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Damage in extrusion additive manufactured parts: effect of environment and cyclic loading

Amirpasha Moetazedian[a], Andrew Gleadall[a]*, Elisa Mele[b], Vadim V. Silberschmidt[a]

[a]*Wolfson School of Mechanical, Electrical and Manufacturing Engineering, Loughborough University, Loughborough, LE11 3TU, UK*
[b]*Department of Materials, Loughborough University, Loughborough, Leicestershire LE11 3TU, UK*

Abstract

With a general cautious attitude regarding the anisotropic properties of upright 3D-printed parts, there is a lack of fundamental understanding of behavior of 3D-printed polymers under cyclic loading condition, which is more representative of real-life applications including biomedical ones. To this date, no study considered the multi cyclic testing of an interface bond between layers. So, to examine this, specially designed specimens were developed with the filament widths varied as printed normal to the direction of printing in order to produce dogbone specimens for cyclic tensile testing with two key aims: (i) to characterise the accumulation of damage adjacent extruded filaments; and (ii) to investigate the effect of testing environment on the degradation of mechanical properties. It was found that cyclic loading of 3D-printed polylactide (PLA) specimens resulted in the accumulation of plastic strain, lowering the ultimate strength and strain at break by less than 10% compared to non-cyclic testing. The strength of specimens tested submerged at 37°C were 50% lower than that of tested in air. PLA was plasticised by water, which increased the strain at fracture by approximately 40%. Incremental loading of specimens increased the energy dissipation as approaching the yield point of the material for both testing environments. Meanwhile, damage estimation from the slope of unloading curves indicated that plasticised polymer accumulated 18.1% more damage at lower strain compared to that of tested in air. Specimens tested in air failed in a brittle manner, while, submerged cyclic testing resulted in an intermediate brittle-ductile fracture by formation of apparent shear lips and striation along the fracture plane. The results of this study provide new understanding of the material behavior under condition close to *in-vivo* environment.

Keywords: Additive manufacturing; Medical polymers; Interface; Cyclic loading; Submerged testing; Damage

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* Corresponding author. Tel.: +44 (0)-1509-227578.
E-mail address: a.gleadall@lboro.ac.uk

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1. Introduction

Biodegradable polymers, such as polylactide (PLA), are actively researched and used for biomedical applications including scaffolds, screws and fixation plates (Gleadall et al. 2018). Material extrusion additive manufacturing (MEAM) is currently considered as one of the most commonly used additive manufacturing process due to its ease of use, low cost, meanwhile allowing to achieve intricate and customised parts compared to conventional methods (Gleadall et al. 2018; Safai et al. 2019). Multiple studies (Ahn et al. 2003; Song et al. 2017) highlighted the anisotropic properties of 3D-printed parts, with regard to the direction of the extruded filament (i.e. interface between layers); therefore, there is a widespread caution regarding the use of parts fabricated upright in load-bearing applications.

As MEAM systems are now more widely employed, the 3D-printed polymeric parts should withstand both environmental and mechanical conditions that may occur during their in-service use. This can be crucial since polymers are more susceptible to the changes in the surrounding environmental conditions, such as temperature, moisture and type of loading, compared to metals and ceramics (Lawrence et al. 2001; Wu et al. 2006). A previous study (Moetazedian et al. 2020) showed that, under non-cyclic uniaxial tension, a 50% reduction in the ultimate tensile strength (UTS) was achieved once 3D-printed PLA was tested submerged at physiological temperature (37°C) compared to that of tested in air.

However, the 3D-printed polymers for biomedical applications are more likely to be subjected to sub-critical repetitive loading/unloading conditions (i.e. below their yield point) once implanted in a human body (Safai et al. 2019), resulting in accumulation of damage and eventually failure of the implant earlier than expected. Previous studies (Safai et al. 2019; Afrose et al. 2016; Senatov et al. 2016) considered the fatigue life of polymeric parts mainly during compression, which may require millions cycles until failure. No study was found in literature that considered the damage evolution during multi cyclic loading of 3D-printed PLA and there was research into the submerged cyclic testing at 37°C.

This is the first study, which directly characterise the damage evolution for interface between 3D-printed layers which has never been studied before. Additionally, this study aims to provide a direct comparison between tests in air and submerged at 37°C in terms of the damage evolution of the polymer once subjected to incremental cyclic loading.

