pISSN 1229-3008 eISSN 2287-6251
Progress in Superconductivity and Cryogenics
Vol.26, No.2, (2024), pp.19~23 https://doi.org/10.9714/psac.2024.26.2.019

Evaluation of mechanical properties of polylactic acid and photopolymer resin processed by 3D printer fused deposition modeling and digital light processing at cryogenic temperature

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(Received 12 June 2024; revised or reviewed 27 June 2024; accepted 28 June 2024)

Abstract

3D printing has the advantage of being able to process various types of parts by layering materials. In addition to these advantages, 3D printing technology allows models to be processed quickly without any special work that can be used in different fields to produce workpieces for various purposes and shapes. This paper deals to not only increase the utilization of 3D printing technology, but also to revitalize 3D printing technology in applications that require similar cryogenic environments. The goal of this study is to identify the mechanical properties of polylactic acid and photopolymer resin processed by Fused Deposition Modeling (FDM) and Digital Light Processing (DLP) respectively. The entire process is meticulously examined, starting from getting the thermal contraction using an extensometer. A uniaxial tensile test is employed, which enables to obtain the mechanical properties of the samples at both room temperature (RT) and cryogenic temperature of 77 K. As the results, photopolymer resin exhibited higher tensile properties than polylactic acid at RT. However, at cryogenic temperatures (77 K), the photopolymer resin became brittle and failure occurred due to thermal contraction, while polylactic acid demonstrated superior tensile properties. Therefore, polylactic acid is more suitable for lower temperatures.

Keywords: additive manufacturing, polylactic acid, photopolymer resin, mechanical property, cryogenic temperature

1. INTRODUCTION

Nowadays, the advancement and optimization of high polymer materials are crucial in industrial production. However, their diversity is limited by fluctuations in thermomechanical properties during heating or cooling. Therefore, thermomechanical analysis (TMA) is essential before their application [1]. Recently, Polylactic acid (PLA) has been recognized for its versatility and performance at cryogenic temperatures, making it useful in cryobiology [2-3]. The thermal stability and tensile strength of PLA are significantly enhanced by incorporating silica nanoparticles into the polymer matrix [4]. PLA, a lactic acid (LA) derivative produced from renewable resources like wheat, straw, corn, and sorghum, is completely biodegradable and environmentally friendly [5-6]. It is considered the most promising biodegradable polymer material on the market and is widely used as a filament material for 3D printing [7]. On the other hand, photopolymer resins also offer a straightforward method for producing custom-made parts from thermosetting plastics. Digital Light Processing (DLP) is a key technique for creating 3D-printed objects with excellent dimensional control and resolution. Liquid acrylic resins used in DLP typically contain molecules with (meth)acrylate functionality, acting as building blocks or reactive diluents, along with a photo initiator. The resulting products exhibit good mechanical performance, thermal stability, and solvent resistance due to the cross-linked network [8].

To revolutionize polymer prototyping and production across various industries, additive manufacturing (AM), also known as 3D printing, was developed to more efficient method. Fused deposition modeling (FDM) is widely used AM technique, utilizes the controlled extrusion of thermoplastic filaments to build objects layer-by-layer [9]. This process offers several advantages, including affordability and compatibility with a wide range of materials [10]. Moreover, Stereolithography Apparatus (SLA), was another foundational AM technique developed. SLA relies on a UV laser to selectively cure liquid photopolymer resins layer-by-layer, creating high-resolution prototypes ideal for biomedical applications such as dental implants and tissue engineering scaffolds. Additionally, DLP utilizes a light projector to achieve similar results to SLA but with faster layer exposure times [8]. Due to the demand of utilizing 3D printing, it is increasingly utilized across various fields, including prototyping, bioprinting, prosthetics, jewelry, art, and architecture, owing to its ability to create complex and customized shapes that traditional methods cannot easily replicate. These technologies employ different materials and methods to construct 3D objects layer by layer [11].

Recently, 3D printing has been introduced to cryobiology, presenting opportunities to develop polymer materials suitable for cryogenic temperatures [2-12]. This advancement has applications in high-temperature superconductors, expanding its relevance from healthcare

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TABLE 1
SPECIFICATION OF 3D PRINTED SAMPLES.

