

Photopolymer Fiber Composite Structures in Microgravity

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Abstract

Deployable structures are the future in space manufacturing. A common approach, are the pneumatically actuated deployables, which deploy using the pressure of a fluid and generally impose a low risk during deployment. However, their greater disadvantage is the low stiffness and damage tolerance. In order to solve this problem, AML-Applied Mechanics Laboratory and the AML Space Group researching the idea of deploying pre-impregnated polymer fibers fabrics into space and curing them, thus creating lightweight, high-stiffness, thin-shelled structures in space. This research was focused around the behavior of photopolymer resins in a micro-gravity environment, by participating in the ESA Fly Your Thesis 2017 campaign. In this experiment, uncured glass fiber reinforced polymer tubes stowed before flight, deployed by compressed air and cured during the micro-gravity intervals of the parabolic flights, using a UV light-source. Identical reference specimens were produced on Earth with the same equipment at 1-g gravity acceleration. The goal was to test and compare the mechanical properties and microstructure between these two batches of specimens produced in the two different gravity acceleration levels. Identifying the effect of different gravity levels on the mechanical properties and microstructure of polymeric materials, shall enable better understanding on how such polymers could be used in space manufacturing.

1. Introduction

The concept of UV polymers in space have been around for a long time, from when it was first conceived in 1963, by the Hughes Aircraft Company for use in fibre reinforced inflatable structures [1]. In different cases, the curing of the inflatable booms was either using internal UV ledstrips or with the aid of solar radiation. Since then, extended research on the use of different materials in space has been performed throughout time. In 1985, NASA compiled a catalogue which also included the behaviour of materials in vacuum chambers, measuring the outgassing [2]. However, on the effect of

micro-gravity on the formation and curing of materials, the research still continues up to this day. In addition, such research is significantly expensive, as it includes research on a micro-gravity experiment platform. Specifically, researching the effect of microgravity on most thermoset polymers involved increased amounts of time, and reduces the available platforms for such research. However, UV polymers require a minimal amount of time that spans from milliseconds to a few seconds depending on the UV radiation intensity, thus making them applicable for a wider variety of micro-gravity experiment platforms – such as the parabolic flights in our case [3; 4; 5].

Over the years, deployable structures has been investigated in parabolic flights, such as the “Spaghetti Tubes” project participating in the ESA Fly Your Thesis! Campaign in 2003, which investigated the use of inflatable deployable tubes [6]. Moreover, research has been performed on UV polymers, but from a biomedical perspective, such as the 2004 project "Composite Photopolymerization for Teeth Repairing" which flew on the ESA parabolic flight campaign [7]. Additionally, the “FOCUS” experiment which flew on a suborbital rocket flight in the 2011 ESA REXUS project, investigated the application of inflatable impregnated fibre reinforced UV polymers tubes in deployable structures [8]. However, in our case, the AML Space Group aims to investigate the material properties in both the microscopic and macroscopic levels, to observe the effects of micro-gravity on the UV polymer composite materials.

2. Manufacture

For the parabolic flight campaign, both the specimens and the UV curing device were designed and manufactured in-house, using the Applied Mechanics Laboratory facilities. The dimensioning of the specimens was decided to be similar to that of literature in order to perform a closer comparison with existing inflatable deployable structure components [14; 15; 16; 17].

2.1. Specimen Materials

For the composite tube specimens manufacturing, the glass fiber sleeve used that was S-glass 34tex, had a base dimensioning of woven $\pm 45^\circ$ at 45 mm diameter and a thickness of 0.4 mm. The polyethylene sleeves used to seal the pre-impregnated glass sleeve, had also a diameter of 45 mm and a thickness of 0.1 mm. Both glass fiber and PE sleeves were provided by Fibermax Composites (Volos, Greece). The photopolymer resin used, was mainly acrylic based (>98% wt) with Phenyl bis(2,4,6-trimethylbenzoyl) phosphine oxide photoinitiators and without inhibitors. The photopolymer was acquired by 3D ink, (Kansas City, USA), and used as received.

