Polymer 145 (2018) 88–94

Contents lists available at ScienceDirect

Polymer

journal homepage: www.elsevier.com/locate/polymer

Experimental investigation of mechanical properties of UV-Curable 3D printing materials

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ARTICLE INFO

Article history:
Received 5 February 2018
Received in revised form
16 April 2018
Accepted 26 April 2018
Available online 30 April 2018

Keywords:
Additive manufacturing
3D printing
Mechanical properties
Photopolymers
Build directions
Anisotropy
Electron beam

ABSTRACT

More recently, three dimensional printing (3D Printing), also known as an additive manufacturing (AM), has been highlighted since it shows a great promise to realize almost any three dimensional parts or structures with computer aided design (CAD). Several different processes are available for 3D printing, which includes fused deposition modeling, selective laser sintering, stereolithography, photopolymerization, and etc. In particular, considerable attention is paid to the 3D printing technique with photopolymerization due to their high resolutions. Unfortunately, the 3D printed products with photopolymerization however possess poor mechanical properties. Understanding of this should be necessary for the advantages of the 3D printing to be fully realized. Here, this study experimentally investigates the mechanical properties of the 3D printed photopolymer through thermomechanical analysis and tensile testing. In this study, it is found that the printed specimens are not fully cured after the 3D printing with photopolymerization. DiBenedetto equation is employed to better understand the relationship between the curing status and tensile properties. In addition to the poor mechanical properties, anisotropic and size dependent tensile properties of the 3D printed photopolymers are also observed. Electron beam treatment is used to ensure the cure of the 3D printed photopolymer and the corresponding tensile properties are characterized and investigated.

1. Introduction

Three Dimensional (3D) printing, also known as an Additive Manufacturing (AM) is one of emerging technologies in our society. Very recently, it has attracted tremendous attention from various industries and academic societies with its great potential to improve traditional manufacturing process in an efficient and economical way as well as to create fundamentally and conceptually new designed products. 3D printing, unlike conventional subtractive manufacturing processes, takes a bottom-up process to create 3 dimensional tangible parts from 3D model data, designed by Computer aided Design (CAD) [1]. This technology could be employed in a wide range of engineering applications, including do-it-yourself 3D printing, tissue engineering, materials for energy, food processing, microfluidics and low-density, high-strength composite materials [2–5].

Since 3D printing technique builds a product by a layer upon layer process [1], it can be expected that the material properties of 3D printed products would depend on the printing parameters

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https://doi.org/10.1016/j.polymer.2018.04.067
0032-3861/

such as ambient temperature, resolution, layer thickness, geometries and building directions [4,6–8]. C.S. Lee et al. [6] and S.H. Ahn et al. [7] experimentally investigated anisotropic mechanical behaviors of 3D printed parts and Tymrak B. M [9] et al. studied the effects of layer thickness and deposition patterns on mechanical properties in fused deposion modeling (FDM) type 3D printers.

Ultraviolet (UV)/laser light curable photopolymers are often chosen as one of the most commonly used 3D printing techniques for Digital Light Processing, Stereolithography Apparatus and Polyjet type 3D printing systems, which can allow for both high resolution and printing speed. Typically, photopolymers are light sensitive thermoset polymeric materials. They can transform their phase from liquid to solid state when exposed to UV light or Laser, whereas unexposed UV or laser-curable polymer region still remains liquidized state and often undesirably unsolidified [10]. Thanks to the nature of photopolymers, it can provide lower viscosity for much easier printing process, and fast curing reactions, which are suitable for 3D printing manufacturing process. Furthermore, high resolution and high fidelity of the products in all 3 dimensional directions can be achieved with such 3D printing systems by precisely manipulating optical sources [2,10,11]. Although it seems to be promising, however, the poor mechanical properties [12,13] of the 3D printed photopolymers can significantly limit its potential only to a few applications including figures or secondary structural parts.

Here, this study investigates the mechanical properties of the 3D printed photopolymers by using Polyjet type 3D printing technique. We report that from the tensile testing results the anisotropic properties of the printed photopolymers are observed with respect to the printing orientation, while the geometry dependent tensile properties are measured by varying its thickness of the specimen. The observed “poor” tensile properties are interpreted by characterizing the thermal properties of the printed photopolymers. This poor properties seem to be mainly attributed to insufficiently cured 3D printed photopolymers. The Electron Beam treatment is employed to ensure full cure of the 3D printed polymers and the mechanical properties are characterized and compared.