Sample	Sample Brand	3D Printer Brand and model	3D printing condition		Printing time per specimen
			Layer thickness	In-fill ratio	
Photopolymer Resin	NOVA3D	LONGER/Orange 30	50 μm	100%	1h 54 min
Polylactic Acid	PURIWAY	Anet/A8 Plus	200 μm	95%	30 min

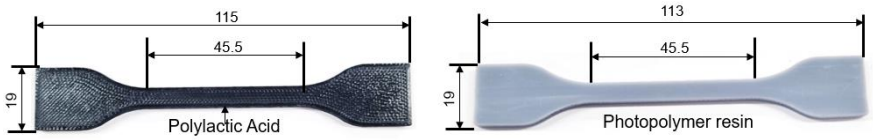

Fig. 1. Tensile specimen geometry made of polylactic acid and photopolymer resin by 3D printing.

to transportation and energy. Despite the high energy density and compact design of superconducting wires and cables, lightweight and complex thermal insulation is essential to minimize thermal and energy losses, particularly in aerospace applications [3]. Therefore, expanding the use of 3D-printed materials, such as plastics, resins, and polymers, can accelerate the production of fields of superconductors by providing complex, lightweight designed materials. However, the mechanical performance of 3D printed parts from thermoplastics depends on a combination of factors beyond material selection. Part design and printing optimization of 3D printers play crucial roles in achieving desired mechanical behavior [13]. This includes parameters like layer thickness, infill density and pattern, printing speed, build orientation, and raster angle. Studies have shown that a higher infill density generally leads to increased strength [12]. Similarly, research has identified optimal printing speeds, temperatures, and build orientations for maximizing tensile strength in FDM-printed PLA parts [14]. Despite the advantages of these polymers in various applications, extrapolating their mechanical properties from room temperature to cryogenic temperatures is challenging for thermoplastics, which can undergo significant changes in macro-scale structure, coefficient of thermal expansion and tensile properties [12]. Therefore, tensile tests are commonly used to evaluate the mechanical properties of polymers [15]. Moreover, evaluating the thermal coefficient of polymers is also crucial in various applications, particularly in superconducting devices. Recently, we assessed the coefficient of thermal expansion of polypropylene laminated paper (PPLP) used as an electrical insulating material in which the thermal-induced strain on HTS tapes and their insulation during cooling from room temperature to 77 K could significantly impact the critical current (Ic) of coated conductor (CC) tapes [16].

This study aims to explore the differences in thermal expansion and mechanical properties of two types of 3D-printed polymer samples: PLA using FDM and photopolymer using DLP. The uniaxial tensile test will be conducted at both room temperature and cryogenic temperatures.

2. EXPERIMENTAL PROCEDURES

2.1. Samples

Commercially available photopolymer resin and polylactic acid filaments were selected for tensile testing. Samples were produced using different 3D printer machines and conditions, resulting in dog-bone shaped specimens. The specimen geometries manufactured by 3D printing methods are shown in Figure 1. The average thickness and width of polylactic acid are 3.3 mm and 6.3 mm, respectively, while the photopolymer has an average thickness of 3.4 mm and width of 6.1 mm. The printing conditions and sample specifications are detailed in Table 1. For the photopolymer resin specimen, the digital light DLP method was used with a layer thickness of 50 um and an infill ratio of 100%. Nova3D's SRAR0-M10-a931 photocurable resin was used as material. To improve the degree of curing, it was washed with ethanol for 30 minutes and then additional curing was performed for 15 minutes using UV rays. For polylactic acid, FDM was employed, it was processed with a layer thickness of 0.2 mm at an infill ratio of 95%. The nozzle temperature set to 190 ºC, the build plate temperature set to 60 ºC, and the nozzle and cooling fan rotation speed set to 60 mm/sec and 100%, respectively. The infill pattern used was a rectilinear pattern, which intersects orthogonally with each layer. As shown in Fig.1, the build orientation was set so that the layers are stacked in the thickness direction of the specimen. Additionally, the raster angle was set to 45 °.

