Dynamic Mechanical Analysis of 3D Printed PETG Material

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Abstract. 3D printing technology through fused filament fabrication has found various industrial applications in the field of rapid manufacturing to fabricate prototypes and concept models. However, being the most popular technology, fused filament fabrication requires understanding about the influence of the process parameters on resulting products. This investigation attempts to provide the terse behavior of the viscoelastic properties of fused filament fabrication processed with Poly Ethylene Terephthalate Glycol (PETG) samples considering the impact of fused filament fabrication process parameters. Dynamic mechanical analyzer is used to perform the dynamic mechanical analysis (DMA) and the dynamic response of fused filament fabrication PETG specimens is studied while they are subjected to dual cantilever loading under periodic stress. The fused filament fabrication process parameters such as, feed rate, layer thickness and infill density are considered. PETG parts are fabricated using 100% infill density at a feed rate of 50 mm/sec with three different layer thicknesses of 0.17 mm, 0.23 mm and 0.3 mm. DMA is performed with temperature ranging from room temperature to 130°C at five different frequency values of 1 Hz, 2 Hz, 5 Hz, 7 Hz & 10 Hz. The effect of process parameters of fused filament fabrication and frequencies on the viscoelastic properties of 3D printed PETG specimens is explored. The results revealed that, the storage module and loss module values are better for the specimens prepared with a layer thickness of 0.17 mm irrespective of the variation in the frequency values.

1. Introduction

Because of its low cost and accessibility, fused filament fabrication technique has dramatically enforced the reduction in the cost of printing and various open source designs reduced the cost of printing from thousands of dollars to hundreds. Due to which, the technology has brought...
into the field of academics and research [1]. 3D printing through fused filament fabrication technology is an ecological process as it consumes 90% of the material and creates near-net-shape components with less wastage. Also, this process doesn’t require extravagant molds for producing the complex and sophisticated parts [2]. In general, compression tests, model tests and nano-indentation tests are performed to characterize the 3D printed polyactic acid (PLA) with graphene nano-composites consists of 10% graphene (by weight) in the PLA matrix. Besides, the effects due to the incorporation of graphene on the strength, modulus and hardness of 3D printed nano-composites can be revealed by performing the mechanical tests. Also, the damping behavior which is examined through cyclic compression and modal tests has also increased with the incorporation of graphene compared with neat/normal PLA specimens [3]. However, the maximum thermal degradation temperature of PLA/graphene nano-composites has been decreased compared to the unfilled photopolymer. Instead, the storage module of actual part (G) and the glass transition temperature ($T_g$) have increased and has yielded to higher values for 20% (w/v) titanium oxide ($T_o$) due to the better reinforcing effect and dispersion [4].

Furthermore, infill density, print speed and layer height with the values of 80%, 60 mm/sec and 200 µ respectively have emerged as the significant machine process parameters. Optimized machine parameter values may be useful while identifying the working ranges to PETG 3D print material for various applications [5] & [6]. Mansour et al. [7] studied the dynamic and mechanical behavior of 3D printed polyethylene terephthalate glycol with carbon fibers reinforced by fused filament fabrication. The authors observed that, there is reduction in the values of compressive strain (as much as 66%) and the hardness and modulus values have increased by an amount of 27% & 30%, respectively, with the inclusion of carbon fibers. Besides, the values of loss factor and damping behavior values have decreased, which is evaluated from the cyclic compression and model tests [7]. Ferreira et al. [8] have performed experimental studies and observed that, there is an increase in the values of Young’s modulus (by 70.10%) with the addition of fibers, but, the stress with standing capacity has been lowered by an amount of 28.21%. Shuheng et al. [9] have investigated the effect of process parameters on the dynamic mechanical properties of fused filament fabricated PLA specimens while the temperature of the environment changes often. Doddamani [10] performed dynamic mechanical analysis on 3D printed high-density polyethylene (HDPE) composite and observed that, the storage module, loss module and damping behavior values have increased with the increase in the cenosphere content of a HDPE composite. Galeja et al. [11] studied the effect of raster angle on the static and dynamic properties of various acrylonitrile butadiene styrene (ABS) specimens and it was observed that, the angle of 55° has yielded for better durability and mechanical performance.

There exists a dearth of literature about the dynamic mechanical properties of Poly Ethylene Terephthalate Glycol (PETG) specimens processed by fused filament fabrication technique. This investigation attempts to explore the effect of fused filament fabrication process parameters such as, layer thickness, infill density and frequencies on the viscoelastic properties of 3D printed PETG specimens, namely loss module, storage module and damping behavior using DMA with the temperature variation from room temperature to 130°C. The feed rate is maintained constant (50mm/sec) throughout the process and the effect of frequency variation on the viscoelastic properties of PETG specimens is also investigated.

