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Microstructural characterization of additively manufactured multi-directional preforms and composites via X-ray micro-computed tomography

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Additive manufacturing features "direct" and "layer-by-layer" fabrication and has significantly facilitated the microstructural design and fabrication of a wide range of highly complex parts. To enable the application of additive manufacturing in major industries and composites, it is necessary to evaluate the microstructural features of additively manufactured parts. Among the various advanced characterization techniques, X-ray micro-computed tomography (μ-CT) showed unique advantages as a high resolution, nondestructive and 3D visualization and measurement technique for material characterization. In the research reported in this article, we have fabricated an array of multi-directional preforms and composites and have characterized their microstructural features via X-ray μ-CT. First, a solid specimen as well as 3D orthogonal and 3D braided preforms have been fabricated using fused filament fabrication (FFF) and inspected with X-ray μ-CT. Then, the fabricated preforms have been infused with silicone matrix and the multi-directional preforms and composites have been tested in compression at different strain levels, to reveal their damage evolution under compressive loading. The preliminary effort made in this research demonstrates the feasibility of characterizing microstructure of additively manufactured parts via X-ray μ-CT technique and enables an investigation of the microstructural features and damage evolution of multi-directional preforms and composites.

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1. Introduction

In contrast to conventional manufacturing methods, additive manufacturing features "direct" and "layer-by-layer" fabrication [1], which greatly facilitates the design of parts with intricate structure [2–16], provides a versatile technique for small-batch fabrication of customized objects [17–22] and, thus, enables a wide range of engineering and biomedical applications [23–29]. In order to explore opportunities for additive manufacturing of multi-directional preforms for composites, Quan et al. recently demonstrated the fabrication of an array of preforms via fused filament fabrication (FFF) [30] and reported their results on microstructural design and additive manufacturing and characterization of short carbon fiber/acrylonitrile-butadiene-styrene (ABS) 3D orthogonal preforms and composites [31]. Although high model-to-part fidelity was observed, the microstructure and interfaces of additively manufactured preforms and composites were not characterized.

Some efforts have been made by researchers in dimensional and structural characterization of additively manufactured parts. For example, Pei et al. [32] and Galantucci et al. [33] used visual inspection and optical microscopy to evaluate part dimensional accuracy. Kumar and Kumar [34] utilized contact surface profilometer to measure part surface roughness. Dimitrov et al. [35] and Polzin et al. [36] adopted a coordinate measuring machine to determine part dimensional precision. However, the above approaches have mainly focused on dimensional and external evaluation of additively manufactured parts.

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Unlike techniques reviewed above, X-ray micro-computed tomography (μ-CT), a nondestructive 3D visualization and measurement technique for material characterization, is capable to provide not only exterior features of parts at high resolution, but also their internal structural information [37]. Principally, the X-ray μ-CT technique is based on measurement of the attenuation of X-ray photons. When incident photons pass through an object, they will be attenuated and the attenuation is critically dependent on the composition (physical density) of the object. If there are more than one composition existing in the object, attenuation contrast will occur. In X-ray μ-CT measurement, the attenuation contrast is recorded at multiple angles. Then, the recorded contrast data are processed and converted into a stack of slices to reconstruct the 3D volume [38,39]. Recently, μ-CT has been adopted to characterize the microstructure of additively manufactured parts [40–43]. du Plessis et al. [40,41] evaluated the dimensional accuracy and internal features (such as macro- and micro-porosity) of 3D printed poly lactic acid (PLA) and ABS parts as well as laser sintered parts. Tammas-Williams [42] characterized the size, volume fraction and spatial distribution of pores in selective electron beam melting samples. Guessasma et al. [43] studied the influence of printing orientation on specimen porosity and observed weak inter-line adhesion in fusion deposition modelling (FDM) parts.

In this article, we report the characterization of microstructure of additively manufactured multi-directional preforms and their matrix-infused composites via X-ray μ-CT. First, three types of pores were identified in as-fabricated solid pure ABS specimen. Next, pure ABS and short carbon fiber/ABS multi-directional preforms were fabricated using FFF and the influence of preform structural parameters (i.e., yarn diameter and inter-yarn spacing) on structural clarity of fabricated preforms has been studied. Then, the as-fabricated multi-directional preforms were infused with silicone matrix and the influence of preform structure clarity on matrix infusion was checked. Lastly, compressive performances of the multi-directional preforms and their composites have been examined under different strain levels, to reveal their damage evolution.