2.2. Specimen Preparation

The specimens preparation procedure for the inflatable deployable tubes is described below. Initially, two Glass-fiber sleeves as well as two polyethylene sleeves were cut at the length of 800 mm for every specimen. The edges of the glass-fiber sleeve were fixated using tape, so as to minimize attrition and loose fiber strands. The tube laminate was created by placing one Glass-fiber sleeve over the other and enclosing these two fabric layers between the two polyethylene sleeves. The tube laminate was placed on top of a metallic mandrel, so as to stretch the laminate into tubular shape. A circular metallic disk was apposed at the base of the structure, inside the inner bag and fixated with a metallic clamp. Any excess polyethylene bag folded between circular disc and bottom plate. The specimen bottom plate was apposed above the circular disk, with the interjection of an O-ring, so as to achieve air-tightness at the bottom interface. The specimen bottom plate was apposed with the circular disc through a bolt dressed in Teflon tape. The top plate differs in that the circular disk bears an one-way valve. Finally, the specimens were filled with UV resin up to a vf of 0.5, folded using the zigzag folding method [14] and packed for transportation. During transportation and resin filling, the specimens were kept in light-tight storage, to ensure that no polymerization occurred before curing in the parabolic flight.

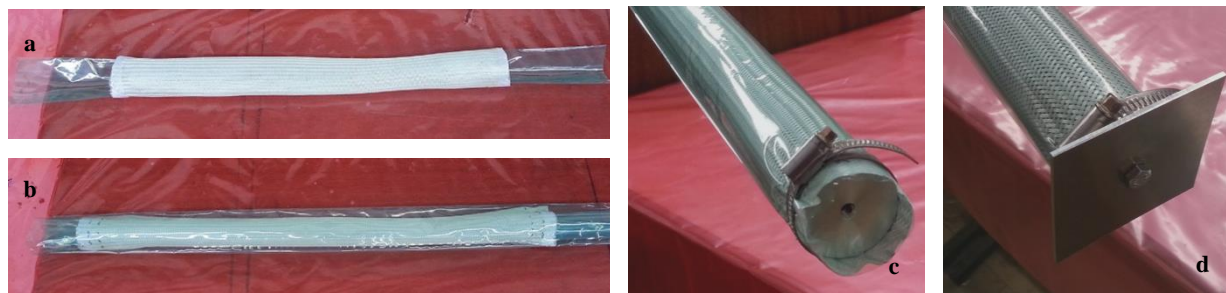


Figure 1. Specimen preparation process. a, b) glass fiber sleeves between PE sleeves. c) mounting of the lower metallic disc and clamp. d) Installation of the bottom plate after O-ring placement.

2.3. Specimen Curing

An in-house device was designed and manufactured in the Applied Mechanics Laboratory, at the University of Patras, which fulfilled the requirements set by Novespace for the participation in parabolic flights [18]. The device included a curing chamber which consisted of a UV-light LED source for curing and an oil-less air compressor for the inflation and deployment of the specimens. The interior of the chamber was large enough for a batch of 4 specimens to be inflated and cured simultaneously. Prior and after curing, the specimens were stored in light-tight storage, to ensure that no polymerization occurred outside the curing chamber.

The curing process took place during the 0-g phase only, of the parabolic flight that spanned a timeframe of 20 seconds. The air compressor provided an overpressure of 0.5 bars inside the specimens, deploying them via inflation over a timeframe of 5 seconds and providing the desired cylindrical shape for the curing phase, acting like an inflatable mould. Afterwards, the UV-light source was switched on, providing 80 W/m^2 quasi-uniform irradiation at 395-405 nm of wavelength for the remaining of the 0-g phase of the flight. After the first parabola, each batch would remain inside the chamber for an additional 4 parabolas (additional 80 secs of curing), for curing only during the 0-g flight phases. To prevent deflection of the deploying end of the tube specimens during deployment, a sliding plate mechanism was used to fixate the deploying end, as shown in figure 2.