2. Materials and Methods
The experimental details and the various steps of methodology are described in this section.

2.1. filament Material
The specimens are fabricated using thermoplastic type Poly Ethylene Terephthalate Glycol (PETG) as the PETG is very strong, has less shrinkage and has remarkable layer adhesion
properties. The additional properties of PETG material at printing condition are specified in Table 1. Nozzle material selection is done based on the filament material. Nozzle made with brass is a favorable choice for PETG filament material. Later, the filament material is allowed to pass through an extruder which results in a thin molten state material which is coming out through the nozzle. This thin material forms thin layer and these thin layers are sandwiched over another to obtain the final form of the specimen. Similar procedure is followed while fabricating the complex specimens and a support filament material is required to support the structure.

2.2. Fabrication of Specimens

The test specimen is initially designed by using as per ASTM D4065 standard using ‘Solidworks’ software and this 3D part model is then saved in ‘.STL’ format. The process parameters are obtained through the slicing software (CURA ULTIMAKER). The parameters such as layer thickness, infill density, temperature of extruder, temperature of bed, feed rate, orientation of specimen etc., are obtained through the slicing software. Finally, the fused filament fabrication makes use of the G-code file to fabricate the ASTM standard specimens. The values of process parameters are:

- 3D printing technique : Fused filament fabrication
- Material used : PETG (Poly Ethylene Terephthalate Glycol)
- Operating temperature : 220°C-250°C
- Layer thickness : 0.17 mm, 0.23 mm & 0.3 mm
- Feed rate : 50mm/sec
- Infill density : 100%
- Extruder temperature : 235°C
- Bed temperature : 60°C

2.3. Dynamic Mechanical Analysis

Dynamic Mechanical Analysis (DMA) is performed to analyze and classify the materials and beneficial especially to analyze the viscous behavior of the polymers. Initially, stress (in the form of sinusoidal) is imposed and the value of applied sinusoidal stress is measured in a sample of the temperature material. Later, the rate of stress is modified which further yields to develop the variations in the damping values. Subsequently, the temperature at which the product transition happens from the region of glass to the region of rubber can be identified through DMA. In addition to that, the viscoelastic nature of the polymer can be examined through DMA, by examining the displacement (strain) resulted due to the resistance offered by the material against the applied sinusoidal force. This displacement of the viscous material can be completely deferred in relation to stress during the orthogonal phase of stress. While performing the DMA testing of elastic viscous polymers, various features can be retrieved during the occurrence of phase lag. Storage module measures the energy stored which corresponds to the elastic part and the loss of energy in the form of heat dissipation corresponds to the viscous part. The following experimental conditions are used to analyze the PETZ sample.

| Table 1. Properties of the PETG material |
|------------------------------------------|
| Filament diameter                       | 1.75 mm  |
| Density of the filament                 | 1.27 g/cc |
| Favorable working temperature           | 235°C    |
| Bed temperature to be maintained        | 60°C     |
Figure 1. Behaviour of storage module with layer thickness - at frequency of 1Hz

- Instrument used : Diamond DMA with a heating rate of 5°C/sec
- Temperature range : 25°C to 130°C
- Frequencies : 1, 2, 5, 7 & 10 Hz
- Deformation mode : 3-Point Bending
- Sample dimensions : 60x10x3 mm (ASTM D4065)

3. Results & Discussions

3.1. Storage module

The variation in the values of storage module (E') of PETG specimens produced by fused filament fabrication at a frequency of 1 Hz and with different values of layer thickness and temperature is displayed in Figure 1. It can be observed that, the storage module values of PETG specimens are inversely proportional to the increase in the layer thickness due to adhesion between the layers of the material. The value of storage module is found to be higher for the specimen having the layer thickness of 0.17 mm and less for the specimen having the layer thickness of 0.3 mm. Also, the storage module of the specimens decreases with the growth of temperature values. This can be due to the loss in the stiffness values of the material with the growth of temperatures. It is worth noting that, the specimen consisting of 0.17 mm layer height has resulted in the highest value of storage module, because of the better interface bonding when compared with the other specimens. Also, due to the molecular mobility at peak temperatures, the specimen consisting of 0.3 mm layer height has yielded to the less values of storage module. As the peak temperature doesn’t have much influence on the layer height, the storage module values are almost very near to each other. Besides, the variation in the storage module values against the temperatures at different frequency values are shown in Figure 2, 3 & 4. There exists a minute variation in the storage module values with the variation of frequencies for the specimen consisting of 0.17 mm layer height. Similar trends in the results are obtained for the other specimens having the layer heights of 0.23 mm and 0.3 mm.
3.2. Loss module

