Composites Meet Sustainability – Proceedings of the 20th European Conference on Composite Materials, ECCM20. 26-30 June, 2022, Lausanne, Switzerland

INVESTIGATION OF 3D PRINTED POLYMERS AND MOULDED COMPOSITES IN HOT CONCENTRATED ACID

Damilola Aje[a], Liu Yang[a]

a: Department of Mechanical and Aerospace Engineering, University of Strathclyde, 75 Montrose Street, Glasgow G1 1XJ, UK – damilola.aje@strath.ac.uk

Abstract: *This paper investigates the resistance of polymer composites after being soaked in a hot (85°C) concentrated (85%wt) phosphoric acid solution, it compares the rate of acid intake by different selected polymeric materials, and conduct an analysis on the impact of acid intake and elevated temperature on the baseline mechanical properties of the polymer composites. Four polymers and polymer composites were selected among the common high-performing polymers used in structural applications in this investigation, two of which were 3D printed pure polymers while the other two was an injection moulded polymer composite. Investigation proved that mechanical properties of the selected polymers was not significantly affected by the hot acid. The strength and modulus remain unchanged, however, there is a significant drop in strength and modulus for the selected polymers when tested under elevated heat chamber of 95°C.*

Keywords: Polymers; Polymer Composites; Additive Manufacturing; Chemical-aging; Mechanical Properties.

1. Introduction

Polymers and polymer composites are gaining more acceptance in the construction industry due to the numerous advantages they possess over the traditional metallic materials. The rising cost of maintenance and repair of existing infrastructural systems is driving the push for a more reliable and cost-effective materials. Polymers are regarded to be a lightweight material and coupled with their high strength, are being regarded as the material needed to drive down the ever-rising cost of production and maintenance [1]. This polymer advantage is very vital, especially in the Aerospace industry where cost saving is desired without jeopardizing the structural integrity of the components [2]. Major companies are now switching into polymer technologies to benefits from its versatile cost saving advantage. Effort is ongoing within most industries in replacing metallic-based components with advanced polymer composites combining with additive manufacturing technology as a productive approach for the next generation of engineering components for mining applications [3].

Having observed the role of additive manufacturing in other industries such as in aerospace industry, 3D printing has now become a mainstream tool for producing composite parts quickly and efficiently without the usual waste as we have it in traditional manufacturing [3]. Apart from the common reasons of cost-effectiveness, streamlined and efficient production processes and the time-saving nature of the technology as observed in other industries where this technology have been proven, there are other specific advantages the mining industry has identified to benefit from 3D polymer technologies. Some of these specific advantages includes better supply chain management, quick turn-around of replacement parts, and reduction in manufacturing lead time [3].

Despite the advancement of this non-metallic material, and the fact it is being currently used in engineering applications such as in pipes, pipelines, and tanks, long-term resistance against hot concentrated chemicals remain extremely challenging for most engineering polymers today. Design for this type of applications can be further complicated when the polymers will be used to process chemical fluids at elevated temperature. Understanding the structural behaviour of polymers and polymer composites in different working environments is a vital requirement needed to qualify the integrity of polymeric materials in structural applications.

Many researchers have carried out several test on different high performing polymers and have reported polymers to have good corrosion resistance, high strength-to-weight ratio, adequate mechanical properties and have ease of handling. But, little or nothing has been said in the literatures how hot concentrated acid can affect mechanical performance of the polymers after an extended period of time. M. Amini et all [4] conducted an experimental study of the effect of prolonged corrosive media on the glass fiber/polyester at different temperature levels of 25 oC, 50 oC and 70oC. The polymer specimen was completely submerged into hydrochloric acid solution (10%wt) for various time period. The results indicated that the bending strength, ultimate tensile strength, Young's modulus and hardness of the samples decreased when exposed to longer exposure duration and/or higher temperature.

