FATIGUE CHARACTERISATION AND MONITORING IN 3D PRINTED SHORT FIBRES REINFORCED POLYAMIDE

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Abstract: *3D printed composites are rapidly gaining a growing interest in several industrial sectors. However, for their widespread adoption, it is necessary to characterise their fatigue response. In this work, several 3D printed Onyx specimens were tested under tension-tension fatigue load with two main objectives. The first one was the assessment of different monitoring techniques for the fatigue damage evolution in the Onyx specimens. It was observed that the strain calculations from the machine crosshead displacement led to better results than strains from digital image correlation or extensometers. The second objective was the characterisation of the fatigue response of the Onyx specimens: SEM fractography was able to identify the fatigue damage initiation and propagation regions. Moreover, modulus and temperature trends were observed to assume a non-monotonic trend, which is unusual for composites. It was thus suggested that these quantities can be potential indicators of the fatigue damage in the specimens.*

Keywords: Short fibres; fatigue; 3D printed; damage monitoring; experimental mechanics.

1. Introduction

Fused filament fabrication (FFF), or fused deposition modelling, is one of the most popular additive manufacturing techniques for polymeric composites. Academic and industrial interest for FFF has recently increased, since this technology allows cheap manufacturing of light parts with complex geometries. Several studies have investigated the effects of the manufacturing parameters on the static behaviour of 3D printed composites [1]. Among these parameters, printing orientation has a high influence on the stiffness and strength, as it coincides with the fibre orientation [1].

A few works have also investigated the fatigue response of 3D printed polymers [2] and continuous fibre reinforced polymers [3,4]. However, there is a striking lack of works investigating the fatigue performance of 3D printed short fibres reinforced composites. The topic is of great relevance considering the fast spread of materials like Onyx, a micro-carbon fibres reinforced polyamide developed by Markforged [5]. Moreover, Onyx and similar materials are also frequently used as matrix for continuous fibre reinforced polymers [3]: this makes the characterisation and understanding of their fatigue response even more critical, as it may affect also the higher performing continuous fibres composites.

Given the importance of the topic, this work aims to evaluate different fatigue testing and fatigue damage monitoring techniques in 3D printed short fibres reinforced polymers. The work was performed on Onyx specimens, whose fatigue behaviour was therefore also studied.

Evaluating the possible monitoring techniques and establishing a proper one for 3D printed short fibres polymers will set the basis for future fatigue characterisations of 3D printed polymers and composites.

2. Materials and methods

2.1 Specimens fabrication

All test specimens were additively manufactured in Onyx with a FFF printer, namely the Markforged Onyx Pro. The layer height and filament width were 0.1 mm and 0.4 mm, respectively.

Since it was not possible to print unidirectional specimens, the specimens were fabricated by alternating layers with a 0° orientation and with a 90° orientation with respect to the longitudinal axis. Moreover, the printer also imposed a contour shell made of two concentric Onyx rings around the specimens. Overall, the final specimens' dimensions were 140 mm x 30 mm x 3.2 mm.

Onyx end tabs were printed to be bonded to the specimens. These tabs had dimensions of 40 mm x 30 mm x 2 mm. A commercially available bicomponent epoxy glue by Pattex was used for their bonding. As a result, the gauge length of each specimen was 60 mm.

A fatigue test campaign requires the knowledge of the static properties of the analysed specimens. A static characterisation was performed by the authors in another study [6], but the data useful for this study are reported in Table 1.

Table 1 : Static properties of the Onyx specimens with a 0°/90° orientation from the static characterisation of [6]

Young's modulus (MPa)	Ultimate tensile stress (MPa)	Strain at failure (%)
967	23.7	11.8

2.2 Fatigue tests and monitoring techniques

Load-controlled cyclic tests were performed according to the ASTM D3479 standard [7]. The machine used was an MTS Landmark Stigma, equipped with a 100 kN load cell. The cyclic frequency was set to 2 Hz. The machine was set to acquire data for the first 100 cycles, then for every 10 cycles between 10^2 and 10^3 cycles, for every 100 cycles between 10^3 and 10^4 cycles and so on. Specimens that did not fail within one million cycles were considered runouts. R-ratio of minimum to maximum load was R=0.1. Crosshead displacement was available for all tested specimens.

