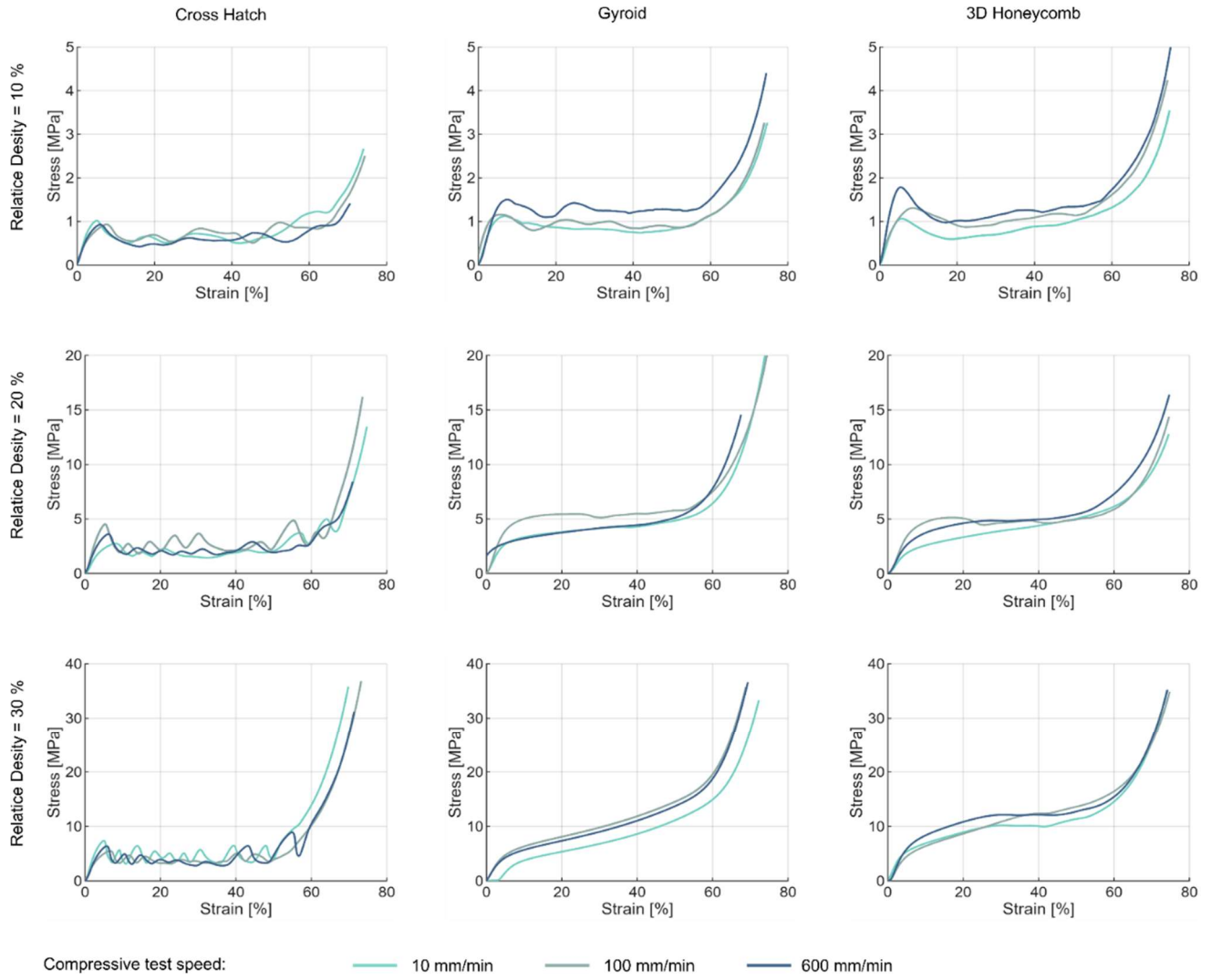


Fig. 3. 45: Compressive stress–strain curves for the three topologies at 10 mm/min, 100, and 600 mm/min. Matrix shows density levels (rows) vs infill patterns (columns)

PA6-CF shows minimal dependence on strain rate, with all topologies displaying nearly overlapping curves at the three tested speeds. For completeness, Fig. 3. 46 reports the Young's modulus and yield strength extracted at three speeds, confirming that stiffness, collapse stress and densification strain remain essentially unchanged.

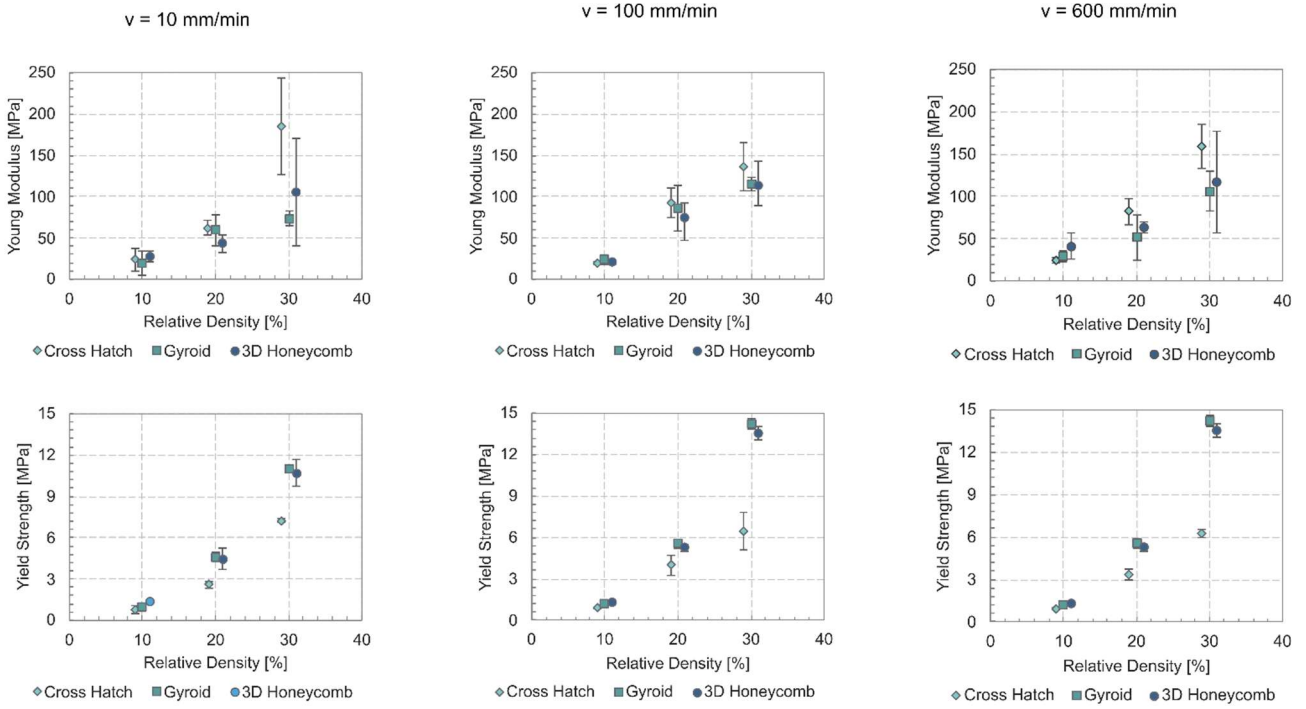


Fig. 3.46: Young's modulus (top) and yield strength (bottom) of PA6CF lattice structures as a function of relative density at testing speeds of 10, 100, and 600 mm/min. The negligible variation across strain rates indicates a weak rate sensitivity, with mechanical properties mainly governed by lattice topology and relative density.

Mechanical behavior and deformation mechanics of PA6-CF lattice: Gibson and Ashby model

The mechanical response of the PA6-CF lattice architectures were interpreted using the Gibson & Ashby scaling laws. These relationships relate the relative stiffness and strength of cellular materials to their relative density through power-law relationships. The two power law for relative stiffness and strength are listed below in the Eq. 18: and Eq. 19:

$$\frac{E}{E_s} = C_1 \left(\frac{\rho}{\rho_s} \right)^{n_1} \quad \text{Eq. 18}$$

$$\frac{\sigma}{\sigma_s} = C_2 \left(\frac{\rho}{\rho_s} \right)^{n_2} \quad \text{Eq. 19}$$

The fitted coefficients for the three topologies are enumerated in Tab. III.7, that provides important insight into the dominant deformation mechanisms and the role of local instabilities in surface-based lattices manufactured by FDM.

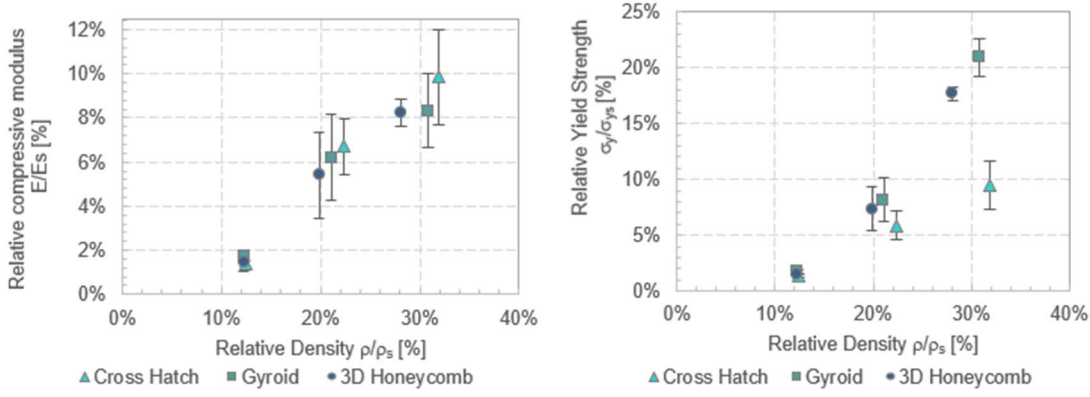


Fig. 3. 47: The value of E/E_s (relative modulus) and σ_y/σ_{ys} (relative yield strength) alongside the three topologies and the three levels of densities

In the image in Fig. 3. 47 is depicted the value of E/E_s (relative modulus) and σ_y/σ_{ys} (relative yield strength) alongside the three topologies and the three levels of densities. The value of E_s and σ_{ys} are the reference values obtained from the compression test performed on full-density cylinders, and the reference values are those illustrated in Tab III. 6 for the 10 mm/min speed.

Tab III. 7: Values of the Gibson and Ashby model fitting-coefficients for the three structures

	n1	C1	n2	C2
Cross hatch	2,14	1,32	2,14	1,23
Gyroid	1,75	0,76	2,72	5,32
3D Honeycomb	2,11	1,36	3,03	8,80

For the cross-hatch lattice, both the relative elastic modulus E/E_s and the relative yield strength σ_y/σ_{ys} scale with an exponent $n \approx 2$, together with comparable coefficients $C1$ and $C2$. This behavior is characteristic of bending dominated architecture, in which the cell walls deform primarily through flexural bending rather than axial stretching. This scaling is consistent with classical Gibson & Ashby predictions for foams and honeycomb structures, where bending is the predominant mechanism at low relative densities [74]. Moreover, for the crosshatch structure the influence of relative density is therefore regular and predictable: as density decreases, both stiffness and strength decline proportionally, and as density increases, both properties grow quadratically without qualitative changes in the collapse mode. The onset of buckling is gradual and primarily controlled by the slenderness of the printed walls, with failure governed by flexural instability rather than material yielding.

The Gyroid topology exhibited super-quadratic yield strength scaling with $n_2 \approx 2.72$ and coefficient $C_2 = 5.32$, indicating bending-dominated behavior with significant influence from elastic instability. This power-law scaling reflects a transition from buckling-dominated failure at low densities (large, thin-walled unit cells) to plastic yielding at higher densities. As unit cell size decreases while maintaining constant wall thickness, the critical buckling stress of TPMS surfaces increases more rapidly than quadratically. The super-quadratic exponent ($n_2 > 2$) quantifies this efficiency gain: at 30% density, the Gyroid achieves yield strengths

significantly exceeding those predicted by classical bending theory due to suppression of elastic buckling instabilities.

The 3D Honeycomb structure showed the strongest density dependence with $n_2 \approx 3.03$ and coefficient $C_2 = 8.80$, also reflecting bending-dominated behavior with pronounced buckling effects. The cubic scaling indicates severe strength penalties at low densities where thin cell walls buckle elastically well below material yield strength. However, the exceptionally high coefficient $C_2 = 8.80$ demonstrates that once relative density reaches 20-30% and cell walls gain sufficient thickness to resist buckling, the Honeycomb becomes highly efficient structurally, achieving the highest normalized yield strengths among the three topologies.

