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Thermal characterization of recycled polymer for additive manufacturing applications

K.S. Boparai [a], R. Singh [b], F. Fabbrocino [c], F. Fraternali [d, *]

[a] Mechanical Engineering Dept., MRRSPTU Bathinda, India
[b] Department of Production Engineering, GNDEC Ludhiana, 141006, India
[c] Pegaso University, Department of Engineering, Piazza Trieste e Trento, 48, 80132 Naples, Italy
[d] Department of Civil Engineering, University of Salerno, Italy

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ABSTRACT

This work is focused on the thermal characterization of Nylon 6 based nano-composite (NC) material. Initially, melt flow index (MFI) test confirms the qualification of this material, as an alternative material for the fabrication of FDM filament. The differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA) measurements characterize the material by recording their phase and mass changes as a function of temperature. The DSC results confirmed the decrease of crystallinity with the inclusion of nano fillers but also realized that these filler particles act as a thermodynamic sink and improves its stability. The TGA analysis also demonstrated the increase in thermal stability and flame retardancy level of NC material. In addition to above scanning electron microscopy analysis visualized the dispersion of filler materials in Nylon 6 matrix.

1. Introduction

The reinforcement of glass, fibers, metal and ceramics etc. in polymeric matrix have proved to be advantageous in terms of the enhanced mechanical, thermal and tribological properties [1]. Such reinforced polymers are superior to conventional polymers and can be considered for wide industrial applications. Before the selection of these reinforced polymers for particular application, it is the matter of interest to characterize the material by rheological, mechanical, morphological, thermal, chemical and tribological testing [2–4]. The various analytical methods for the thermal characterization of polymeric based composite material are Dynamic mechanical analysis (DMA), differential scanning calorimeter (DSC), Thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR) and thermal conductivity tests as shown in Fig. 1. All these methods are destructive type and found to be very useful for the assessment of the degradation of material [4].

Among all these methods, DSC and TGA are most frequently used techniques in thermal analysis [5,6]. DSC is used to study the behavior of material as a function of temperature and time. Melting point, crystallization behavior and chemical reactions are just some of the many properties and processes that can be measured by DSC [5]. DSC measured the heat flow produced in a sample when it is heated, cooled or held isothermally at constant temperature. A sample may undergo one or more phase changes during heating and cooling. The DSC measurement curve shows the peak whose area corresponds to the enthalpy involved in the process. The DSC curve shows typical thermal effects when amorphous plastic heated. These includes glass transition temperature, peak due to cold crystallization, melting and finally decomposition. In DSC experiments, the heat flow from the furnace to the samples is measured relative to the heat flow to reference material. The sample and reference crucibles are identical except that the reference crucible is usually empty. Both the sample and reference crucibles are surrounded by heater or furnace. The sensor, which is the heart of DSC, detects the heat flow. For quantitative heat flow measurements, the crucible containing the sample must have excellent thermal conductivity and be an optimum contact with the sensors. Thus the geometry of crucible and material used are very important irrespective of the kind of sample that is measured. Due to this various types of crucibles to suit different samples are available. Most commonly used are standard aluminum crucible with normal lid, gold crucibles for samples reactive with aluminum, high pressure crucible for measurement in closed atmosphere and other different type of crucible are available to match the

* Corresponding author.
E-mail address: f.fraternali@unisa.it (F. Fraternali).

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requirement set by sample and application.

DSC measurements can be performed dynamically using a linear temperature ramp, isothermally or with temperature modulation [5,7]. Temperature scans are used to investigate temperature dependent processes such as glass transition temperature, crystallization, melting and curing reactions [8]. Isothermal temperature experiments are mainly used to determine the oxidation induction time of materials or to study chemical reactions. Temperature modulated experiments enabled to separate reversing and non-reversing effects such as glass transition temperature for simultaneous occurring reactions or evaporation. Special atmosphere such as pure oxygen and nitrogen are often used for applications to accelerate and prevent decomposition of samples.

Stark [9] demonstrated the correlation of DMA and DSC experiments of carbon fiber epoxy prepreg (CFC) and indications were found for gelation and vitrification. Cho and Bahadur [3] conducted DMA and DSC analysis on polyphenylene sulphid (PPS) composites reinforced with carbon nano fiber (CNF) to study the viscoelastic properties and the thermal transitions. There is no significant effect observed with the reinforcement on the mechanical properties and effect of damping is also negligible. However the crystallinity of PPS decreases with the increase in reinforcement of CNF.