To evaluate different monitoring techniques, the specimens were equipped with several instrumentations. All the adopted techniques per each specimen are reported in Table 2, while each technique is described below.

An MTS extensometer with a 20 mm gauge length was used for some specimens (EXT in Table 2). The acquisition rate was 200 Hz, so it was not necessary to alter the load history of the specimens. Extensometer data were acquired in the same cycles acquired by the testing machine.

Digital Image Correlation (DIC in Table 2 and the rest of the paper) was also considered for some specimens. In particular, one of the surfaces of these specimens was spray painted with a white paint and then a black speckle was applied. The image acquisition was performed with a Canon EOS-RP. The camera was mounted on a tripod and a remote control was used to acquire the pictures: this was done not to modify the field of view between the acquisitions. The load history of these specimens was adjusted to allow image acquisition. In particular, static ramps were introduced, ranging from 0 kN to the maximum applied level of fatigue load, at an applied speed of 5 mm/min. Load was then held, and a picture was taken at this maximum load. The load was then set back to zero and cycling could continue. These ramps were performed:

- every 20 cycles in the first 200 cycles;
- every 100 cycles from cycle 200 to cycle 10^3;
- every 200 cycles from cycle 10^3 to cycle 10^4;
- every 250 cycles from cycle 10^4 to cycle $7{\cdot}10^4$;
- every 10^3 cycles from cycle $7{\cdot}10^4$ to cycle 10^5;
- every $2{\cdot}10^3$ cycles from cycle 10^5 until the end.

GOM Correlate was used to post-process the images.

Finally, some specimens were also equipped with a thermocouple (TEMP in Table 2) pressed with a clip on the analysed specimens. This device measured the temperature fluctuations in the specimens due to the cyclic loading. The tests were performed in a controlled environment, so that environmental conditions did not played a significant role in these measurements.

Table 2: Monitoring techniques adopted for each tested specimen.

Specimen number	Load level (% of UTS)	Monitoring techniques	Cycles to failure
1	85%	EXT	246
2	85%	DIC	265
3	70%	EXT	234074
4	70%	EXT	26740
5	70%	EXT	220572
6	70%	EXT + TEMP	160081
7	70%	DIC	241334
8	60%	EXT + TEMP	Runout

2.3 Microscopy

The fracture surface of four specimens, two EXT and two DIC, was observed on a Zeiss EVO 50XVP Scanning Electron Microscope (SEM). Due to their length, the specimens were cut to fit into the SEM. A minimum distance of 10 mm between the cut and the fracture surface was maintained to avoid altering the fracture features. A gold coating was applied prior to the scanning. Moreover, a ZEISS Stereo Discovery V12 microscope was also used in the analysis.

3. Results and discussion

3.1 Fatigue response of Onyx specimens

Figure 1 shows the results of the specimens tested with the different monitoring techniques. The figure is meant as an overview of the tests results: since the specimens featured different measuring equipment (extensometers, DIC, etc.), this curve cannot be considered as an indication of the durability of the material.