2. Experimental

2.1. Material

Material of interest in this study is 3D printable UV-curable resin, which is composed of Acrylic monomer/Isobornyl acrylate/Phenol, 4,4'-(1-methylethylidene) bis-, polymer with (chloromethyl) oxirane, 2-propenoate/Diphenyl-2,4,6-trimethylbenzoyl phosphine oxide/Titanium dioxide/Acrylic acid ester/Propylene glycol monomethyl ether acetate/Phosphoric acid (Israel). All tensile test specimens and Differential Photocalorimetry (DPC) samples were fabricated and prepared with this chosen material.

2.2. Sample preparation with 3D printing

According to ASTM D638 V, tensile specimens were printed with the commercially available Polyjet type 3D printer, Objet350 Connex (Stratasys Inc, Israel). 3D CAD models were generated by Autodesk 123D Design. In Polyjet type 3D printing, the each specimen is printed by three main processes. Firstly, in pre-processing step built-in software automatically calculates the placement of photopolymers and support materials from 3D CAD file. In printing step, the model and support materials, are jetted onto a build tray and cured immediately by the UV-light to realize 3D products. After printing step the final samples were obtained by removal of supporting materials.

2.3. Mechanical analysis

The tensile specimens with five different thicknesses 0.5, 0.8, 1.0, 3.0, and 5.0 mm are printed, respectively. In particular, the specimens with 0.8 mm in thickness printed in two different printing directions, longitudinal and transverse to the length of the tensile specimen, respectively. The tensile properties are measured with Instron Electro pulse E3000 (Instron Ltd, High Wycombe, UK). The uniaxial tensile tests were conducted under rate of 1 mm/min with the 3 kN load cell. At least 8 samples for each thickness and direction were prepared and characterized.

2.4. Thermal analysis

Curing behaviors and glass transition temperatures (T_g) of the photopolymers (Objet VeroWhiteplus RGD835) prior to 3D printing were characterized with Differential Photocalorimetry (DPC) technique. Compared to traditional Differential Scanning Calorimeter (DSC) technique, DPC is designed to detect enthalpy change (ΔH) of materials by using a UV generator. The DPC measurements were conducted to confirm ΔH of curing reaction of the photopolymer with UV generator (Photocalorimetry Accessory, PCA). The DSC-Q1000 (TA Instrument Co., Delaware, USA) was used for both thermos-mechanical characterization. The DPC measurements were conducted under isothermal ambient conditions at −10.0, 0, 10, 25, 40, 50, 60, and 70 °C, respectively. The average intensity of UV light source was selected to be 66 mW/cm^2. Then, dynamic DSC measurements were performed to determine glass transition temperature (T_g) of each sample.

2.5. Electron beam irradiation

Post to the printing, to ensure full cure, the electron beam treatment on 3D printed specimens was applied. A cart type electron beam accelerator, whose width was 110 cm and conveyor velocity was 10 m/min, were used. Electron beam with 8.5 mA of beam current and 2.5 MeV of beam energy was irradiated.

3. Results and discussion

3.1. Mechanical characterization

The tensile test results and the values provided from the manufacturer are compared along with the corresponding properties of Epon 828®, which is briefly summarized in Table 1. Note that Epon 828® is a commonly used an epoxy polymer. The maximum measured value for each tensile property is taken from the test results of the printed specimens with 5 mm in thickness. It is noted that the specimens with 5 mm in thickness exhibit the greatest value for each tensile property among all the specimens with each thickness. Although the manufacturer's provided values are found to be quite close to those of Epon828®, the tensile properties of the printed specimens investigated in this study are measured to be up to 45% less than the provided values for all three of tensile strength, Young's modulus, and elongation at break. This unexpected poor mechanical properties are studied and discussed in details in the section of Thermal Characterization in this paper.

In order to investigate anisotropic behaviors of the 3D printed specimen, the tensile specimens with 0.8 mm in thickness are chosen to be built in two different printing directions, which are longitudinal and transverse direction to the length of the specimen, respectively. The schematic of both longitudinally and transversely printed tensile specimens is illustrated in Fig. 1 (a) and (b) and their optical images at the magnification of x 500 are shown in Fig. 1. (c) and (d) respectively. The tensile testing results are presented in

Table 1
Comparison of manufacturer provided and experimental tensile properties along with corresponding properties with Epon 828®.

