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Optimization of the 3D Printing Parameters on Dimensional Accuracy and Surface Finishing for New Polyamide 6 and Its Composite Used in Fused Deposition Modeling (FDM) Process

Tuan Noraihan Azila Tuan Rahim, Hazizan Md Akil*
School of Materials and Mineral Resources Engineering, Engineering Campus, Universiti Sains Malaysia, 14300 Nibong Tebal, Pulau Pinang, Malaysia

Abdul Manaf Abdullah, Dasmawati Mohamad
School of Dental Sciences, Health Campus, Universiti Sains Malaysia, 16150 Kubang Kerian, Kelantan, Malaysia

Zainul Ahmad Rajion
College of Dentistry, King Saud Bin Abdulaziz University for Health Sciences (KSAU-HS), Riyadh, Kingdom of Saudi Arabia

**azila.trahim@gmail.com*

ABSTRACT

Currently, 3D printing is emerged as one of the attractive manufacturing option to build intricate parts without human intervention. However, the users must be aware of the inherent printer's limitation, particularly related with geometric tolerance of printed materials. This study aims to determine the optimum parameters to fabricate polyamide 6 and filled polyamide through fused deposition modeling (FDM). The studied parameters including printing speed (10-100 mm/s), temperature (240-260°C) and addition of fillers (10 wt%). The rheological properties were evaluated to estimate the behaviour of polyamides through the FDM system. In order to obtain a part with achievable accuracy between the designed and actual dimension, the accuracy of printed part manufactured using production-grade FDM 3D printer by Statasys Ltd. was used as a benchmark and also assigned in a statistical equation. The surface finishing was also studied under

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stereomicroscope. It was found that all parameters have a substantial influence on the rheological behaviours. Generally, increasing the printing speed improves its dimensional accuracy however higher temperatures than 250°C was not desired as it lowers the melt viscosity and reduce the geometric precision. The presence of fillers modifies the rheological behavior of the materials with respect to the unfilled polymer, in which the viscosity was slightly reduced particularly at higher shear rates. It can be concluded that high geometric precision and surface finishing can be achieved if the printing settings were correctly set up especially for new materials in FDM process.

Keywords: *Fused Deposition Modeling, 3D Printing, Dimensional Accuracy, Surface Finishing, Viscosity*

Introduction

Fused deposition modeling (FDM) is one of the 3D printing technique that gains such popularity in medical field since the past few years. With the capabilities of building intricate parts without human intervention, this technology has opened up for product customization, which is very useful for fabrication of biomedical implant. Acrylonitrile butadiene styrene (ABS) and polylactic acid (PLA) have traditionally been used as the feedstocks for FDM 3D printing. But more recently, there are a number of studies investigating the printed ABS and PLA reinforced with graphene, short fiber, montmorillonite, metals, TCP etc. [1–6] to improve the mechanical properties of printed part. Nevertheless, there have been recent concern over the toxicity of ABS and the durability of PLA, thus limit the accessibility of FDM 3D printing to produce a biomedical implant.

Therefore, there is an interest in developing a new material for FDM 3D printing to be feasible for use in biomedical implant. One of the new potential material to be used in FDM is polyamide, as it exhibits excellent mechanical properties, good biocompatibility as well as high processability in FDM technique. More recently, the properties of polyamide can also be enhanced by incorporating with some bioceramic fillers. A study by Wang et al.[7] showed the mechanical properties of hydroxyapatite filled polyamide composites were similar to the natural bone, while in vivo results that these composites had good biocompatibility to the bone and could bond to the hard tissue directly. Another study by Tuan Rahim et al. [8] have demonstrated the potential of polyamide 12 reinforced up to 40 wt% ceramic fillers in FDM 3D printing with enhanced mechanical properties. While most studies were focused on how to improve the mechanical properties of printed parts, too little attention has been paid to select the appropriate conditions for new materials.

Undoubtedly, many challenges and issues may encounter to achieve a high quality of printing especially when developing a new material. The process parameters such as layer thickness, temperature and speeds are largely influence on surface finishing and dimensional accuracy. While surface finishing can be qualitatively determined, dimensional accuracy of built parts can be expressed as a difference in length, width and thickness. Previously, Sood et al. [9] reported the value of thickness was always more than the desired value although a polymer may shrink after cooling which can reduce a part dimension. Moreover, a study by Melenka et al. [10] showed the percentage error of part thickness was significantly higher compared with other dimensions. Vasudevarao et al. [11] found the print temperature and layer of height had no significant effect on surface finishing. It can be concluded that, for obtaining the best surface finishing with high dimensional accuracy, the parameters should be systematically optimized.

