Advanced Structured Materials

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Additive Manufacturing Hybrid Processes for Composites Systems

Advanced Structured Materials

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João P. T. Pereira · Luis Miguel Oliveira
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Additive Manufacturing Hybrid Processes for Composites Systems

Springer

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Preface

This book is centred on the emergent technology of additive manufacturing (AM) and its application beyond the state of the art in fibre reinforcement thermoplastics (F RTP). It includes the development of a hybrid and integrated process that combines, into a single-step platform, additive and subtractive operations and allows CAD-to-Part productions with freeform shapes using long or continuous F RTP. Moreover, it addresses the following engineering issues:

- Design rules for hybrid additive manufacturing (hAM).
- Thermoplastics compounds for AM processing appropriate to high temperature and strength applications.
- Advanced extrusion heads and process concepts for AM of F RTP.
- Hybridization strategies regarding AM specifications (supports, slicing, filling, etc.) and material in-process properties (rheology, interfacial adhesion, layer consolidation, etc.).
- Software ecosystem for hAM design, pre-processing, process planning, emulating and multi-axis processing.
- Three-dimensional path generator for hAM based on a multi-objective optimization algorithm that matches the recent curved adaptive slicing method with a new transversal scheme.
- hAM parameters real-time monitoring and closed-loop control.
- Multi-parametric nondestructive testing (NDT) tool customized for F RTP AM parts.
- Sustainable manufacturing process validated by advanced LCA/LCC models.

Development of a constitutive model to predict the elasto-plastic behaviour of 3D-Printed thermoplastics using a meshless formulation. Covering the whole value chain, this next-generation technology is presented starting with part design, simulation and materials composition; then going through transformation stages; and finishing with the product evaluation and end-of-life studies.

Additive manufacturing (AM) is one of the most promising manufacturing technologies nowadays. Aeronautics and aerospace surrendered to the advantages of AM. The sale of AM professional industrial equipment increased regularly and is

expected the worldwide turnover on AM to quadruple between 2015 and 2020. Numbers are even more impressive for metal additive manufacturing showing the higher growth rates within the different available additive technologies.

Similarly, the composite materials industry assists a movement of great progression and penetration into new sectors, exploring the main advantages of high performance allied to lightweight designs. In order to reduce the labour intensive and manual operations typically associated with composite fabrication and to satisfy the needs for flexible automated composite processes, research is committed in investigating the feasibility of highly automated, integrated and reproductive processes based in principles such as extrusion, automated tape placement or automated fibre placement.

Several scientific initiatives are known to intent the implementation of composite manufacturing processes through AM, but these attempts collide with a number of shortcomings that limit their usability. Identified issues are related to the layer-by-layer approach of AM without reinforcements between layers (composites anisotropy that decreases through-thickness properties), the use of short fibres, the high roughness low-quality surface finishing, the added complexity of algorithms and motion paths. The poor performance of raw materials when directly used in AM processes without appropriate properties optimization and the dependence on experimental equipment based on available commercial machines (mainly SLS and FDM) without proper design for the processes to be implemented is also an important issue.

Still, there are insufficient or no exploration of certain required scientific fields starting by the balancing of properties when composing raw materials for AM processes. The fabrication of the fibre-reinforced composite filaments or laminates is required as a pre-step before AM processing, necessitating the need for materials to be composed and developed. A fibre-reinforced thermoplastic raw material for AM proposes should present an adequate rheological profile (viscosity), compatibility with the heat sources (softening/melting temperature ranges) and suitable mechanical behaviour in terms of ductility and flexibility avoiding brittleness. Using long or continuous fibres instead of short fibres is difficult to incorporate into processing and additional processing functions have to be merged like the fibre cutting systems.

The purpose of this book is to walk through the challenging scientific route to develop an advanced hybrid additive manufacturing process beyond the state of the art, which enables the lightweight design and manufacture of fibre-reinforced thermoplastics products under ecological friendly conditions.

The research developments presented in this book include a high potential manufacture process, the additive manufacturing hybridized with subtractive technologies and an innovative product and high-performance composite parts produced without moulds and with tailored properties. Both process and products hold a high potential of, in future, converting into tradable goods fostering the industry in particular and the economy in general.

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Acronyms

3D	Three Dimensions
ABS	Acrylonitrile butadiene styrene
AM	Additive manufacturing
AR	Aramid fibre
ASTM	American Society for Testing and Materials
ATC	Automatic trajectory control
ATRP	Atom transfer radical polymerization
BiCGStab	Bi-conjugated gradient stabilized
CAD	Computer-aided design
CAM	Computer-aided manufacturing
CBAM	Composite-based additive manufacturing
CCD	Charge-coupled device
CFD	Computational fluid dynamics
cFR	Continuous fibre reinforced
CFRP	Carbon fibre-reinforced polymer
cFRTP	Continuous fibre-reinforced thermoplastics
CIRP	College International pour la Recherche en Productique
CLFDM	Curved Layered Fused Deposition Modelling
CMOS	Complementary metal-oxide semiconductor
CNC	Computer numeric control
CNT	Carbon nanotube
DCB	Double cantilever beam
DED	Direct energy deposition
DFM	Design for manufacturing
DfMA	Design for additive manufacturing
DIC	Diagonal incomplete-Cholesky
DILU	Diagonal incomplete-LU
DIY	Do-it-yourself
DMA	Dynamic mechanical analysis
DMD	Direct metal deposition

DOE	Design of experiments
DSC	Differential scanning calorimetry
EBM	Electron beam melting
EC	Eddy current
EHS	Experimental hybrid system
EM	Electromagnetism-like
EPDs	Environmental report declarations
ER	Experimental rig
ERS	Experimental rig system
FBG	Fibre Bragg gratings
FDM	Fused deposition modelling
FEA	Finite element analysis
FEM	Finite element modelling
FFF	Fused filament fabrication
FGM	Functionally graded materials
FP	Fabry–Perot
FRTP	Fibre-reinforced thermoplastics
G-Code	G-code protocol (ISO/DIN 66025 standard)
GnP	Graphene nanoplatelets
GS	Granty speed
HVAC	Heating, ventilation and air conditioning
IPC	Institute of Polymers and Composites
IR	Infrared
ISO	International Organization for Standardization
L/C-FRTP	Long and continuous fibre-reinforced thermoplastics
LC	Life cycle
LCA	Life cycle assessment
LCC	Life cycle cost
LENS	Laser Engineering Net Shape
LM	Layer-by-layer manufacturing
LMD	Laser metal deposition
MAT	Medial axis transformation
MEMS	Microelectromechanical system
MFG	Multi-functional and graded features
MFI	Melt Flow Index
MIP	Mathematical integer programming
MIT	Massachusetts Institute of Technology
MMC	Metal matrix composites
MOPSO	Multi-objective particle swarm optimization
MQ	Multi-quadric
MT	Magnetic particle testing
MWCNT	Multi-walled carbon nanotube
NASA	National Aeronautics and Space Administration
NDT	Nondestructive testing
NiTi	Nitinol