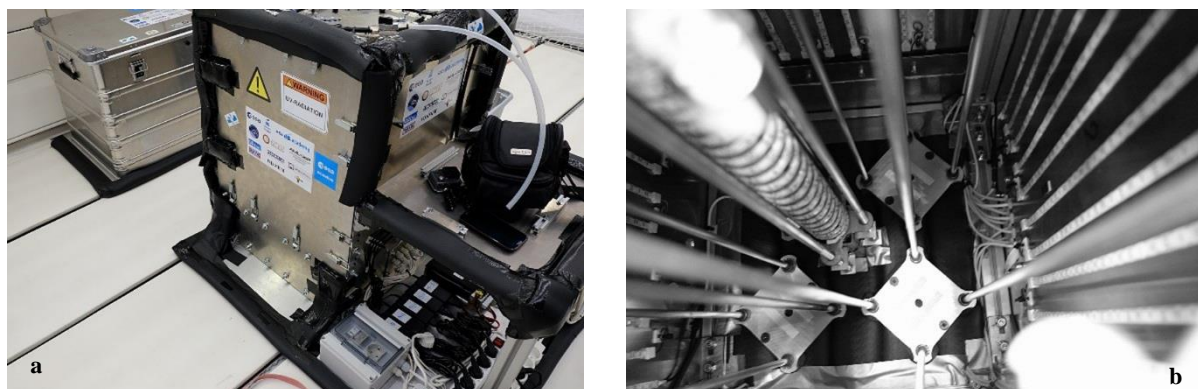


Figure 2. a) Curing device (front) and storage rack (back). b) Curing chamber as shown from the specimen installation opening, showing the sliding plate mechanism.

3. Experiments and Testing

The specimens produced during the parabolic flight campaign were processed, cut into specimens and tested back in the laboratory. The specimens were tested in SEM – Scanning Electron Microscopy, DMA – Dynamic Mechanical Analysis, DSC – Differential Scanning Calorimetry, XRD – X-Ray Diffraction, Void Calculation and Mechanical Testing.

3.1. SEM

The microstructure of the composite tubes manufactured in 0-g conditions was compared with identical tubes manufactured in 1-g conditions. The morphology of the UV-cured composite deployables was investigated by SEM observations on the cross-sections of the composites. In figure 3, the cross-section of a deployable manufactured in 0-g phase reveals a uniform structure, where the resin soaked the fiber reinforcement throughout the overall thickness. However, the resin seems to cannot sustain cutting and left fibers exposed, in both cases. Figure 4, shows a cross-section of the 1-g manufactured deployable, where fiber reinforcement appeared to be more exposed than the 0-g sample with much less resin residues left on the fibers.

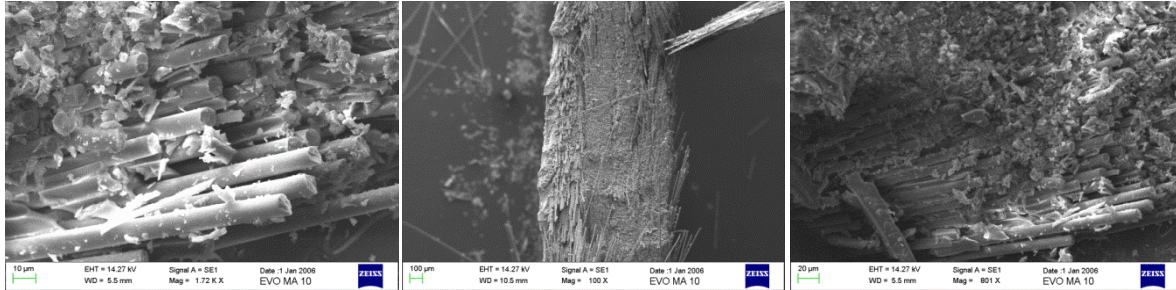


Figure 3. Cross-section of 0-g deployable shell, images of cut edges. 137-1.88K magnification.

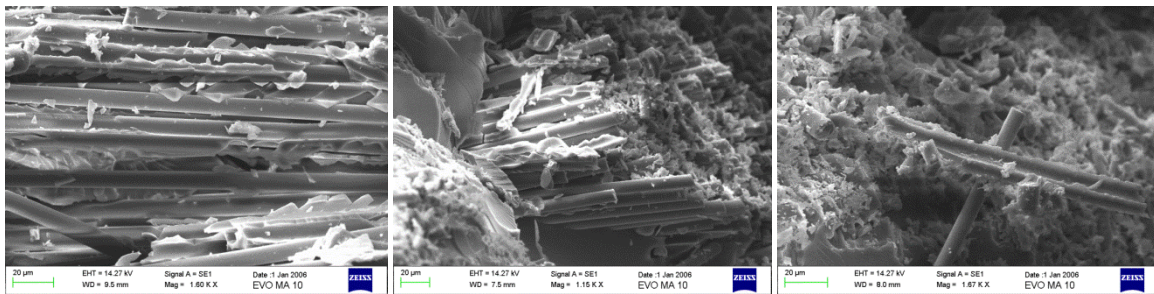


Figure 4. Cross-section of 1-g deployable shell. Fibers with resin remaining on their surface across the cross-section. 380-2.27K magnification.