TGA is also mainly used to characterize the polymeric based materials by measuring their change in mass as a function of temperature [5,10,11]. The properties and behavior that can be measured by TGA include composition, purity, decomposition reactions, decomposition temperature and absorbed moisture contents [11]. The TGA measures the mass of the sample as it heated, cooled or held at a constant temperature in a defined atmosphere.

The use of recycled polymer based nano composite (NC) material as a feedstock material for Fused Deposition Modeling (FDM) system is a cost effective approach which also leads towards green technology. Moreover customized properties can be inculcated with the reinforcement of different filler materials. In this work, Nylon6-Al-Al_2O_3 based NC material for FDM system in place of commercial ABS material were characterize in terms of their thermal properties. The thermal properties were studied by DSC and TGA analysis. The work starts with development of NC material, followed by rheological testing (MFI value) and then thermal testing. The literature reveals that, hitherto very little work have been reported on the investigations of the thermal properties of alternatively developed material (Nylon6-Al-Al_2O_3) for the fabrication of FDM filament, which is necessary before the loading in commercial FDM system.

2. Experimentation

2.1. Materials and samples preparation

The used plastic waste material Nylon6 (average density of 1.13 g/cm^3) was initially chopped in a pulverizer and then reduced to a particle size of 450–750 μm by cryogenic grinding. The nanofillers such as Al (average density of 2.7 g/cm^3) and Al_2O_3 (average density of 3.9 g/cm^3) were supplied by Thomas Bakers, India. Based upon the previous work done by Boparai et al. [12], the volume % proportions of NC material was selected as shown in Table 1. Prior to processing, Nylon 6 material was dried for 8 h at 50 °C in a vacuum oven, in order to remove humidity and oil traces. The different weight proportions of composition material were then mixed in tumbler mixer by rotating it with 200 rpm for 2 h. Samples for MFI, DSC and TGA measurements were prepared by melt mixing using single screw extruder (SS-11-E, 25 mm extruder, M/s Binflex Plastic Industry, Punjab, India) followed by granulation. The single screw extruder barrel, consist of three heaters, each having a capacity of 1 KW and equipped with zone temperature control. The heater towards hoper side set at lower temperature and heater on die side sets at higher temperature. The mean barrel temperature and die temperatures were 170, 205 °C, the screw rotated at 35 rpm and the pressure was 22 MPa.

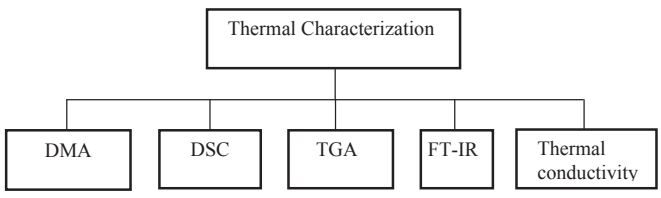

Fig. 1. Thermal Characterization techniques for polymeric based composite materials.

The NC density was calculated by using following formula [13]:

$$\rho_c = 1 \Big/ \left\{ \left(\frac{W_s}{\rho_s}\right) + \left(\frac{W_m}{\rho_m}\right) + \left(\frac{W_p}{\rho_p}\right) \right\}$$

where

ρ_c = density of composition; ρ_s = density of Al; ρ_p = density of Nylon 6; ρ_m = density of Al_2O_3
W_s = weight proportions of Al; W_m = weight proportions of Al_2O_3; W_p = weight proportions of Nylon 6

2.2. Experimentation

2.2.1. Melt flow index value

The melt flow index (MFI) values of materials such as NC material, waste unfilled Nylon6 and standard ABS were determined by using MFI tester (SE-MFI-I, Shanta Engineering, Mumbai India). The test was performed by maintaining cylinder temperature of melt flow tester at 230 °C and weight 3.8 Kg (as per ASTM D 1238). The average value of ten observations was recorded for each sample. This is generally a comparative study of flow behavior under similar processing conditions and limits the practical level, up to which filler materials can be incorporated into the thermoplastic polymers.

