Experimental Evaluation of Additively Manufactured Continuous Fiber Reinforced Nylon Composites

M. Mohammadizadeh and I. Fidan

Abstract Continuous fiber reinforced additive manufacturing (CFRAM) is a promising fabrication technology with a wide range of potential applications in different industries. The potential applications of CFRAM components justify the need for investigation of their thermomechanical properties. In this study, Dynamic Mechanical Analysis (DMA), tensile, and thermal properties of CFRAM components was studied and the effect of fiber percentage on properties was investigated. Nylon was used as thermoplastic polymer matrix and carbon fiber (CF), and fiber glass (FG) as reinforcing agents. It is found that fiber reinforcement improves storage modulus, loss modulus, tensile strength, elastic modulus, thermal conductivity, and heat conduction of nylon. Scanning Electron Microscope (SEM) was used to study printing quality, fiber–matrix interface, and microstructure of composite. The final results in this research study present the basis for industrial applications of fiber reinforced thermoplastic polymers for industrial applications.

Keywords Additive manufacturing · Fiber reinforcement · Mechanical properties

Introduction

Continuous fiber reinforced additive manufacturing (CFRAM) has attracted much attention due to the wide range of potential engineering applications [1–4]. Using CFRAM, complex parts can be produced much easier, and with low cost [5, 6]. Among AM methods, Fused Filament Fabrication (FFF) has found a great position due to low cost, ease of use, and low wastage of materials [6, 7]. However, the main challenge facing FFF method is that mechanical properties of the printed parts are not

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sufficient for engineering applications [8]. Continuous fiber reinforcement is used to improve thermomechanical properties of 3D printed polymer materials [9, 10]. Using CFRAM technology, reinforcing fiber is added to the structure to improve the mechanical properties.

Due to the increasing number of industrial applications of CFRAM components, in recent years several research studies are conducted to investigate the thermal and mechanical properties of CFRAM components [8–11]. Researchers mostly focused on the investigation of tensile [12–14], strength [15, 16], inter-laminar strength [17], and fracture mechanism [17]. To best of our knowledge, a few number of research has been conducted on DMA analysis of CFRAM components. DMA results including storage modulus and loss modulus demonstrate viscoelastic behavior of materials under cyclic load and different temperatures. Storage modulus indicates energy stored in material, and represents an elastic portion of materials. Loss modulus represents viscous portion and indicates energy dissipation by material as heat and molecular movements. Tensile test is one of the most important tests to study mechanical properties of materials. Also, thermal analysis is important to understand thermal stability of material for engineering applications like the car parts under the hood.

Thermoplastic polymers due to the low cost, easy process, low density, and recyclability are being increasingly used for a number of industrial applications [18–21], while their thermomechanical properties are not generally good enough for engineering applications. Using continuous fiber reinforcement technology can improve thermomechanical properties of polymers while maintaining low density and processability. In this paper, nylon as a thermoplastic polymer was used as matrix and carbon fiber (CF) and fiberglass (FG) were used for fiber reinforcement. Storage modulus and loss modulus as two major DMA parameters were measured and the effect of fiber reinforcement was investigated. Tensile test was conducted to measure tensile strength and elastic modulus. Thermal analysis was conducted to study the effect of fiber reinforcement on thermal conductivity and heat capacity of specimens. SEM technique was used to further study microstructure of printed specimen, bonding strength, interfacial strength, and printing quality.

Materials and Methods

Filaments of nylon as matrix and filaments of carbon fiber (CF) and Fiberglass (FG) as reinforcement were purchased from Markforged. Filaments were placed in a Pelican box to prevent water absorption. Fibers were made of bundles infused with sizing agent. Fiber diameter for nylon was 1.75 mm, for Carbon fiber was 0.35 mm, and for fiberglass was 0.3 mm. A Markforged Mark Two machine was used for manufacturing the specimens. The printer has two separate printing heads for matrix and reinforcement filaments. The printing head temperature was 265–270 °C with a non-heated bed. Filaments are sliced layer by layer to complete the part according to the 3D CAD model. Other printing parameters are filling density of 50%, matrix fill pattern of rectangular, fiber fill type of isotropic, and fiber angle of 0°. Samples were prepared according to the test machine manufacturer and with different fiber layers and consequently different fiber contents of CF and FG.