Mechanical Properties of OBJET VeroWhitePlus RGD835			
Properties	Manufacturer Provided value	Experimental value	Epon 828®
Tensile strength (MPa)	55–60	32	69
Young's modulus (MPa)	2000–3000	1434	2750
Elongation at break (%)	10–25	60	10
Isotropy	Not mentioned	Anisotropic	Isotropic

Fig. 2. It was clearly observed that the printed specimens exhibit anisotropic characteristics in their tensile behavior (Fig. 2(c) and (d)). Slight difference (less than 4.3%) in Young's modulus and the tensile strength is observed between the longitudinally and transversely printed specimens (Fig. 2(a) and (b)). In a sharp contrast, the tensile toughness of the longitudinal specimens, which is measured by about 3.28 MPa, has more than 300% greater than one (0.99 MPa) of the transverse specimens. Correspondingly, the longitudinal specimens also exhibit 300% greater elongation at break (0.32) over the transverse specimens (0.10) as seen in Fig. 2(c) and (d). Expectedly, this observed anisotropy of the printed specimens can be attributed to the nature of the 3D printing of UV curable photopolymers, which creates layers by orthogonal movement of printing heads. These behaviors could be analogous to ones of unidirectional continuous fiber reinforced composites, which are inherently anisotropic and designed to have far much higher strength/stiffness in longitudinal than ones in transverse direction under external loadings [14].

According to the definitions of stress and strain, the tensile properties of any materials are supposed to be independent of the geometry and dimensions of test specimens. However, it can be often found that the greater size of a material can have more chance to possess defects or voids, thereby providing poorer mechanical properties. That is why many materials are often much stronger and stiffer in fiber form than they are in bulk form [14].

Unexpectedly, however, it is interesting noted that the 3D printed photopolymer showed tendency of increasing in Young's modulus (Fig. 3 (a)) and tensile strength (Fig. 3 (b)) with the increase in their thickness. The significant differences, in Young's modulus and tensile strength with respect to thickness were statistically confirmed by using analysis of variance (ANOVA) tests (P-values of ANOVA were 9.09e-11 and 7.06e-11 respectively). In fact, it is believed that those intriguing results are associated, at least in part, to the nature of the photopolymer 3D printing technique, which builds designed objects in a layer-by-layer stacking process [15]. Upon printing a new layer, the extra UV-light can be exposed to pre-printed layers and this extra UV-irradiation could promote to further polymerization of residual unreacted monomers *i.e.* post-cure. The irradiation time of UV-light (T_{irr}) might play a key contributing factor to the size dependent tensile properties observed in this study, since the curing degree (α) of the photopolymers highly depends on T_{irr} under constant UV intensity at ambient temperature condition. Here, the Differential Photocalorimetry (DPC) measurement were conducted to study the dependence of T_{irr} on the curing behaviors of the photopolymer investigated.

The curing behaviors of the photopolymer were monitored by the use of DPC technique, under the given condition of ambient temperature and constant UV irradiation intensity, 70 °C and 66 mW/cm [2], respectively. The curing degree of the photopolymer with respect to irradiation time (T_{irr}) was characterized with kinetic model, as seen in Fig. 4. A previous study already showed that curing reactions followed step polymerization and it can be expressed with simple autocatalytic Equation (1) (plotted as dash line in Fig. 4.), where α indicates curing degree and t is time, $k = 10.0 \pm 0.06$, $m = 0.52 \pm 0.02$ and $n = 1.65 \pm 0.05$ [16]. The results show that the curing degree is proportionally increasing up to 90% for 10 s of the T_{irr}, and with the further irradiation time, the cure is gradually developed and fully completed at around 30 s of the UV-irradiation time to reach the full cure of the photopolymer.