3.4.3.3 Anisotropy in PA6-CF Lattice Structures

The anisotropic nature of the FDM process becomes particularly evident when evaluating the compressive response of PA6-CF lattice structures. To quantify this effect, each lattice topology—Cross-hatch, Gyroid, and 3D Honeycomb—was tested at three relative densities (10%, 20%, 30%) under two loading orientations:

- (i) UP, aligned with the build direction
- (ii) SIDE, perpendicular to it.

Representative stress–strain curves for the three topologies are reported in Fig. 3. 48, where UP and SIDE responses at equal density levels are directly compared.

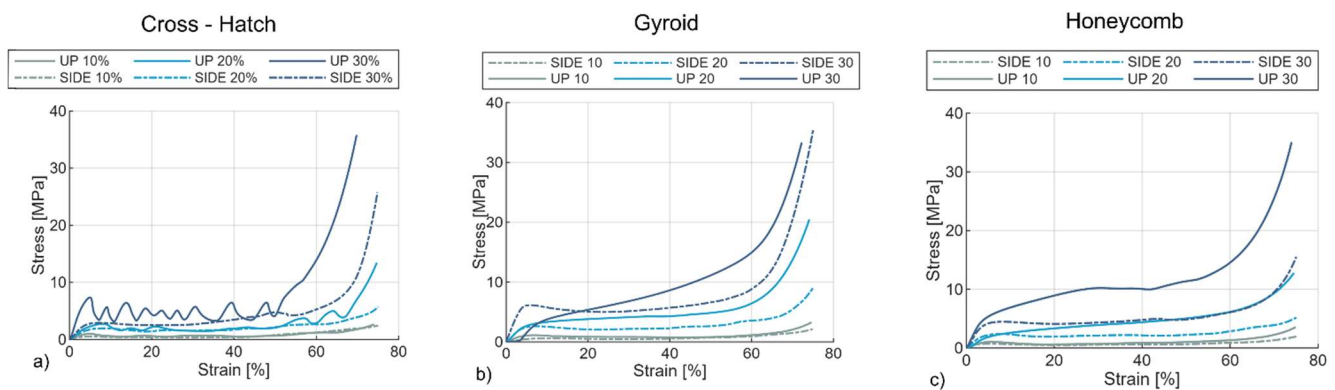


Fig. 3. 48: Representative compressive stress–strain curves of PA6-CF lattice structures (a) Cross-hatch, (b) Gyroid, and (c) 3D Honeycomb at relative densities of 10%, 20%, and 30%, tested along the build direction (UP) and perpendicular to it (SIDE).

Across all topologies, tests conducted in the UP direction generally exhibited higher peak stresses and earlier transition to the densification regime. This behavior is consistent with the intrinsic layering of FDM, where filaments stacked along the build direction provide higher resistance when loaded axially, while SIDE loading induces shear across interlayer interfaces—typically the weakest direction in short-fiber FDM composites. Similar observations have been reported for short-fiber reinforced PA6 systems by [126].

The mechanical properties extracted from the curves—Young's modulus, yield strength, and specific energy absorption (SEA) are summarized in Fig. 3. 49, showing clear distinctions between UP and SIDE loading.

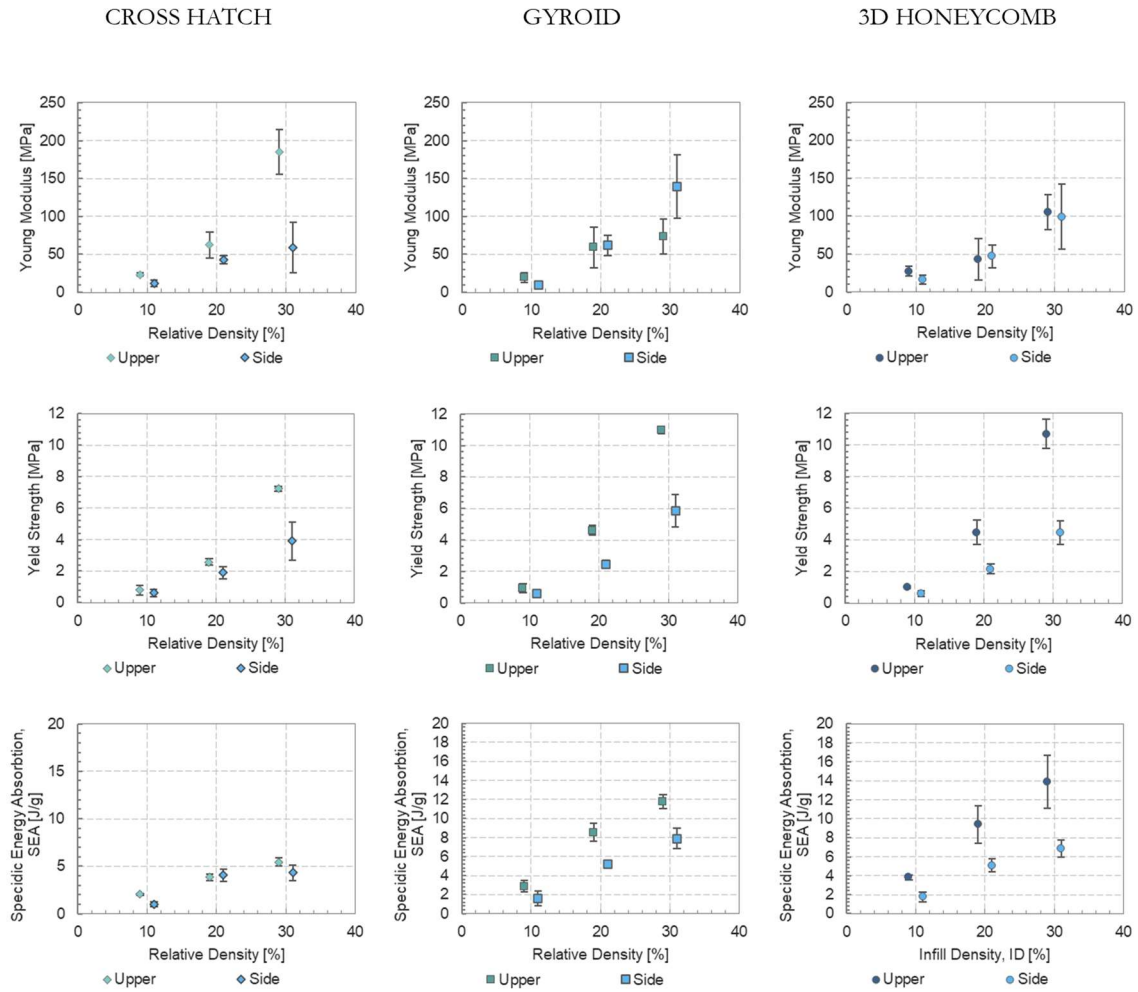


Fig. 3. 49: Summary of mechanical properties of PA6-CF lattice structures tested along the build direction (UP) and perpendicular to it (SIDE): Young's modulus (top row), yield strength (middle row), and specific energy absorption, SEA (bottom row), as a function of relative density for Cross-hatch, Gyroid, and 3D Honeycomb topologies

Differences in elastic modulus remain relatively modest for Gyroid and 3D Honeycomb at 10% and 20% RD, indicating a partially isotropic stiffness response. At 30% RD, however, the Cross-hatch structure shows a dramatic increase in anisotropy (185 MPa in UP vs. 59 MPa in SIDE), driven by the geometric alignment of long struts with the build direction.

Yield strength exhibits a more pronounced anisotropy than modulus across all topologies. Gyroid structures show a consistent ~40–60% increase in UP direction, with anisotropy strengthening with RD. Honeycomb structures display the highest anisotropy at yield, especially at 20% and 30% RD, with coefficients exceeding 2.0. These trends reflect the higher sensitivity of collapse mechanisms—buckling or shear-delamination—to orientation compared with the purely elastic regime.

SEA shows the least orientation dependence. For most configurations, UP and SIDE values differ only by 10–20%, and all topologies fall within $ASEA \approx 1.5\text{--}2.0$ except for some low-density Cross-hatch samples. The Gyroid demonstrates the most isotropic energy-absorbing behaviour, reflecting its continuous curvature and progressive collapse mode.

Anisotropy coefficients

The anisotropy index A_{Ip} , is calculated accordingly to the Eq. A as the ratio between UP and SIDE properties, while the dispersion is calculated as illustrated in the Eq. A+1. The values of indices, namely A_E , A_{σ_y} and A_{SEA} , are reported in Fig. 3. 50. The dashed line at $A = 1$ corresponds to ideal isotropy.

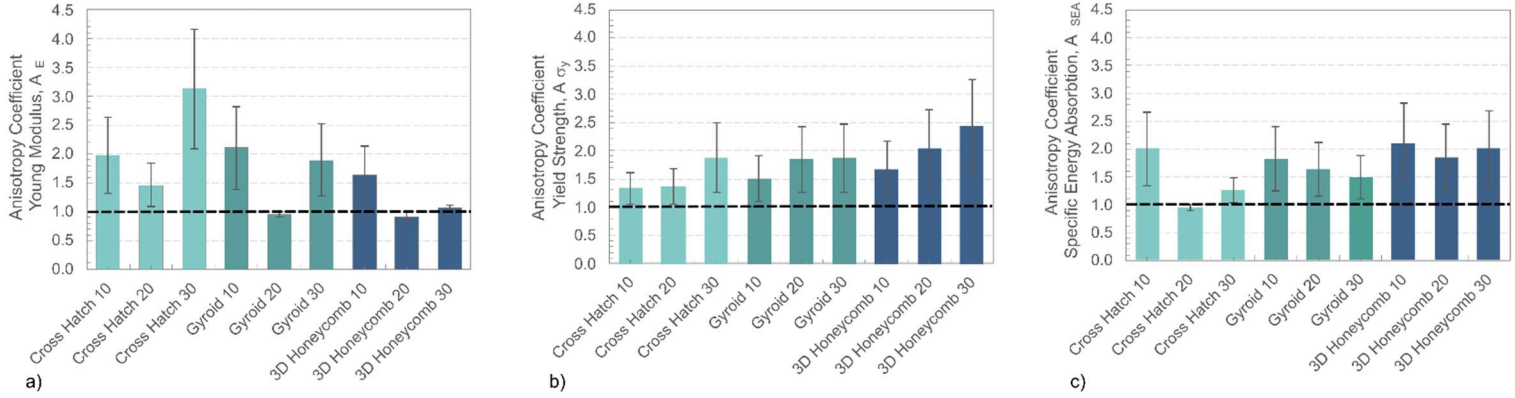


Fig. 3. 50: Anisotropy coefficients of PA6-CF lattice structures as a function of topology and relative density: (a) Young's modulus anisotropy A_E , (b) yield strength anisotropy A_{σ_y} , and (c) specific energy absorption anisotropy A_{SEA} , defined as the ratio between properties measured in the UP and SIDE loading directions. The dashed line at $A = 1$ indicates ideal isotropic behavior.

Several trends emerge:

- Crosshatch is the most anisotropic topology, especially at 30% RD, where A_E exceeds 3.0. The layered nature of geometry amplifies process-induced anisotropy.
- Gyroid displays the lowest anisotropy, with A_E and A_{σ_y} typically between 1.3 and 1.9 and decreasing with RD for SEA. Its smooth, continuous surface mitigates inter-layer weakness.
- 3D Honeycomb sits between the other two, with near-isotropic stiffness but strongly anisotropic yield strength, consistent with collapse governed by cell-wall buckling.

Interestingly, anisotropy increases with relative density for stiffness and yield strength, but decreases for energy absorption, especially for the gyroid. This occurs because thicker walls in denser samples intensify layer orientation effects for modulus and yield strength, but SEA integrates the entire stress strain response, including the densification phase, which is less direction dependent.

Overall, the anisotropy behavior indicates a key design implication: energy-absorbing performance of PA6-CF lattices is comparatively robust to orientation effects, whereas strength driven applications must carefully align load direction with the build direction.

3.4.4 Mechanical behavior of TPU lattice structures manufactured by MJF

The mechanical response of TPU lattice structures produced through Multi Jet Fusion (MJF) was investigated to evaluate the stability, deformation modes and energy absorption capabilities of elastomeric cellular materials. The study focused on two widely adopted truss-based topologies—Face-Centered Cubic (FCC) and Diamond—manufactured across four relative densities. This enabled a systematic comparison of how architecture and relative density influence quasi-static, rate-dependent, and cyclic behavior.

Fig. 3. 501 depicts the stress-strain curves of full-density samples tested under different loading speeds (0.1 mm/s to 10 mm/s). The comparison of the trends indicates a poor influence of the deformation rate on stress.