2.2.2. Thermal analysis

Thermal analysis of NC material, waste unfilled Nylon6 and standard ABS material was carried out by DSC and TGA measurements. DSC curves were registered on DSC1 (Mettler Toledo) with HSS8 sensors and Huber TC intercooler (Refrigerated cooling). No pretreatment of samples was required and weighted about 6–8 mg sample in 40 μl Al standard crucible with pinhole on Mettler Toledo 5 decimal balance. Samples were heated from 25 °C to 350 °C with 1 min isothermal curing at 350 °C and cooled to 25 °C at a rate of 10 K/min. A second heating scan was then performed at 10 K/min. the DSC measurement curves shows the peak whose area corresponds to the enthalpy involved in the process. The DSC curves highlight typical thermal effects when amorphous material heated. These include exothermic and endothermic peaks namely glass transition temperature (Tg), peak due to cold crystallization, melting enthalpy and finally decomposition. The crystallinity value for both on melting (X_m) and cooling (X_c) was determined by using following equations [7,11].

$$X_m = \frac{\Delta H_m}{f \cdot \Delta H^\circ} \times 100\% \quad (1)$$

Table 1
Volume % proportions of NC.

S. No.	Nylon6	Al	Al_2O_3	Density (g/cm^3)
1	80	16	4	1.50

$$X_c = \frac{\Delta H_c}{f \cdot \Delta H^\circ} \times 100\% \quad (2)$$

whereas f is the weight fraction of Nylon 6 in the composite into account and the melting enthalpy (ΔH°) of 100% crystalline Nylon 6 is 176.4 J/g [14].

Thermogravimetric analysis measurements were carried out on Mettler Toledo TGA/DSC1 with DSC sensor. The sample weight was approximately 15 mg and heated by imposing a temperature ramp between 30 °C and 800 °C at a rate of 10 K/min. Both DSC and TGA experiments were performed under a constant nitrogen flow of 40 ml/min.

3. Results and discussion

3.1. Melt flow index value

In the process of alternative material development for FDM system without changing its hardware and software, the necessary condition is to compare the MFI value of this material with standard FDM material (as recommended by Stratasys). Presently U-Print SE model of the FDM uses P430XL ABS (IVR) plastic as a model material. Initially, the MFI value of waste Nylon 6 is 10.6, which decreased to 2.30 due to nano filler loading in Nylon 6 matrix. The selected NC material has nearly same MFI value as the standard ABS (2.4) has, as shown in Table 2. Therefore, selected NC material can be processed in FDM system with same set of processing conditions as established by the manufacture for standard material.

3.2. Differential scanning calorimeter analysis

The DSC measurements were performed dynamically using a liner temperature ramp on DSC1 [15]. Here the samples was heated and cooled at a constant rate (10 K/min) and the different states of the samples were measured as a function of temperature. The DSC curves as shown in Fig. 2, illustrates the typical temperature scan of waste Nylon 6 material. The sample quantity taken for each measurement was <9 mg so as to achieve a uniform heating/cooling. The multiple thermopiles offered by DSC1 are shown in Fig. 2. As the DSC1 was equipped by HSS8 sensor, it offers high sensitivity and flat baseline permits measurement of weak effects like glass transition temperature (T_g) even with smaller quantity of samples [15]. Huber TC100 Intercooler (max. −85 °C lowest min. achievable temp.) was also configured with DSC1.

The black curve highlights first heating run. It illustrates the typical effect observed on heating. The first event is the glass transition temperature, which can be seen as a step in curve. This is followed by exothermic cold crystallization peak and endothermic melting peak. If the samples were heated at higher temperature, it will start to decompose. The temperatures at which these effects are likely takes place are characteristic for each particular material. The DSC curves can therefore be used as a finger prints in quality control [15,16]. Generally the first heating measurement is performed to remove the thermal history of the sample (Polymer) which may be due to the processing conditions induced during sample preparation [4,16]. In general it is often very useful to measure the cooling curve of the sample and then recording second heating run. These additional measurements provide more information about the behavior of the material [3]. The second heating curve (Blue) shows the measurement after removal of thermal history of the sample. The melting peak of waste Nylon6 is at 219.03 °C with enthalpy value (heat of melting) of 81.37 J/g. The glass transition do not accompany endothermic peak due to enthalpy relaxation. The crystallization peak can be seen in the cooling run shown green line in Fig. 2. In contrast the waste Nylon6 was almost completely amorphous because the cooling process during manufacturing was too fast for crystallization to occur [13,14].