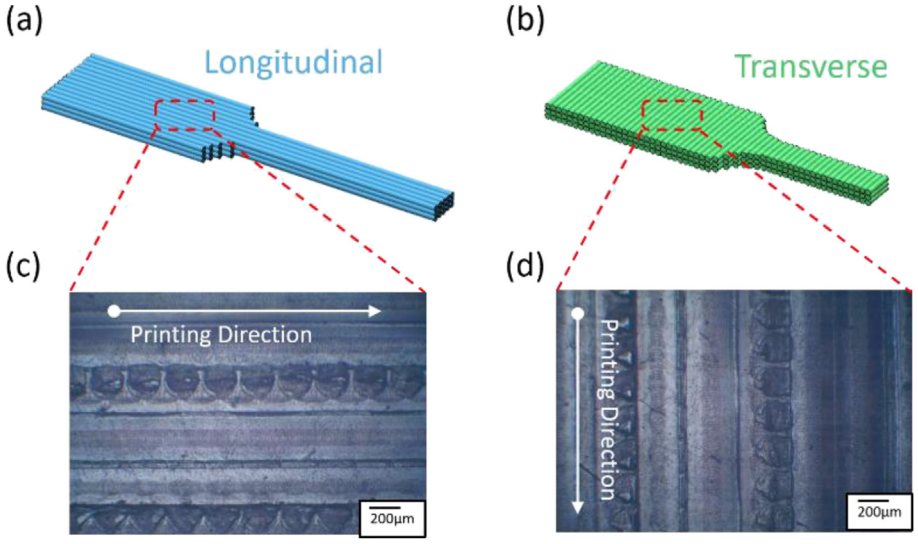

Fig. 1. Schematic illustration of (a) longitudinally printed and (b) transversely printed specimens followed by ASTM D638 V and optical microscopic images of (c) longitudinally printed and (d) transversely printed specimens at the magnification of x 500.

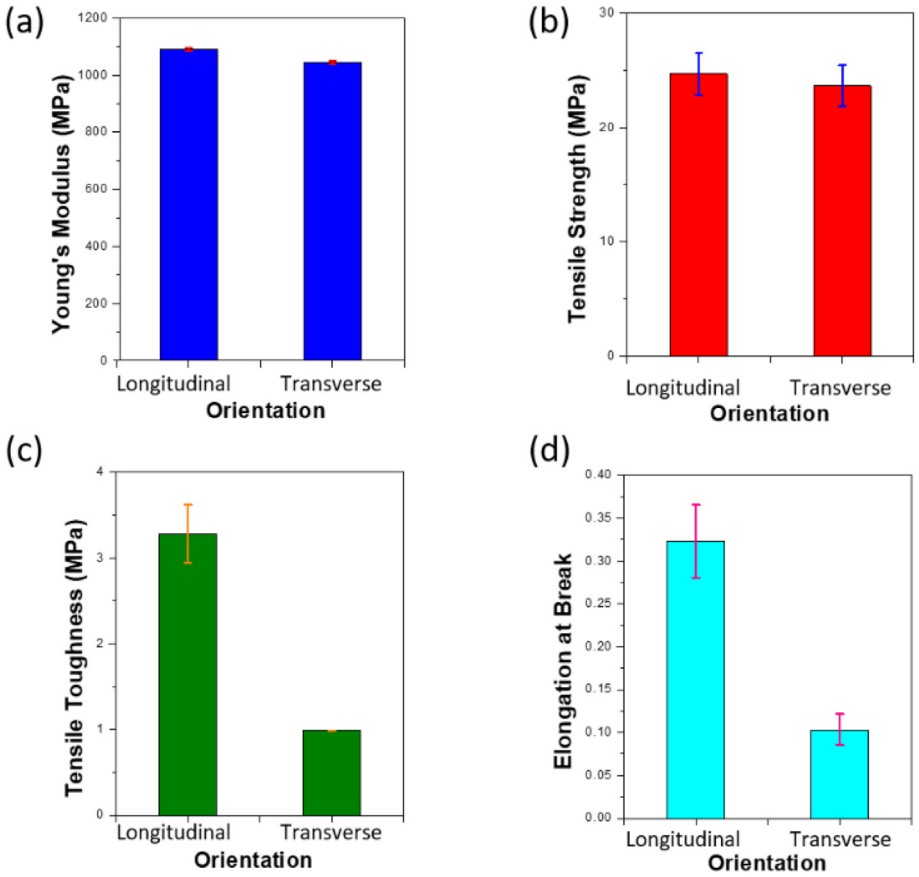

Fig. 2. The comparisons of the tensile test results of 3D printed specimens in longitudinal and transverse directions. (a) Young's modulus, (b) tensile strength, (c) tensile toughness, and (d) elongation at break.

$$\frac{d\alpha}{dt} = k\alpha^m(1-\alpha^n) \tag{1}$$

It can indicate that if the T_{irr} is not long enough provided in printing each layer, the "intrinsic" tensile properties of the printed photopolymers are not likely to be achieved due to the corresponding insufficient cure of the polymer. Furthermore, for the layer-by-layer stacking printing process, it could imply that more number of layers of the printed products would have higher cure degree over the less number of layers, thinner printed ones. This is because during the printing process, the more layers will have more chance to expose to the UV irradiation, which consequently leads to closer to the intrinsic mechanical properties. Therefore, it is important to know whether the printed polymer is fully cured or, if not, the cure degrees at a given condition.