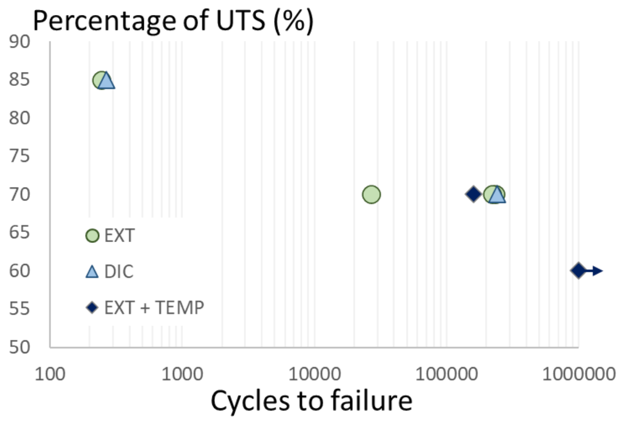

Figure 1: Results of the different fatigue tests

Nevertheless, valuable information was obtained on the fatigue failure behaviour of the tested specimens. In particular, the SEM fractography of the analysed specimens revealed the presence of two different micro-mechanical behaviour in the same fracture surface: a micro-ductile and a micro-brittle failure behaviour. The entire cross-section of specimen 7 is shown in Figure 2a, as an example, with close-ups on micro-brittle and micro-ductile region shown respectively in Figure 2b and Figure 2c. As shown, the micro-ductile fractured surface is identifiable by the presence of high plastic deformation, with strong presence of polymeric “filaments”; on the other hand, the micro-brittle fractured surface presents significantly lower deformation. Using DIC, the strain distribution along the fracture surface at 99.4% of the specimen’s life was obtained and reported in Figure 2a. As shown, the region with the higher strain corresponds to the micro-ductile region in the fracture surface. It seems likely that, in this area, fatigue damage nucleated and propagated, as it is the case for injection moulded composites [9]. Fatigue damage thus seems to occur in a ductile fashion. Once the remaining cross section can no longer sustain the applied load, static failure occurs abruptly: this high-strain-rate failure causes the micro-brittleness in the remaining region. This behaviour was also observed under static load conditions [6].

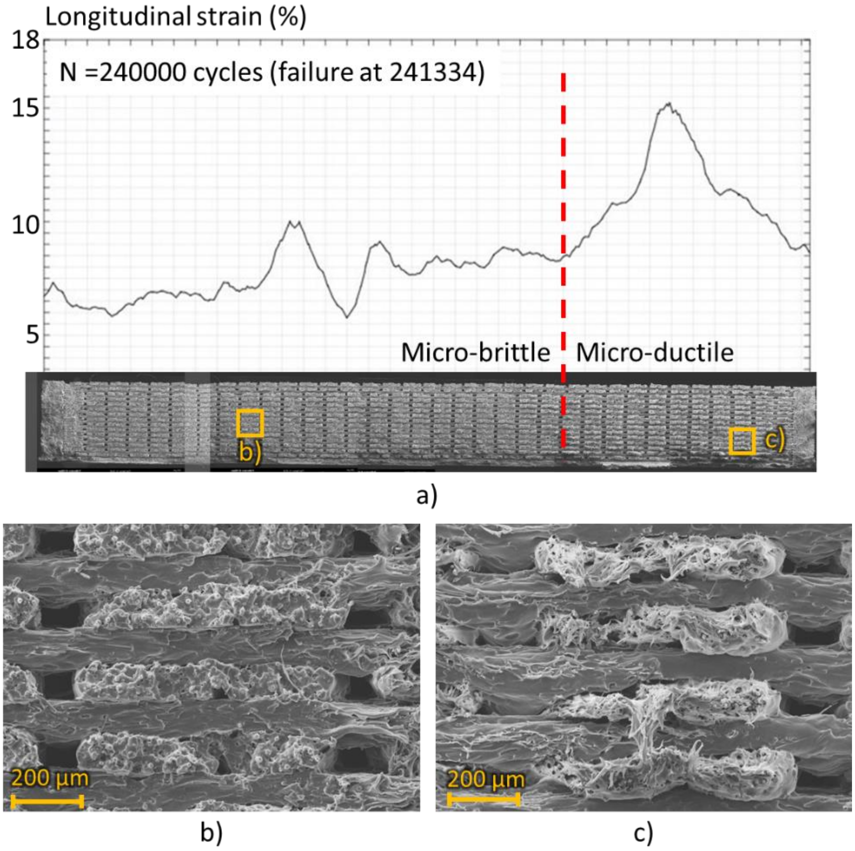

Figure 2: a) Fracture surface of specimen 7 with DIC strain measurement and detail of b) the micro-brittle region and c) the micro-ductile region.

3.2 Displacement and strain measurement during cyclic load

The stiffness evolution during the cyclic load is usually considered an important indicator of the fatigue damage in composite materials [8]. It is therefore crucial to adopt an adequate strain monitoring technique that does not affect the fatigue response of the tested material. In this work, strains were measured or calculated using DIC, extensometer and the crosshead displacement. These techniques are here compared and evaluated.