2.2. Set-up for thermal expansion measurement at 77 K

The thermal expansion of photopolymer and polylactic acid was measured at 77 K using the coefficient thermal expansion (CTE) determination method from our previous study using double extensometers [16]. To prevent the sample from moving during the measurement, the upper region of the sample was clamped with the gripper hung in the fixture as shown in figure 2(a). The Nyillas double extensometer was attached to the gauge length region of the sample to enable measuring the change of voltage (ΔV) when applying low temperature as shown in figure 2(b). The sample was subsequently submerged into the liquid nitrogen (LN2) as shown in figure 2(b). The measured

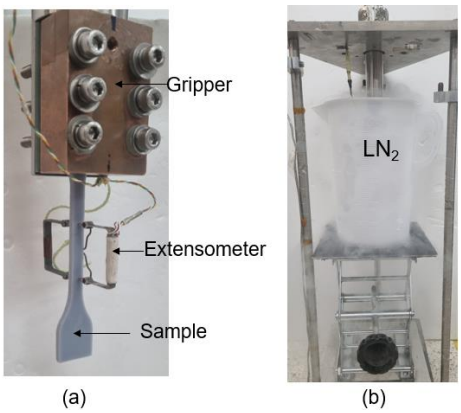

Fig. 2. (a) set-up of sample mounting with extensometer for CTE measurement, (b) Appearance of set-up submerged into LN2 bath.

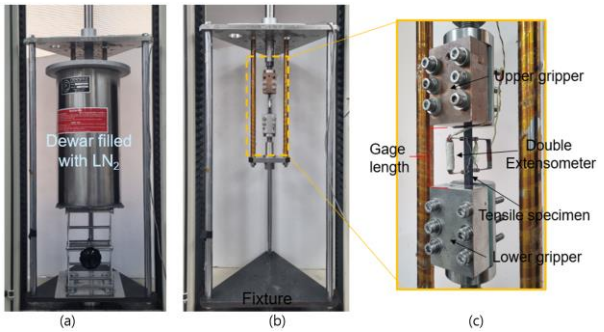

Fig. 3. Setup used for mechanical properties of 3D printed samples at (a) 77 K, (b) RT under uniaxial tensile load, and (c) magnified view of the double extensometer location on the specimen.

change in voltage due to the thermal contraction, which occurred at 77K, was used to determine the coefficient of thermal expansion (α) as shown in Equation 1. CF@T, the calibration factor, was measured using double extensometers at specified temperatures (77 K). L@RT is the initial length at RT, corresponding to the gauge length of 25 mm adopted by the extensometer. ΔT is the difference between the final temperature (77 K) and the initial temperature (297 K). The thermal expansion of the samples was obtained using equation 2, where the change in sample length (ΔL) corresponds to change in voltage (ΔV) over the calibration factor of the strain gauge at 77 K (CF@T), the L@T, is the length at specified temperature (77 K). Thus, the thermal contraction of the sample at 77 K was calculated by the product of the coefficient of thermal expansion ($\varepsilon_{thermal}$) and change in temperature. The derived equation 1 and 2 in this study was adopted from J.W. Ekin [17].

$$\alpha = \frac{\Delta V}{CF@T(L@RT)\Delta T} \qquad Eq.1$$

$$\varepsilon_{thermal} = \frac{\Delta L}{L@RT} = \frac{L@RT - L@T}{L@RT} = \alpha \Delta T \qquad Eq.2$$

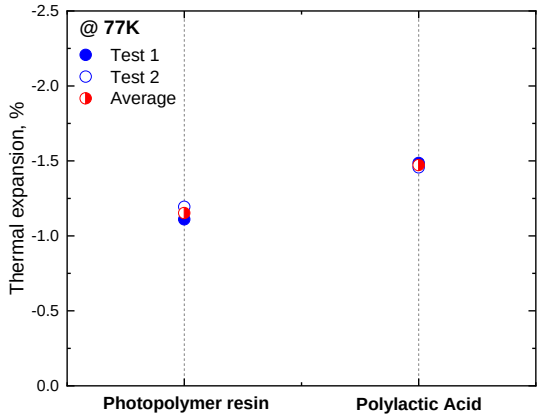

Fig. 4. Thermal expansion induced on the of photopolymer resin and polylactic acid by cooling down from RT to 77 K.