3.2. DMA

By performing DMA, the data were provided to determine the glass transition temperature T_g for the composite manufactured in 0-g conditions. From the Storage modulus and Tan-delta graphs, the storage modulus was calculated at **1,17 GPa** and the T_g between **63-73 °C**.

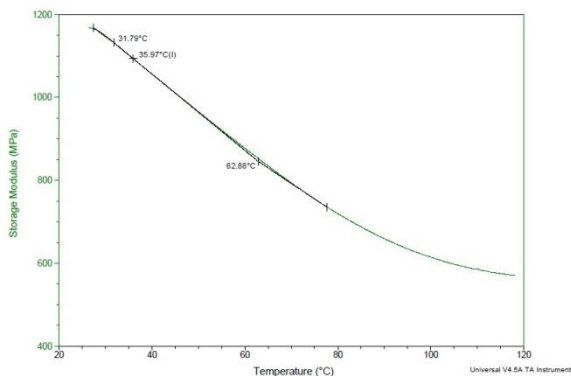


Figure 5. Storage modulus plot of the 0-g specimen

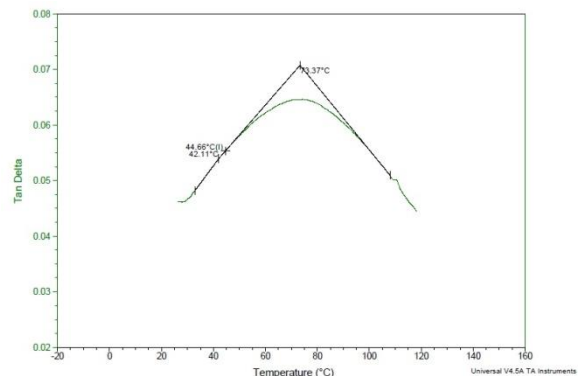


Figure 6. Tan-delta graph of the 0-g specimen

3.3. DSC

The T_g temperature for neat resin was determined by a DSC plot. As shown in Figure 7, the T_g for the sample of neat acrylic resin cured in 0-g conditions is about **55,7 °C** while T_g for the neat resin cured in 1-g is about **61,4 °C**. This difference was expected, as photopolymers in microgravity conditions are affected by gravity-sensitive mechanisms [22]. Presence of air bubbles containing oxygen, sedimentation of polymer globules and absence of thermal convection, are the main factors which result in a lower degree of polymerization and non-uniform density distribution of the polymer in micro-gravity conditions [23]. Moreover, the DSC analysis reveals that the resin primary microstructure is at an amorphous state.

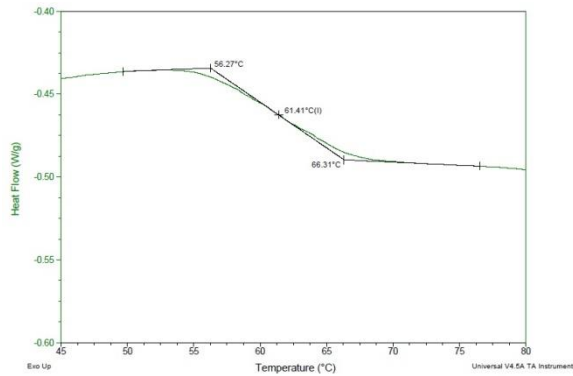


Figure 7. DSC plot of the 0-g specimens.