Temperature is another factor that affect the selection of polymers for structural application as they are more sensitive to elevated temperatures. In general, load-supporting polymers are not suited for long-term use at a temperature higher than their glass transition temperatures (Tg). Glass transition temperature is the temperature at which a polymer turns from a rigid state into a flexible state. Tg of polymers varies and it is determined by the grades, the curing process, and the moisture content. significant performance drop can still occur to polymer composites at above Tg. In addition, creep will become more remarked at above Tg, although fiber reinforcement can mitigate the creep to large extent if the temperature is not too far above Tg [2].

Fluid-structure interaction of polymers and polymer composites was investigated as part of this project through laboratory experiment and testing under coupled thermo-chemical and thermo-mechanical environments. The objective of this work is to investigate the resistance of polymer composites to 85%wt concentrated phosphoric acid solution with elevated temperature at 85oC, compare the rate of acid intake by different polymeric materials, and conduct an analysis on the impact of acid intake and elevated temperature on the baseline mechanical properties of the polymer composites.

2. Experimental

2.1 Material

The detailed review of different polymer materials database was carried out to generate a list of material candidates. These include material software tool known as GRANTA, various material handbooks, and online data sources, as well consultations and discussions with polymer composites suppliers. A list of selected materials was however selected to be tested. Four different high-performing pure polymers and polymer composites were selected and presented in this report. They include.

1. Antero 800 NA – This is a PolyEtherKetoneKetone (PEKK) based thermoplastic with excellent mechanical properties and excellent resistant to most industrial chemicals. They are manufactured by Additive Manufacturing (AM) method known as Fused Deposited Modelling (FDM).

2. Ultem 1010 – This are high performing Polyetherimide (PEI) thermoplastic with high heat resistant and excellent mechanical properties. They are manufactured by Additive Manufacturing method known as FDM.

3. Ketron 30% CF – This is a PolyEtherEtherKetone (PEEK) based thermoplastic polymer. Manufactured by injection moulding methods, Ketron is a 30% carbon fiber reinforced grade with excellent stiffness and strength.

4. Victrex 30% CF – This is a high-performance thermoplastic material which is an Aerospace grade material. It contains 30% carbon fibre reinforced PolyEtherEtherKetone (PEEK) and can be manufactured by injection moulding and extrusion. It is regarded to have a much higher strength and modulus than Ketron.

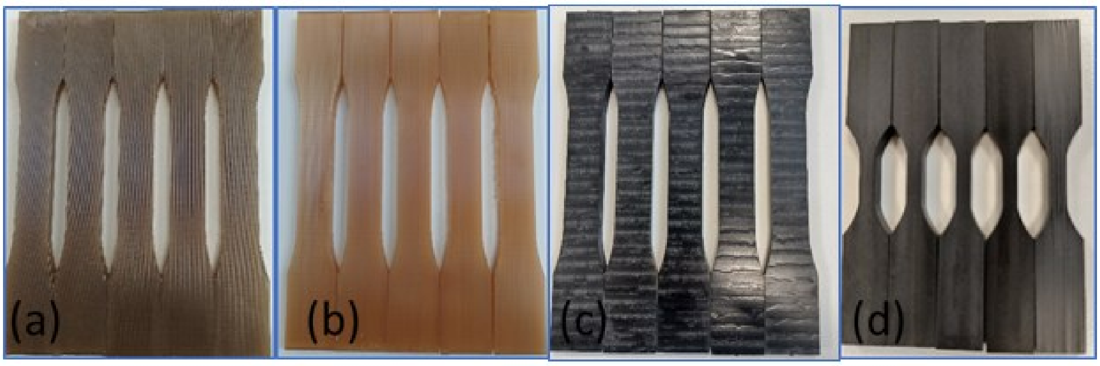

Figure 1 – Polymers under investigation (a) Antero 800NA (b) Ultem 1010 (c) Ketron 30%CF (d) Victrex 30%CF.