2. Methodology

Natural PLA (3DXTECH® branded NatureWorks® polylactide 4043D, Sigma Aldrich) was used to produce novel filament-scale micro-tensile specimens using a RepRap x400 machine. A custom G-CODE (series of commands) was utilised to control the movement of the print head and extrusion rate. The nozzle's temperature was set at 210°C to deposit four single filaments in the form of square (Fig. 1a) in XY plane (along the print bed). This process was repeated for 225 layers as print bed moves down in Z direction. The height and width of the hollow box were 45mm x 45 mm respectively; which was printed without any support material. The filament widths varied from 0.75 mm (shoulder region) to 0.5 mm (gauge region) to produce dogbone specimens suitable for tensile testing, while the layer height was kept at 0.2 mm. To characterise the manufacturing-induced mechanical behaviour of 3D-printed specimens, the extruded filaments were orientated normal to the direction of load (Fig. 1b). A customised rig and blades were used to cut the corners of the box to yield four walls (Fig. 1b). The walls were then cut using another customised rig with blades into 5-mm wide specimens to provide twenty-four specimens per box. The dogbone specimens were tested under two main testing conditions: (i) dry specimens tested in air at room temperature, 20°C (denoted as 'air'), and (ii) hydrated specimens in phosphate buffer saline (PBS) for 2 days (to become saturated) and then tested submerged in PBS at physiological conditions; 37°C, which was denoted as 'submerged'. The specimens ($n = 3$) were subjected to incremental (5, 10, 20, 30, 40, 50, 60, 70 and 80% of UTS) cyclic loading at strain rate of 4.0×10^{4} s^{-1} (displacement of 0.5 $mm.min^{-1}$) using a universal mechanical testing machine (Instron 5944, USA) equipped with a temperature-controlled bath (Instron BioPlus, Instron, USA) and a 2 kN load cell. The level of energy dissipation and damage were calculated from the hysteresis curves. A Zeiss Primotech microscope was used to analyse the fracture surface of mechanically tested specimens. The average strength was calculated for each specimen using the average pre-fracture bond width (measured by microscopy) and specimen width (measured by caliper) to calculate the cross-sectional area.

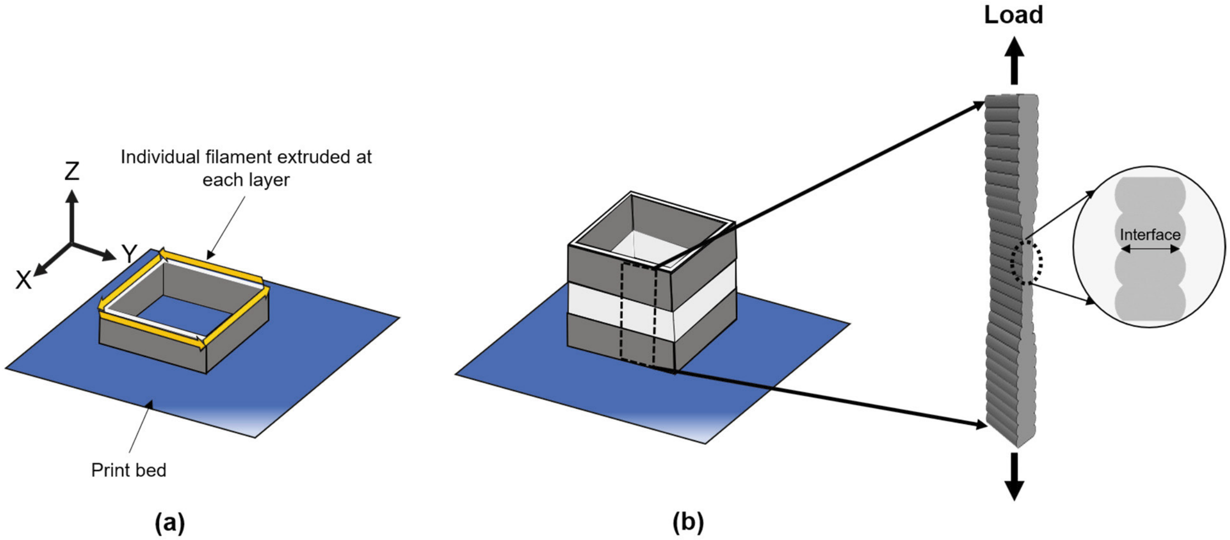

Fig. 1. (a) Schematic diagram indicating deposition of a stack of single extruded filament to produce the hollow box. (b) Dogbone specimens produced by extrusion of filament normal to print bed which were cut into 5-mm wide specimens to test cyclic tensile properties of the interface between layers.