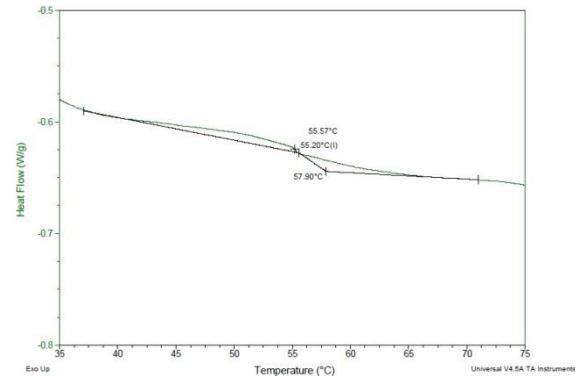


Figure 8. DSC plot of the 1-g specimens.

3.4. XRD

The XRD analysis for neat resin cured in 0-g conditions is given in figure 9. The curve reveals a totally amorphous polymer, with a wide peak at **18°** corresponded to the amorphous phase of the resin. In figure 10, the pattern of a 1-g curing conditions sample is shown, where the wide peak was situated closer to **19°**, characterizing its arrangements of macromolecules [23].

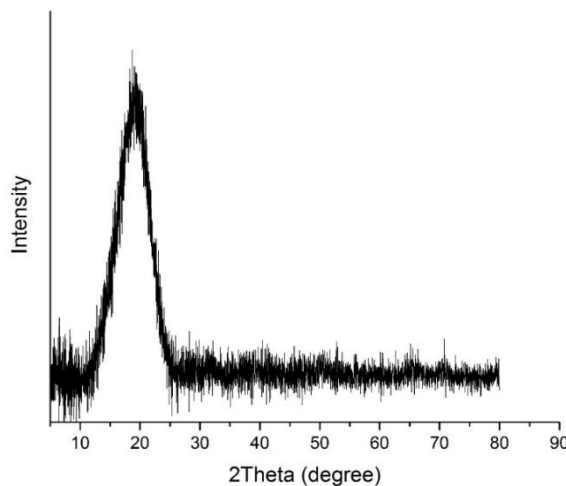


Figure 9. XRD plot of the 0-g specimens.

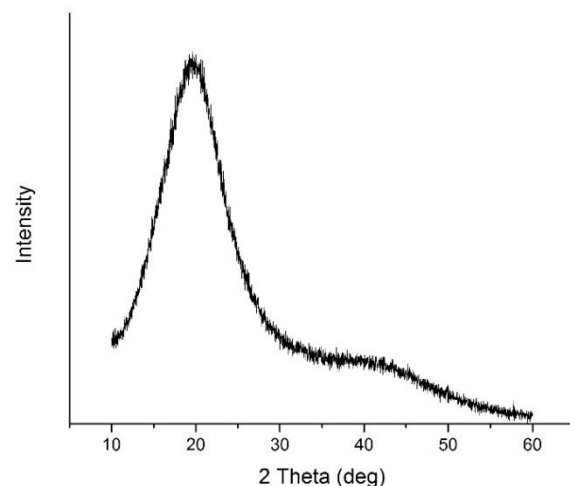


Figure 10. XRD plot of the 1-g specimens.

3.5. Void Calculations

In order to quantify the surface porosity of the 1-g and 0-g of double layer woven glass fibers and evaluate the possible reduction in mechanical properties, measurements were performed using post-processing software able to track the contours of the recorded maps. These images clearly showed the

location, size and evolution of voids on the surface of the deployable. A typical example of the methodology used is presented in Figure 11. Initially, a raw image was taken and uploaded into an image analysis software. Then the areas of surface porosity were outlined and converted into a binary image, where black pixels represent areas of surface porosity. The black pixels in the image represent 12.07% of the total pixels in the image, translating thus to a 12.07% of this area being surface porosity.

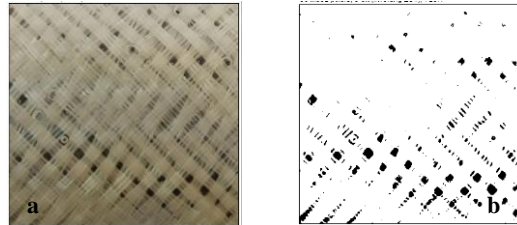


Figure 11. Sequence of actions used to quantify surface porosity. (a) Raw image taken and uploaded into image analysis software (b) Post-processed image.