DMA analysis was conducted using a TA Instrument tester model Q800. The tests were conducted in tensile mode with a default frequency of 1 Hz and temperature range of 30–150 °C. Storage and loss modulus of specimens were measured. Tensile test was conducted in accordance with the American Standard Test Model (ASTM) D638, standard test method for tensile properties of plastics. Tensile specimens were tested using INSTRON testing machine. The testing variables were all controlled and input through Bluhill interface. For tensile testing, the speed of the test was 5 mm/min according to the standard. Thermal conductivity of specimens was measured using a KD2 Pro thermal analyzer.

Results and Discussion

DMA analysis was conducted to study viscoelastic behavior of 3D printed composite parts under dynamic load. Our previous research showed that CFRAM components present viscoelastic behavior [9]. Storage modulus of nylon and fiber reinforced nylon in temperature range of 30–150 °C is shown in Fig. 1. Results demonstrate that CF inclusion improves the storage modulus of nylon 46 times at room temperature. Enhancement of storage modulus is observed for all temperature range, while as temperature increases, storage modulus decreases gradually. As can be seen in Fig. 1, storage modulus reduces as the temperature increases. As the temperature increases material passes glass transition temperature (Tg) and the behavior of polymer changes from glassy to rubbery, cause polymer chains to gain enough energy

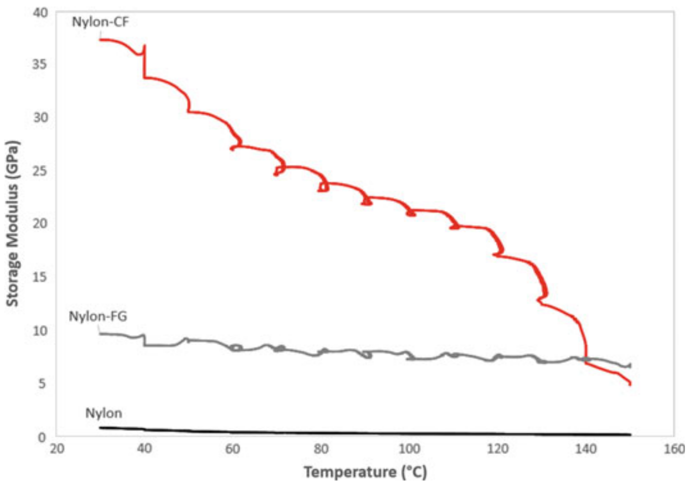

Fig. 1 Effect of carbon fiber reinforcement of storage modulus of nylon at different temperatures

Table 1 Effect of fiber reinforcement on storage modulus at room temperature

Material	Storage modulus (Gpa) At room temperature	Loss modulus (Mpa) At room temperature
Nylon	0.81	120.45
Nylon-FG	9.64	510.67
Nylon-CF	37.34	5263.29

to move. Increasing the temperature cause loosening chemical bonding of polymer matrix at temperatures higher than Tg of polymer, and loosening interface between the fiber and matrix [18].

Table 1 demonstrates the effect of fiber reinforcement by CF and FG on storage modulus and loss modulus of nylon specimen. As can be seen, inclusion of CF and FG improves storage modulus of nylon matrix by 12 and 64 times at room temperature, respectively. This can be explained by the reinforcing effect of the fibers leading to increased stiffness of the matrix [22]. This enhancement of storage modulus demonstrates that reinforced composites have higher energy storage capacity compared to pure polymer specimens. Also, inclusion of CF and FG improves loss modulus by 43 and 4 times at room temperature, respectively, which demonstrate that the materials become tougher compared to the unreinforced 3D printed nylon.

The effect of the fiber volume percentage on storage modulus of the nylon specimens reinforced with FG at room temperature was studied and the results are shown in Table 2. Fiber glass with different fiber layers of two, four, and eight layers and consequently volume percentages of 16, 20, and 64% were added to the nylon. As shown, increasing the fiber volume percentage from 16 to 64% improves the storage modulus from 2.5 to 13.37 GPa. The effect of fiber volume percentage on loss modulus of nylon specimens reinforced with FG at room temperature was studied and the results are shown in Table 2. As shown, increasing fiber volume percentage from 16 to 64% improves the loss modulus from 121.92 to 1580 MPa. The fiber reinforcement improves loss modulus values, which means more energy is dissipated as fiber content increases.