3.2. Thermal characterization

In order to estimate the curing degree of the printed photopolymer, an empirical model which can predict the cure degree of the photopolymer with the glass transition temperatures was employed to investigate the cure degree with varying the ambient temperature. It is well-known that the glass transition temperature of thermoset polymers is strongly related to the cure degree. Subsequently, their material properties, including mechanical properties, of thermoset polymers mainly depend on degree of cure (α), because mobility of polymer can be decreased by increasing crosslinking density [17]. In this study, the correlation between T_g and curing degree were analyzed via the empirically derived DiBenedetto equation (Equation (2)), which considers entropic characteristics from monomer ($\alpha = 0$) to fully cured network ($\alpha = 1$) [17–19]:

$$\frac{T_g - T_0}{T_{g\infty} - T_{g0}} = \frac{\lambda\alpha}{1-(1-\lambda)\alpha} \tag{2}$$

where T_{g0} and $T_{g\infty}$ are the glass transition temperatures of the uncured resin ($\alpha = 0$) and fully cured resin ($\alpha = 1$) respectively, and λ indicates a structure-dependent parameter with value between 0 and 1.

3D printable photopolymer samples were cured on the different but isothermal condition (−10, 0, 25, 30, 40, 50, 60, 70 °C) of chamber equipped in DPC instrument with constant UV irradiation intensity of 66 mW/cm^2. The exothermic heat of reaction (ΔH) were detected to determine curing degree (α) in each temperature condition. The curing degree (α) of each sample was calculated by monitoring heat of reaction (ΔH_T) with following Equation (3), where the maximum heat of reaction (ΔH_{max}) is observed in 70 °C cured sample.

$$\alpha = 1 - \frac{\Delta H_T}{\Delta H_{max}} \tag{3}$$

After every specimen was cured through DPC, the glass transition temperature (T_g) of each specimen was measured by dynamic DSC, respectively. The one-to-one relationship with curing degree variation, from monomer (0%) to fully cured (100%), and corresponding glass transition temperatures (T_g) from the experimental results of DSC and DPC measurement are shown in Fig. 5.The

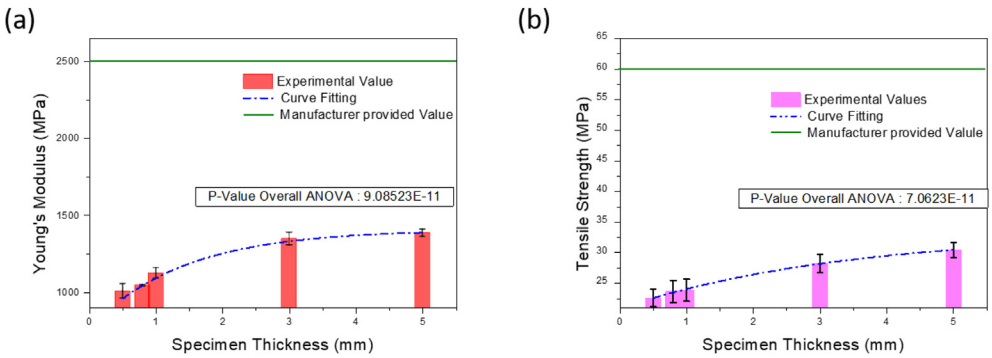

Fig. 3. Size dependently tensile properties and comparison with manufacturer provided values (a) Young's modulus and (b) Tensile strength.