2.3. Test procedures for measuring mechanical property at RT and 77 K

Figure 3(a) and (b) shows the setup for uniaxial tension tests of dog-bone shape tensile specimen of photopolymer resin and polylactic acid at 77 K and RT respectively. Tensile testing was carried out based on the ASTM D638 standard [18]. Gripping blocks were utilized to fix the upper and lower regions of the sample to upper/lower grips at both ends, maintaining a set gage length of 45.5 mm for both samples as shown in figure 3(c). Tensile loading was performed using a universal material testing machine (Shimadzu AG-IS, load cell capacity: 5 kN) at a constant crosshead speed of 1 mm/min. Nyilas-type double extensometers with a gauge length of 25 mm were positioned at the central part of the sample to measure the induced strain during the tension test as shown in figure 3c [19]. These extensometers were directly linked to the signal conditioner (Kyowa, CDV-700, sampling rate: 500 kHz). For testing at 77 K, the sample was immersed in a liquid nitrogen (LN2) bath and allowed to equilibrate thermally for 10 minutes before applying the tensile load. The mechanical properties, such as elastic modulus and yield strength, were determined based on the stress-strain curves obtained using the double extensometers.

3. RESULTS AND DISCUSSION

3.1. Thermal contraction at 77 K

Figure 4 presents the thermal expansion (%) results for photopolymer resin and polylactic acid at 77 K. Two tests were conducted in each sample, denoted by close and open blue symbol and red symbol representing an average value. To calculate the voltage signal difference between room temperature (RT) and 77 K using a double extensometer, the voltage signal was initially set to 0 V at RT, and the change in voltage signal upon submersion in LN2 was utilized as the final voltage. This approach allowed for the determination of the thermal coefficient using Equation 1. The polylactic acid specimens exhibited a higher average voltage signal output of -0.509 V compared to the photopolymer resin's average of -0.3985 V, indicating that

TABLE 2
MATERIAL PROPERTIES OF POLYLACTIC ACID AND PHOTOPOLYMER RESIN OBTAINED BY UNIAXIAL TENSILE TEST AT RT AND 77 K.

Temperature (K)	Test No.	E [GPa]	σ_y [MPa]	σ_{uts} [MPa]	δ [%]
		Polylactic acid			
300 K	Test 1	2.34	30	34	3.9
	Test 2	2.28	26	30	3.3
	Average	**2.31**	**28**	**32**	**3.6**
77 K	Test 1	5.4	43	80	3.6
	Test 2	5.3	44	72	3.1
	Average	**5.35**	**43.5**	**76**	**3.35**
		Photopolymer resin			
300 K	Test 1	2.37	26	40	8
	Test 2	2.37	25	38	9
	Average	**2.37**	**25.5**	**39**	**8.5**

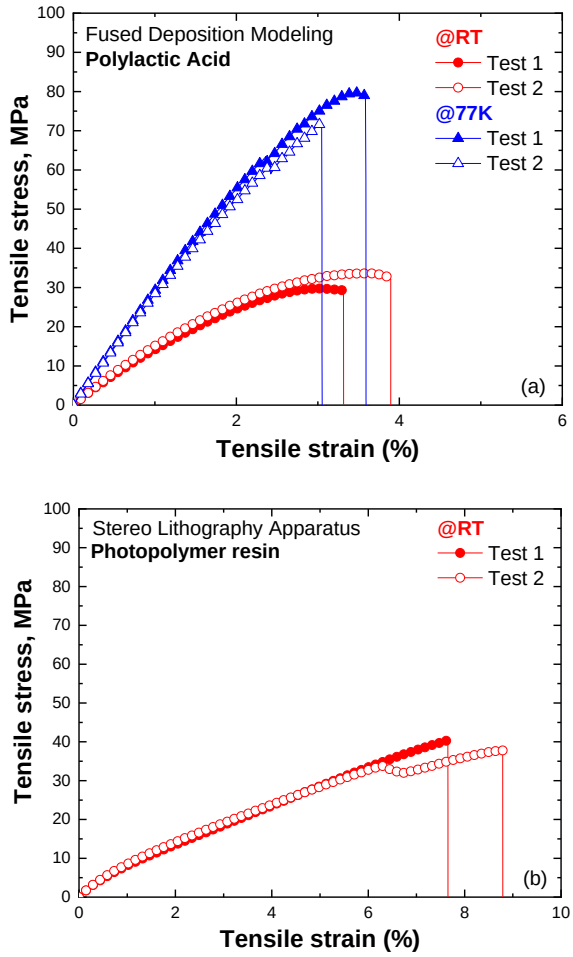

Fig. 5. (a) Stress-strain curves of polylactic acid obtained at RT and 77 K (b) Photopolymer resin at RT.