2.2 Sample Preparation

A Type 1 dog-bone tensile bar produced in accordance with standard dimensions as per ASTM D638-14 standard. Five different samples were tested for each set of experiment. Each of the test samples were taped at the tab section with PTFE tape to isolate the tab section from the acid solution. This was to ensure that the chemical acid does not affect the tab area for mechanical testing purposes. Sample dimensions (length, breath and thickness) was taken by Spi digital Calliper, and the weight was measured by desktop-based Mettler Toledo weighing machine. The test specimen was conditioned at standard laboratory atmosphere 23 ± 2°C and 50 ± 10 % relative humidity prior to the test in accordance with Procedure A of Practice D618. The test was conducted in the standard laboratory atmosphere of 23 ± 2°C and 50 ± 10 % relative humidity.

2.3 Exposure Condition

The specimens were completely submerged into hot concentrated acid (85% wt.) for up to 2000hrs. Some sets of samples were monitored every 24hours to capture the time the fluid intake become saturated. Some others monitored at the end of every 168hours. The sealed container which contained the acid solution and samples was placed in the water bath with constant temperature of 85°C.

2.4 Exposure Condition

Visual inspection of conditioned specimen was observed according to ASTM D638-14. Discolouration of the test medium, the formation of sediments of the samples and surface crack, softening of specimen were physically observed after chemical immersion. An initial discolouration will demonstrate extraction of soluble composites.

2.5 Tensile Testing

The tensile test was carried out on the baseline samples as well as on the chemical conditioned samples. Test was carried out using the 50kN Mechanical Tester (Instron Electromechanical 5969) electromechanical system with a maximum loading capacity of 50kN and equipped with fully integrated video extensometer for contactless measurements. Both Type 1 and Type V tensile testing bars was tested using the appropriate strain rate as per ASTM D638-14. Three samples were tested for each condition, and the average values are presented. See Figure 2b for a typical tensile test machine.

2.6 Microstructural Characterization

The surface topology of the samples was observed in a scanning electron microscope (Leitz Ergolux microscope) from Microscope Systems Limited, UK for detecting physical/surface change due to chemical exposure. The microscope was able to pick the solution absorptions on the surface of the specimens.

2.7 3D Printing Orientation

It is worth noting that, the behaviour of a 3D-printed polymer materials in chemical solutions is affected by the printing orientations adopted during the printing process. There are three printing orientations applicable in every additive manufactured product as shown in Figure 2a.

2.8 Thermo-chemical Testing

The four polymers were conditioned in a hot concentrated acid solution up to 2000 hours. The Weight changes were monitored daily and weekly, the weight percentage versus conditioning time are presented in the results section. Initial weight of the samples was taken prior to conditioning and the weight after were taken immediately the coupons were removed from the container jar and after they were rinsed and dried. Similar behaviour of absorption was observed

Composites Meet Sustainability – Proceedings of the 20th European Conference on Composite Materials, ECCM20. 26-30 June, 2022, Lausanne, Switzerland

for after rinsing/drying and before rinsing/drying as presented in Figure 4 and 5. The thickness and width change remain insignificant. No loss of substrate was observed for the four polymers and the colour of the acid solution did not change.

2.9 Thermo-mechanical Testing

Understanding the behaviour of polymer composites in extreme temperature environment was also considered in this work, this is to evaluate how tensile properties, such as strength and modulus have been affected by increased temperature. Tensile testing of the polymers at room temperature was carried out using the 50kN Mechanical Tester (Instron Electromechanical 5969) and the same tester with a heating chamber was used to perform the tensile test at 95°C. The variation in mechanical properties in response to the increased temperature was recorded. Figure 6 presents the graph which shows how strength of polymer materials was affected by elevated temperature.

3 Test Results

3.1 Chemical Solution Absorption Rate

Figure 3 presents the graph which represents rates of chemical intake by the four selected materials with all materials tested to at least 2000hrs of chemical conditioning. Acid intake for polymer composites is seen to be less than that in the pure polymers as observed in the absorption graphs. The increased fluid intake observed in the pure polymers is due to the presence of voids in the crystals of the pure polymers. Also, comparing the two pure polymers, it is seen that Antero (PEKK) behaved better in the acid solution, in terms of acid intake than, its PEI (Ultem 1010) counterparts. Moreover, for both pure polymers, it is observed that the OnEdge (XZ) printing orientation absorbs less fluid than the Upright (ZX) orientation. Hence, the acid intake is affected by the printing orientation.