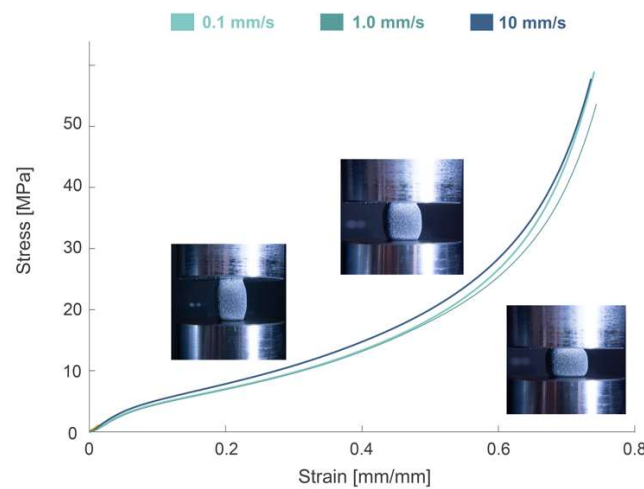


Fig. 3. 51: Stress-strain curves of the full density samples tested under different loading rates.

Fig. 3. 52 depicts the samples produced using different architectures and densities. As can be inferred, the geometries differed by the trusses thickness while the unit cell dimension was identical over the different densities. The geometry of the samples (except for the thickness of the trusses) was identical for FCC structures and for Diamond Structures. Manufacturing repeatability was evaluated through dimensional accuracy and mass consistency across all specimen configurations. Mass characterization revealed distinct patterns between lattice types (Fig. 3. 52), with strong linear correlations between theoretical and experimental values ($R^2 = 0.9932$ for FCC; $R^2 = 0.9692$ for diamond structures). Consistency varied with architectural topology and density level. FCC structures exhibited coefficients of variation ranging from 1.4% to 3.7%, with greater variability at lower densities. Diamond structures maintained more consistent coefficients of variation (1.1%–1.8%) across all density levels, indicating superior manufacturing precision. Experimental samples weight exceeded theoretical predictions for both architectures. In FCC structures, the discrepancy ranged from 23% to 35% (density-dependent), while diamond structures showed 20% to 50% deviations. This divergence was structure- and density-dependent, ruling out systematic software density input errors as the underlying cause.

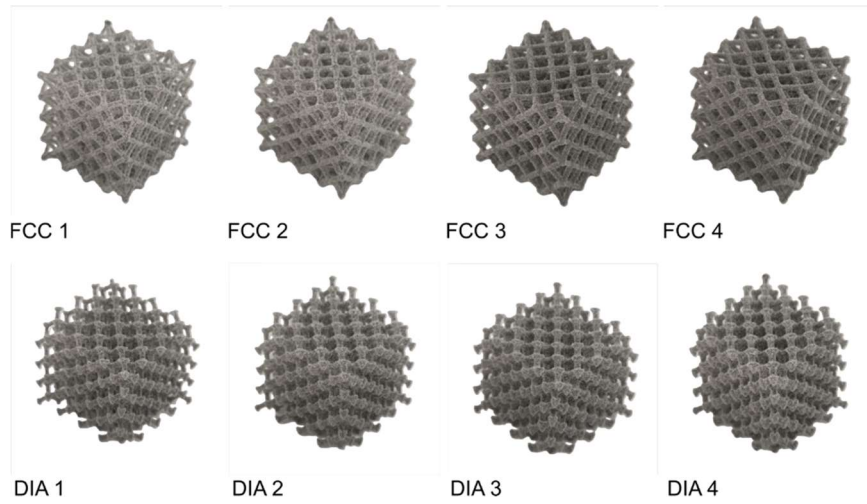


Fig. 3.52: Picture of FCC and Diamond samples produced using different relative 12.5%, 15.5%, 19% and 22%.

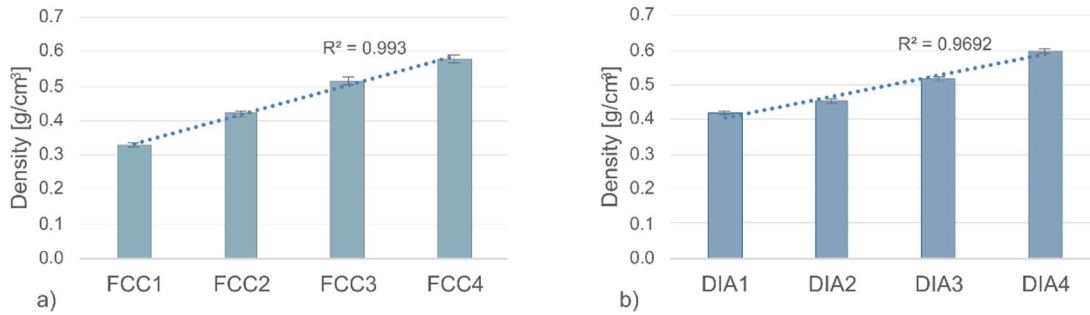


Fig. 3.53: Experimental density of lattice specimens as a function of nominal density level for (a) FCC and (b) Diamond architectures

Tab III. 8: Manufacturing consistency metrics with statistical analysis

Specimen	Mean Mass [g]	Standard Deviation [g]	Coefficient of Variation [%]	Theoretical mass values – software [g]	Relative Density [%]
FCC -1	2.15	0.08	3.7	1.74	12.5
FCC -2	2.67	0.06	2.2	1.98	15.5
FCC -3	3.21	0.07	2.2	2.37	18.7
FCC -4	3.58	0.05	1.4	2.8	20.8
DIA-1	2.71	0.04	1.5	1.8	15.8
DIA-2	2.89	0.05	1.7	2	16.8
DIA-3	3.35	0.06	1.8	2.5	19.5
DIA-4	3.72	0.04	1.1	3.1	21.6

Scanning electron microscopy, reported in Fig. 3.54, revealed powder particles adhering to the structural trusses, accounting for the experimental weight excess. This effect was more pronounced in low-density structures due to their higher surface-to-volume ratio, and less significant in denser structures. Critically, this additional mass does not contribute to load-bearing capacity.

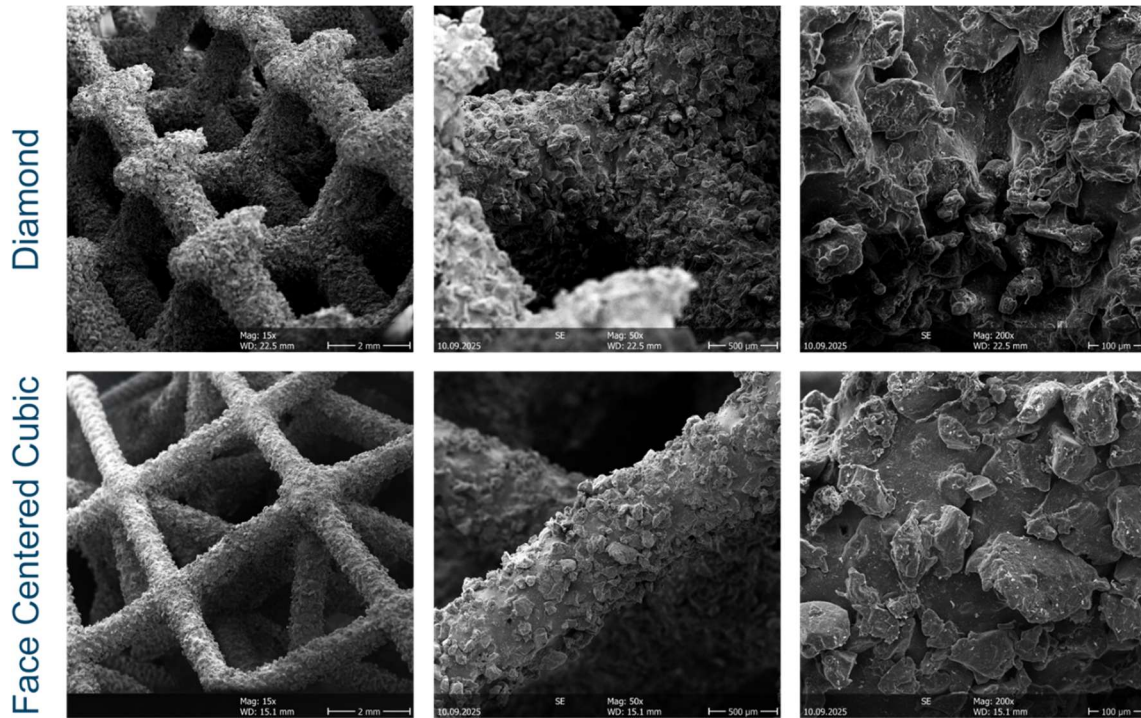


Fig. 3. 54: SEM macrographs of Diamond and Square structures produced by Binder Jetting at different magnifications

Nominal stress and strain curves were calculated by dividing force by nominal sample cross section (25 mm x 25 mm), while the strain was calculated as deformation divided by initial height (25 mm) of the samples. Engineering stress-strain curves of some reference tests are reported in Fig. 3. 55. FCC specimens exhibited characteristic cellular material behavior with distinct three-phase deformation: initial linear elastic region (5-8% strain), plateau phase with relatively constant stress levels (up to ~50% strain), and rapid densification with sharp load increase. Instead, diamond specimens showed smoother load increase throughout the deformation range, with gradual transitions between phases rather than the distinct boundaries observed in FCC structures. In addition, densification strain was delayed as compared to FCC structures while steeper load increase once reached densification was observed. Such deformations were further investigated through Digital Image Correlation Analysis, as reported in Fig. 3. 56 (FCC) and Fig. 3. 57 (Diamond). Diamond structure effectively undergoes more uniform deformation during the compression, with exception of small, localized regions owing to friction with the compression plates.

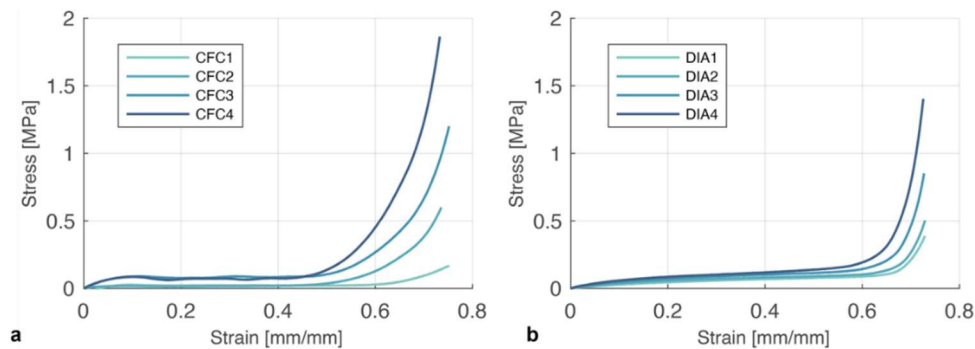


Fig. 3. 55: Engineering stress-strain curves demonstrating: (a) FCC structures with distinct three-phase behavior and sharp transitions, (b) Diamond structures with gradual progressive deformation throughout loading range

On the other hand, FCC4 structure undergoes instabilities just at the early stage of deformation. For a displacement of 2.4 mm (corresponding to an average strain of almost 0.1 mm/mm), strain localization is evident. The trusses undergo severe instabilities (since the hyperstatic nature of this structure captured by the high Maxwell number) and the strain localizes in a narrow horizontal region of the sample (Fig. 3. 56 b)). After cell collapsing (and early localized densification), the deformation proceeds with another region until the entire structure undergoes densification. These different behaviors are consistent with the bending dominated nature of diamond structure and stretched dominated nature of struct based FCC.

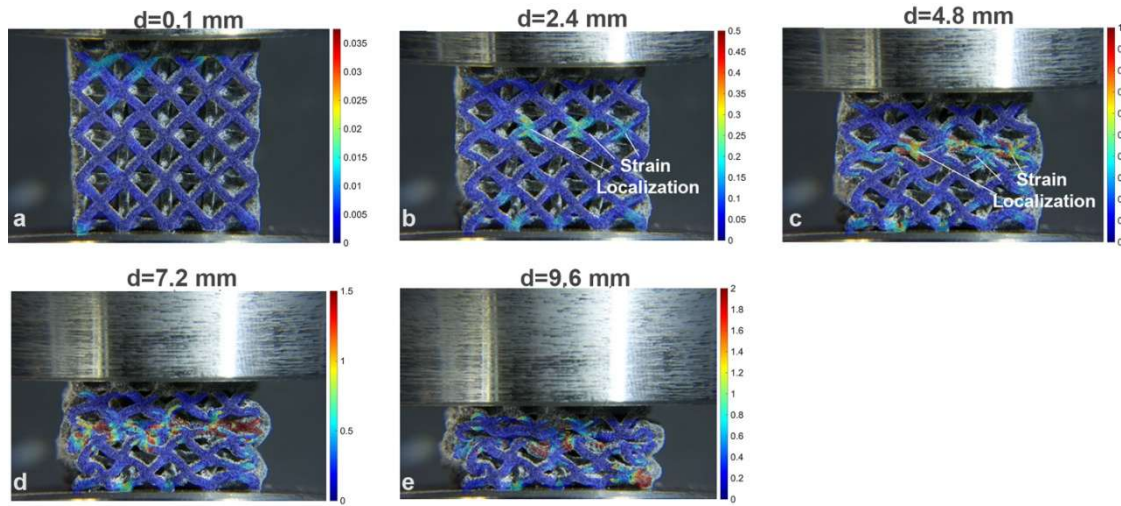


Fig. 3. 56: Sequence of pictures with equivalent strain calculated by means of Digital Image Correlation (DIC) for Face Centered Cubic structure (FCC4) at different displacements d .