In order to examine the extent to which Nano fillers influence the crystallization and melting behavior of waste Nylon 6 material, the crystallization and melting curves of unfilled waste Nylon 6 and reinforced waste Nylon 6 were realized during cooling and second heating processes of temperature variable DSC measurements (Fig. 3). Table 2 shows the summery of DSC results, including crystallization peak temperature (T_c), melting peak temperature (T_m), melting enthalpy (ΔH°_m), crystallization enthalpy (ΔH°_c), degree of crystallinity on heating (X_m), degree of crystallinity on cooling (X_c).

The unfilled waste Nylon6 have a slightly low T_c (176.1 °C) than reinforced waste Nylon 6 during cooling process. This indicates a small enhancement in the crystallization rate due to the nucleation effect of nanoparticles in Nylon 6 matrix [13]. However, the inclusion of nano-fillers in Nylon 6 matrix decreased T_m, X_m and X_c to 218.18 °C, 23.55% and 26.0% respectively. The unfilled waste Nylon 6 have T_m 219.03 °C, X_m 46.12% and X_c 41.4%. This is due to the fact that besides the nucleation effect, Nano-fillers retard the movement and diffusion of Nylon 6 molecular chains to the surface of the nucleus in the composite material [5,10,16,17]. The melting temperature of polymeric based composite material, established their processing conditions and thermal properties [10]. A slight decrease in T_m was observed by reinforcing Nano particles in Nylon 6 matrix. This result realized that although filler contents acts as the nucleation agents, promoted the crystallization and growth but it interfered the perfect crystallization of Nylon 6 [16]. This resulted in shift of T_m to a lower temperature. As reported by many researchers [5,10,16], the decrease of matrix crystallinity adversely affect the mechanical properties of NC material.

3.3. Thermogravimetric analysis (TGA)

The TGA curves realized the thermal degradation behavior of polymeric based materials. In this work two different curves are generated, mass loss curve and first derivative of the mass loss curve (DTG). The temperatures associated with a weight loss of 20% ($T_{20\%wt}$) and weight loss of 50% ($T_{50\%wt}$) were also identified. Moreover, the first derivative of TGA peak (T_{DP}) highlights the decomposition pattern in much details than the weight loss signal and quantified thermo-oxidative stability of the material by residue value.

As shown in Fig. 4, the neat Nylon6, NC and ABS exhibited a single stage degradation having peaks at 449 °C, 462 °C and 430 °C respectively. The process initially starts with removal of volatile contents such as moisture, followed by thermal decomposition. The ABS material shows less thermal stability than neat Nylon6, as the percentage of mass loss occurs at lower peak temperature. A significant increase in T_{DP} temperature for NC was noted compared to neat Nylon6 and ABS. This indicates that filler materials affect the thermal behavior of Nylon6 matrix. Additionally, the results also revealed that there is no change in the onset temperature of the degradation. The onset temperature of waste Nylon6, ABS and NC was ~41 °C which indicates the absence of any interaction (physical or surface) between Nylon6 and filler materials.

Table 2
MFI and DSC data.

Sample No	Material	MFI (gm/10min)	T_c (°C)	T_m (°C)	$\Delta H°_c$ (J/g)	$\Delta H°_m$ (J/g)	X_c (%)	X_m (%)
1	Waste Nylon6	10.61	176.10	219.03	73.16	81.37	41.4	46.12
2	NC	2.30	178.36	218.18	27.61	24.93	26.0	23.55