NPV	Net present value
NSGA-II	Non-dominated sorting genetic algorithm
OCT	Optical coherent tomography
OEM	Original equipment manufacturer
OM	Origami mechanism
PA	Polyamide
PA 12	Polyamide 12
PA 66	Polyamide 66
PAEK	Polyaryletherketone
PAI	Polyamide-imide
PBF	Powder bed fusion
PC	Polycarbonate
PCG	Pre-conditioned conjugate gradient
PCL	Polycaprolactone
PCR	Product Category Rules
PDMS	Poly (dimethylsiloxane)
PE	Polyester
PEEK	Polyetheretherketone
PEI	Polyetherimide
PEKK	Poly Ether Ketone Ketone
PES	Polyethersulphone
PI	Polyimide
PLA	Polylactic acid
PMMA	Polymethyl methacrylate
POF	Polymeric optical fibres
PPPA	Phenylphosphonic acid
PPS	Polyphenylene sulphide
PPSU	Polyphenylsulphone
PS	Polystyrene
PT	Penetrant testing
PTFE	Polytetrafluorethylene
PVC	Polyvinyl chloride
R&D	Research and development
RA	Raster angle
RBF	Radial basis function
RP	Rapid prototype
RPIM	Radial point interpolation method
RTM	Resin transfer moulding
RVE	Representative volume element
SAFE	Semi-analytical finite element method
sCF	Short carbon fibres
sCFPA12	Short carbon fibre polyamide 12
SEM	Scanning electron microscopy
SETAC	Society for Environmental Toxicology and Chemistry
sFRTP	Short fibre-reinforced thermoplastics

SHS	Selective heat sintering
SLA	Stereolithography (Chap. 2)
S-LCA	Social life cycle assessment
SLE	Selective laser erosion
SLM	Selective laser melting
SLS	Selective laser sintering
SMA	Shape memory alloys
SMP	Shape memory polymers
SROI	Social return on investment
STL	Stereolithography (Chap. 1)
STL	Standard Tessellation Language (Chap. 2)
TGA	Thermogravimetric analysis
TMA	Thermomechanical analysis
TO	Topology optimization
TPO	Thermoplastic olefin
TW	Welding time
UAV	Unmanned aerial vehicle
UD	Unidirectional
UNEP	United Nations Environmental Programme
US	Ultrasound
UT	Ultrasonic tests
UTS	Ultimate tensile strength
VE	Vinylester
WLF	Williams–Landel–Ferry
Xc	Degree of crystallinity

Symbols and Units

ρ	Density
γ'	Shear rate
$\emptyset$	Diameter
$\varphi_j(\mathbf{x}_1)$	Interpolations functions (Chap. 6)
$\varphi^T(\mathbf{x}_1) = \{\varphi_1(\mathbf{x}_1), \varphi_2(\mathbf{x}_1), \dots, \varphi_n(\mathbf{x}_1)\}$	Interpolation function calculated at the interest point
σ	Stress tensor (Chap. 6)
$\sigma_Y _{\text{tensile}}, \sigma_Y _{\text{comp}}$	Yield stresses of the same material when subjected to tensile or compression loads, respectively (Chap. 6)
ε	Strain tensor (Chap. 6)
$\delta\varepsilon$	Virtual strain tensor (Chap. 6)
δu	Virtual displacement (Chap. 6)
$d\lambda$	Plastic strain multiplier (Chap. 6)
$d\varepsilon^e$	Infinitesimal elastic strain increments (Chap. 6)
$d\varepsilon^p$	Infinitesimal plastic strain increments (Chap. 6)
$d\sigma$	Stress increment (Chap. 6)
σ_{Y0}	Initial yield stress (Chap. 6)
λ	Relaxation time
τ	Deviatoric stress tensor
$\square$	E-step values
∇	Gradient
Ω	Domain (Chap. 6)
Γ	Boundary (Chap. 6)
$\mathbf{a}$	Normal vector (Chap. 6)
$a_i(\mathbf{x}_1), b_j(\mathbf{x}_1)$	Non-constant coefficients of $R_i(\mathbf{x}_1)$ and $p_j(\mathbf{x}_1)$, the polynomial basis, respectively, with m being the basis monomial number (Chap. 6)

a_T	Shift factor
A	Hardening parameter (Chap. 6)
$\mathbf{b}$	Body forces per unit volume (Chap. 6)
$\mathbf{B}$	Deformation matrix (Chap. 6)
BT	Building time
c, p	Shape parameters (Chap. 6)
c_1	WLF parameter
c_2	WLF parameter
cm	Centimetre
c_p	Heat capacity
$^{\circ}\text{C}$	Degree Celsius
$^{\circ}\text{C}/\text{min}$	Degree Celsius per minute
$\mathbf{D}_{\text{ep}}$	Elasto-plastic constitutive matrix given (Chap. 6)
$\Delta H_{\text{f}0}$	Standard enthalpy of formation
ΔH_{m}	Melting enthalpy
$\Delta\lambda_{\text{FBG}}$	FBG wavelength shift
$\Delta\lambda_{\text{FP}}$	FP wavelength shift
$\Delta\varepsilon$	Strain shift
ΔT	Temperature shift
Δt	Periods of time
f	Frequency
F, G and H	Material constants and characterize the anisotropy (Chap. 6)
g/10 min	Gram per ten minutes
$\mathbf{G}$	Matrix (Chap. 6)
G'	Storage modulus
G''	Loss modulus
GPa	Gigapascal
h	Hour
h	Convection coefficient
H'	Proportionality parameter used to update the yield stress based on strain hardening (Chap. 6)
$\mathbf{H}$	Blocks of diagonal matrixes, $\mathbf{H}^j$, containing the shape function of each node j of a given ‘influence domain’, with $\mathbf{H}^j = \varphi_j(\mathbf{x}_1)\mathbf{I}$ (Chap. 6)
H_{c}	Enthalpy of combustion
Hz	Hertz
i	Node calculated at the interest point $\mathbf{x}_1$ (Chap. 6)
I	Period

<i>I</i>	Identity matrix with dimension $[d \times d]$, where d is the number of degrees of freedom of the analysed problem (Chap. 6)
J	Joule
J/g	Joule per gram
<i>k</i>	Thermal conductivity
$k_{\text{FBG}\epsilon}$	Strain sensitivity
k_{FBGT}	Temperature sensitivity
kg	Kilogram
kg/h	Kilogram per hour
kN	Kilonewton
K_0	Initial stiffness calculated using the elastic constitutive matrix, <i>D</i> (Chap. 6)
<i>L</i>	Differential operator (Chap. 6)
L_0	Length of filament at the pre-set extruder speed
L_{arb}	Arbitrary Length
L_{real}	Real Length
L_{ref}	Reference mark on the filament
L_{rem}	Remaining Length
μm	Micrometre
<i>m</i>	Mass
m	Metre
m/min	Metre per minute
mg	Milligram
mL/min	Millilitre per minute
mm	Millimetre
mm/s	Millimetre per second
$\dot{M}_i$	Throughput
MPa	Megapascal
<i>n</i>	Power law exponent
<i>n</i>	Number of nodes within the ‘influence domain’ of x_1 (Chap. 6)
η^*	Complex viscosity
η_∞	Viscosity at the lower Newtonian plateau
η_0	Viscosity at the upper Newtonian plateau
N	Newton
<i>p</i>	Pressure
<i>P</i>	Polynomial matrix (Chap. 6)
Pa	Pascal
Pa s	Pascal second
<i>R</i>	Matrix (Chap. 6)
Ra	Surface roughness measure
$R_i(x_1)$	Radial basis function RBF (Chap. 6)
rpm	Revolution per minute

s	Second
SA	Support area measure
SE	Staircase effect measure
SA	Adapted support area measure
SE	Adapted staircase effect measure
t	Time
$\bar{t}$	Traction forces acting on the natural boundary Γ_1 (Chap. 6)
t_1, t_2	Initial and final time (Chap. 6)
T	Temperature
T_0	Reference temperature
T_c	Crystallization temperature
T_g	Glass transition temperature
T_m	Melting temperature
u	Velocity vector
$\mathbf{u}$	Displacement field (Chap. 6)
$\mathbf{u}$	Kinematically admissible displacement field (Chap. 6)
u_i	Value of the field variable in the node i and $\varphi_i(\mathbf{x}_1)$ (Chap. 6)
$\dot{\mathbf{u}}$	Velocity (Chap. 6)
$\mathbf{u}(\mathbf{x}_1) = \sum_{i=1}^n \varphi_i(\mathbf{x}_1) u_i$	Interpolation functions, being n the number of nodes inside the ‘influence domain’ of the interest point $\mathbf{x}_1$ (Chap. 6)
v_{ext}	Pre-set extruder speed
V	Volt
$\mathbf{x}_1$	Integration point (Chap. 6)
$\mathbf{x}_I, \mathbf{x}_J$	Interest points containing the same number of nodes (sixteen), but a different radius ($r_I \neq r_J$) (Chap. 6)
W	Relative water content
W/g	Watt per gram
W/m K	Watt per metre Kelvin