Figure 3a shows a comparison of the maximum and minimum measured strains via crosshead displacement and extensometer for specimen 3. As shown, the two measurements are initially very close and coherent with each other. However, after several cycles, the two measured trends diverge and are not anymore correlated. This behaviour was observed for all specimens equipped with the extensometer. Considering the accurate initial performance, it is unlikely that this was caused by an intrinsic difference between the two measuring techniques. It is arguable that the crosshead displacement is usually affected by the compliance of the machine itself, and thus often considered an unreliable strain measuring technique. However, in this case the low stiffness of the material (around 1 GPa) makes this error negligible, as confirmed by the initial accurate estimation. More likely, the difference arises when diffused fatigue damage reduces the material stiffness also outside the shorter gauge length of the extensometer. This is also coherent with the lower measured strain by the extensometer. Moreover, Figure 3b shows a microscopic magnification of the region close to the extensometer's arms of one of the specimens: as shown, these arms left an imprint on the surface of the specimen. These imprints were observed in all specimens equipped with extensometer; moreover; all these specimens failed very close to these imprints and in the extensometer's arms area. This failure behaviour strongly supports the fact that strain calculated via crosshead displacement is to be preferred to extensometer measurements.

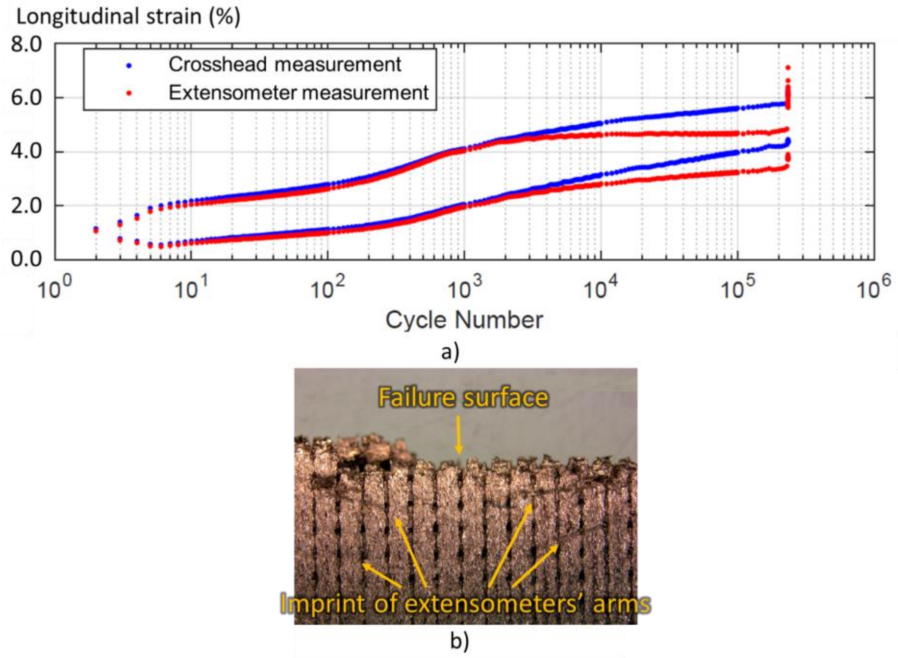

Figure 3: a) Maximum and minimum strain measured by the extensometer and calculated via crosshead displacement for specimen 3 and b) imprints of the extensometers' arms

DIC is not at risk of influencing the local failure behaviour of the specimens, thanks to its contactless nature. However, this technique is affected by two major issues. The first one is the need for a modified load history that includes static ramps for a correct image acquisition. Especially for higher loads (like for specimen 2), the holding time required to acquire the picture was long enough for the material to be subjected to creep. This is shown in Figure 4a, where strain increase is clearly visible in the ramps. This significantly affected the stiffness monitoring, as explained in section 3.3. In addition, the white paint used for the speckle was observed to penetrate several layers of specimen 2. This was observed via SEM of the fracture surface, of which Figure 4b reports a close-up. This likely influenced the fatigue response of the Onyx specimens, although a deeper investigation is required to assess how. While this is not necessarily a consistent phenomenon (it was not observed for specimen 8), the intrinsic discontinuous and porous morphology of 3D printed composite specimens makes this risk non negligible.