In particular, the average value of the measured samples shows **2.54%** of porosity in 0-g and **12.07%** in 1-g specimens respectively, a difference in results consistent with the hypothesis that that in microgravity the surface tension of the liquids dominates over gravity, thus providing better wettability of the fibers. Because polymerization occurred in the presence of air, bubbles were distributed uniformly throughout the whole surface of the composite deployable.

3.6. Mechanical Testing

3.6.1 Specimen tensile testing

For the mechanical tensile testing, the ASTM D 3039/D 3039M-00^{e1} standard [19] for tension testing of composites was followed as closely as possible. The specimens to be tested, were taken off from several double layered tube specimens in the axial direction at 200x15 mm dimensions, producing $[\pm 45^\circ]_2$ samples with a minimal curvature. The tests were performed at an INSTRON 8872 with a 25 kN loadcell, under controlled temperature and ambient moisture conditions and a rate of grip separation of 1.5 mm/min.



Figure 12. Tensile coupons fixated on the testing machine. (a) prior to failure (b) post-failure

The results from the 1-g control samples showed a maximum stress of 7.07 ± 1.27 MPa, ultimate strain of 3.71 ± 0.58 % and toughness 326.9 ± 58.0 kJ/m³. In contrast, the results from the 0-g samples were better, showing a maximum stress of 11.59 ± 1.60 MPa, ultimate strain of 4.81 ± 0.48 % and toughness 741.5 ± 168.2 kJ/m³. Comparing the 0-g samples with the 1-g control, an increase in maximum stress of

63.9% is observed. In addition, the tests displayed an increase of 29.6% in the ultimate strain and 126.8% in toughness

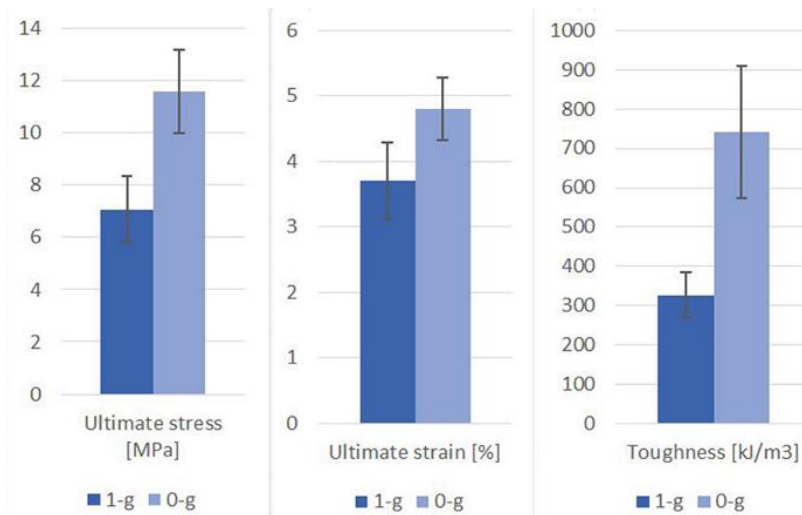


Figure 13. Tensile properties comparison of the 1-g and 0-g coupon specimens.

3.6.1 Specimen compression testing

For the mechanical compression testing, the ASTM D 3410/D 3410M-03 standard [20] for compression testing of composites was used as reference. For the preparation of samples, a bandsaw was used to cut several double-layer tubes in the axial direction at 90x15 mm coupon dimensions. The same testing and loading conditions were used as the tensile tests.