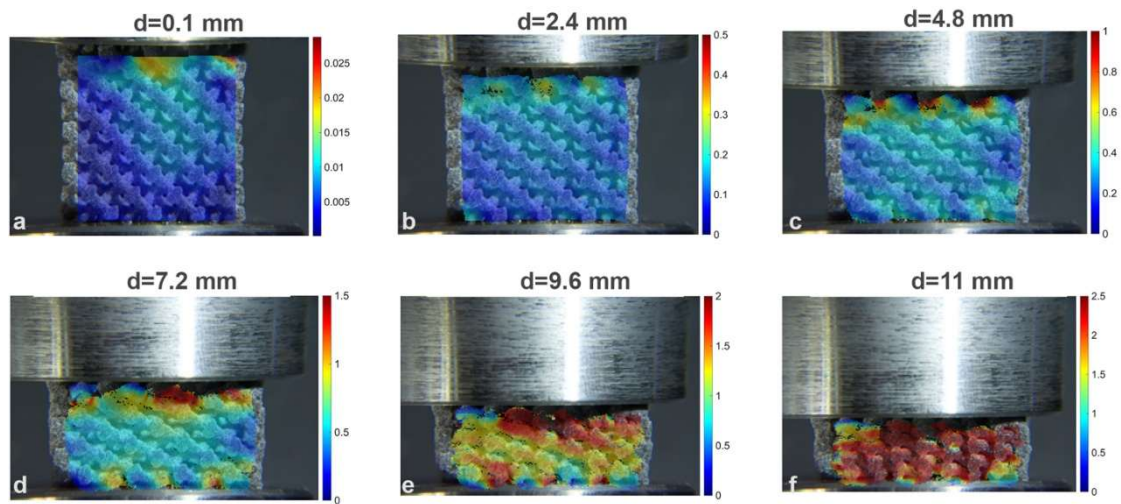


Fig. 3. 57: Sequence of pictures with equivalent strain calculated by means of Digital Image Correlation (DIC) for diamond structure (DIA4) for different displacements d .

Energy absorption efficiency curves revealed contrasting patterns between architectures (Fig. 3. 58 (a)). FCC structures exhibited irregular efficiency trends with significant oscillations throughout the deformation range, reflecting sequential cell collapsing mechanisms. Diamond architecture demonstrated smoother, more predictable efficiency curves with consistent patterns. Both configurations showed systematic reduction in

densification strain with increasing relative density. The densification strain values are presented in Fig. 3. 59.

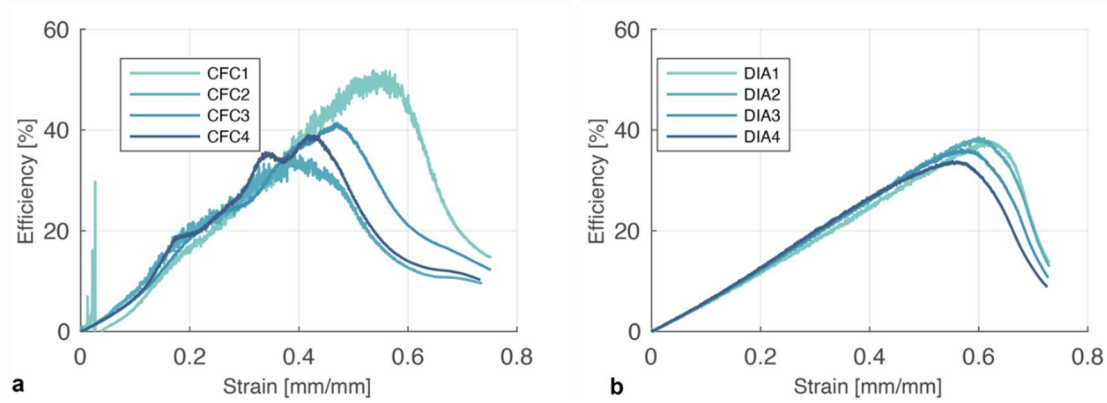


Fig. 3. 58: Energy absorption efficiency curves and densification strain analysis Energy absorption efficiency $\eta(\epsilon)$ versus strain for (a) FCC and (b) Diamond structures. Efficiency calculated using Equation 16: $\eta(\epsilon) = \frac{1}{\sigma(\epsilon)} \int_0^\epsilon \sigma(\epsilon) d\epsilon$; .

FCC structures achieved densification strains ranging from 41% to 48%, with variability ranging from 1.3% to 4.8% coefficient of variation. Diamond structures reached densification strains of 56% to 61%, with consistently low variability (standard deviation <1%) across all configurations.

The densification strain behavior exhibited distinct characteristics between the two structural configurations. For FCC structures, the densification strain demonstrated an approximately linear decrease from 0.48 to 0.45 mm/mm as relative density increased from 12.5% to 18.7%. However, a subsequent increase in relative density to 20.8% resulted in a marked discontinuity, with the densification strain decreasing abruptly to 0.41 mm/mm. This non-linear response suggests a potential transition in the deformation mechanism beyond the 18.7% relative density threshold.

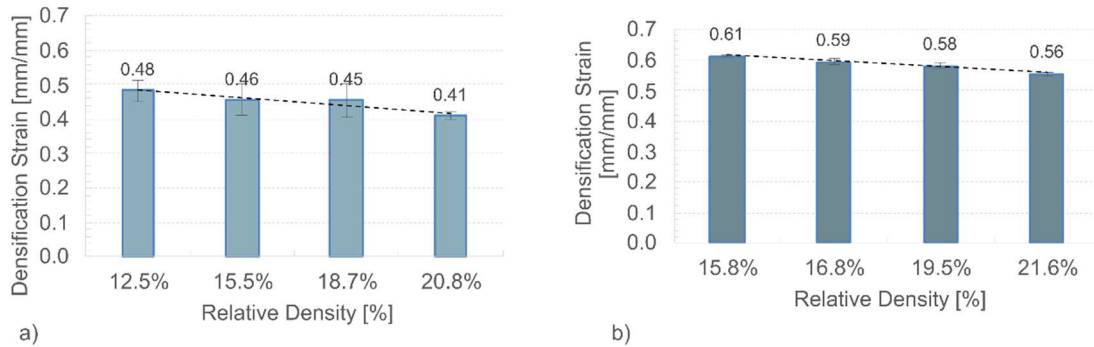


Fig. 3. 59: Densification strain trends for (a) FCC and (b) Diamond structures calculated using Eq. 17: $\epsilon_d = \left. \frac{d\eta(\epsilon)}{d\epsilon} \right|_{\epsilon=\epsilon_d} = 0$

In contrast, diamond structures displayed a more consistent trend across the tested density range. The densification strain decreased progressively from 0.61 mm/mm at 16% relative density to 0.56 mm/mm at 22% relative density, following a smoother, nearly linear trend. This more regular behavior indicates a stable deformation mode throughout the investigated range of relative densities for the diamond lattice configuration.

Energy absorption characteristics differed between the two architectures across all density levels (Fig. 3. 60 and Fig. 3. 61). FCC structures exhibited peak energy absorption at density level 3 (2.4×10^{-5} J/mm³),

followed by reduced performance at the highest density level ($1.8 \times 10^{-5} \text{ J/mm}^3$). This non-monotonic behavior resulted in energy absorption values ranging from 8.98×10^{-6} to $2.43 \times 10^{-5} \text{ J/mm}^3$. The presence of the peak at intermediate levels of relative density seems counterintuitive. The trends observed on the FCC structures of the energy is due the instability of the load during sample compression that produces fluctuations. Such instabilities induce a reduction of energy absorbed by the structure and affected the reliability of the structures (the FCC structures were characterized by higher dispersion/standard deviation) of the energy to densification as compared to diamond structures. Furthermore, the abrupt reduction of the energy densification observed in the transition of the relative density from 18.7% to 20.8% further contributed to reduce the energy to densification of the highest relative density (20.8%) as compared to 18.7% density.

Energy trends of diamond structures were characterized by more regularity (the structures with higher relative densities were always characterized by higher energies as compared to those pertaining to lower relative densities samples (Fig. 3. 61 (b)).

Besides, in diamond structures the increase in the relative density from the lowest to the highest value involved a reduction of a lower densification strain of 10.6% as compared to that measured on FCC (17.7%). This concurred in achieving higher energy densification on diamond structures as compared to FCC structures.

Diamond structures demonstrated monotonic increases in energy absorption across all density levels, ranging from 8.4×10^{-5} to $9.7 \times 10^{-5} \text{ J/mm}^3$. The variability in performance was minimal (standard deviation $< 0.006 \text{ J/mm}^3$) across all configurations.

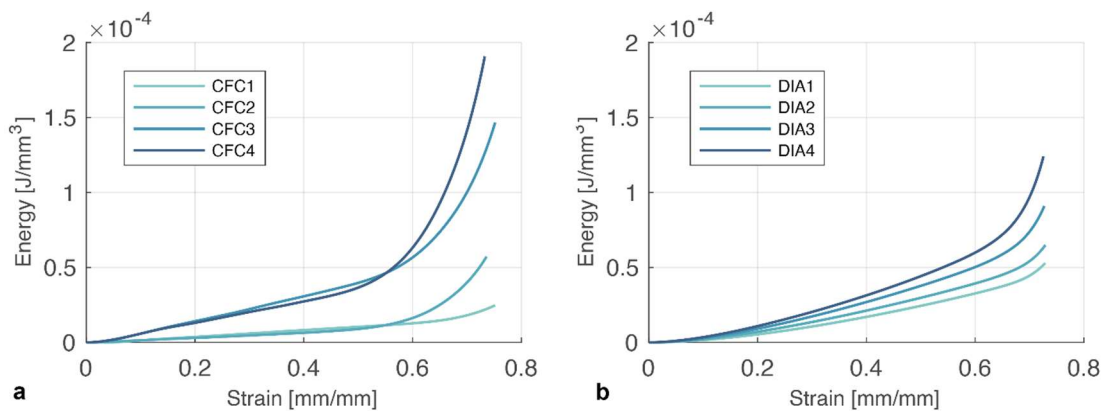


Fig. 3. 60: Specific energy absorption versus strain for different topology and relative densities (a) CFC structures and (b) Diamond structures.

Specific energy absorption (SEA) values for FCC structures ranged from $2.7 \cdot 10^{-2}$ to $4.7 \cdot 10^{-2} \text{ J/g}$, with maximum values achieved at intermediate density levels. Diamond architecture exhibited values from $8.4 \cdot 10^{-2}$ to $9.7 \cdot 10^{-2} \text{ J/g}$, with minimal sensitivity to density variations (13% range across all levels).

FCC structures showed 43% variation between minimum and maximum specific energy values, while Diamond structures-maintained values within a 13% range. Manufacturing consistency analysis revealed

higher standard deviations for FCC structures across all density levels, while Diamond structures maintained low variability (coefficient of variation $<2\%$) for all mechanical properties.