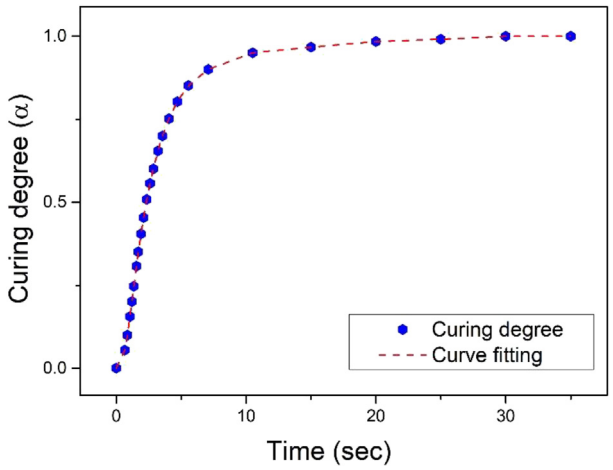

Fig. 4. Curing degree (α) as function of time (sec) and kinetic curve fitting.

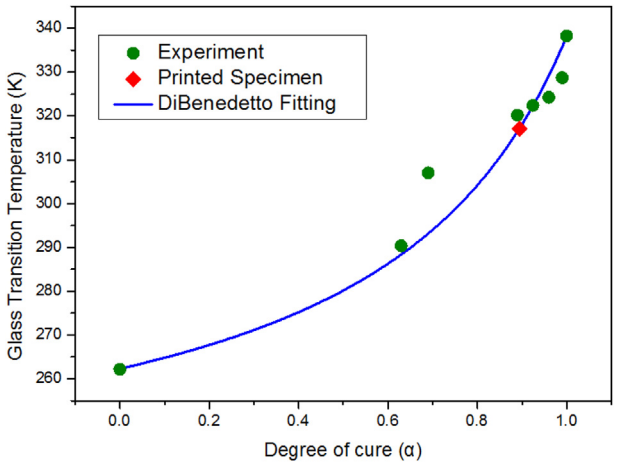

Fig. 5. The glass transition temperature (Tg) as a function of curing degree (α) for 3D printed photopolymer and DiBenedetto fitting.

DiBenedetto curve fitting were also plotted in Fig. 5. The maximum glass transition temperature ($T_{g\infty}$) and minimum glass transition temperature (T_{g0}) of the photopolymer are measured as 326.45 K and 262.31 K. In this analysis, the structure dependent parameter (λ) was determined as 0.39. By these works, the curing degree (α) of 3D printed specimen was estimated as 0.89 b y projecting glass transition temperature of 3D printed photopolymer on DiBenedetto fitting. Meanwhile, the DiBenedetto fitting shows a dramatic increase of glass transition temperature in the range between 0.9 and 1.0, which could be attributed to significant enhancement of mechanical properties. It seems that the critical curing degree to achieve intrinsic mechanical properties should be above 0.9 (90%). However, the 3D printed photopolymer showed less curing degree than the critical point of the polymer material investigated in this study. We believe that this under-cure phenomenon is one of the major factors to exhibit the observed poor mechanical properties of the 3D printed specimens (Fig. 6).

3.3. Electron beam treatment results

The extra UV-light treatment by DPC instrument with an elevated temperatures from room temperature to 80 °C, was performed to post-cure of 3D printed photopolymer. However, the results showed neither changes in ΔH nor in glass transition temperature. It is suspicious that the 3D printed photopolymer is already vitrified and the extra UV-light exposure cannot be sufficient way to achieve post-cure. With increasing in conversion of monomers, the polymerization medium becomes more viscous and polymer radicals become more crowded and entangled with already reacted oligomers or polymers, which leads to decrease in segmental mobility and to hinder further conversion of unreacted monomers, the vitrification effect. Typically, to overcome this vitrification effect, *i.e.*, post-cure, thermal treatment over glass transition temperature is applied to stimulate unreacted monomers. Several studies reported and emphasized the significant improvement in their thermal and mechanical properties of thermoset polymers through post-cure [20–23]. However, the UV-cured photopolymers seem to need other post cure treatments to promote to further conversion for the full cure. In this study, the electron beam treatment was chosen to investigate the cure characteristics in order to ensure the full cure of the printed photopolymers.

As the same manner of curing reactions, the post curing mechanism also initiates with the breaking of a chemical bond. In UV curing process, since the UV photons have lower energy (~3.5 eV) compared to bond energy of alkene (~6.5 eV) in acrylates, the existence of photoinitiators is necessary to activate monomers. In contrast, the energy of electron beam is high enough (70 keV) to activate double bond of acrylates in photopolymer without the help from photoinitiator [10].