Chapter 7

Experimental Testing and Process Parametrization

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Abstract In this chapter, a characterization to the resulting FDM-printed parts and hybrid manufactured, essentially in terms of mechanical properties, is exposed and discussed. Once evaluated the polymeric thermal and/or mechanical response of the neat filaments, we were able to move forward with the mechanical characterization of the different printed parts developed under different methodologies and distinct purposes. After all this, the performance of hybrid trials in order to evaluate system functionalities, as well as hybridization strategies associated with the presence of AM supports during milling and layer adhesion on the top of a completely cured and machined surface was pursued. Additionally, it was also studied advanced pre-processing methods such as adaptive or curved slicing assessed in the experimental hybrid system with a special attention to the constraints of using long or continuous carbon fibres. All the experimental methodologies carried out and obtained results are described in detail herein.

Keywords Fusion deposition modelling · Fibre-reinforced thermoplastic polymers · Hybrid manufacturing · Mechanical properties

7.1 Introduction

FDM process depends on the local bonding of individual deposited layers, thus the degree of welding between the layers and raster as well as the consequent mesostructural-layered structures are very important to the mechanical performance of the resultant printed parts. Consequently, the most important weakness of FDM-printed parts focuses on the non-homogeneity of the structure, with subsequent

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bonds and voids between layers which depends on the deposition process [1]. Hence, mechanical properties of parts fabricated by FDM are mainly affected by the FDM production parameters, for instance, the printing temperature, printing speed, raster height and thickness, filling density, nozzle diameter, chamber temperature and raster angle [2]. The anisotropic characteristics of the fabricated components, associated with the layering and influenced by the directionally deposition process, were also found as important for the mechanical behaviour of the parts [3]. In addition, poor mechanical properties can also be related to the uncontrolled shrinkage during the cooling, bad levelling of the building plate, insignificant discontinuities in filament extrusion and imprecision of extruder motion [4]. Taking into account that the main objective of this study is to consider the usage of continuous carbon fibre-reinforced polymers FDM-printed parts in aeronautical, automobile and aerospace industries, mechanical evaluations are quite important to apply to the final FDM-printed/hybrid products. Therefore, some experimental approaches were investigated in order to understand the mechanical behaviour of the different printed parts produced with different materials using typical dogbone specimens, and further studied the intra- and inter-layer bonding by the DCB approach, taking into account the initiation and involved stress around the crack of all the DCB specimens.

In literature, it is being stated that the main advantages of using hybrid systems include surface finish, precision, repair, multi-material 3D printing and addition of complex features [5]. However, the implantation of hybrid machines still has some challenges to surpass, resultant from the interaction between additive and subtractive processes such as process planning, adjustment of process parameters, accessibility of machining tool and alternation sequence between additive and subtractive processes [6, 7]. Considering all of this, to validate the hybrid system, it will be required to follow some essential stages including the design of the part and geometry constraints; determination of additive and machining parameters; process sequence; material properties and specifications (see Fig. 7.1).

The design of the part is related to the dimensions, geometry and material information, which all a combined result in CAD model and then converted in a STL format file. Part geometry is an important factor for hybrid manufacturing since it will determine the accessibility of the machining tool to the surfaces or the need or not of additional support materials, for example, in the case of present free-form/curved surfaces or overhang features (Γ -shape object). Thus, geometry can imply the existence of a variety of macro-level features, namely holes, channels, narrow cavities, pockets, sharp edges not to mention perpendicular, parallel and sloped surfaces at different angles [8]. Thereafter, this geometry information contained on the CAD model is then used to generate the manufacturing code for the additive process as well as the offset geometry to be used during the subtractive process.

The input of material properties and specifications, for instance, elasticity modulus, ductility, viscosity, thermal conductivity, hardness, melting point temperature, glass transition temperature, microstructure and shrinkage characteristics, is also required since it directly affects manufacturing process parameters [9].

The selection of appropriate additive and machining process parameters is the next step to consider in the hybrid process validation and the most challenging one.

Fig. 7.1 Process planning for the hybrid system validation with the most important parameters to consider

Thereafter, for the additive manufacturing process, the parameters that can be taken into account are layer thickness, nozzle diameter, raster angle, printing speed, part building orientation, part location, bed temperature, printing temperature, envelope temperature, infill pattern, presence of support material, contour number (shell), filling percentage, retraction, material flow, raft/skin presence and many others. Concerning machining parameters, tool path, tool type, tool size, machining tool speed, feed speed, stepdown, sidestep, profile stepdown (for holes, pockets and vertical walls), corner radius, cooling/cleaning and tolerance [6, 10, 11] are the parameters to consider. All of these parameters will be essential to find out an optimal combination of the significant control parameter to achieve good surface finish, minimum process time and minimum material wastage cost effectively. The layer thickness is related to the lower surface finish due to the staircase effect, a known issue of additive process, meaning that to achieve a good surface finish and minimize the material waste, a slower deposition of thinner layers is the option, besides the increase of production time. In addition, layer thickness will best represent the part geometry in an efficient manner by enabling contours in each slice and consequently it will allow that part geometry be machined effectively [10]. It is also important to refer that machining too-thin material is undesirable once thin material can suffer from deflection under large cutting forces resulting in vibration, bad surface finish and dimension accuracy, chipping of the cutter teeth, breaking of materials or damage of machining tool [10]. Additionally, once subtractive manufacturing process applies cutting forces, there is a need of a fixing base, such as vacuum or a sacrificial material, in order to part withstand these forces. In the last case, the sacrificial base material

can also serve as a raising structure of the part, providing the possibility of machining surfaces without the collision of the subtractive tool with the build plate [12]. So, this factor must be considered as a process parameter as well.

Regarding process sequence, it is defined the sequence of the hybrid part manufacturing by a set of additive and machining operations to produce high-quality final parts correctly. This sequence can be settled as machining after deposition or interchangeable between deposition/machining and inspection. However, in general, the sequence of operations deeply depends on machine configurations and part geometry [10, 12]. After setting all the process parameters and planning, tool paths are generated for FDM and machining, respectively, resulting in the final part manufacturing. Lastly, the part is ready to be inspected identifying the dimensional tolerance, surface finish and mechanical properties such as tensile, flexural and Charpy impact. Consequently, at least three standard parts are produced to evaluate the repeatability of the hybrid manufacturing process for each evaluation test.