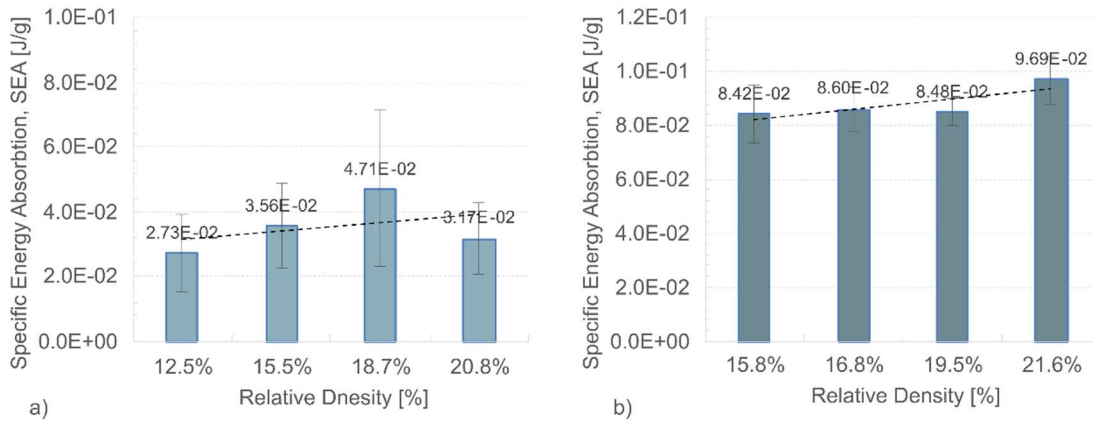


Fig. 3. 61: Specific energy absorption comparison between FCC and Diamond structures which reveal superior manufacturing consistency

The mechanical response of the Diamond lattice exhibited a pronounced dependence on the applied strain rate. Fig. 3. 62 reports the specific energy absorption (SEA) measured at 0.1, 1, and 10 mm/s for the four relative densities investigated (15.8%, 16.8%, 19.5%, 21.6%). While at quasi-static loading (0.1 mm/s) SEA values remained comparatively low, a substantial increase was observed when the loading rate increased to 1 mm/s and, more markedly, to 10 mm/s. At the highest strain rate, SEA increased by approximately a factor of 4–5 relative to the quasi-static condition, with the largest enhancement occurring at the highest relative density ($\approx 21.6\%$). This behavior is consistent with the viscoelastic nature of thermoplastic polyurethane processed via MJF: as the deformation rate increases, segmental mobility becomes increasingly restricted and the material stiffens, resulting in higher plateau stresses and delayed cell wall buckling. The increase in SEA with strain rate also reflects changes in the deformation mode of the Diamond topology. At low velocity, collapse progresses through stable bending of struts, producing a relatively flat plateau and limited energy dissipation. At higher rates, however, the structure exhibits more abrupt stress rises and localized buckling events, which amplify the area under the stress–strain curve. This effect becomes more pronounced as relative density increases, indicating a synergistic interaction between geometric stiffness and rate-dependent material hardening. Mechanical behavior of TPU diamond lattice under cyclic loading.

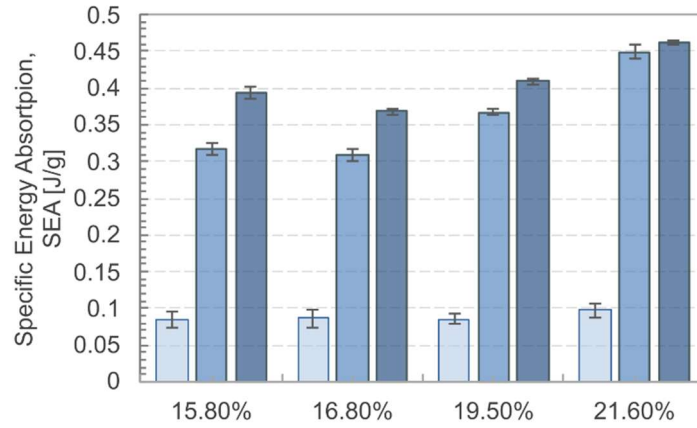


Fig. 3. 62: Specific energy absorption of Diamond lattice at 0.1, 1, and 10 mm/s for the four relative densities

3.4.4.1 Mechanical Behaviour of TPU Lattice Structures under Cyclic Loading

After establishing the monotonic compressive behaviour of FCC and Diamond topologies, additional analyses were performed to evaluate the repeatability, stability, and long-term functional performance of elastomeric Diamond lattices under cyclic loading. TPU lattices are typically employed in applications requiring repeated deformation, such as wearable protection, cushioning systems, and biomedical supports making cyclic performance a critical aspect of their qualification.

Diamond structures across the four levels of densities were subjected to repeated loading–unloading cycles up to two prescribed strain levels, namely 48% and 68%. For each density and strain level, multiple specimens were tested to ensure statistical robustness. Across all configurations, a consistent qualitative response was observed. Representative engineering stress–strain hysteresis loops are shown in Fig. 3. 63, highlighting the characteristic viscoelastic behavior of elastomeric TPU lattices.

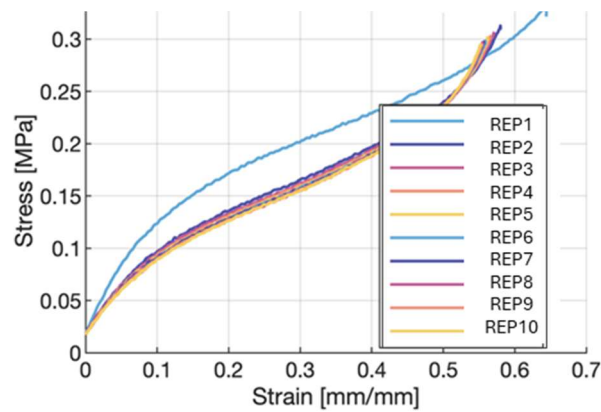


Fig. 3. 633: Representative cyclic compressive stress–strain response of a TPU Diamond lattice with 21.6% relative density subjected to repeated loading–unloading cycles. The curves show an initial strain-softening (Mullins effect) between the first and second cycles, followed by a stable and repeatable response up to the tenth cycle, with progressively reduced hysteresis and no evidence of mechanical instability or permanent damage.

The cyclic response is marked by:

- Stable and smooth loading path, with no serrations or instability.
- A pronounced reduction of hysteresis between the first and second loading cycles;
- Subsequent stabilization of both plateau stress and dissipated energy, with only minor variations up to the tenth cycle.

This behavior is characteristic of thermoplastic elastomers and can be interpreted as a Mullins-type softening effect, commonly associated with microstructural rearrangement and partial damage of the polymer network during the initial large deformation. After this initial conditioning phase, the material reaches a quasi-steady cyclic regime, in which the stress–strain curves become nearly superimposable. The absence of progressive degradation or stiffness indicates excellent repeatability and resistance to fatigue-induced damage.

This softening trend displayed in Fig. 3. 63 is typical of TPU elastomers and is associated with:

1. Microstructural rearrangement of the polymer network,
2. Localized bending of struts that become more compliant after the initial cycles,
3. Reduction of internal friction at the nodal junctions.

The stabilized response is consistent with prior reports on elastomeric TPUs produced via powder-bed fusion technologies [127,128]

The energy-based analysis is summarized in Fig. 3. 64. For each specimen, the specific energy absorption at the first cycle was taken as reference, and the mean SEA of cycles 2–10 was expressed as a percentage of that value. At 48% strain, the Diamond lattices remained between 76% and 89% of the initial SEA, depending on relative density. The lowest density (15.8%) and the highest density (21.6%) showed the highest retention ($\approx 89\%$ and $\approx 86\%$, respectively), while intermediate densities were slightly more affected, with SEA reductions of about 20–25%. At the higher strain level of 68%, the degradation was more pronounced, as expected: the residual SEA lay between 71% and 79% of the first-cycle value, corresponding to an energy loss of roughly 20–30%. Standard deviations over the four replicas ranged from 3% to 11%, indicating satisfactory repeatability of the cyclic response.

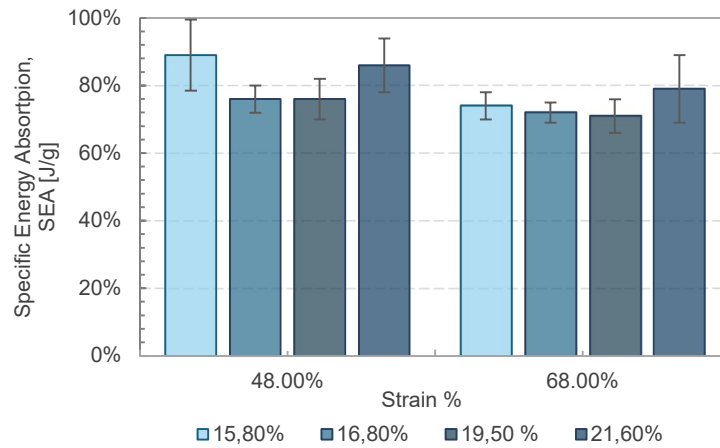


Fig. 3. 64: Normalized specific energy absorption (SEA) during cyclic compression of Diamond TPU lattices. The mean SEA over cycles 2–10 is reported as a percentage of the first-cycle value for two maximum strain levels (48% and 68%) and four relative densities, highlighting the stability and repeatability of the energy-absorbing response after initial Mullins-type softening.

Across the whole TPU campaign, the Diamond architecture consistently outperformed the FCC lattice in terms of energy absorption and stability of the collapse plateau. At quasi-static rate (0.1 mm/s) DIA specimens already showed higher SEA for a given relative density; when the loading rate was increased to 1 and 10 mm/s this advantage became even more pronounced, with SEA rising markedly while the deformation mode remained progressive and well controlled. Cyclic tests at 10 mm/s further confirmed the robustness of the Diamond topology: after the first loading cycle, which induces a limited Mullins-type softening, SEA stabilized with only 20–30% loss and good repeatability across replicas and densities.

Based on these results, the configuration with the highest relative density, DIA-4 (21.60% relative density), was selected as the reference design for the industrial application. This lattice combines the largest specific energy absorption in both quasi-static and dynamic conditions with a stable, repeatable response under multiple loading cycles. DIA-4 therefore represents the optimal compromise between mass, comfort and protective capability, and is the lattice architecture recommended to the company for the fabrication of the final shin-guard prototype.

4. DISCUSSION

4.1 Mechanisms Governing Interlayer Adhesion

The combined thermal, mechanical, and microstructural results obtained on PLA clearly demonstrate that the mechanical performance of MEX-printed polymers is governed primarily by the quality of interlayer bonding. Across all investigated configurations, tensile strength and elongation at break closely followed the substrate temperature and the interlayer time, confirming that polymer chain interdiffusion—rather than porosity or elastic stiffness—controls the dominant failure mechanisms. Directly printed specimens consistently exhibited higher strength and more ductile fracture behavior, owing to shorter interlayer times and higher consolidation temperatures. In contrast, specimens extracted from printed plates showed reduced performance and prevalent interlayer delamination, associated with longer cooling times and less effective bonding. Variations in nozzle diameter and layer height further highlighted the coupled influence of filament geometry and cooling kinetics on void morphology and interfacial adhesion.

These findings support a unified process–structure–property framework in which thermal history plays a central role. Porosity contributes to mechanical degradation only when amplified by large filament volumes or insufficient compaction, while the elastic modulus remains largely insensitive to processing conditions. The methodology developed using PLA is therefore validated as a robust approach for interpreting the mechanical response of MEX-printed components through combined thermal and microstructural evidence.

When extended to aerospace-grade PAEK materials, the same framework remains applicable while revealing additional material-specific mechanisms. As observed for PLA, directly printed PAEK specimens exhibited reduced ductility and a more brittle response, primarily due to surface defects and non-uniform interlayer bonding. In contrast, specimens extracted from bulk components displayed higher strength and significantly greater elongation at break. However, unlike PLA, PAEK introduces crystallinity as an additional governing factor. Directly printed specimens showed partial crystallization already in the as-printed state, whereas plate-cut specimens remained largely amorphous unless subjected to post-processing heat treatment. The substantial enhancement in mechanical performance following annealing confirms that, for semicrystalline high-performance polymers, interlayer adhesion and crystallization kinetics act concurrently in determining tensile behavior.

Overall, these results demonstrate that the methodology established on PLA is transferable to engineering-grade polymers, provided that material-specific effects are properly accounted for. For amorphous or low-crystallinity polymers, interlayer adhesion remains the dominant mechanism, whereas for semicrystalline materials such as PAEK, adhesion and crystallization must be interpreted jointly. This provides a coherent foundation for extending the proposed framework to more complex loading conditions and structural

configurations, including the compressive and thermo-mechanical analyses required for aerospace qualification.

4.2 Discussion – Compressive Behavior of Architected Polymeric Lattice Structures

The compressive characterization of architected lattice structures confirms that the mechanical response of additively manufactured cellular materials is governed by the combined effects of topology, relative density, material class, and process-induced features. While the general three-stage compressive behaviour typical of cellular solids is preserved across all investigated systems, namely elastic response, collapse plateau and densification, the underlying deformation mechanisms and performance metrics differ markedly depending on both architecture and manufacturing route.