The effects of electron beam treatment on tensile properties of 3D printed specimens were shown in Fig. 7. The Young's modulus and tensile strength with respects to specimen thickness and irradiation dose of electron beam were compared as seen in Fig. 7 (a) and (b). A remarkable increase in Young's modulus and tensile strength is observed from 100 kGy to 700 kGy in irradiation dose

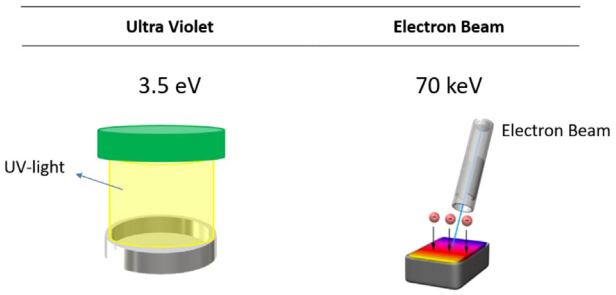

Fig. 6. Comparison of energy levels with UV-light and Electron Beam.

investigated in this study. Importantly, it should be noted here that the effect of size and printing direction dependence on the mechanical properties clearly disappear after the electron beam treatment, showing Young's modulus and tensile strength of 2.21 GPa and 58.84 MPa, respectively (Fig. 7 (c) and (d)). The P-value of Levene's test confirmed that there were no significant differences in both Young's modulus (P-value = 0.37) and the tensile strength (P-value = 0.752) of the photopolymers with the post cure treatment.

To study the effect of the electron beam treatment under 100 kGy in irradiation dose, the 3 mm thick specimens were exposed to irradiation by varying electron beam irradiation dose from 0 kGy to 100 kGy. The tensile properties are characterized and compared in Fig. 8. A sudden increase in Young's modulus (156%) and tensile strength (207%) was observed at 20 kGy irradiation dose over the specimens at 0 kGy irradiation dose (without the electron beam treatment) as seen in Fig. 8. The maximum values in Young's modulus (2.40 GPa) and tensile modulus (65.25 MPa) are measured at 300 kGy dose, which are up to 80% and 108% compared to manufacturer's provided values, respectively. In addition, the gradual increase in the tensile properties with respect to the irradiation dose between 20 and 300 kGy dose is found while a slight decrease in the properties beyond 300 kGy is seen. This could indicate that the post-cure reaction in the printed photopolymers is progressively developed with the increase of irradiation dose until 300 kG y, and with the greater than that, however, the polymers could be degraded in their mechanical properties.

4. Conclusion

In this study, the mechanical properties of 3D printed photopolymers are investigated, and unexpectedly, poor mechanical properties along with their anisotropic as well as size dependent behaviors are found. The anisotropic properties are clearly seen in tensile toughness and elongation at break with respect to a printing direction, while it is not seen in their Young's modulus and tensile strength. It is found in the tensile test results that the longitudinally printed specimens exhibit 300% greater tensile toughness than one of the transversely printed specimens. This could be analogous to unidirectional continuous fiber reinforced composites with much higher strength/stiffness in longitudinal than ones in transverse direction. With the increase in thickness of 3D printed photopolymer specimens, the increase in Young's modulus and tensile strength are observed and statistically confirmed. It might result from the nature of the 3D printing technique which builds a designed object in a layer-by-layer stacking process. For thicker specimens, upon printing a subsequent layer, the corresponding extra UV-light exposure to the previously printed layers will promote to further polymerization of residual unreacted monomers. The isothermal Differential Photocalorimetery (DPC) and dynamic Differential Scanning Calorimeter (DSC) measurements can confirm that the curing conditions, such as ambient temperature and irradiation time, are the key factors to determine the observed mechanical properties of the 3D printed photopolymers. By employing DiBenedetto equation, the curing degree of the 3D photopolymers at a given printing condition chosen in this study was estimated to be about 89.3%. It also indicates that the critical point of curing degree is found to be above 90% to achieve intrinsic tensile properties of the polymers that are supposed to possess. Furthermore, electron beam treatment was applied as a post-cure treatment in