Overall, both extensometer and DIC are likely to affect the mechanical performance and/or fatigue failure behaviour of the specimens. Therefore, they both proved to be inadequate measuring techniques for the 3D printed Onyx specimens undergoing fatigue loads. On the other hand, the crosshead displacement led to more reliable strain calculations, also due to the high compliance of the Onyx material.

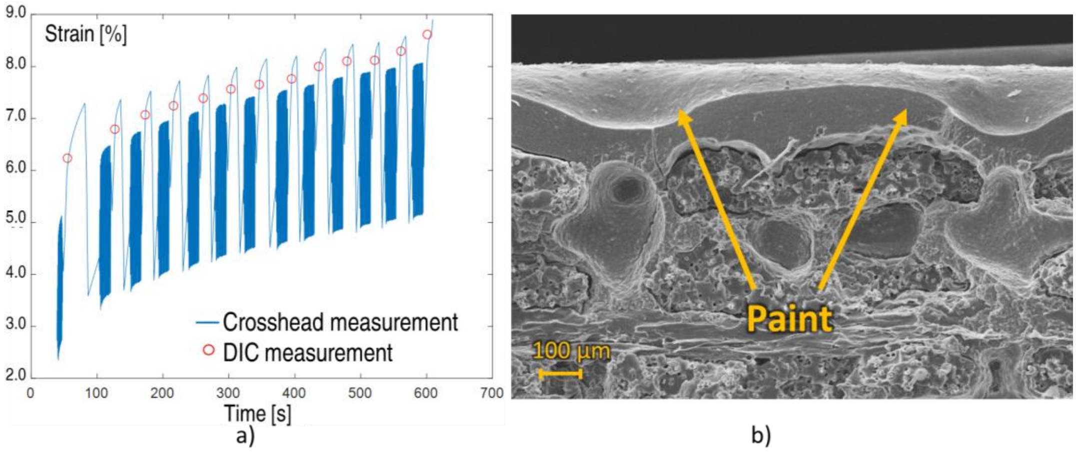

Figure 4: a) DIC and crosshead comparison on specimen 2 and b) detail of the SEM fracture surface of the same specimen

3.3 Modulus and temperature trends

Figure 5a shows the modulus trends for the tested specimens. As explained in section 3.2, these were obtained with the strain calculated via crosshead displacement; moreover, the trends of the specimens on which DIC measurements were performed were heavily affected by the different load history and were thus not included. Interestingly, all moduli do not decrease monotonically due to damage, but start to increase after around 10^3 cycles. A similar trend change is observed in the temperature measured on the specimens. As shown in Figure 5b, the temperature increases due to the cyclic loading until 10^3 cycles, after which it starts to decrease (fluctuations of the measured temperature in the high-cycle part of the load history are attributed to large period fluctuations in the room temperature).