For PEEK lattices manufactured by high-temperature FDM, the observed stress–strain responses highlight the strong influence of unit-cell topology on collapse stability and energy absorption. Cross-type lattices exhibit pronounced stress oscillations associated with localized buckling and layer-by-layer collapse, indicating a predominantly bending-dominated behaviour with limited redistribution of stresses. In contrast, Gyroid architectures display a smooth and extended plateau region, characterized by distributed micro-buckling of continuous curved surfaces. This deformation mode effectively suppresses stress localization and results in superior energy absorption capability. Octet structures show the highest elastic stiffness and plateau stress, consistent with their stretch-dominated nature, but are more prone to localized instability once collapse initiates. These observations align with classical and extended Gibson–Ashby frameworks, while also emphasizing that, for surface-based lattices fabricated by FDM, local wall instability and manufacturing-induced imperfections play a critical role in determining the effective collapse mode. Importantly, the fitting of experimental data reveals that the classification into bending- or stretching-dominated behaviour based solely on scaling exponents is insufficient. The absolute values of the Gibson–Ashby coefficients significantly influence the attainable stiffness and strength, explaining why bending-dominated architectures such as the Gyroid can outperform nominally stretch-dominated lattices in practical compressive applications.

Across all investigated PEEK lattices, increasing relative density leads to higher stiffness, yield strength and energy absorption, while reducing the densification strain. However, the rate at which these properties evolve with density is topology-dependent. Gyroid and Octet lattices exhibit a more-than-linear increase in strength and energy absorption with density, reflecting improved stability of thicker load-bearing surfaces. In contrast, Cross lattices show a more limited performance gain, confirming their reduced efficiency for energy-absorbing applications. When energy absorption is normalized by mass, Gyroid lattices consistently emerge as the most efficient solution, combining high plateau stress with stable collapse over a wide strain range. This finding is particularly relevant for lightweight design, where mass efficiency rather than absolute load capacity is often the primary design driver.

The thermo-mechanical characterization of PEEK provides a critical link between material-level viscoelastic response and structural performance. The DSC and DMA analyses demonstrate that annealing significantly increases crystallinity, stiffens the glassy response, and shifts the viscoelastic transition to higher temperatures. These effects are directly reflected in the compressive behaviour of lattice structures tested at elevated temperatures. Below the glass transition region, compressive stiffness and strength degrade moderately with temperature, while collapse mechanisms remain qualitatively similar to those observed at room temperature. Above the transition region, a pronounced softening occurs, leading to smoother plateaus and reduced load-bearing capacity. The bilinear temperature-dependent laws identified for both Young's modulus and yield strength provide a compact and physically meaningful representation of this behaviour, capturing the transition from a relatively stable regime to a highly temperature-sensitive one.

The qualitative agreement between normalized compressive modulus and DMA storage modulus confirms that the thermo-mechanical response of lattice structures is strongly governed by the viscoelastic behaviour of the base material, despite the additional complexity introduced by geometry and porosity.

The high-temperature constant-load compression tests further extend this analysis by addressing time-dependent deformation under conditions representative of autoclave curing. The results demonstrate that permanent compressive strain and residual load-bearing capability are strongly temperature- and density-dependent. Higher-density lattices consistently exhibit lower permanent deformation and retain a larger fraction of the applied compressive pressure after cooling, indicating superior dimensional stability.

The non-linear relationship between permanent strain and residual pressure highlights that irreversible deformation alone does not fully describe structural degradation. Instead, lattice density and topology govern how deformation translates into loss of functional performance. These findings are particularly relevant for the use of AM lattices as tooling or support structures in high-temperature environments, where repeated thermal cycles and sustained loads are expected.

When compared with PA6-CF and TPU lattice systems, the PEEK results underline the role of material rheology and viscoelasticity. PA6-CF lattices exhibit limited strain-rate sensitivity but pronounced anisotropy due to fiber orientation and interlayer bonding. TPU lattices, manufactured by MJF, show strong rate-dependent energy absorption and excellent repeatability under cyclic loading, reflecting the elastomeric nature of the material and the near-isotropic microstructure enabled by powder-bed fusion.

Together, these results demonstrate that while lattice topology governs the qualitative deformation mechanisms, material class and manufacturing process dictate rate sensitivity, anisotropy and thermal stability. A unified design approach therefore requires integrating topology optimization with material-specific thermo-mechanical characterization.

5. CONCLUSIONS

This thesis investigated the mechanical behavior of polymer components manufactured by additive manufacturing, with particular emphasis on process-induced anisotropy, interlayer adhesion and thermo-mechanical performance. A multiscale and multi-material experimental framework was developed to link manufacturing conditions, material response and structural performance, with direct relevance for industrial applications.

The methodology was first established on PLA using open-architecture FDM systems, enabling a controlled investigation of deposition strategies, thermal history and interlayer time. The results demonstrated that:

- interlayer adhesion, governed by polymer chain interdiffusion, is the primary factor controlling tensile strength and fracture behavior.
- Thermal history emerged as the dominant process signature, while porosity played a secondary role unless amplified by unfavorable filament geometry or insufficient compaction.
- The combined use of infrared thermography, mechanical testing and microstructural analysis proved effective in rationalizing process–structure–property relationships.

When the methodology was extended to aerospace-grade semicrystalline polymers such as PAEK and PEEK, the same experimental framework proved effective, while highlighting additional material-specific mechanisms. In this case, the results showed that:

- Interlayer adhesion and crystallization kinetics act jointly in governing mechanical behavior, requiring a coupled interpretation rather than a purely interfacial one.
- Thermal post-treatments (annealing) strongly modified crystallinity, stiffness and damping response, as demonstrated by DSC and DMA, leading to substantial improvements in both tensile and compressive performance.
- Component geometry and characteristic dimensions emerged as key process variables, as they directly affect interlayer time, cooling rates and substrate temperature during deposition.
- Directly printed specimens, characterized by shorter interlayer times and higher substrate temperatures, underwent incipient crystallization during deposition, which inhibited polymer chain interdiffusion and resulted in reduced tensile strength and ductility compared to specimens extracted from larger printed components.

- The observed geometric–thermal coupling confirms that, for semicrystalline polymers, mechanical performance cannot be decoupled from part scale and deposition history, and must be interpreted within an integrated process–structure–property framework.

The compressive characterization of architected lattice structures extended the investigation from bulk material behavior to structural performance across different materials and additive manufacturing technologies. The main outcomes can be summarized as follows:

- Lattice structures manufactured in PEEK, PA6-CF and TPU exhibited distinct compressive responses depending on topology, relative density, strain rate, temperature and load orientation, confirming the strong interplay between material properties and architectural design.
- Gibson–Ashby scaling laws provided a valuable interpretative framework; however, the results demonstrated that surface-based lattices require explicit consideration of elastic buckling and stability effects, particularly at low relative densities where thin walls are prone to premature instability.
- In FDM-produced lattices, process-induced anisotropy persisted as a direct consequence of build direction and layer-wise deposition, significantly affecting stiffness, yield strength and energy absorption, even when global geometry was kept constant.
- High-temperature compression and creep-like tests on PEEK lattice structures revealed a clear temperature-dependent degradation of stiffness and strength, well captured by bilinear constitutive laws and fully consistent with DMA-derived viscoelastic trends.
- The combined analysis of permanent deformation and residual load-bearing capability demonstrated that higher relative densities improve dimensional stability under sustained load, supporting the use of PEEK lattices in thermally demanding environments.

Overall, these findings support the feasibility of additively manufactured PEEK lattice structures as lightweight tooling solutions for composite curing processes. Compared to conventional subtractive manufacturing, this approach offers reduced material waste, lower production costs and increased design freedom, while maintaining adequate thermo-mechanical stability under autoclave-like conditions. However, the results also highlight an intrinsic limitation related to the glass transition temperature of PEEK, which lies in the range of 150–160 °C. When tooling applications involve operating temperatures approaching or exceeding 200 °C, the progressive viscoelastic softening observed in PEEK lattices may become critical. In such cases, polymers with higher thermal capability, such as PEI (Ultem), are more suitable candidates, offering improved dimensional stability and mechanical retention at elevated temperatures.

Beyond the specific results discussed in this thesis, a broader outcome concerns the definition of a rigorous and transferable methodology for the mechanical qualification of additively manufactured polymers and structures. In aerospace and other safety-critical sectors, where certification requirements are particularly stringent, the adoption of additive manufacturing cannot rely on nominal material data or simplified testing protocols. Instead, it requires advanced characterization strategies capable of capturing process signatures, anisotropy, thermal history and scale effects. The experimental framework developed in this work contributes

to this need, providing a structured approach that links material behavior, manufacturing conditions and structural performance in a way that is compatible with qualification and validation pathways.

More generally, the characterization of advanced polymers and architected structures enables the informed design of components across a wide range of industrial sectors. Additive manufacturing is undergoing a clear transition, moving from a tool primarily associated with rapid prototyping and small-scale production towards an established manufacturing technology for high-value, functional components. This shift is evident not only in aerospace, but also in tooling, biomedical devices, energy absorption systems and consumer products, where design freedom, lightweighting and material efficiency are increasingly decisive factors. In this context, robust experimental methodologies such as those presented in this thesis are essential to support the reliable and responsible integration of additive manufacturing into industrial practice, bridging the gap between laboratory-scale studies and real-world applications.

BIBLIOGRAPHY

1. Attaran M (2017) The rise of 3-D printing: The advantages of additive manufacturing over traditional manufacturing. *Business horizons* 60:677-688.
2. Leung Y-SaK, Tsz-Ho and Li, Xiangjia and Yang, Yang and Wang, Charlie CL and Chen, Yong (2019) Challenges and status on design and computation for emerging additive manufacturing technologies. *Journal of Computing and Information Science in Engineering* 19.
3. Hopping EPaH, Wensheng and Xu, Kunning G (2021) Small hall effect thruster with 3d printed discharge channel: Design and thrust measurements. *Aerospace* 8.
4. Ghomi ERaK, Fatemeh and Neisiany, Rasoul Esmaeely and Singh, Sunpreet and Ramakrishna, Seeram (2021) Future of additive manufacturing in healthcare. *Current Opinion in Biomedical Engineering* 17.
5. Mohajeri BaP, J{"u}rgen and Kauranen, Ilkka and Nyberg, Timo (2016) Shift to social manufacturing: Applications of additive manufacturing for consumer products. Paper presented at the 2016 IEEE International Conference on Service Operations and Logistics, and Informatics (SOLI),
6. Bordegoni SGaFRaRCaPSaMVaM (2017) Designing for Metal Additive Manufacturing: A Case Study in the Professional Sports Equipment Field. **Procedia Manufacturing**.
7. Weyhrich CWaL, Timothy E (2022) Additive manufacturing of high-performance engineering polymers: present and future. *Polymer International* 71:532 - 536.
8. Stansbury JWaL, Mike J (2016) 3D printing with polymers: Challenges among expanding options and opportunities. *Dental materials* 32.
9. Davoudinejad AaDP, LC and Quagliotti, D and Pedersen, David B and Albajez Garc{ (2018) Geometric and feature size design effect on vat photopolymerization micro additively manufactured surface features. *Special Interest Group Meeting: Additive Manufacturing: Advancing Precision in Additive Manufacturing*.
10. Zhao LaY, Ran and He, Yangyang and Zhang, Manwen and Tian, Feng and Wang, Lei and Zhao, Yulei and Huang, Wei (2024) 3D printed epoxy/acrylate hybrid polymers with excellent mechanical and shape memory properties via UV and thermal cationic dual-curing mechanism. *Additive Manufacturing* 79.
11. Rosa R (2023) Synthesis and characterization of bio-based polymers for innovative additive manufacturing applications via stereolithography 3D printing.
12. Krizsma SaS (2025) Applying high-performance resins in stereolithography printing to produce prototype injection moulds. *Scientific Reports* 15.
13. LIQCREATE Elastomer-X. https://www.liqcreate.com/product/elastomer-x/?utm_source=chatgpt.com.
14. TOPP RAPID PROTOTYPING technology. https://www.rp.topp.it/prod/en/dlp-digital-light-processing_l4?utm_source=chatgpt.com
15. Meenakshisundaram VaF, Keyton and Chartrain, Nicholas and Long, Timothy and Williams, Christopher (2024) Characterizing photopolymer resins for high-temperature vat photopolymerization. *Progress in Additive Manufacturing* 9:2061 - 2071.
16. Steyrer BaB, Bernhard and Harak{'a}ly, Gy{'o}rgy and Liska, Robert and Stampfl (2018) Hot Lithography vs. room temperature DLP 3D-printing of a dimethacrylate. *Additive Manufacturing* 21.
17. Bove AaC, Flaviana and Galati, Manuela and Iuliano, Luca (2022) Photopolymerization of ceramic resins by stereolithography process: a review. *Applied sciences* 12.