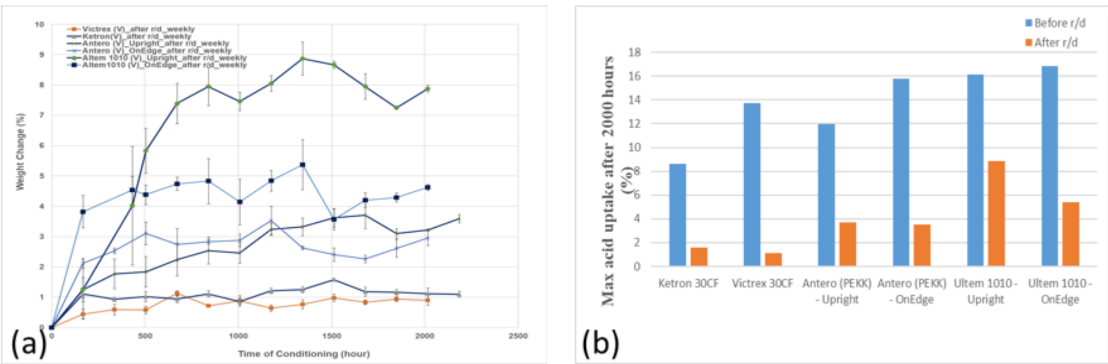

Figure 3 – Chemical Intake Graphs of selected polymers after 2000hrs conditioning (a) weight change versus conditioning time, (b) maximum acid intake before specimen rinsing/after specimen rinsing

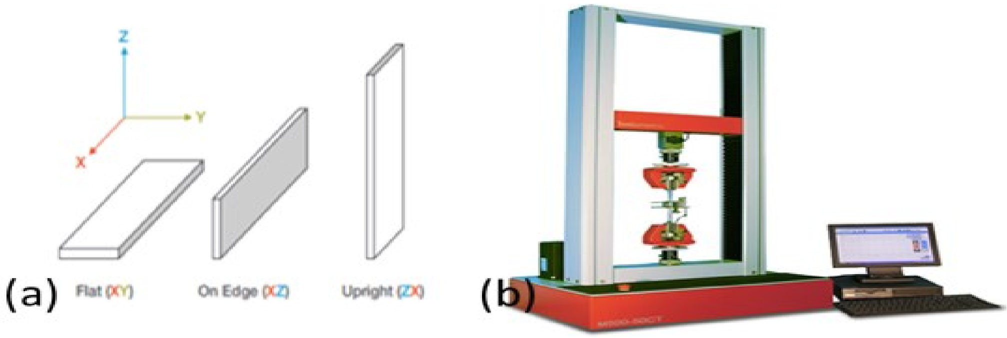

Figure 2 – (a) 3D Printing orientation, (b) Instron Mechanical Test Machine

3.2 Performance Retention Rate

Mechanical testing of the baseline materials and the chemical conditioned materials were done using the 50kN Instron tensile testing machine, (Instron Electromechanical 5969). Baseline data (strength and modulus) for all four selected materials was obtained from the tensile testing and were recorded. Conditioned samples of the four selected materials were also tested after reaching the targeted conditioned period of 2000 hours. The variation in strength and modulus for both the baseline and the conditioned samples were obtained and recorded. The data was used to determine the chemical effects on the selected materials in term of performance retention rate.