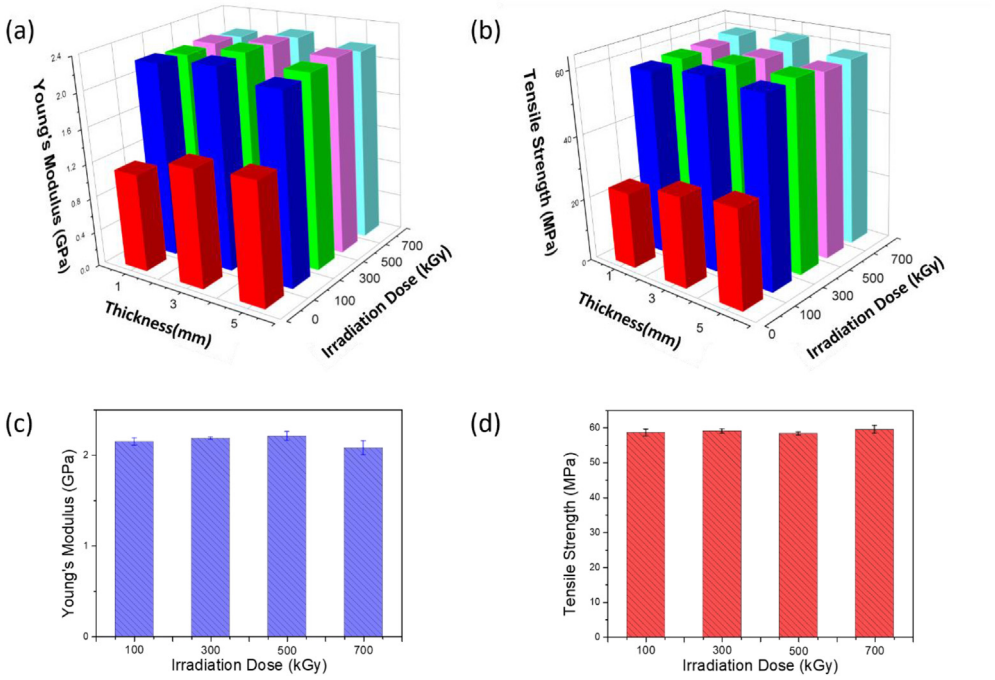

Fig. 7. The Electron Beam Treatment Effects on tensile properties of 3D printed photopolymer.

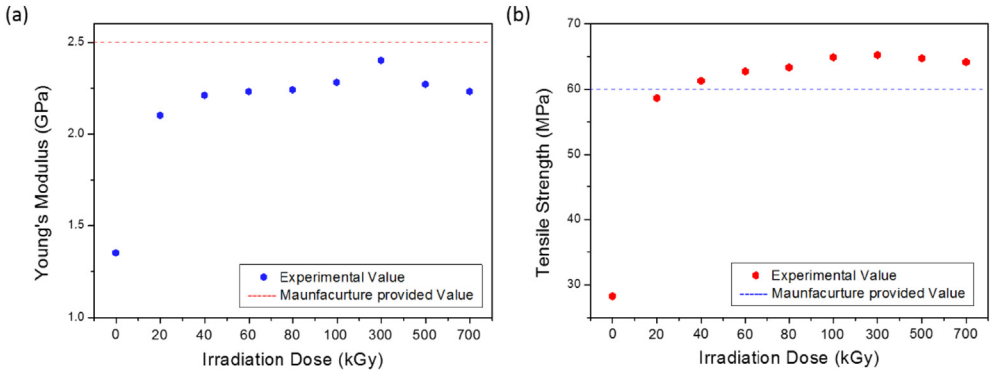

Fig. 8. The Electron Beam Treatment Effect on 3 mm thick specimens, a) Young's modulus and (b) Tensile strength.

order to ensure full cure of the 3D printed photopolymers. With the test results, the dramatic enhancement of mechanical properties at the 300 kGy electron beam dose were observed, showing 2.4 GPa of Young's Modulus and 65.23 MPa of tensile strength, which are close to the properties of epoxy polymers commonly used. It should be also noted that the size dependent and anisotropic behaviors also disappear in the tensile properties after the electron beam treatment. This work reports that it is critically important to understand the thermo-mechanical behaviors of 3D printing materials, particularly photopolymers for the use of the emerging manufacturing technique, 3D printing, in a wide variety of engineering applications. Otherwise, it limits only to prototyping or mock-up design.

Acknowledgment

This work is supported by the “Development of manufacturing technology of nuclear fuel assembly supporting structures and safety class 1 valve items based on 3D printing and standardizations” of the Korea Institute of Energy Technology Evaluation and Planning (KETEP) granted financial resource from the Ministry of Trade, Industry & Energy, Republic of Korea (No. 20171510102030) and National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP) (2018R1A2B2001565).

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