Loss module describes the maximum energy emitted by the PETG specimens during the deformation process. Loss module ($E''$) provides the response about the viscous materials which are resulted due to the molecular motion in the specimens. The variation in the values of loss module of PETG specimens produced by fused filament fabrication at a frequency of 1 Hz and with different values of layer thickness and temperature is illustrated in Figure 5. The peak value in the loss module curve corresponds to the glass transition temperature ($T_g$) of specimens. It can be observed that, the loss module values are increased with the growth in the temperature until $T_g$ and then decreased. The value of loss module is found to be higher for the specimen
Figure 4. Behaviour of storage module with frequency - at layer thickness of 0.3 mm

Figure 5. Behaviour of loss module with layer thickness - at frequency of 1 Hz

having the layer thickness of 0.17 mm followed by 0.23 mm and 0.3 mm. The peak value of loss module is found to be 0.293 GPa at the transition temperature of 71°C and this shows that the thermal stability of specimen made with 0.17 mm layer thickness is better than the other PETG specimens. In addition to that, the variation in the loss module values of specimens against the temperatures at different frequency values are shown in Figure 6, 7 & 8. There exists a considerable difference between the values of loss module of PETG specimens with the variation in the values of frequency. It can be observed that, there is a reduction in the values of loss module trend with the increase in the frequency values and the specimen at 1 Hz has registered the highest peak of loss module followed by 2 Hz, 5 Hz, 7 Hz and 10 Hz, respectively. This can be
3.3. Damping behavior

Damping behavior (Tan $\delta$) can be described as the ratio of loss module to the storage module and its value depends upon the adhesive nature of the layers of specimen. Higher damping behavior values result in poor adhesive nature of the material and less damping behavior values favor the load carrying capacity of the specimen. This can be due to the fact that, the strong due to the enhancement in the molecular mobility due to the reduction in the frequency values. Similar trends in the results are obtained for the other specimens having the layer heights of 0.23 mm and 0.3 mm.

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adhesive property may lead to reduce the mobility of polymer chain which in turn reduces the damping. The variation in the values of damping behavior ($\tan \delta$) of PETG specimens produced by fused filament fabrication at a frequency of 1 Hz and with different values of layer thickness and temperature is illustrated in Figure 9. It can be observed that, the damping behavior curve of specimen having the layer thickness of 0.3 mm has registered better damping behavior values. In contrast, specimen having the layer thickness of 0.17 mm has the less damping behavior values and thus it possess the better load carrying capacity. This can be attributed to the fact that, the strong adhesive property yields for an adequate transfer of stress. The peak of damping behavior ($\tan \delta$) curve provides the value of glass transition temperature ($T_g$) and those values

**Figure 8.** Behaviour of loss module with frequency - at layer thickness of 0.3 mm

**Figure 9.** Behaviour of damping with layer thickness - at frequency of 1Hz
are found to be $79^\circ$C, $76^\circ$C and $76^\circ$C for the layer thickness of 0.17 mm, 0.23 mm and 0.3 mm respectively, refer Figure 9. Besides, the glass transition temperature ($T_g$) values have shifted slightly and registered higher values for the case of specimen with the layer thickness of 0.3 mm. This could be due to the reduction in the molecular mobility of polymer chain by the high strength offered by the material due to the increase in the layer thickness. In addition to that, the variation in the damping behavior values of specimens against the temperatures at different frequency values are shown in Figure 10, 11 & 12.

Figure 10. Behaviour of damping with frequency - at layer thickness of 0.17 mm

Figure 11. Behaviour of damping with frequency - at layer thickness of 0.17 mm
4. Conclusions
The primary focus of the current investigation is to perform the dynamic mechanical analysis on the fused filament fabricated PETG specimens and to observe the effect of the process parameters on the viscoelastic properties of PETG specimens. The feed rate is maintained constant (50mm/sec) throughout the process and the effect of frequency variation on the viscoelastic properties of PETG specimens is also investigated. It is observed that, the PETG specimens fabricated with different layer thickness (0.17 mm, 0.23 mm & 0.3mm), the storage module values are gradually decreasing with the increase of temperatures. Whereas the values of loss module and damping behaviour values increase to some extent (up to glass transition temperature $T_g$) and then falls. Storage module and loss module values are maximum at 0.17mm layer thickness irrespective of frequency variance. Damping behaviour values are maximum for the specimen prepared with 0.3 mm layer thickness. Frequency shows minute effect on storage module, considerable effect on loss module as the loss module values are decreased with the increase in the frequency values. The glass transition temperature ($T_g$) value increases with the increase in frequency.

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