From figure 4a & 4b, it is seen that the tensile strength and young modulus of the selected materials does not significantly reduce after 2000 hours of chemical conditioning. Also, Young's modulus unchanged for selected materials after 2000 hours conditioning. There was about 20% increase in modulus for Ketron material after 2000 hours conditioning, further test of Ketron also confirm this increase. Further test was carried out on Ketron material to be sure that the unexpected increase in modulus was not due to measurement error. The ketron material was left in the chemical solution under constant elevated temperature of 85°C for 2000hrs without interruption. The results show similar trend with the previous measurements. Figure 4c presents the graph which includes the data for the interrupted Ketron measurement.

3.2 Tensile Testing with Heating Chamber

Understanding the behaviour of polymer composites in extreme temperature environment was also considered in this work, this is to evaluate how tensile properties, such as strength and modulus have been affected by increased temperature. Tensile testing of the polymers at room temperature was carried out using the 50kN Mechanical Tester (Instron Electromechanical 5969) and the same tester with a heating chamber was used to perform the tensile test at 95oC. The variation in mechanical properties in response to the increased temperature was recorded. Figure 4c presents the graph which shows how strength of polymer materials was affected by elevated temperature.

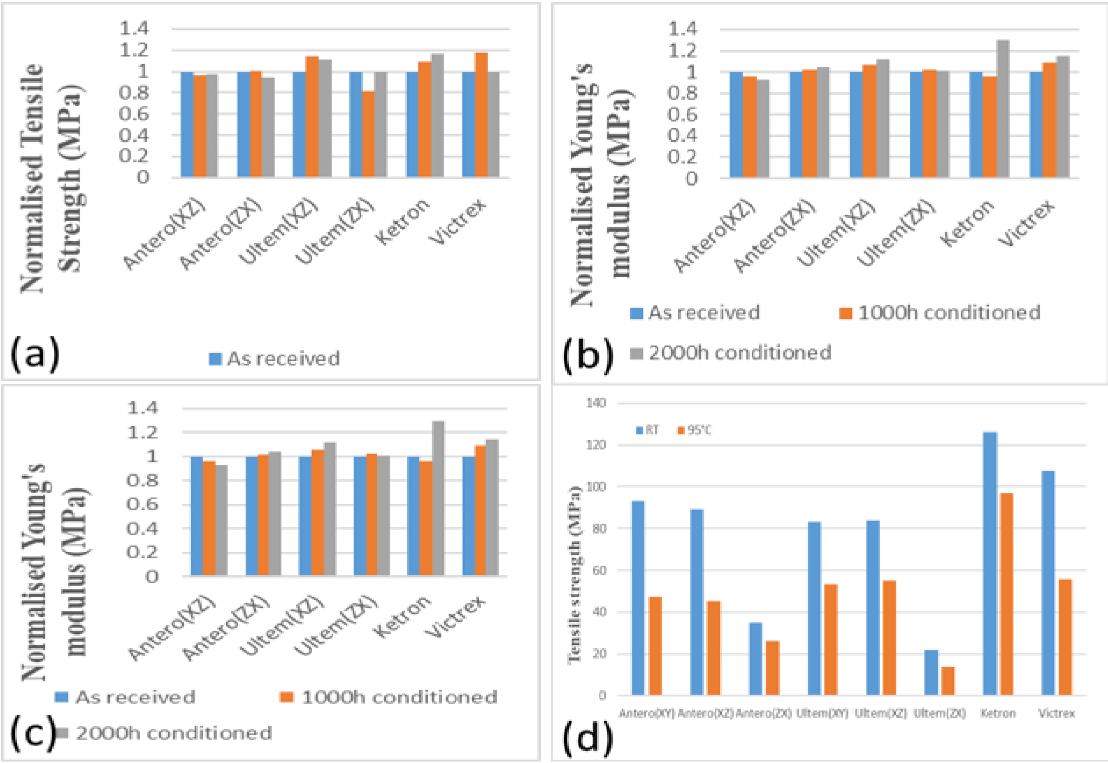

Figure 4 – Test Results after 2000hrs of chemical conditioning (a) Tensile strength, (b) Young Modulus, (c)Young modulus- uninterrupted measurement, (d)Tensile strength at room and elevated temperature.