Table 3 shows the effects of fiber volume fraction on the tensile strength and elastic modulus of the specimen for continuous CF specimens. As shown, fiber reinforcement of 60% volume percentage improves tensile strength and elastic modulus of unreinforced nylon specimens 18 times and 53 times, respectively. The tensile strength of sample with volume fraction of 60% reached 360.50 MPa, which is

Table 2 Effect of fiber volume percentage on storage and loss modulus of nylon-FG at room temperatures

Fiber layers	Nylon layers	Fiber volume (%)	Storage modulus (Gpa)	Loss modulus (Mpa)
2	8	16	2.51	121.92
4	6	20	6.15	421.99
8	2	64	13.38	1580.12

Table 3 Effect of carbon fiber volume percentage on tensile strength and elastic modulus of AM nylon

Fiber layers	Nylon layers	Fiber volume (%)	Tensile strength (MPa)	Elastic modulus (GPa)
0	40	0	19.45	0.29
18	22	33	272.19	26.12
36	4	60	360.50	50.30

close to tensile strength reported for Aluminum 6061-T6 [16]. Similar performance was reported by Dickson et al. [8], who presented composite specimens with yield strengths of up to 368 MPa with volume fraction of 35% carbon fiber.

Figure 2 demonstrates the effect of fiber reinforcement on thermal conductivity and heat capacity of fiber reinforced 3D printed nylon specimen. Nylon was reinforced with CF and FG containing 50% volume fraction. As shown, fiber reinforcement improves thermal conductivity of nylon specimen from 0.114 W/(m K) to 0.406 with CF and 0.277 W/(m K) with FG, respectively. Also, fiber reinforcement with CF and FG improves heat capacity of nylon from 1.039 to 1.714 and 1.105 (KJ/Kg.K), respectively.

SEM analysis was used to study microstructural morphology, interlayer adhesion, printing quality, and fracture mechanisms. As shown in Fig. 3, SEM images demonstrate that there is a good adhesion between the nylon matrix and carbon fiber layer because voids are rarely observed between two phases. Also, it can be seen that fiber breakage and fiber pull-out are the main failure mechanism for the specimen. Voids are usually observed due to the weak adhesion between fiber and the matrix. Increasing the storage modulus by fiber inclusion is due to increasing the stress transfer between fiber and the nylon [18–20].

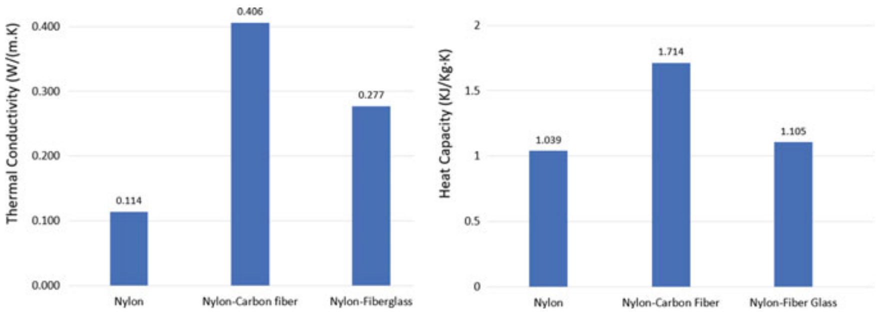

Fig. 2 Effect of carbon fiber reinforcement on thermal conductivity and heat capacity of CFRAM components