Reinforced additive manufacturing is an emerging relatively new field of the additive manufacturing, in which it is encompassed the development of fibre-reinforced thermoplastic (FRTTP) composites. Discontinuous and continuous fibres can be introduced in a fused deposition modelling process [13]. Continuous fibre-reinforced thermoplastics composites offer great advantages including excellent mechanical and chemical performance, design tailor ability, recycling and lightweight. Manufacturing complex functional and structural parts with continuous FRTTP is now possible by FDM [14]. Carbon, glass and aramid fibres are the most used in FDM although in this work the focus was mainly on carbon fibres. However, the combination of the fibres into the thermoplastic matrix with strength, good adhesion between fibres and polymer matrix, good consolidation and control of fibres orientation is still a challenge [15]. To print continuous fibre thermoplastic composites, there are the following approaches:

- (1) in-nozzle, direct impregnation of fibres with molten thermoplastic at the nozzle;
- (2) post-nozzle, to implement the continuous fibres after the polymer matrix passed the nozzle while the part is being manufactured (necessary double-nozzle for separate fibre and polymer filament);
- (3) pre-nozzle, extrusion of pre-impregnated fibres, which is the case of this project. In addition, cases 1 and 3 require a mechanical cutting, laser cutting or resistive heating in order to cut the fibres at the end of each layer or when it is not needed [12, 16].

By adding reinforcements to polymer matrixes, the rheology of the raw material increases thus introducing printability issues [17]. Thereupon, process planning for a hybrid system with both additive and subtractive processes for carbon-reinforced thermoplastics is much more complex than the ones for just additive or hybrid manufacturing of non-reinforced thermoplastics [12]. The presence of fibre reinforcement introduces other parameters to consider in the process planning and in the creation of new models for hybrid process. These are the composition of material, distribution, alignment of fibres, anisotropy of continuous FRTTP composite, amount of fibres, fibre layers, regions of the layers where fibres should be placed, to assure good quality

and improved performance [17]. Furthermore, some of the aforementioned process parameters also need to be appreciated due to their high importance in the presence of fibres, namely the raster angle, infill pattern, printing temperature, printing speed, layer thickness, nozzle diameter, milling tool speed, feed speed, stepdown and corner radius.

The deposition of carbon fibres is still limited to some infill patterns in order to the fibres not break due to tight angles or narrow areas during the deposition. This means the fibres can mainly be deposited in a concentric pattern, in which the shape is filled from outside inward in a spiral-shaped laydown. It implies that the part is oriented along the outer perimeter of the part, forming annular rings or through an isotropic pattern, which consists of parallel lines with areas without fibre and consequently filled by the thermoplastic non-reinforced before the subsequent layer is printed, resulting in a unidirectional anisotropic part [15, 18]. Another thing to take into account is that to avoid exposed fibres on the outer surface (harder to machine than the raw polymer), bottom and top layers need to be printed with 100% infill percentage as well as the outer periphery for each layer with unreinforced polymer [15]. There will also be the need of information regarding the layers/regions in which the fibres should be placed and the layers in which the subtractive tool should actuate. In the end, the process planning will provide the contours and infill for the additive, tool paths that generate sacrificial and excess material for subtractive process and the tool paths for the fibre placement regions/layers, as well as the setting of the other parameters.

Afterwards, the process planning for hybrid manufacturing of continuous FRTP can be applied thus producing the composite parts according to all the acquired information, regarding the optimal additive and subtractive process parameters, the processing strategies, the assessment of a whole set of tools custom-designed for hybrid AM, including design methods and software applications such as algorithms and paths. The second phase will consist in the inspection and characterization of these reinforced 3D-printed specimens. The same tests as used during the previous subchapters, such as dimensional tolerance, surface finish and mechanical properties are used in pursuance of a comparison of AM parts of polymer and composite materials, hybrid AM/SM parts of raw materials produced by commercial printers and/or experimental rigs.

Thus, this chapter involved the studies of polymer-based-printed parts, composite parts with multi-wall carbon nanotubes and carbon fibre by means of single FDM technology and further by hybrid manufacturing (FDM and milling) using adequate methodologies for each process.

7.2 Experimental

7.2.1 Material Filaments

The commercial and developed filaments used as raw materials for these preliminary tests and further printing of parts and consequent evaluation are stated in Table 7.1.

7.2.2 Material Properties

The evaluation of the neat materials before printing is very important to understand the behaviour of the materials in order to set the printing parameters and improve their printability. This information comprised by complex viscosity (η^*), glass transition temperature (T_g), melting temperature (T_m), crystallization temperature (T_c), melt flow index (MFI), strain at break (ε_b), ultimate tensile stress (σ_u) and tensile modulus (E), is discussed in more detail in Chap. 3 and further summarized in Table 7.2. Thermal tests will give information, for example, about glass transition temperature which is very important for FDM printing, since values of printing bed temperature slightly above glass transition of the materials are required for an optimal adhesion of the printed sample to the printing bed [19]. Additionally, through rheological tests, it is possible to obtain data about viscosity of the materials and it is known that viscosity plays a role in the coalescence/sintering of polymers, thus a high viscosity material will result in printed parts with low bonding quality [20].

Table 7.1 Material specifications

Material	Commercial reference	Reinforcement	Manufacturer	Printing temperature (°C)	Diameter (mm)
PLA	Smartfil PLA	–	SmartMaterials 3D	200–220	2.79 ± 0.01
PA 12	STYX-12	–	FormFutura	240–270	2.78 ± 0.02
PA 12	Nylon FX 256	–	Fillamentum (Parzlich s.r.o.)	235–250	1.68 ± 0.01
PA 6/69	Alloy 910	–	Taulman 3D	250–255	1.65 ± 0.05
PA 12	Nylon CF15	Short fibre 15% (w/w)	Fillamentum (Parzlich s.r.o.)	235–260	1.75 ± 0.01
PEEK/MWCNT	Victrex PEEK 450G	Nanotubes 2% (w/w) or 4% (w/w)	Victrex (filament produced)	400–430	1.70 ± 0.07 or 1.73 ± 0.06
PA66/MWCNT	Zytel E42A NC010	Nanotubes 4% (w/w)	Dupont (filament produced)	250–275	1.72 ± 0.11

Table 7.2 Material properties

Material	$\eta^* (\text{Pa s}) (\gamma = 100 \text{ s}^{-1})$	T_g (°C)	T_m (°C)	T_c (°C)	MFI (g/10 min)	ϵ_b (%)	σ_u (MPa)	E (GPa)
PLA Smartfil	173.1	60.2 ± 1.4	151.6 ± 1.3	115.8 ± 6.2	21.1 ± 0.6	7.2 ± 0.5	60.4 ± 3.3	1.8 ± 0.1
PA STYX-12	292.3	134.5 ± 0.4	245.7 ± 0.1	173.7 ± 0.1	25.0 ± 0.4	314.4 ± 34.0	49.4 ± 1.9	0.9 ± 0.1
Nylon FX256	132.7	52.7 ± 3.8	177.1 ± 0.5	144.3 ± 1.5	16.9 ± 0.9			
Alloy 910	325.4	59.0 ± 0.1	198.4 ± 0.1	151.0 ± 0.1	8.4 ± 0.1	109	246.4	224.3
Nylon CFI5	228	152.8 ± 12.1	178.5 ± 0.2	150.3 ± 0.1	14.6 ± 1.4			
PEEK/MWCNT 2%	–	146.5 ± 5.1	342.5 ± 0.1	293.5 ± 0.4	9.8 ± 0.0	36.0 ± 10.7	91.3 ± 2.1	2.6 ± 0.3
PEEK/MWCNT 4%	–	147.3 ± 1.3	342.2 ± 0.1	293.8 ± 0.6	2.5 ± 0.0	29.3 ± 6.6	98.8 ± 8.2	2.9 ± 0.2
PA66/MWCNT 4%	114.9	148.6 ± 0.2	264.4 ± 0.9	225.3 ± 2.3	9.3 ± 0.8	68.1 ± 6.2	57.6 ± 5.2	0.7 ± 0.1

7.2.3 Experimental Methodology for FDM Printing

The characterization of FDM printed parts aiming their structural integrity and inter-layer adhesion evaluation was achieved by using two types of specimens, such as dogbone and double cantilever beam (DCB). Two different methods, method A and method B were used to print specimens, since two research groups were involved in this work, which are explained below. The materials used by both research groups were also different. One used the commercial materials, Smartfil PLA and PA STYX-12 as well as the produced PEEK/MWCNT 2% or 4% composite filaments to print the specimens; the other studied 3D-printed parts using Nylon FX 256 and PA Alloy 910 as well as the produced composites PEEK/MWCNT 2% or 4% and PA 66/MWCNT 4%.