3. Results and discussion

The results and discussion are separated into two sections: the first section presents the mechanical properties obtained from the incremental cyclic tensile loading to compare the damage evolution under two main testing environments and possible underlying mechanisms. The second section relates the fracture features observed with the optical microscopy to the mechanical-testing results to provide useful information about the material's fracture behavior.

3.1. Mechanical characterisation

PLA is considered as a viscoelastic material, which exhibits different features compared to purely elastic materials. As a result, during deformation of PLA, it can dissipate some of this energy, this process is called "energy dissipation" and quantified by difference between the areas below the loading and unloading curves. The loading criterion selected in this study allowed to capture the mechanical behavior of the material both below and beyond the yield point as well as calculating the energy dissipation of the 3D printed PLA. The first interesting point was a relatively low difference between non-cyclic and cyclic loading for air and submerged environments -only 7.2 and 3.1%-, respectively, despite the accumulation of damage during cyclic loading. In addition, the cyclic loading reduced the strain at break of specimens by only 2.3 and 8.2% for air and submerged, respectively. At the same time, the cyclic loading curve for specimens tested in air showed some degree of cyclic softening. While, the curve for submerged testing followed the master curve (non-cyclic). This feature could not be identified unless the characterisation was carried out under condition close to *in-vivo*. All this information can be useful for designing new polymeric medical implants, which are more likely to be subjected to cyclic loading. Although, the type of loading had little effect, the testing environment greatly influenced the properties of the PLA. Specimens tested submerged (Fig. 2b) had a significantly lower strength but higher strain at break: by approximately 55% and 40%, respectively, compared to those in air (Fig. 2a). The results agreed with the previous study (Moetazedian et al. 2020), which showed the similar trend for the non-cyclic tensile testing of the 3D-printed PLA under the same testing condition.

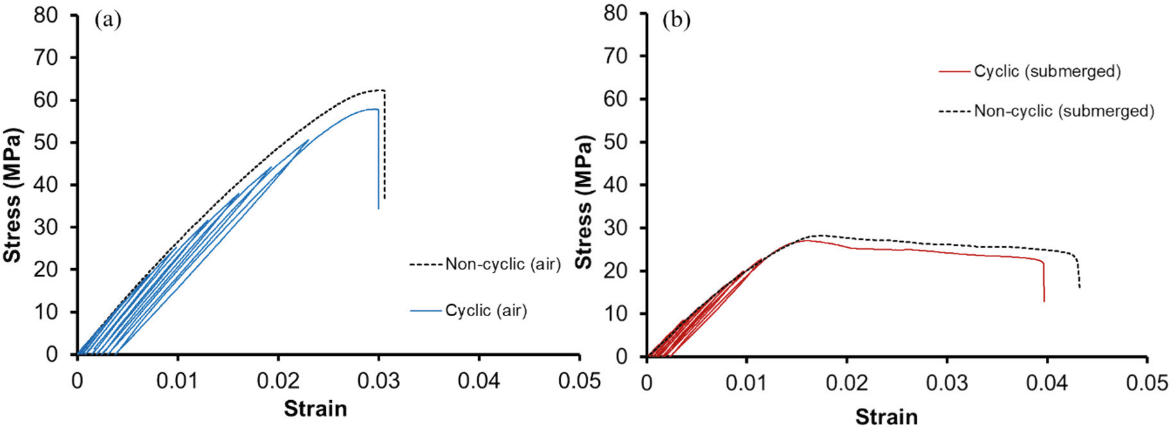

Fig. 2. Quasi-static incremental cyclic and non-cyclic loading stress-strain curves of specimens tested: dry in air at room temperature (a) and hydrated and submerged in PBS at 37°C (b).