2. Experimental

2.1. Additive manufacturing

Four types of specimens as shown in Table 1 were additively manufactured using fused filament fabrication approach [30,31]. The structural parameters were depicted in Fig. 1. All specimens had the dimensions of 12.7 mm (length) × 12.7 mm (width) × 25.4 mm (height). For the fabrication of pure ABS specimens, a *Print® SE Plus* 3D printer (*Stratasys Inc*., Minnesota, U.S.) was adopted with a nominal nozzle diameter of 0.254 mm (0.01 inch); ABS thermoplastic wires were utilized as feedstock, and a water-soluble filament was used as support material. For the fabrication of short carbon fiber/ABS specimens, an *nScrypt® 3Dn-300* (*nScrypt Inc*., Florida, U.S.) micro-dispensing platform was utilized with the processing parameters given in Table 2; a short carbon fiber/ABS wire *3DXMaxTM CFR* (*3DXTech Inc*., Michigan, U.S.) was adopted as feedstock. Unlike the *Print® SE Plus* 3D printer, an *nScrypt® 3Dn-300* micro-dispensing platform does not use support material. It should be noted that the term “yarn” referred to here for 3D multi-directional preforms is borrowed from textile engineering, where a preform is an assembly of individual textile yarns. However, in additive manufacturing, a preform is printed layer-by-layer; and within each layer, a single “yarn” is consisted of a series of printing “lines”.

Table 1
Specimen types and their structural parameters.

Solid specimen type (Pure ABS)			
Specimen type#	Length L (mm)	Width W (mm)	Height H (mm)
1	12.7	12.7	25.4
3D orthogonal preform types (Pure ABS)			
Specimen type#	Yarn diameter D_y (mm)	Inter-yarn spacing G_y (mm)	Yarn number N_1, N_2, N_3
1	1.0	0.10	5, 5, 11
2	1.2	0.12	4, 4, 9
3	1.5	0.15	3, 3, 7
4	2.0	0.20	2, 2, 5
3D braided preform types (Pure ABS)			
Specimen type#	Yarn diameter D_y (mm)		Braiding angle θ
1	0.9		Arctan 5
2	1.5		Arctan 5
3	1.5		Arctan 7
3D orthogonal preform type (short carbon fiber/ABS)			
Specimen type#	Yarn diameter D_y (mm)	Inter-yarn spacing G_y (mm)	Yarn number N_1, N_2, N_3
1	1.0	0.10	5, 5, 11

2.2. Matrix infusion

One specimen of each type of 3D preform in Table 1 was infused with a *Dow Corning 184 silicone elastomer* system (*Dow Corning Inc*., Michigan, U.S.) to fabricate 3D preform based composites. The 3D preforms were immersed in matrix and vacuum was applied for 5 h to ensure thorough matrix infusion. Then, matrix curing was conducted in an oven (100 °C, 2.5 h).

2.3. Compression tests of preforms and composites

The selected preforms (Type 1 and 3 of pure ABS 3D orthogonal preforms, Type 2 of pure ABS 3D braided preforms, and short carbon fiber/ABS 3D orthogonal preforms) and their silicone matrix-infused composites were tested in compression, in accordance with ASTM standard D695-10, using an *Instron TTC 45D* testing system (*Instru-Met Corporation*, New Jersey, U.S.). The nominal specimen dimensions (height × width × thickness) were 25.4 mm (1.0 inch) × 12.7 mm (0.5 inch) × 12.7 mm (0.5 inch), and the compression speed was 1.00 mm/min. Two specimens were prepared for each type of specimen: one was tested continuously up to a high strain level (>50%); the other one was tested repeatedly by loading and unloading at different strain levels as listed in Table 3. For repeated tests, the specimen was scanned with X-ray μ-CT after each loading-unloading cycle, to observe the damage evolution. Compared to 3D orthogonal preforms and composites, 3D braided ones showed higher ductility and, thus, were compressed up to higher strain levels (i.e., 35% and 45%).

2.4. X-ray micro-computed tomography

Imaging of additively manufactured specimens was conducted using a *SkyScan 1172* X-ray μ-CT system (*Bruker Corp*., Billerica, Massachusetts, U.S.). The X-ray power was 10 W with a voltage of 40 KV and a current of 250 μA. The image size of 4000 × 2672 pixels was selected. The resolutions in the three dimensions were the same and closely related to the maximum vertical cross-section size of sample. For as-printed and as-infused specimens, the scanning resolution was around 5 μm/pixel and the image acquisition time was about 3 h per specimen. For post-compression

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