7.2.3.1 Tensile Testing Samples

Method A Methodology A was used to assess structural integrity of 3D-printed parts and this one consisted in the production of typical dogbone models designed by means of SolidWorks software, according to ASTM D638 (Fig. 7.2—left) when using the commercial filaments, Smartfil PLA and PA STYX-12 with diameters of ± 2.85 mm. However, other dogbone specimens were designed according to the ISO 527-2 Type 1BA (Fig. 7.2—right) to print PEEK/MWCNT 2% filament with ± 1.75 mm, considering that this CAD model is smaller and requires less amount of PEEK filament (expensive material with complex printability) than the ASTM D638.

The planning to print all these dogbone samples was made by recurrence to one L8 Design of experiments (DOE), resulting in eight different experiments, for each material, playing with printing parameters such as printing temperature (Print. T —A); bed temperature (Bed T —B); air gap, the space between paths (AG—D); the gantry speed (GS—C); the raster angle (RA—E). The resultant DOE to test Smartfil PLA, PA STYX-12 and PEEK/MWCNT 2% dogbone-printed parts is summarized in Table 7.3, respectively.

This set of printing specifications was settled to the *stl* file obtained from CAD software, SolidWorks, by using Ultimaker Cura software, with the purpose to slice

Fig. 7.2 Dogbone samples designed according to ASTM D638 (left) and to ISO 527-2 type 1BA (right)

Table 7.3 Values of the factors according to L8 orthogonal array for PLA/PA12/PEEK composite dogbone specimens

Exp.	Print. <i>T</i> (°C)	Bed <i>T</i> (°C)	AG (mm)	GS (mm/s)	RA (°)
	A	B	D	C	E
1	200/255/400	50/60/140	0.0000	20/20/10	−45°/−45°
2	220/265/410	50/60/140	−0.0254	20/20/10	−45°/45°
3	200/255/400	70/80/160	−0.0254	20/20/10	0°/90°
4	220/265/410	70/80/160	0.0000	20/20/10	0°/90°
5	200/255/400	50/60/140	0.0000	40/40/15	0°/90°
6	220/265/410	50/60/140	−0.0254	40/40/15	0°/90°
7	200/255/400	70/80/160	−0.0254	40/40/15	−45°/45°
8	220/265/410	70/80/160	0.0000	40/40/15	−45°/45°

these parts into layers according to each corresponding experiment specifications and creating a *gcode* file, which will be read by the printer. For instance, one model of a dogbone specimen (ASTM D638) in Cura software as well as some other constant printing parameters for using with commercial materials is demonstrated in Fig. 7.3 (top). Both commercial materials, Smartfil PLA and PA STYX-12 were printed in an FDM machine with double-nozzle of 0.4 mm, Ultimaker 3. This printer was used to fabricate a set of three samples for all the eight experiments for both materials. The other model of a dogbone sample designed according to ISO 527-2 type 1BA in Ultimaker Cura software is exhibited in Fig. 7.3 (bottom), including the other constant printing parameters to print with PEEK/MWCNT 2%. This composite filament was printed using an FDM printer prepared to print PEEK in a closed chamber, namely the APIUM P155 printer. Three samples for all the eight experiments were also produced. Upon completion, the printed tensile specimens were subjected to a uniaxial load, which is provided by an Instron 5969 with a 5 kN load cell, at 23 °C. The machine displaced the specimens at a constant crosshead speed of 5 mm/min.

Method B Experiment number two, performed by the other research group, consisted in the analysis of the tensile properties of printed samples and by changing the parts tray orientation and internal pattern, a comparison of the results defining the effects of these conditions in the properties was made. For this experiment, the

Fig. 7.3 Slicing pattern of ASTM D638 (top) and ISO 527-2 type 1BA (bottom) dogbone specimens

Fig. 7.4 **a** XY $\pm 45^\circ$ infill, **b** XZ $\pm 45^\circ$ infill, **c** XY concentric and **d** XY concentric at 0°

materials considered were PA6/69 alloy 910 from taulman 3D, PA12 FX256 and PA12 CF15 CARBON from Fillamentum.

Figure 7.4 shows the four types of samples produced. Figure 7.4a presents a flat sample (XY) with an internal infill of $\pm 45^\circ$. Figure 7.4b the internal pattern is the same as the previous; however, this sample is printed on edge (XZ). In addition, the last two types of samples Fig. 7.4(c and d) were printed horizontally, with a concentric pattern that disposes the filament beads along the sample instead of a cross pattern as found in the first type of sample. The main difference between the concentric samples is that the entry point of the extruder in the first one (c) is located in the beginning of the neck, while the second concentric sample extruder entry is made at 0° in the gripping area. For this test, dogbone ISO 527 geometry was considered, and an 100% infill percentage was used all samples.

7.2.3.2 DCB Samples

Method A Concerning the analysis of the adhesion between layers based on a fracture mechanics approach, DCB parts with a pre-crack set at the interface of the layers were also printed and designed by using SolidWorks software, according to the test method of Aliheidari et al. [21] for commercial polymers (Smartfil PLA and PA STYX-12). To study the inter- and intra-layer adhesion of the composite filament PEEK/MWCNT 4%, the DCB parts were designed according to a different model. Considering that PEEK is an expensive material, the amount of produced filament

was limited and mainly because PEEK printing is not very easy, since this material has high retraction, causing warpage and thus reducing adhesion between layers and the printing bed. The final design to test DCB made of PEEK/MWCNT 4% was achieved after pre-tests and consequent verification of the resulting parts until obtain a drawing with resultant good printed DCB part.

Equally to tensile testing, L8 DOE matrixes for evaluating the same printing parameters were also prepared for each material, Smartfil PLA, PA STYX-12 and PEEK/MWCNT 4%, taking into account their processing temperatures and printing constraints.

For all the DCB experiments, a 100% solid linear pattern infill with a double perimeter was used to print the longitudinal layers. Each planner layer was constituted of continuous parallel lines to the printer *x*-axis (raster angle of 90°) resulting in specimens with all the layers oriented in the longitudinal direction of printed DCB. The samples were printed with sacrificial support structures between the DCB arms already designed in the CAD model. Then, these bridge supports used to create the pre-crack in the DCB samples were removed using a sharp blade after the printing. The DCB CAD models for commercial polymers and for the PEEK composite are presented in Fig. 7.5.

As well as dogbone specimens, *gcode files* were obtained by Cura software. Examples of one-sliced DCB specimen for the different models are illustrated in Fig. 7.6.