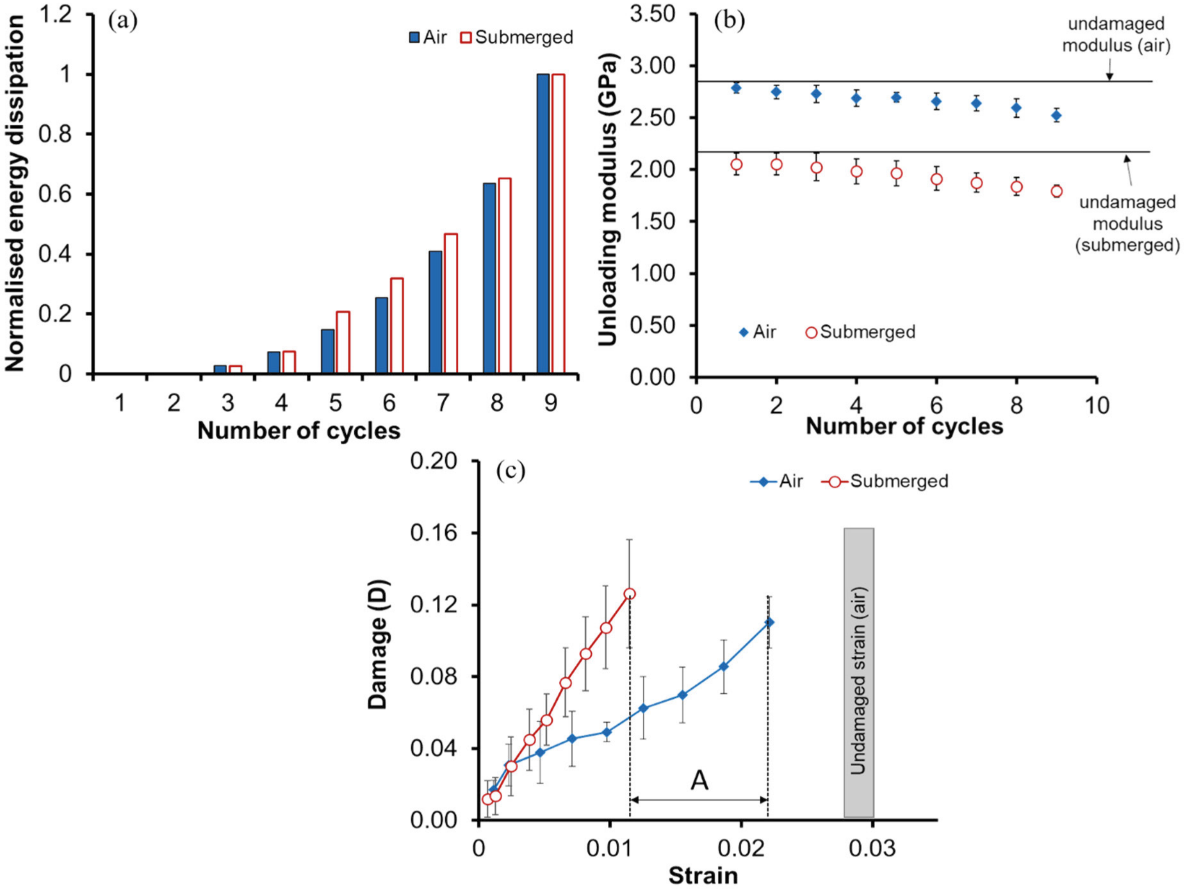

Fig. 3. (a) Normalised energy dissipation values for first 9 cycles for specimens tested in air and submerged. (b) Degradation of unloading modulus became apparent after 5th cycle. (c) Damage level was 18% greater for specimens tested submerged compared to that of tested in air. Region A represents the overestimation of strain capacity when PLA was tested in air.

Further analysis of the results demonstrated that the energy-dissipation values increased as the number of cycles grew as expected, due to accumulation of damage throughout the structure. The data for both testing environments were normalized to the 9th cycle (80% UTS) to allow a direct comparison between the two conditions (Fig. 3a). The calculated energy dissipated was similar in the beginning until the 4th cycle for both testing environments. After the 5th cycle, it appeared that presence of water molecules during cyclic loading resulted in higher relative energy dissipation per cycle compared to those tested in air. This could happen due to the plasticisation effect of water molecules during testing, which could interact with polymer chains of the deformed material (Moetazedian et al. 2020). This interaction could also explain a significant reduction of modulus values for submerged tests (Fig. 3b). The degradation of modulus during the unloading stage indicated its gradual decrease, as more damage accumulated (Fig. 3b).