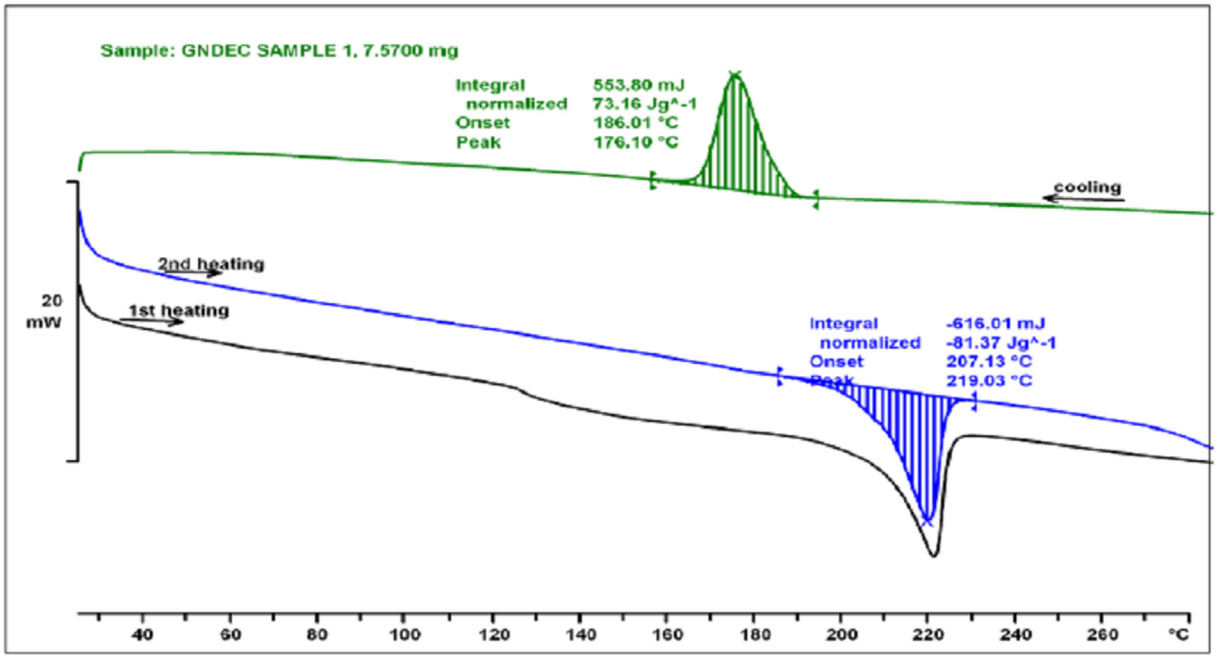

Fig. 2. DSC thermograms for waste Nylon6.

It was observed from Table 3 that neat Nylon6 has high values of $T_{20\%wt}$ and $T_{50\%wt}$, which indicates that Nylon6 has slow degradation than ABS. The addition of Al and Al2O3 in Nylon6 matrix, further increase the value of both these temperatures. This shows the high efficiency of NC material than neat Nylon6 and ABS. The incorporation of filler materials in Nylon6 matrix, improves the thermal resistance parameters of NC material.

Moreover char residue of NC is very larger than neat Nylon6 and ABS. This result suggested that through the addition of fillers, the flame retardancy of Nylon6 is leveled up by 46.67%. As reported by various researchers [10,13,16], that char residue directly indicates the potency of flame retardation of polymeric based materials. The nano filler materials improves the thermal and thermal oxidative stability. This is due to the barrier effect of nano particles which