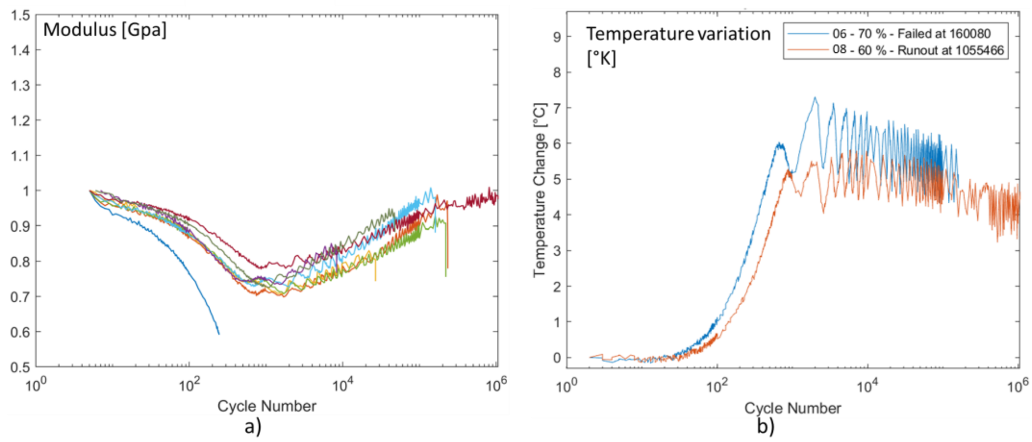

Figure 5: a) Modulus and b) temperature trends

This behaviour is highly unusual for short fibre composites, that simply show a modulus decrease due to damage [8]. The modulus and temperature trends seem to be highly correlated, although a deeper investigation is necessary to fully understand this correlation. Nevertheless, both the specimens' stiffness and temperature were successfully measured during the cyclic tests. The obtained results suggest that these quantities may be used to monitor damage in 3D printed Onyx, although not as straightforwardly as done for injection moulded composites [8].

4. Conclusions and future works

3D printed Onyx specimens with a 0°/90° raster orientation were tested under tension-tension fatigue load. Several monitoring techniques were adopted for different specimens to assess their applicability to this material. It was shown that both extensometer and DIC likely alter the fatigue response of the Onyx specimens. Therefore, it was suggested to calculate strains from the crosshead displacement of the testing machine. Regarding the fatigue response of the material, fatigue damage initiates and propagates in a micro-ductile fashion. This makes it easier to identify the fatigue initiation and propagation regions by SEM fractography. Moreover, the stiffness of the specimens was shown to have a non-monotonic behaviour, with a first decreasing part followed by an increasing one. A strong inverse correlation was found between modulus and specimens' temperature, with the latter increasing and decreasing accordingly. This work is therefore a useful guide for fatigue testing of short fibres 3D printed short fibre reinforced polymers.

Moreover, it also highlighted important open questions regarding the fatigue response of these materials. In particular, the modulus and temperature trends shown by the specimens has still to be fully understood, as well as their relationship. This work thus set some basis for a full characterisation of the fatigue response of 3D printed short fibres reinforced polymers, that will also require the testing of specimens with different raster orientation and under different loading and environmental conditions.

5. References

1. Brenken B, Barocio E, Favaloro A, Kunc V, Byron Pipes R, Fused filament fabrication of fiber-reinforced polymers: a review. Add Man 21, 1-16 (2018).
2. Safai L, Cuellar JS, Smit G, Zadpoor AA, A review of the fatigue behaviour of 3D printed polymers. Additive manufacturing 28, 87-97 (2019).
3. Shanmugan V, Das O, Babu K, Marimuthu U, Veerasimman A, Johnson DJ, Neisiany RE, Hedenqvist MS, Ramakrishna S, Berto F, Fatigue behaviour of FDM-3D printed polymers, polymeric composites and architected cellular materials. Int J Fat 143, 106007 (2021).
4. Pertuz AD, Díaz-Cardona S, González-Estrada OA, Static and fatigue behaviour of continuous fibre reinforced thermoplastic composites manufactured by fused deposition modelling technique, Int J Fat 130, 105275 (2020).
5. Markforged, Onyx datasheet 2020: https://markforged.com/materials/plastics/onyx
6. Canegrati A, Martulli LM, Bolzoni G, Kostovic M, Rollo G, Sorrentino A, Carboni M, Bernasconi A, 3D printed short carbon fibres reinforced polyamide: tensile and compressive characterisation and multiscale failure analysis. 20th European Conference on Composite Materials (ECCM20), 26-30 June 2022, Lausanne (CH).
7. ASTM D3479. Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite. vol. 08; 2002.
8. Monte M, Moosbrugger E, Quaresimin M. Influence of temperature and thickness on the off-axis behaviour of short glass fibre reinforced polyamide 6.6 cyclic loading. Compos Part A Appl Sci Manuf 2012;41:136
9. Horst JJ, Spoormaker JL. Mechanisms of fatigue in short glass fiber reinforced polyamide 6. Polym Eng Sci 1996;36:2718–26.