18. Guttridge CaS, Alice and O'Sullivan, Aidan and O'Sullivan, Kevin J and O'Sullivan, Leonard W (2022) Biocompatible 3D printing resins for medical applications: A review of marketed intended use, biocompatibility certification, and post-processing guidance. *Annals of 3D Printed Medicine* 5.
19. Ngo TDaK, Alireza and Imbalzano, Gabriele and Nguyen, Kate TQ and Hui, David (2018) Additive manufacturing (3D printing): A review of materials, methods, applications and challenges. *Composites Part B: Engineering* 143:172 - 196.
20. Peng HaH, Bo and Tong, Tianjian and Jin, Xin and Peng, Yanbo and Guo, Meitong and Li, Bian and Ding, Jiaxin and Kong, Qingfei and Wang, Qun (2024) 3D printing processes in precise drug delivery for personalized medicine. *Biofabrication*.
21. Hui DaG, Ruth D and Scotchford, CA and Grant, David M (2018) Laser sintering of nano-hydroxyapatite coated polyamide 12 powders. *Additive Manufacturing*:560 - 570.
22. Caulfield BaM, Peter E and Lohfeld, S (2007) Dependence of mechanical properties of polyamide components on build parameters in the SLS process. *Journal of Materials Processing Technology* 182.
23. Pavan MaF, M and Strobbe, D and Van Hooreweder, B and Craeghs, T and Moens, D and Dewulf, W (2017) On the influence of inter-layer time and energy density on selected critical-to-quality properties of PA12 parts produced via laser sintering. *Polymer testing* 61.
24. Gunpath UFaW, Gavin and Pawlik, Marzena and Lu, Yiling and Wood, Paul (2023) Effect of Powder Bed Fusion Laser Sintering on Dimensional Accuracy and Tensile Properties of Reused Polyamide 11. *Polymers* 15.
25. Zhang CaL, Teng and Deng, Qingtian and Li, Xinbo (2022) Compression behavior of 3D printed polymer TPU cubic lattice structure. *Materials Research* 25.
26. Czelusniak TaA, Fred L (2020) Influence of energy density on selective laser sintering of carbon fiber-reinforced PA12. *The International Journal of Advanced Manufacturing Technology* 111.
27. Berretta SaE, K and Ghita, O (2018) Additive manufacture of PEEK cranial implants: Manufacturing considerations versus accuracy and mechanical performance. *Materials \& Design* 139.
28. Alomarah AaR, Dong and Masood, Syed and Gao, Zhanyuan (2019) Compressive properties of a novel additively manufactured 3D auxetic structure. *Smart Materials and Structures* 28.
29. Xu ZaW, Yue and Wu, Dingdi and Ananth, K Prem and Bai, Jiaming (2019) The process and performance comparison of polyamide 12 manufactured by multi jet fusion and selective laser sintering. *Journal of Manufacturing Processes* 47.
30. Vaes DaVP, Peter (2021) Semi-crystalline feedstock for filament-based 3D printing of polymers. *Progress in Polymer Science* 118.
31. Bellehumeur CelaL, Longmei and Sun, Qian and Gu, Peihua (2004) Modeling of bond formation between polymer filaments in the fused deposition modeling process. *Journal of manufacturing processes* 6.
32. Inkjet Printing and Adhesion of Low Surface Energy Polymers.
33. Shanmugam VaP, Munagoti Venkata and Babu, Karthik and Karnan, Balamurugan (2021) Fused deposition modeling based polymeric materials and their performance: A review. *Polymer Composites* 42.
34. Cicala GaL, Alberta and Del Curto, Barbara and Lo Russo, Alessio and Recca, Giuseppe and Far{\`e}, Silvia (2017) Engineering thermoplastics for additive manufacturing: A critical perspective with experimental evidence to support functional applications. {*Journal of applied biomaterials \& functional materials* 15.
35. Matsuzaki RaU, Masahito and Namiki, Masaki and Jeong, Tae-Kun and Asahara, Hirosuke and Horiguchi, Keisuke and Nakamura, Taishi and Todoroki, Akira and Hirano, Yoshiyasu (2016) Three-dimensional printing of continuous-fiber composites by in-nozzle impregnation. *Scientific reports* 6.

36. Lee AaW, Mathew and Quigley, Liam and Salviato, Marco and Zobeiry, Navid (2022) Effect of temperature history during additive manufacturing on crystalline morphology of PEEK. *Advances in Industrial and Manufacturing Engineering* 4.
37. {Vatanda{\c{s}}} BBicsaGumuk, Recep Additive manufacturing and mechanical performance of short fiber reinforced PEEK thermoplastic composites in a vacuum environment. *The International Journal of Advanced Manufacturing Technology* 134:1677 - 1698.
38. Aliberti FaL, Raffaele and Raimondo, Marialuigia and Pantani, Roberto and Vertuccio, Luigi and Guadagno, Liberata (2025) Additive manufacturing of polymers and composites for applications in aerospace and aeronautics. *Materials Horizons*.
39. El Khadiri IaZ, Maria and Nguyen, Nhan-Quy and Abouelmajd, Mohamed and Hmina, Nabil and Belhouideg, Soufiane (2023) Topology optimization methods for additive manufacturing: a review. *International Journal for Simulation and Multidisciplinary Design Optimization* 14.
40. Wu TaT, Andres (2019) Multiscale, thermomechanical topology optimization of self-supporting cellular structures for porous injection molds. *Rapid Prototyping Journal* 25.
41. Jihong ZaH, ZHOU and Chuang, WANG and Lu, ZHOU and Shangqin, YUAN and others (2021) A review of topology optimization for additive manufacturing: Status and challenges. *Chinese Journal of Aeronautics* 34:91 - 110
42. Thompson MKaM, Giovanni and Vaneker, Tom and Fadel, Georges and Campbell, R Ian and Gibson, Ian and Bernard, Alain and Schulz, Joachim and Graf, Patricia and Ahuja, Bhriugu and others (2016) Design for Additive Manufacturing: Trends, opportunities, considerations, and constraints. *CIRP annals* 65:737 - 760.
43. Rinaldi MaC, Federico and Pigliaru, Lucia and Ghidini, Tommaso and Lumaca, Francesco and Nanni, Francesca (2020) Additive manufacturing of polyether ether ketone (PEEK) for space applications: A nanosat polymeric structure. *Polymers* 13.
44. Merema BBaS, Frederik KL and Kraeima, Joep and Witjes, Max JH (2025) A non-metallic PEEK topology optimization reconstruction implant for large mandibular continuity defects, validated using the MANDYBILATOR apparatus. Merema, Bram BJ and Spijkervet, Frederik KL and Kraeima, Joep and Witjes, Max JH 15.
45. Carter WaE, DJ and Abbott, DH and Bruck, CE and Wilson, GH and Wolfe, JB and Finkhousen, DM and Tepper, A and Stevens, RG (2014) The GE aircraft engine bracket challenge: an experiment in crowdsourcing for mechanical design concepts.
46. Fasel UaK, Dominic and Baumann, Leo and Cavolina, Giovanni and Eichenhofer, Martin and Ermanni, Paolo (2020) Composite additive manufacturing of morphing aerospace structures. *Manufacturing Letters* 23:85 - 88.
47. Liu SaL, Yingguang and Li, Nanya (2018) A novel free-hanging 3D printing method for continuous carbon fiber reinforced thermoplastic lattice truss core structures. *Materials \& Design* 137.
48. Belleumeur CelaL, Longmei and Sun, Qian and Gu, Peihua (2004) Modeling of bond formation between polymer filaments in the fused deposition modeling process. *Journal of manufacturing processes* 6:170 - 178
49. Thumsorn SaP, Wattanachai and Kurose, Takashi and Ishigami, Akira and Kobayashi, Yutaka and Ito, Hiroshi (2022) Rheological behavior and dynamic mechanical properties for interpretation of layer adhesion in FDM 3D printing. *Polymers* 14.
50. Ahn S-HaM, Michael and Odell, Dan and Roundy, Shad and Wright, Paul K (2002) Anisotropic material properties of fused deposition modeling ABS. *Rapid prototyping journal* 8:248 - 257.
51. Ahn D-KaK, Soon-Man and Lee, Seok-Hee (2008) Expression for surface roughness distribution of FDM processed parts. Paper presented at the 2008 International Conference on Smart Manufacturing Application,
52. Goshtasbi AaG, Luca and Sadeghi, Ali (2025) Bio-inspired 3D printing approach for bonding soft and rigid materials through underextrusion. *Scientific reports* 15.
53. Alkunte SaF, Ismail and Naikwadi, Vivekanand and Gudavasov, Shamil and Ali, Mohammad Alshaikh and Mahmudov, Mushfig and Hasanov, Seymour and Cheepu, Muralimohan (2024)

- {Advancements and challenges in additively manufactured functionally graded materials: A comprehensive review. *Journal of Manufacturing and Materials Processing* 8.
54. Brure de Carvalho Paiva VM (2022) Under-extrusion challenges for elastic filaments: the influence of moisture on additive manufacturing. *Deutsche Nationalbibliothek*.
 55. Lawrence BDaC, Michael D and Phillips, Francis and Henry, Todd C and Nikishkov, Yuri and Makeev, Andrew (2022) Evaluation of the mechanical properties and performance cost of additively manufactured continuous glass and carbon fiber composites. *The International Journal of Advanced Manufacturing Technology* 120.
 56. Pace FaS, Antonios G and Eckl, Michael and Senck, Sascha and Glinz, Jonathan (2023) Analysis of the manufacturing porosity in fused filament fabricated onyx/long fiber reinforced composites using X-ray computed tomography. *Journal of Nondestructive Evaluation* 42.
 57. Glinz JaP, Francesco and Maurer, Julia and Holzleitner, Martin and Eckl, Michael and Vop{\a}lensk{\y}, Michal and Kumpov{\a}, Ivana and Kastner, Johann and Stamopoulos, Antonios and Senck, Sascha (2023) Influence of continuous fiber reinforcement on tensile properties in fused filament fabricated specimens.
 58. Armillotta AaB, Mattia and Cavallaro, Marco (2018) Warpage of FDM parts: Experimental tests and analytic model. *Robotics and Computer-Integrated Manufacturing* 50:140 - 152.
 59. Huynh TTaN, Tien VT and Nguyen, Quoc Manh and Nguyen, Trieu Khoa (2021) Minimizing Warpage for Macro-Size Fused Deposition Modeling Parts. *Computers, Materials \& Continua* 68.
 60. Cavallaro MaA, A (2017) Edge quality in Fused Deposition Modeling: I. Definition and analysis.
 61. Turner BNaG, Scott A (2015) A review of melt extrusion additive manufacturing processes: II. Materials, dimensional accuracy, and surface roughness. *Rapid Prototyping Journal* 21:250 - 261.
 62. Sun XaM, Maciej and Cheng, Chi-Tsun (2023) A review of void reduction strategies in material extrusion-based additive manufacturing. *Additive Manufacturing* 67.
 63. Spoerk MaG-G, Joamin and Sapkota, Janak and Schuschnigg, Stephan and Holzer, Clemens (2018) Effect of the printing bed temperature on the adhesion of parts produced by fused filament fabrication. *Plastics, Rubber and Composites* 47:17 - 24.
 64. Adarsh SHaN, Mahadevappa (2025) Effect of Printing Parameters on Mechanical Properties and Warpage of 3D-Printed PEEK/CF-PEEK Composites Using Multi-Objective Optimization Technique. *Journal of Composites Science* 9.
 65. Chacón JM, Caminero, M. A., García-Plaza, E., & Núñez, P. J. (2017) Additive manufacturing of PLA structures using fused deposition modelling: Effect of process parameters on mechanical properties and their optimal selection. *Materials & Design* 124:143-157.
 66. Tymrak BM, Megan Kreiger, and Joshua M. Pearce (2014) Mechanical properties of components fabricated with open-source 3-D printers under realistic environmental conditions. *Materials & Design* 58:242 - 246.
 67. ASTM International: West Conshohocken P, USA, 2013. (2013) Standard Terminology for Additive Manufacturing-Coordinate Systems and Test MethodologieS.
 68. Rankouhi BaJ, Sina and Delfanian, Fereidoon and Letcher, Todd (2016) Failure analysis and mechanical characterization of 3D printed ABS with respect to layer thickness and orientation. *Journal of Failure Analysis and Prevention* 16:467--481.
 69. Sikder PaC, Bharath Tej and Gummadi, Sudeep Kumar (2022) A comprehensive analysis on the processing-structure-property relationships of FDM-based 3-D printed polyetheretherketone (PEEK) structures. *Materialia* 22.
 70. Hassanifard SaB, Kamran (2022) Effects of voids and raster orientations on fatigue life of notched additively manufactured PLA components. *The International Journal of Advanced Manufacturing Technology* 120:6241--6250.
 71. Drozdov AadC, Jesper (2021) Thermo-mechanical behavior of poly (ether ether ketone): Experiments and modeling. *Polymers* 13.