A batch of three sequentially printed samples of all the eight experiments for both specimens of Smartfil PLA and PA STYX-12 were printed in an FDM machine

Fig. 7.5 DCB CAD models for commodity polymers (left) and for composite filament (right)

Fig. 7.6 DCB specimens sliced by Cura software and the values of the constant printing parameters for commercial polymers (left) and for PEEK composite material (right)

Table 7.4 Values of the factors according to L8 orthogonal array for PLA/PA12/PEEK composite DCB specimens

Exp.	Print. <i>T</i> (°C)	Bed <i>T</i> (°C)	AG (mm)	GS (mm/s)
	A	B	D	C
1	200/255/435	50/60/150	0.0000	20/20/10
2	220/265/445	50/60/150	−0.0254	20/20/10
3	200/255/435	70/80/160	−0.0254	20/20/10
4	220/265/445	70/80/160	0.0000	20/20/10
5	200/255/435	50/60/150	0.0000	40/40/15
6	220/265/445	50/60/150	−0.0254	40/40/15
7	200/255/435	70/80/160	−0.0254	40/40/15
8	220/265/445	70/80/160	0.0000	40/40/15

Ultimaker 3 printer with double-nozzle of 0.4 mm. Regarding PEEK/MWCNT 4% DCB specimens, the printer used was the APIUM P155. Upon completion, the tensile testing of all DCB specimens was executed applying the same method as for dogbone tests. We use an Instron 5969 with a 5 kN load cell for Smartfil PLA and PA STYX-12 and a 1 kN load cell for PEEK/MWCNT 4% samples, at 23 °C and with a constant crosshead speed of 5 mm/min and 2 mm/min, respectively. To these DCB samples, a relatively rigid steel wire (0.5 mm or 0.1 mm diameter) was run through the loading holes or strategically placed just around the arms (in case of PEEK specimens) of DCB to facilitate loading. The resulting load was used to calculate maximum load and crack’s initiation. Table 7.4 exposes the resultant DOE for Smartfil PLA, PA STYX-12 and composite PEEK/MWCNT 4% DCB samples.

7.3 Results and Discussion

The results and discussion compile the results of the two mechanical characterization tests (i.e. tensile testing and DCB approach) on the fully dense 3D-printed parts produced by single FDM process under the different methodologies and using the materials discussed previously.

7.3.1 Tensile Testing Samples

The characterization of 3D dogbone-printed parts involved the printing of samples in several orientations and with distinct internal structures, with the goal of understanding the influence of these features on mechanical properties.

Regarding mechanical properties of PLA Smartfil dogbone samples printed with method A, Fig. 7.7 shows the representative stress–strain curves obtained for the

Fig. 7.7 Smartfil PLA dogbone experiments representative stress–strain diagram

different experiments. Experiments 1, 2, 7 and 8 besides the elastic behaviour, they additionally exhibit plastic behaviour. For these experiments, in the plastic region, their stress linearly increased with the applied strain, reaching to a maximum and then their stress slightly relaxed and remained almost unchanged until the fracture.

Comparatively, Experiments 3, 4, 5 and 6 only showed elastic behaviour, being brittle, since after reaching the maximum stress, the samples for these experiments suddenly suffered the final failure. The PLA Smartfil filament undergoes the same performance. In addition, ultimate tensile stress (UTS), strain at break and tensile modulus were calculated from the stress–strain curves and displayed in Fig. 7.7. Practically, all the experiments showed great resistance to deformation compared to the filament, except experiments 5 and 7, which presented very low tensile strength values, 17–20 MPa, against the over 40 MPa of the other experiments. The most similar in terms of tensile strength to the filament is the Experiment 4. When it comes to matters of strain at break, Experiments 1, 2, 7 and 8 have better values meaning that after maximum load they support higher elongations mainly due to their ductile nature. However, the values of the printed parts cannot reach the filament values for this property. Moreover, Experiments 1, 6 and 8 demonstrated greater tensile modulus even when compared to the filament, specially 1 and 8. This means that they are slightly stiffer than the filament and the other experiments.

Unlike the ductile neat PA filament (STYX-12), all the 8 PA STYX-12 dogbone specimens, printed with Methodology A, behaved as brittle, considering that after yield, they suffered the fracture (see Fig. 7.8). By Fig. 7.9, it is feasible to affirm that all the experiments have similar strengths, with values within 30 MPa, except Experiment 4 that stands out for the lower strength, 21 MPa. Notwithstanding, printed PA STYX-12 dogbone parts are worse than the filament (49.5 MPa). Towards elongation at break values, experiments are once again very much alike, not extending over more than 8%, but Experiments 2 and 3 followed by Experiments 5 and 6 are slightly better than the others. Notably, filament has a significant elongation under tension,

Fig. 7.8 Smartfil PLA dogbones tensile results (left), strain at break (middle) and tensile modulus (right)

Fig. 7.9 PA-STYX-12 dogbones representative stress–strain diagram

compared to the printed parts essentially because it is ductile whereas the dogbone samples are brittle.

Furthermore, tensile modulus is better in Experiment 5 (1 GPa) and poor in Experiment 4 and 2 (around 0.7 GPa), Fig. 7.10. However, Experiments (5, 6, 7 and 8) printed with a printing speed of 40 mm/s are quite constant and greater than the ones

Fig. 7.10 PA STYX-12 dogbone experiments tensile strength (left) and elongation at break (middle) and tensile modulus (right)

printed at 20 mm/s (1, 2, 3 and 4). It is noteworthy that most of the experiments exhibit a higher tensile modulus in relation to filament tensile modulus.

Nylon FX 256 dogbone samples were produced by using Method B, thus printed in the same $XY \pm 45^\circ$ orientation, $XZ \pm 45^\circ$ and in addition, the XY concentric with the entry at 0° (XYCE). Through Fig. 7.13, it is possible to observe that the increased mechanical behaviour found in XZ for the Alloy 910 is not reflected for this new material. However, by comparing the stress–strain curves in Fig. 7.11, it is possible to understand that, both XYCE and XZ present almost similar characteristics. Figure 7.12 presents the average values obtained for the testing of these three types of samples. Considering test loads, the values are similar in XY and XZ, however, the XZ value presents a higher value of deviation. Young’s Modulus values are higher in XY samples and the inferior value is found in the XZ sample in contrast with PA Alloy 910 material.

Maximum stress values are also higher in XY sample and even in both other orientations. The strain at break parameter shows a higher value again in XY, then XZ and finally, XYCE. This analysis shows that the best behaviour is found in the XY sample, contradicting the behaviour in the previous material. In addition, it is found that there is a similarity in behaviour in XZ and XYCE. This can be explained by the fact that, even though one is printed on edge and the other flat on the tray, there

Fig. 7.11 PA 12 FX256 stress strain curves XY (left), XZ (right) and XYCE (bottom)

is a similarity in the number of longitudinal lines (along the sample—solicitation direction) creating this resemblance in behaviour.

Following the Methodology B, used as well for Nylon FX 256, tensile tests were also performed for several orientations. Figure 7.13 presents the stress–strain curves

Fig. 7.12 PA 12 FX256 XY versus XZ versus XYCE mechanical properties

Fig. 7.13 PA Alloy 910 stress–strain curves XZ (left) and XY (right)

Fig. 7.14 PA Alloy 910 XY versus XZ mechanical properties

for XZ and XY samples of the PA Alloy 910. From Fig. 7.13, it is noticeable that the on edge (XZ) samples present a better performance than the flat (XY). Both sets of curves present a slight waviness when the plasticity area is reached, which may indicate that inter-layer detachment has occurred.