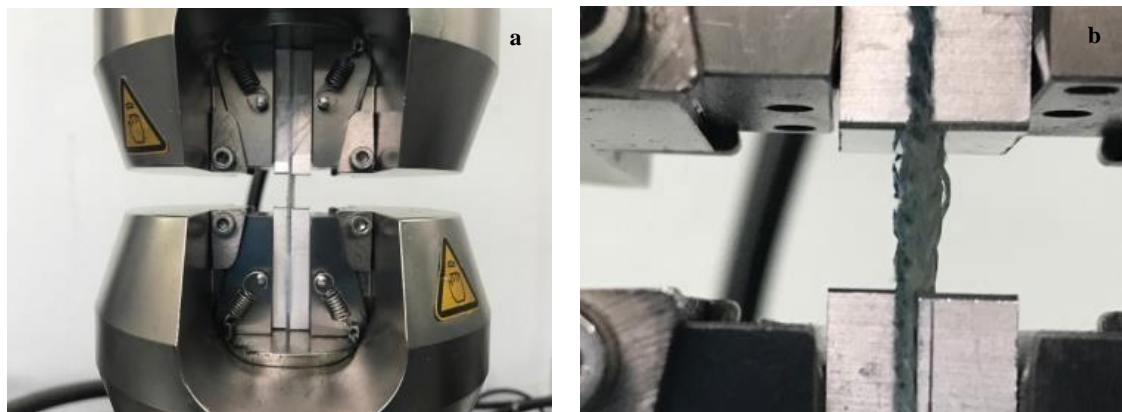


Figure 14. Compression coupons fixated on the testing machine. (a) prior to failure (b) post-failure

The results from the 1-g control samples showed a maximum stress of 19.96 ± 9.51 MPa, ultimate strain of 1.51 ± 0.48 % and toughness 923.6 ± 757.9 kJ/m³. In contrast, the results from the 0-g samples performed better, showing a maximum stress of 24.21 ± 4.33 MPa, ultimate strain of 1.38 ± 0.19 % and toughness 1398.1 ± 473.4 kJ/m³. When the 0-g samples are put to comparison with the 1-g control, an increase in maximum stress of 21.3% is observed. As for the ultimate strain, a reduction of 8.6% is observed and the toughness is increased by 51.4% compared to the control.

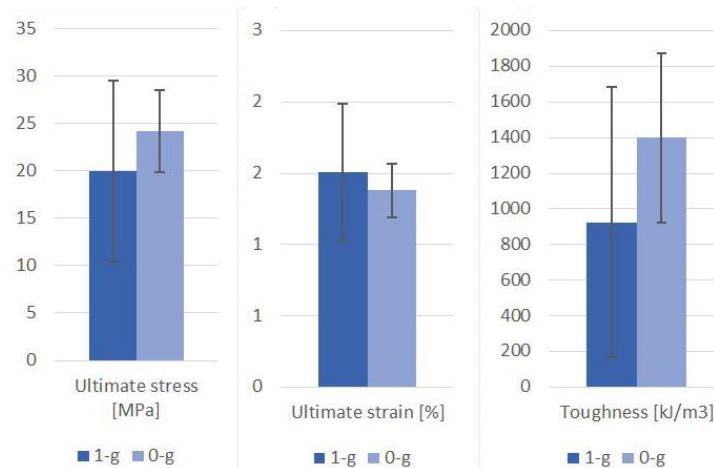


Figure 15. Compression properties comparison of the 1-g and 0-g coupon specimens.

4. Conclusions

The testing performed in the laboratory showed that gravity affects the microscopic and macroscopic properties of fiber reinforced polymer materials differently. Comparing the 1-g gravity acceleration to the 0-g conditions, it is concluded that the 0-g specimens display a small superior behavior in terms of macroscopic behavior, and a slight decrease in terms of microscopic behavior.

In the microstructure level, the wettability of the fibers has improved in the 0-g specimens in comparison with the 1-g control. This finding could support the hypothesis that in microgravity the surface tension of the liquids (resin in this case) dominates over gravity, thus providing better wettability of the fibers. However, on a smaller scale, the sedimentation of polymer globules and the presence of air bubbles containing oxygen, are the main causes that the thermal properties of the material are reduced slightly, with a decrease in the degree of polymerization and the reduction of the material's T_g by a few degrees.

For the mechanical testing, the results displayed a large scatter in all cases and the conclusions reached are not definitive. More specific, the results from the coupon testing in both tensile and compression show that the composite manufactured in microgravity has better mechanical properties in general, comparing with the 1-g control composite. The ultimate stress increase is quantified at roughly about 60% for tension and 20% for compression, in reference of the control specimens. The ultimate strain shows a slight increase of 20% in tension and negligible change in compression comparing 0-g to 1-g control specimens, while the 0-g specimens toughness increase is up to 2.5 times for tension and 1.5 times for compression compared to the reference. More testing and investigation is foreseen in the future, including the mechanical behavior of the tubes under internal pressure conditions.

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