72. Basgul CaT, Florian M and Kurtz, Steven M (2021) Heat transfer-based non-isothermal healing model for the interfacial bonding strength of fused filament fabricated polyetheretherketone. *Additive manufacturing* 46.
73. Wang XaZ, Liping and Fuh, Jerry Ying Hsi and Lee, Heow Pueh (2019) Effect of porosity on mechanical properties of 3D printed polymers: Experiments and micromechanical modeling based on X-ray computed tomography analysis. *Polymers* 11.
74. Ashby MFaG, Lorna J (1997) *Cellular solids: structure and properties*. Press Syndicate of the University of Cambridge, Cambridge, UK:175 - 231.
75. Maskery IaS, Logan and Aremu, Adedeji O and Panesar, Ajit and Williams, Christopher B and Tuck, Christopher J and Wildman, Ricky D and Ashcroft, Ian A and Hague, Richard JM (2018) Insights into the mechanical properties of several triply periodic minimal surface lattice structures made by polymer additive manufacturing. *Polymer Composites* 152:92 - 71.
76. Benedetti MaDP, Anton and Ritchie, Robert O and Dallago, Michele and Razavi, Nima and Berto, Filippo (2021) Architected cellular materials: A review on their mechanical properties towards fatigue-tolerant design and fabrication. *Materials Science and Engineering: R: Reports* 144.
77. Deshpande VSaF, Norman A and Ashby, Michael F (2001) Effective properties of the octet-truss lattice material. *Journal of the Mechanics and Physics of Solids*.
78. Ashby MFaG, Lorna J (2006) The properties of foams and lattices. *Philosophical Transactions of the Royal Society A: Mathematical, Physical and Engineering Sciences* 364:15 - 30.
79. Qiu CaY, Sheng and Adkins, Nicholas JE and Ward, Mark and Hassanin, Hany and Lee, Peter D and Withers, Philip J and Attallah, Moataz M (2015) Influence of processing conditions on strut structure and compressive properties of cellular lattice structures fabricated by selective laser melting. *Materials Science and Engineering: A* 628:188 - 197.
80. Liu WaS, Lin and Zhang, Zihui and Ju, Shanglian and Wang, Fei and Zhao, Yiping (2023) Compression and resilient behavior of graded triply periodic minimal surface structures with soft materials fabricated by fused filament fabrication. *Journal of Manufacturing Processes* 105:1 - 13.
81. Bose SaD, Aditi and Bandyopadhyay, AmitBose, Susmita and Dahiya, Aditi and Bandyopadhyay, Amit (Additive Manufacturing) *Additive Manufacturing of Biomaterials*.169 - 197.
82. Murr LEaG, Sara M and Ramirez, Diana A and Martinez, Edwin and Hernandez, Jennifer and Amato, Krista N and Shindo, Patrick W and Medina, Francisco R and Wicker, Ryan B (2012) Metal fabrication by additive manufacturing using laser and electron beam melting technologies. *Journal of Materials Science \& Technology* 28:1 - 14.
83. Ryan GaP, Abhay and Apatsidis, Dimitrios Panagiotis Fabrication methods of porous metals for use in orthopaedic applications. *Biomaterials* 27:2651--2670.
84. Arabnejad SaJ, R Burnett and Pura, Jenny Ann and Singh, Baljinder and Tanzer, Michael and Pasini, Damiano (2016) High-strength porous biomaterials for bone replacement: A strategy to assess the interplay between cell morphology, mechanical properties, bone ingrowth and manufacturing constraints. *Acta biomaterialia* 30:345--356.
85. Kurtz SMaD, John N (2007) PEEK biomaterials in trauma, orthopedic, and spinal implants. *Biomaterials* 28:4845--4869.
86. Toth JMaW, Mei and Estes, Bradley T and Scifert, Jeffrey L and Seim III, Howard B and Turner, A Simon (2006) Polyetheretherketone as a biomaterial for spinal applications. *Biomaterials* 27:324--334.
87. Berretta SaE, K and Ghita, O (2018) Additive manufacture of PEEK cranial implants: Manufacturing considerations versus accuracy and mechanical performance. *Materials & Design* 139:141 - 152.
88. Hou WaH, Pan and Yang, Yi and Sang, Lin (2023) Crashworthiness optimization of crash box with 3D-printed lattice structures. *International Journal of Mechanical Sciences* 247.

89. Wang KaL, Yisen and Wang, Jin and Xiang, Jiangyang and Yao, Song and Peng, Yong (2022) On crashworthiness behaviors of 3D printed multi-cell filled thin-walled structures. *Engineering Structures* 254.
90. Um H-JaJ, Na-Hyun and Shin, Ji-Hwan and Kim, Hak-Sung (2023) High-performance multifunctional energy storage-corrugated lattice core sandwich structure via continuous carbon fiber (CCF)/polyamide 6 (PA6) 3D printing. 5.
91. Yuan SaS, Fei and Bai, Jiaming and Chua, Chee Kai and Wei, Jun and Zhou, Kun (2017) Tancogne-Dejean stiffness and SEA of AM BCC], [Yuan 3D soft auxetic lattice structures fabricated by selective laser sintering. *Materials & Design* 120.
92. Pelin GaS, Maria and Pelin, Cristina-Elisabeta (2024) The use of additive manufacturing techniques in the development of polymeric molds: a review. *Polymers* 16.
93. Additively Manufactured Composite Tooling. https://www.stratasys.com/contentassets/57bc4d217c224cababb9990ec9b5f210/wp_fdm_additivelymanufacturedct_0220a.pdf?v=4a0b10.
94. Hassen AAaL, John and Chen, Xun and Post, Brian and Love, Lonnie and Kunc, Vlastimil (2016) Additive manufacturing of composite tooling using high temperature thermoplastic materials. Paper presented at the SAMPE Conference Proceedings, Long Beach, CA,
95. Kunc VaL, John and Dinwiddie, Ralph and Post, Brian and Love, Lonnie and Matlack, M and Fahey Jr, RL and Hassen, Ahmed A (2016) Investigation of in-autoclave additive manufacturing composite tooling. Paper presented at the CAMX Conference, Anaheim, CA,
96. Kunc VaH, Ahmed Arabi and Lindahl, John and Kim, Seokpum and Post, Brian and Love, Lonnie (2017) Large scale additively manufactured tooling for composites. Paper presented at the Proceedings of 15th Japan international SAMPE symposium and exhibition,
97. Carbon Fiber Parts Manufacturing With 3D Printed Molds. <https://3d.formlabs.com/white-paper-carbon-fiber-parts-manufacturing-with-3d-printed-molds/>.
98. Dynamism: How to 3D Print Molds for Carbon Fiber Parts. <https://www.dynamism.com/learn/automotive/how-to-3d-print-carbon-fiber-molds.html>.
99. Campbell Jr FC (2003) Manufacturing processes for advanced composites.
100. Mallick P (2017) Processing of polymer matrix composites.
101. Korat MaA, GD (2014) A review on current research and development in abrasive waterjet machining. *International Journal of Engineering Research and Applications*.
102. Hashish M (2024) Abrasive waterjet machining. *Materials* 17.
103. ASTM International: West Conshohocken P, USA, 2013. (2007) AC09036782, Anonymus, ed. Standard test method for mode I interlaminar fracture toughness of unidirectional fiber-reinforced polymer matrix composites.
104. Young D, Wetmore, N., & Czabaj, M. (2018) Interlayer fracture toughness of additively manufactured unreinforced and carbon-fiber-reinforced acrylonitrile butadiene styrene. *Additive Manufacturing* 22:508-515.
105. Khudiakova A, Arbeiter, F., Spoerk, M., Wolfahrt, M., Godec, D., & Pinter, G (2019) Inter-layer bonding characterisation between materials with different degrees of stiffness processed by fused filament fabrication. *Additive Manufacturing* 28:184 - 193.
106. Fonseca J, Ferreira, I. A., De Moura, M. F. S. F., Machado, M., & Alves, J. L. (2019) Study of the interlaminar fracture under mode I loading on FFF printed parts. *Composite Structures* 214:316-324.
107. Marşavina L, Stoia, D. I., & Emanoil, L. (2021) Fracture toughness in additive manufacturing by selective laser sintering: an overview. *Material Design & Processing Communications* 3.
108. Stamopoulos AG, Scipioni, S. I., & Lambiase, F (2023) Experimental characterization of the interlayer fracture toughness of FDM components. *Composite Structures* 320.
109. Spoerk M, Gonzalez-Gutierrez, J., Sapkota, J., Schuschnigg, S., & Holzer, C (2018) Effect of the printing bed temperature on the adhesion of parts produced by fused filament fabrication. *Plastics, Rubber and Composites* 47:17-24.

110. Zhao Y, Chen, Y., & Zhou, Y. (2019) Novel mechanical models of tensile strength and elastic property of FDM AM PLA materials: Experimental and theoretical analyses. *Materials & Design* 181.
111. International A ASTM D638: Standard test method for tensile properties of plastics.
112. (ISO) IOfS (2002) ISO 604:2002: Plastics — Determination of compressive properties.
113. Doumeng MaM, Lotfi and Berthet, Florentin and Marsan, Olivier and Delb{\e}, Karl and Denape, Jean and Chabert, France (2021) A comparative study of the crystallinity of polyetheretherketone by using density, DSC, XRD, and Raman spectroscopy techniques. *Polymer Testing* 93.
114. Deshpande VSaF, Norman A and Ashby, Michael F (2001) Effective properties of the octet-truss lattice material. Deshpande, Vikram S and Fleck, Norman A and Ashby, Michael F 49.
115. Zheng XaS, William and Jackson, Julie and Moran, Bryan and Cui, Huachen and Chen, Da and Ye, Jianchao and Fang, Nicholas and Rodriguez, Nicholas and Weisgraber, Todd and others (2016) Multiscale metallic metamaterials. *Nature materials* 15.
116. Burg KaS, SW (2001) PES and PEEK. *Encyclopedia of materials: Science and Technology*:6837--6839.
117. Viswanath AaK, M and Khan, MKA and Cantwell, WJ and Khan, KA (2025) Hierarchical cubic lattice structures with bending-and stretching-dominated cellular designs for enhanced buckling resistance. *International Journal of Lightweight Materials and Manufacture* 8:310 - 320.
118. Tardif XaP, Baptiste and Boyard, Nicolas and Schmelzer, J{"u}rn WP and Sobotka, Vincent and Delaunay, Didier and Schick, Christoph (2014) Experimental study of crystallization of PolyEtherEtherKetone (PEEK) over a large temperature range using a nano-calorimeter. *Polymer Testing* 36:10 - 19.
119. Wei ZaS, Ping and Zhou, Cheng and Chen, Guangyi and Chang, Ying and Li, Jianfeng and Zhang, Wanxi and Liang, Jicai (2013) Insight into the annealing peak and microstructural changes of poly (l-lactic acid) by annealing at elevated temperatures. *Polymer Composites* 54:3377--3384.
120. Wunderlich B (2012) *Macromolecular physics* V2.
121. Kolesov I, & Androsch, R. (2012) The rigid amorphous fraction of cold-crystallized polyamide 6. *Polymer Composites* 53:4770-4777.
122. Mössinger IaS, Yannick and Salerno, Dario Emanuele and Chadwick (2024) Refined determination of dsc-measured crystallinity for thermoplastic composite materials. Paper presented at the 21st European Conference on Composite Materials, Nantes,
123. Das AaE, Martin and Davis, Bradley A and McKnight, Steven H and Williams, Christopher B and Case, Scott W and Bortner, Michael J (2021) Rheological investigation of nylon-carbon fiber composites fabricated using material extrusion-based additive manufacturing. *Polymer Composites* 42:6010 - 6024.
124. Millot CaF, Louise-Anne and Lame, Olivier and Sotta, Paul and Seguela, Roland (2015) Assessment of polyamide-6 crystallinity by DSC: Temperature dependence of the melting enthalpy. *Journal of Thermal Analysis and Calorimetry* 122:307 - 314.
125. Karsli NGaA, Ayse (2013) Tensile and thermomechanical properties of short carbon fiber reinforced polyamide 6 composites. *Composites Part B: Engineering* 51:270 - 275.
126. Dizon JRCaEJ, Alejandro H and Chen, Qiyi and Advincula, Rigoberto C (2018) Mechanical characterization of 3D-printed polymers. *Additive manufacturing* 20.
127. Kummert CaS, Hans-Joachim and Risse, Lena and Kullmer, Gunter (2021) Mechanical characterization and numerical modeling of laser-sintered TPE lattice structures. *Journal of Materials Research and Technology* 36:3182--3193.
128. Bates SRaF, Ian R and Trask, Richard S (2019) Compressive behaviour of 3D printed thermoplastic polyurethane honeycombs with graded densities. *Materials & Design*:130 - 142.

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