Figure 7.14 presents the average values obtained for this test. Maximum load values were higher for the XZ samples, and in concordance, the maximum stress was also superior as can be seen in the previous figure. XZ sample Young modulus value surpassed double the one of the XY-printed part. The strain at break values was similar for both orientations. These results clearly indicate that the better performance was found in the XZ samples. Since the MFI values for this material is low compared to the other polyamides and the filamentary test shows high values, the adhesion between the layers might not be the best. This explains the lower values when the pattern is +45 (XY). Since the other sample is built on edge, there are more layers/perimeters deposited in the solicitation direction prevailing the material strength.

One representative stress-strain curve for each of the eight experiments of PEEK/MWCNT 2% composite dogbone specimens is shown in Fig. 7.15. These dogbone specimens were printed by using Method A. All the eight experiments showed a brittle behaviour, since after yield the rupture occurs, as it happens with all the PA STYX-12 specimens.

Through Fig. 7.16, it is possible to observe that some experiments for PEEK/MWCNT 2% specimens are more resistant to deformation than others, such

Fig. 7.15 PEEK/MWCNT 2% dogbone specimens representative stress–strain diagram

Fig. 7.16 PEEK/MWCNT 2% dogbone specimens tensile stress (left), strain at break (middle) and tensile modulus (left)

as the Experiments 1, 2, 3 and 8, with values around 40 MPa, but essentially the second with 56 MPa. However, the experiments UTS results do not approach the filament UTS result (90 MPa), which means that they are weaker. Regarding strain at break results, practically, all the experiments have similar elongations under stress, except Experiments 2 and 4, which extend 2%, when the other only elongates almost 1%. Nevertheless, composite filament on its own can extend until 30% being able to support longer elongations after maximum load. These experiments are all more brittle than the other materials.

Comparing the tensile modulus values of the eight experiments prepared with the composite filament PEEK/MWCNT 2%, the results are within 1.5 and 2.6 GPa (Fig. 7.15—right). Although, as it happens with the other mechanical properties, some experiments are better than others, such as the Experiment 1 that incredibly almost equals the tensile modulus of its raw composite filament as well as the Experiments 2 and 7, with values around 2.1 GPa. These two experiments (2 and 7) were printed with the same air gap (-0.0254) and raster angle ($-45^{\circ}/45^{\circ}$). Additionally, it

is feasible to estimate that all the experiments printed with the raster angle of $0^\circ/90^\circ$ (3, 4, 5 and 6) present lower tensile modulus than the others printed with $-45^\circ/45^\circ$, so we can point out that raster angle really influences the mechanical properties. Experiment 8 probably had other aspects influencing these results, like possibly a poorer adhesion to printing bed or a different cooling process, but these are just suspicions. It is also important to mention that PEEK composite has a higher melting point (342.5°C —Chap. 3), so it may trigger excessive thermal stress (non-equally distributed inter and intra-layers) and thermal cracks [2].

Morphological Analysis After all the 8 Smartfil PLA dogbone specimens being tensile tested under Methodology A, the resulted fracture zones were observed in a digital microscope Leica DMS1000 which combines an optical zoom system with an advanced digital camera. Portions of the specimens were cut with the help of a little saw, just to fit in the microscope. Figure 7.17 demonstrates the fracture's surfaces (YZ plane) of all experiments. In Experiment 1 is possible to see the diagonal layers with no sign of voids. Experiment 2 seemingly exhibit a great union between paths and layers mainly due to the higher printing temperature compared to Experiment 1, that has the same bed temperature, raster angle and printing speed. In relation to Experiment 3, the layers $0^\circ/90^\circ$ are very visible as well as the consequent presence of voids. However, Experiment 4 with equal bed temperature, raster angle ($0^\circ/90^\circ$) and printing speed as Experiment 3 presents the best intra- and inter-layer adhesion as the voids are quite inexistent which means that the morphology is significantly affected by the print temperature. Experiment 6 also shows good morphology, having the same print temperature, but a different speed, bed temperature (higher) and air gap (closer). So, in this case, the air gap and mostly bed temperature have a big role.

The fracture zones (YZ plane) of PA STYX-12 dogbone specimens are exposed in Fig. 7.18. From micrographs of Experiments 1 and 2, hardly recognize the layers, paths or voids existence, once the raster angles are $-45^\circ/45^\circ$. The colour of the polyamide does not help in this recognition. Nevertheless, the layers and paths are very visible in Experiments 3 and 4 micrographs, but apparently the layers in Experiment 4 are closer and tighter than in Experiment 3 (greater adhesion), essentially due

Fig. 7.17 Smartfil PLA dogbone fracture zones morphological analysis, Experiments 1–8

Fig. 7.18 PA STYX-12 dogbone fracture zones morphology analysis, Experiments 1–8

to the higher printing temperature they tended to approach, diminishing the voids. Experiments 5 and 6 also printed with the same raster angle ($0^\circ/90^\circ$) as specimens 3 and 4 and for that reason the micrographs are similar with prominent layers and paths. Finally, micrographs of Experiments 7 and 8 when compared with micrographs from specimens 1 and 2 (same raster angle) allow us to see the layers and the crossing of the paths that seem to present bigger adhesion.

The resultant fracture zones (YZ plane) of each experiment' PEEK/MWCNT 2% dogbones are exhibited in Fig. 7.19. Part of some layers can be well recognized by this method, being possible to see the crystallinity of the filaments, the raster's direction and the presence of voids that seem to be just a few. Experiments 1 and 2 have raster angles of $-45^\circ/45^\circ$ and that is possible to confirm by the pictures, as well as the $0^\circ/90^\circ$ direction of the Experiments 3 and 4. They have almost no visible voids and Experiment 3 presents layers that are more compact. Through the micrographs of Experiments 5 and 6, it is not possible to confirm so well the raster directions as the specimens 3 and 4 considering that they were all printed with the same raster angle ($0^\circ/90^\circ$). However, it is noteworthy that these experiments have really close and compact layers. By comparison with Experiments 1 and 2 that present the same raster

Fig. 7.19 PEEK/MWCNT 2% dogbone fracture zones morphology analysis, Experiments 1–8

Fig. 7.20 Smartfil PLA DCB experiments representative load–displacement diagram

angle ($-45^{\circ}/45^{\circ}$), Experiments 7 and 8 micrographs allow us to see that Experiment 7 seems to have bigger adhesion than experiment 1 and 8, maybe because it was printed in a hotter bed and with an inferior air gap.

7.3.2 DCB Samples

Concerning the 3D printed Smartfil PLA DCB samples, Fig. 7.20 depicts the representative load-displacement curves. Some nonlinearity can be observed at the beginning of most of the experiments that might be related to the settling of the samples. Despite that, in all of the cases, the load increased relatively linearly with an increase in the crosshead displacement, until the crack growth started, except experiment 1 that firstly showed a relaxation and consequent elongation after yield and crack's initiation.

The maximum load reached is about 150 N by experiment 8, the best result and around 95 N for the worst case, experiment 1 (see Fig. 7.21—left). These results are very good and comparable to the ones obtained by Aliheidari et al. [21] with ABS material. Relatively to crack initiation, the best result belongs to experiment 2 with 4.2 mm of displacement, followed by experiment 1, 3, 7 and 8, presenting crack initiations after around 3.5 mm. The weaker experiments are 4, 5 and 6 because their crack started after nearly 3 mm of displacement. All these results can be attributed to better layer-to-layer fusion as well as the decreased size of inter-layer voids due to printing temperature, speed and angle.