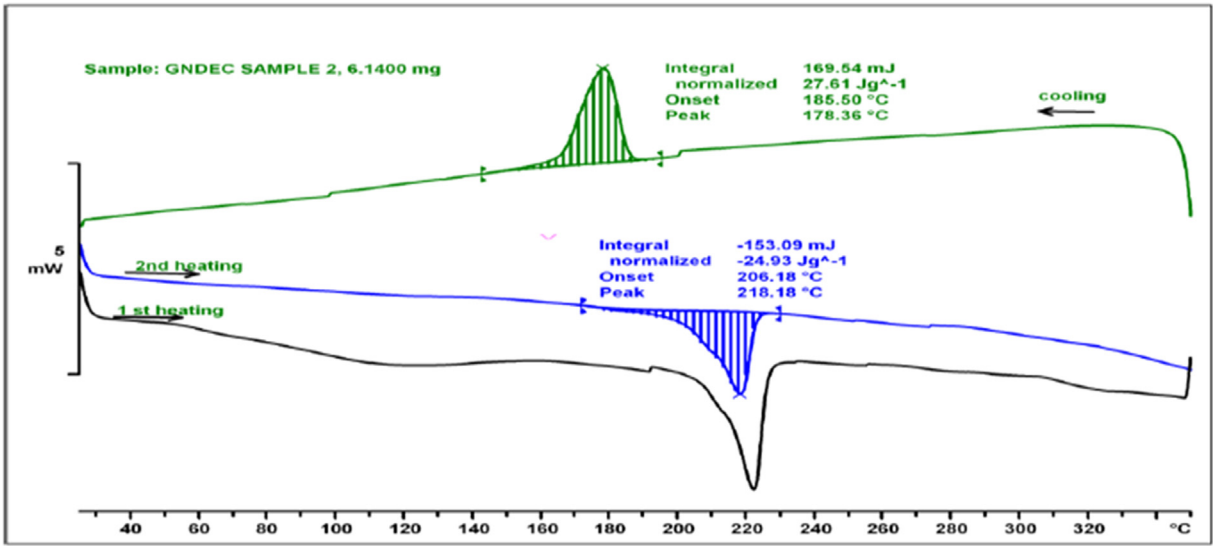

Fig. 3. DSC thermograms for NC.

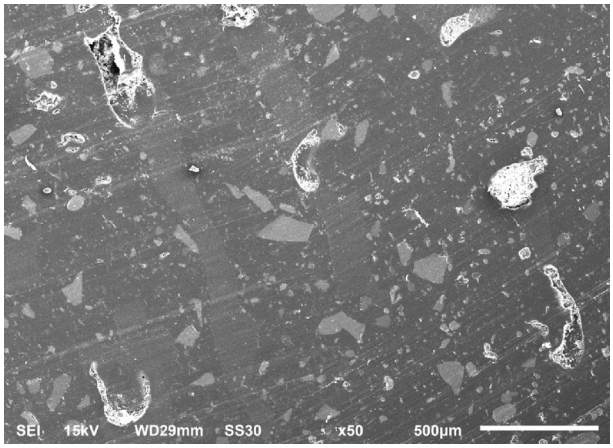

Fig. 4. SEM image (50×) of NC material.

Table 3
Thermogravimetric data of waste Nylon6, NC and ABS material.

Sample no.	Material	Temperature °C			Residue (%)
		$T_{20\%wt}$	$T_{50\%wt}$	T_{DP}	
1	Waste Nylon6	416	445	449	1.8775
2	NC	419	458	462	46.6722
3	ABS	413	428	430	2.5953

limits the production of combustible gases, decreases the exothermic reaction and confines the thermal conductivity of burning material [16,18–20].

The SEM image as shown in Fig. 4 illustrates the dispersion of filler material in Nylon6 matrix. Although the distribution of particles are uniform but some agglomeration of particles are also observed. The distribution of particles can further be improved by mixing with twin screw extruder.

4. Conclusions

In this work, the waste Nylon6-Al-Al_2O_3 based alternative NC material has been successfully synthesized by controlled loading of filler material in Nylon6 matrix and can be used as a FDM feedstock filament material. A significant improvement in the thermal properties were observed. The various experimentation outputs are summarized as follows:

- Al-Al_2O_3 based Nano materials were demonstrated as an effective fillers for waste Nylon6 material. The nano filler loading act as thermodynamic sink which absorb heat of crystallization and hinders the crystal nucleation and growth. The DSC results indicate the decreased of T_m, X_m and X_c to 218.18 °C, 23.55% and 26.0% respectively with the inclusion of nano-fillers in waste Nylon 6 matrix.
- TGA thermopiles realized the potency of flame retardation of NC. The char residue of NC is very large as compared to neat Nylon6 and ABS. A significant increase in $T_{20\%wt}$, $T_{50\%wt}$ and T_{DP} of NC material confirmed that with the reinforcement of filler materials in Nylon6 matrix, improved its thermal resistance parameters.
- SEM analysis indicated that the NC material have uniform dispersion; however it needs further improvement. Overall, Nylon6-Al-Al_2O_3 based NC material shows high potential for promising applications in rapid manufacturing. Thus, it was pointed out that NC material can be used as an alternative material in place of ABS without any modification in FDM system.

Future extensions and generalizations of the current research will be concerned with the use of waste Nylon6-Al-Al_2O_3 based NC filaments for the additive manufacturing of physical models of unconventional materials and structures with properties mainly derived from their geometric design, such as, e.g., reinforcing elements of composite materials [21–29], and soft mechanical metamaterials [30–34].

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