4 Discussion of Test Results

The results presented in this paper clearly shows that, although, the phosphoric acid solution does not significantly reduce the mechanical properties of the polymers under investigation, they however, degrade significantly with increased temperature. Material stiffness and strength is very critical when deciding on the materials of construction in structural applications. Also, the effect of continual use of such materials in an operating environment must be put in consideration when making this important decision. The summary of the tensile results has proved that the, for the materials under investigation, the reduction in strength and modulus can be as high as 50% reduction when used in a condition with temperature up to 95°C. Hence, in designing a component with the polymers and polymer composites under review, under such elevated temperature, a lower value of strength and modulus should be used rather than the baseline strength and modulus data.

5 Further Work Required

This paper has been able to prove that the selected materials have excellent chemical resistance to 85% phosphoric acid at 85°C in static condition. Static condition is a state where there is no load application on the specimens. But, in the actual operating scenario, the material under investigation will be subjected to series of mechanical loads including dynamic loads. Further work is required to see how these materials will behave in the same concentrated solution but under operating load.

6. References

1. Saleh Alsayed, Yousef Al-Salloum, Tarek Almusallam, Sherif El-Gamal, Mohammed Aqel. Performance of glass fiber reinforced polymer bars under elevated temperatures. Department of Civil Engineering, King Saud University, P.O. Box 800, Riyadh 11421, Saudi Arabia.

2. T. W. Clyne and D. Hull. An Introduction to Composite Materials, Third Edition, Cambridge University Press, ISBN 978-0-521-8609-6.

3. Pakshaal S. Shah. 3D Printing Technology and its Impact on the Mining Industry, Article June 2015, BOROE Advantage Procurement.

4. M. Amini, A. Khavandi. Degradation of Polymer-based Composite in Corrosive Media. Experimental Attempt Towards Underlying Mechansm, faculty of Materials Science and Engineering, K.N. Toosi University of Technology, Tehran, Iran 2 School of Metallurgy and Materials Engineering, Iran University of Science and Technology (IUST), Narmak, 16844 Tehran, Iran. Mech Time-Depend Mater (2019) 23 :153–172 https://doi.org/10.1007/s11043-018-09408-7.

5. Vishwas Mahesh, Sharnappa Joladarashi, Satyabodh M. Kulkarni. A comprehensive review on material selection for polymer matrix composites subjected to impact load. Department of Mechanical Engineering. National Institute of Technology Karnataka Surathkal, Mangalore, 575025, India. Journal homepage: www.keaipublishing.com/en/journals/defence-technology.

6. Gurit, Guide to Composites. Delivering the future of composite solution, www.gurit.com.

7. NV Srinivasulu, V. Tejaswi. Mechanical Properties of Polymer Composite Materials. Department of Mechanical Engineering, Chaitanya Bharathi Institute of Technology, Hyderabad, AP, India, IJRET. International Journal of Research in Engineering and Technology, Volume 01, Issue 01, Sept-2012.

8. GRANTA Edupack 2020, an online engineering material database tool.

9. Kimiyoshi Naito, Hiroyuki Oguma, Chiemi Nagai. Temperature-dependent tensile properties of hybrid carbon/glass thermoplastic composite rods. Polymer Composites. 2020, 41 :3985–3995. wileyonlinelibrary.com/journal/pc. © 2020 Society of Plastics Engineers.

10.Rajan Gupta, Shraddha Shinde, Aswani Yella C, Subramaniam b, Sandip K. Saha c. Thermomechanical characterisations of PTFE, PEEK, PEKK as encapsulation materials for medium temperature solar applications. journal homepage: www.elsevier.com/locate/energy.

11. B. M. Primachenko and K. O. Strokin. Study of The Mechanical Properties of Polymer Composite Material with Different Structures. DOI 10.1007/s10692-019-09991-6 Fibre Chemistry, Vol. 50, No. 4, November, 2018 (Russian Original No. 4, July—August, 2018).