Damage evolution was estimated from the degradation of modulus using the well-known relation of continuum damage mechanics ($D = 1- E_D/E_0$), where E_D is the residual modulus of the damaged material and E_0 is the modulus of undamaged material. The damage was plotted as a function of measured strain values for each cycle (Fig. 3c). From the obtained results, the damage occurred earlier for submerged condition than in air, despite both having a similar value at a low strain values (e.g. up to strain of 0.0024). Meanwhile, for higher measured strain, the damage continued to accumulate to a greater extent for submerged condition (≈ 0.13) compared to that in air (≈ 0.11). The rate of damage accumulation for submerged condition was higher than that in air; thus, it reached the highest value at much lower strain (0.012) compared to dry condition (0.023) (Fig. 3c). This means that for testing in air there is an overestimation of strain capacity for biomedical application as labelled by “region A” in Fig. 3c. Once more the obtained results highlighted the adverse effect of water that should be considered for the correct assessment of mechanical properties for biomedical applications, otherwise, these properties can be overestimated.

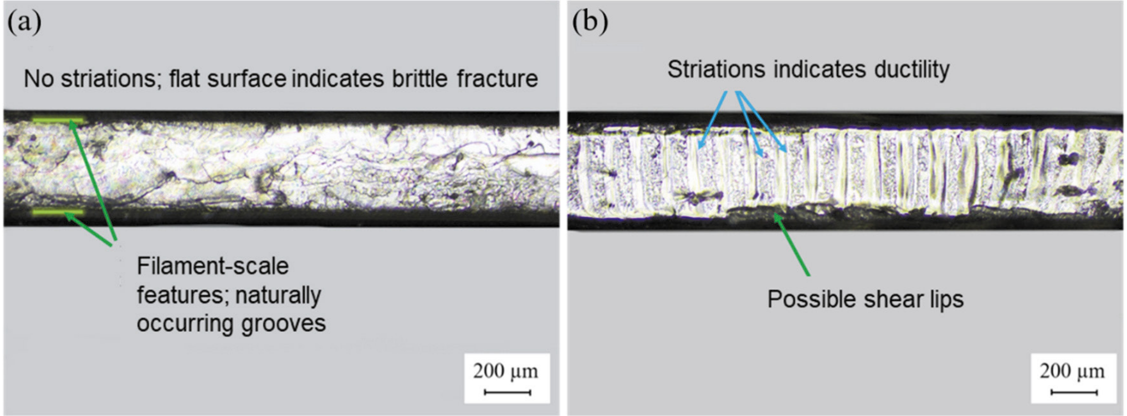

Fig. 4. Optical micrographs of fracture surface of specimens tested dry in air (a) and tested submerged (b). Multiple striations and raised edges were found for submerged condition compared to unsubmerged testing indicating higher ductility.

3.2. Fractography

Analysis of the fracture surface allowed further understanding of the failure of 3D-printed PLA subjected to cyclic loading. The optical micrograph of the specimen tested in air (Fig. 4a) was significantly different to that of the submerged (Fig. 4b). A flat and smooth fracture surface without raised edges (filaments orientated along the direction of fracture) was characteristic for brittle fracture in air (Moetazedian et al. 2020). In contrast, the fracture surface of specimens tested submerged at 37°C resulted in formation of series of striations along the fracture surface as indicated by arrows in Fig. 4b. These striations can be the indication of crack arrest due to the stored energy insufficient to allow the propagation of crack during testing. The appearance of striations was associated with an increase in the strain at fracture (Fig. 2b), due to the plasticising effect of water molecules and higher temperature. Excess material at the edges of the fracture surfaces was also identified for specimens tested submerged (Fig. 4b), which could be sign of shear-lip formation and micro-plasticity of the material. The presence of this feature emphasised once more the significant effect of interaction of water molecules with polymer chains.

4. Conclusions

Filament-scale micro-tensile specimens were fabricated from PLA to assess the mechanical properties of the interface between extruded filaments under incremental cyclic loading. The results showed that despite accumulation of plastic strain under cyclic loading, there was only a 10% difference in UTS and strain at break between cyclic and non-cyclic loading. Considering the testing environment replicating *in-vivo* conditions indicated that the presence of water and heat during mechanical testing significantly changed the mechanical behavior of the material. The possible explanation could be plasticisation of PLA by water molecules and higher temperature since the strain at fracture was increased by approximately 40%, while the strength was halved. At the same time, the specimens tested submerged showed to dissipate comparable or even more energy during the cyclic deformation, which resulted in accumulation of 18.1% more damage at low strain compared to that for the tests in air. Fractography analysis also confirmed the changes in the fracture characteristic from brittle to intermediate brittle-ductile fracture due to formation of shear lip and striations along the fracture surface. The results clearly showed the importance of correct assessment of biomedical polymers, subjected to repetitive loading during their service.

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