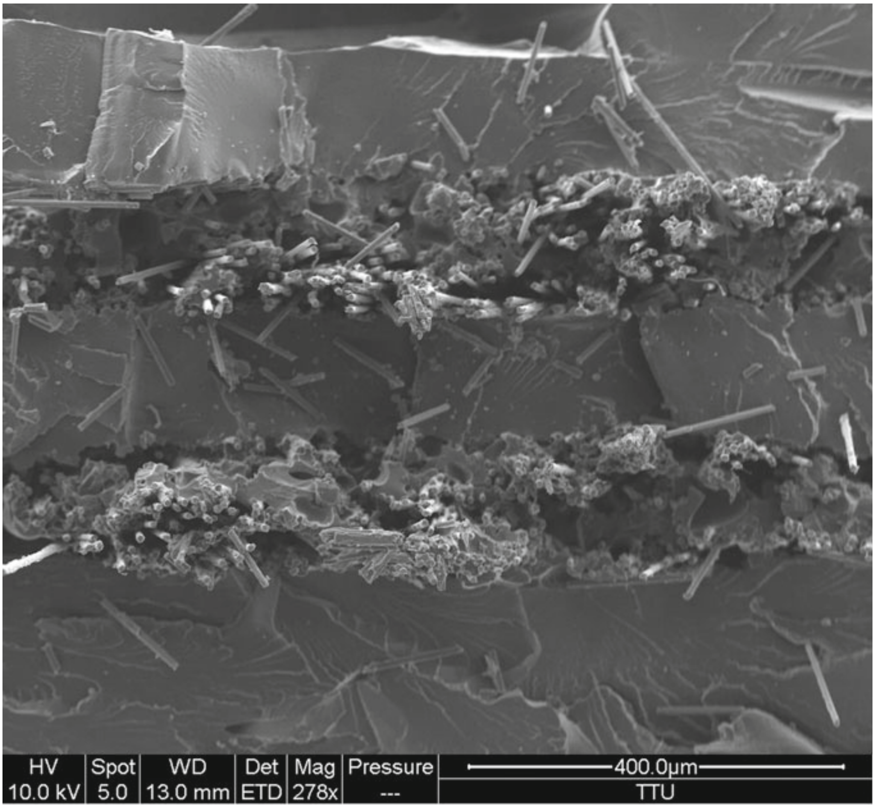

Fig. 3 SEM image from cross-section of nylon-CF 3D printed composite

Discussion

The effect of fiber reinforcement on DMA properties of nylon composites was studied. The results show that increasing fiber content improves storage modulus due to enhanced stiffness of matrices while increasing temperature reduces the storage modulus due to loosening the interface and chemical bonds. By fiber inclusion, the majority of load withstands by the fiber, and storage modulus of composite is improved. By inclusion of fiber, the specimen shows slower flow and consequently a higher loss modulus [18]. Fiber reinforcement restricts molecular mobility of polymer chains, which leads to the enhancement of storage modulus [19, 21, 23]. About the effect of temperature on storage modulus, it can be mentioned that as the temperature increases, fiber and polymer molecules obtain more energy to move away from each other. This causes a weakening interface and reduction of the stress transfer between the fiber and polymer [19]. As temperature increases, loss modulus increases because loss modulus indicates viscous portion of material and as the temperature increases the viscosity decreases because molecules get more energy to move [19].

So, energy loss happens inside the system due to the movement of molecules and friction between the molecules [20]. Another assumption behind the enforcement of properties by the fiber inclusion relates to the strong chemical interaction between fiber and matrix. The surface of CF and FG filament is coated with epoxy resin. Epoxy has functional groups of carboxyl, ether, and hydroxyl that create strong covalent bonds with amide groups of nylon [18].

Tensile test was conducted and as expected, the tensile properties for fiber reinforced composites have much better performance when the amount of fiber is increased. The tensile specimen reinforced with 60% CF volume fraction shows strengths of up to 360 MPa with is higher than the 310 MPa reported for Aluminium 6061-T6 [8]. This enhancement is attributed to the continuous structure of fibers in the matrix, which avoids existence of fiber ends in the structure. It can be confirmed by SEM image that fibers are main element for load-bearing in the composite structure. So, increasing volume fraction of fiber improves composite performance.

Conclusion

In this study, 3D printed nylon reinforced with continuous CF and FG were printed and thermomechanical properties were studied. The effect of fiber reinforcement on storage modulus was investigated and results show that CF reinforcement improves storage modulus of nylon 46 times. Also, the results demonstrate that fiber reinforcement can improve the loss modulus of nylon 43 times. The effect of fiber volume percentage on storage and loss modulus was studied and the results showed that increasing fiber content from 16 to 64% improves the storage modulus by five times. Tensile test results showed that fiber reinforcement with 60% fiber fraction can improve tensile strength and elastic modulus 18 times and 53 times, respectively. Thermal analysis showed that fiber reinforcement with 50% fiber fraction improves thermal conductivity and thermal capacity. SEM analysis demonstrates a good adhesion between the fiber and matrix. The results show that the fiber reinforcement improves thermomechanical properties of nylon, which means that the nylon matrix becomes stronger and tougher after fiber inclusion. This demonstrates the potential of CFRAM composites as an alternative to metals for engineering applications.

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