Figure 7.22 shows representative load-displacement curves for tensile of 3D printed PA STYX-12 DCB specimens. As observed in PLA specimen's curves, at the beginning of all these PA DCB's curves there is a nonlinearity zone probably

Fig. 7.21 Smartfil PLA DCB samples maximum load comparison (left) and crack initiation (right)

Fig. 7.22 PA STYX-12 DCB experiment representative load–displacement diagram

connected to the stabilization of the specimens. All experiments consequently had their load increased by crosshead displacement's increment. Then the crack started and eventually grew, in all experiments.

It is possible to state through Fig. 7.23 that experiments 6 and 8 supported higher loads presenting maximum load values of 74–75 N, experiments 2 and 4 are the next in line with medium values between 66 and 57 N. The weakest experiments seem to be 1 and 5 (supporting around 30 N), and then 3 and 7 (with about 40 N). These results might be associated with the printing temperature, once the strongest specimens (6, 8, 2 and 4) were printed at 265 °C while the worst (1, 5, 3 and 7) were printed at 255 °C. Bed temperature also has influence because when correlating the experiments printed at the same temperature, between experiments 2 and 4 along with 6 and 8, experiments 2 and 6 (bed T : 60 °C) are somewhat greater than 4 and 8

Fig. 7.23 PA STYX-12 DCB specimens' maximum load comparison (left) and crack initiation (right)

(bed T : 80 °C). Contrarily to this, when we compare experiments 1 and 3 as well as 5 and 7, the results are better for the experiments printed at a bed settled at 80 °C (exp. 3 and 7). Towards crack initiation, experiments 1, 2, 6 and 8 appeared to crack after longer crosshead displacements. The cracks initiate after 9.4 mm, 9.1 mm, 8.2 mm, 7 mm, 6.4 mm, 5.7 mm, 4.6 mm and 4 mm for experiments 6, 2, 8, 1, 7, 4, 3 and 5, respectively. Therefore, the weaker specimens are experiments 3 and 5. These results are mainly influenced by air gap and bed temperature.

Representative load-displacement curves of each tested PEEK/MWCNT 4% DCB specimens are exhibited in Fig. 7.24. In all the experiments with the crosshead displacement, the load increases until a certain point where the crack starts to occur, growing and totally separating.

Fig. 7.24 PEEK/MWCNT 4% DCB experiment representative load–displacement diagram

Fig. 7.25 PEEK/MWCNT 4% DCB specimens' maximum load comparison (left) and crack initiation (right)

Analysing Fig. 7.25, it is possible to observe that some experiments undertake more load than others, such as experiments 2, 4, 6, 7, with values around 4 N and mainly experiment 8, with a maximum load value of 5 N. Experiments 2, 3 and 5 are the weakest, exhibiting maximum loads before crack initiation around 2 and 3 N. As it happens with PA STYX-12 specimens, printing temperature can be indicated as the most influent parameter to these results since the strongest specimens were printed at 445 °C, except specimen from experiment 7. Higher bed temperature and higher gantry speed can also influence once comparing specimens with the same printing temperature and air gap, namely experiments 1 and 7, being experiment 7 better than experiment 1. In terms of crack initiation results, experiment 8 stands out from the other experiments, since presents a value around 2.5 mm and the others are around 0.75 and 1.4 mm meaning that its fracture occurs after a longer crosshead displacement. The experiments that seem to present a poor adhesion are experiments 1 and 3. These results are affected by all the printing parameters.

Morphological analysis Some morphological micrographs were taken to the pre-crack zone of PEEK/MWCNT 4% DCB specimens, the area that was used to evaluate the fracture and consequently the adhesion between layers. Through Fig. 7.26, it is possible to distinguish well the deposited layers and the zone where is supposed to occur the fracture between layers. In experiments 1, 4, 5, 7 and 8, the layers of the pre-crack zone seem to be well-adhered to one another. On the contrary, experiments 2, 3 and 6, there is a visible small lack of adhesion between layers in the pre-crack area.

Fig. 7.26 PEEK/MWCNT 4% DCB pre-crack zones morphology analysis, Experiments 1–8

7.4 Conclusions

In general, as expected, the dogbone specimens exhibited brittle behaviour comparing to the raw material filaments with their ductile nature.

Regarding the Smartfil PLA dogbone specimens, Experiments 1, 2, 7 and 8 resulted in ductile dogbone specimens, also able to support higher elongations. Experiments 1 and 8 are stiffer than the neat filament. By morphological analysis, it is possible to state that all the printing parameters, specially printing temperature and bed temperature really influence the existence of voids and the adhesion between paths and layers. The best results were obtained with a printing temperature of 220 °C and bed temperature of 70 °C.

In the matter of PA STYX-12 printed parts mechanical characterization, all dogbone specimens behaved as brittle unlike their raw PA filament. In terms of ultimate tensile stress and strain at break, all the specimens had similar results, but still worse than the filament source by itself. Contrarily, some experiments exhibited higher values of tensile modulus than the own filament. From the fracture zone pictures of the dogbone specimens, it was hard to recognize the layers, paths or voids existence, probably due to the colour of the polyamide which did not help in this recognition.

Method B revealed that both materials present different behaviour and no relationship between printing parameter and performance. This means that, part properties are affected by the part orientation but also by the combination with the material physical and rheological properties. MFI shows that, PA 6/69 is a less fluid material comparing to the PA 12 FX256, and in consequence, a harder material to print, which leads to a difficult bonding process between extruded lines, prevailing the mechanical properties of the material and not structural. This can explain the difference in behaviour in XZ for both materials.

In terms of the results for the mechanical tests of FDM dogbone-printed parts produced with PEEK/MWCNT 2%, some experiments had strength than others, mainly Experiment 1 and all of them presented similar elongations under tension, highlighting Experiments 2 and 4. These composite dogbone specimens showed to be more brittle in comparison to the other tested materials. Experiments 1, 2 and 7

also presented higher tensile modulus. In addition, it can be estimated that the raster angle affected the tensile modulus results. By their morphological analysis, it was possible to observe the crystallinity of the filaments, the raster's direction and the presence of voids that seem to be just a few.

In contrast to the tensile specimens, Smartfil PLA DCB specimens behaved like brittle-printed parts, except Experiment 1 and the weaker were Experiments 4, 5 and 6. Concerning the DCB parts of PA STYX-12, Experiments 3 and 5 are weaker than the others are, so their adhesion is poorer, mostly because of the printing parameters such as air gap and bed temperature. Therefore, it has been proven that the printing temperatures have a big impact in the void's presence and in the intra-layer adhesion. With respect to PEEK/MWCNT 4% DCB parts, Experiments 2, 4, 6, 7 and 8 seem to have better adhesion than the other experiments. In fact, they support bigger loads and the crack occurs later, specially Experiment 8, which can indicate that high values of printing temperature, bed temperature and gantry speed as well as null air gap create parts better adhesion. In addition, by analysing the morphology of the PEEK/MWCNT 4% specimens pre-crack area, it was possible to observe in detail the adhesion/proximity of the layers in that zone. Experiments 1, 4, 5, 7 and 8 as opposed to Experiments 2, 3 and 6 do not exhibit a visible lack of adhesion between the layers of the fracture zone.

In conclusion, both tests, tensile testing and DCB approach, proved to be important to evaluate the mechanical properties of parts fabricated by FDM technique. It was possible to define a relationship between the printing parameters and the mechanical properties as well as the inter- and intra-layer of all the different material specimens. All the tested printing elements showed great influence in the mechanical performance of the experiments mainly the printing temperature. However, a deeper investigation is required in pursuance of refuting and confirming the obtained results for both tests. Although, in general, all the results were within the values reported in the literature.

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