polylactic acid has a greater thermal coefficient with the average value of 6.6888 x 10^{-5} K^{-1} than photopolymer resin which obtained average value of 5.23677 x 10^{-5} K^{-1}. Consequently, by applying Equation 2, the thermal expansion ($\varepsilon_{thermal}$, %) values for each material were derived. The obtained $\varepsilon_{thermal}$ of polylactic acid and photopolymer were -1.472 % and -1.152 % respectively, the negative value shows that all samples exhibit thermal contraction at lower temperature of 77 K, showing the polylactic acid sample exhibiting approximately 22% higher thermal contraction than photopolymer resin. The thermal expansion of the photopolymer and polylactic acid was comparable to various polymers reported in Ekins' study [17]. The photopolymer resin exhibited thermal expansion behavior similar to PMMA (Plexiglass) and epoxy, with values of -1.0941% and -1.028%, respectively. Polylactic acid demonstrated thermal expansion comparable to polyamide (nylon) at -1.256% and TFE (Teflon) at -1.941%. In addition, the CTE of polylactic acid and photopolymer resin were 41% and 24% higher, respectively, than that of PPLP in the previous study [16].

3.2. Mechanical properties of samples at RT and 77 K

To assess the impact of cryogenic temperatures, we compared the tensile properties of 3D printed PLA and photopolymer resin at room temperature (RT) and 77 K. The measured material properties are summarized in Table 2, where E, σ_y, σ_{uts}, and δ represent Young's modulus, yield stress, ultimate tensile stress, and strain, respectively. Figure 5(a) presents the stress-strain curves for PLA at RT and 77 K. At RT, the PLA exhibited an average elastic modulus of 2.31 GPa, with yield and ultimate tensile strengths of approximately 28-32 MPa (similar values). However, at 77 K, the modulus of elasticity, yield strength, and ultimate tensile strength all increased significantly by 57%, 36%, and 58%, respectively. Notably, the elongation remained constant at around 3.5%. This behavior suggests strain hardening at the cryogenic temperature, consistent with observations by Y. Liu et al. study using a cryotensile device [12]. On the other hand, Figure 5(b) displays the stress-strain curve for the photopolymer resin at RT. Its elastic modulus (around 2.3 GPa) was comparable to PLA, while the yield strength was slightly higher (approximately 8% difference). However, the photopolymer displayed superior ultimate tensile strength and elongation, exceeding PLA by 18% and 58%, respectively, indicating its stronger mechanical properties at room temperature. However, at 77 K, the photopolymer exhibited a brittle response, fracturing in the gripper region upon exposure to LN2 temperatures. This suggests the material's inability to

withstand the force exerted by the gripper due to thermal contraction. Consequently, the tensile test could not be performed at 77 K because failure occurred before applying the tensile load. This finding indicates that the photopolymer resin is not suitable for cryogenic environments.

4. CONCLUSIONS

In this study, the coefficient of thermal expansion and mechanical properties of two distinct 3D printed polymers, polylactic acid (PLA) and photopolymer resin, were assessed via uniaxial tensile testing at both room temperature and a cryogenic temperature of 77 K. Results showed that while PLA exhibits higher thermal contraction than photopolymer resin at 77 K, it maintains superior mechanical integrity compared to the resin, which becomes brittle upon exposure to LN2. Conversely, at room temperature, the photopolymer resin demonstrated higher strength and ductility despite exhibiting similar modulus of elasticity to PLA. These findings suggest opposing behavior of the materials at different temperatures. Given its robust mechanical properties at cryogenic temperatures, PLA is particularly promising for cryogenic applications, such as the on-site production of auxiliary tools and support equipment. This capability allows for immediate, tailor-made solutions for the maintenance and operation of cryogenic systems, enhancing efficiency and adaptability in critical environments.

ACKNOWLEDGMENT

This work was supported by a grant from 2023 Research Fund of Andong National University.

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