In this study, an experimental method for obtaining optimized conditions for PA6 and PA6 composites was represented. The potential materials of FDM, polyamide 6 (PA6) and 10wt% filled PA6 were chosen and printed at 5 printing speeds between 20-100 mm/s at 240-260°C. The printing speeds were firstly optimized, followed by temperature and lastly surface finishing. The method can also be used for optimizing of existing or new materials where good surface finishing and high dimensional accuracy was desired.

Materials and Methods

Raw Materials

Polyamide 6 (PA6) manufactured by UBE Industries, Ltd. was used in this study. Due to the hygroscopic property, all polyamide samples were dried in a vacuum oven at 90°C for at least 24 hours prior to any fabrication or test. Hydroxyapatite (No. 21223, Sigma Aldrich) was used as a filler to enhance the mechanical and biological properties. The compositions are shown in Table 1.

Table 1: Compositional of PA6 and PA6 composites

Sample	**Raw material**		**Filler content (wt%)**
	PA6	**HA**	
Unfilled PA6	100	0	0
PA6/10HA	90	10	10

Sample Preparation and Parameters

Polyamide 6 and hydroxyapatite were premixed and subsequently compounded using a twin screw extruder (PSM 30, Sino Alloy Machinery) at

210-225°C with a screw speed of 100 rpm. The pellets were then fed into a single screw extruder and drawn into a filament form through a circular die at the processing temperatures of 210-235°C. The filament of a range between 1.65-1.85 mm was spooled to the winder as it was extruded. The obtained filament was used as a feedstock to the 3D printer (Replicator 2X, Makerbot, USA).

There were several steps that were taken prior to printing the material. It started with constructing the virtual 3D models via SolidWorks 3D computer aided design (CAD) software. In this study, a simple model of 15 mm × 30 mm × 3 mm was designed using SolidWorks 3D Computer Aided Design (CAD) software. The design file was then exported into STL file format and reviewed by the Makerware software program (printer software). In this experiment, fabricated parts with different task were evaluated based on dimensional accuracy and surface finishing. During the study, only speed and temperature parameters were changed, while other parameters were kept constant. The constant parameters of the process are shown in Table 2.

Table 2: Constant parameters in Makerware program

Filling density	100%
Number of shells	2
Layer of height	0.3 mm
Pattern orientation	0°, 90°, 45° & -45°
Platform temperature	150°C

Characterizations

Capillary rheometer analysis

The shear flow properties of PA6 and PA6/10HA composite were measured by a capillary rheometer (Rheograph RG25, Göttfert, Germany). From this measurement, the flow rate is imposed by a piston while the pressure is monitored by a melt pressure transducer. The tests were performed at 240°C, 250°C and 260°C, using two circular dies of 1 mm diameter and different length of 10 and 30 mm. The examined apparent shear rates at the die wall $(\dot{\gamma}_{app})$ ranged from 36 to 1260 s^{-1}. Based on the value of ΔP obtained from a melt pressure transducer, the apparent shear stress (τ_{app}) was determined and apparent viscosity (η_{app}) can be calculated. The expressions are summarized in Equations (1), (2) and (3) and the flow and viscosity curves were constructed.

$$\tau_{app} = \frac{R\Delta P}{2L} \quad (1)$$

$$\dot{\gamma}_{app} = \frac{4Q}{\pi R^3} \quad (2)$$

$$\eta_{app} = \frac{\tau_{app}}{\dot{\gamma}_{app}} \quad (3)$$

Where L is the die length and R is the die radius. Most polymer are pseudoplastic non-Newtonian which obey the power law:

$$\tau = K\dot{\gamma}^n \quad (4)$$

$$\log\tau = n\log\dot{\gamma} + \log K \quad (5)$$

Where ***n*** is the power law index is derived from the slope and ***K*** is the intercept from the plot $\log\tau_{app}$ against $\log\dot{\gamma}_{app}$ Bagley correction was performed to correct the end pressure drop (ΔP_{end}), thus a true shear stress can be obtained which is written as:

$$\tau_{true} = \frac{R(\Delta P - \Delta P_{end})}{2L} \quad (6)$$

The Rabinowitch-Weissenberg correction was also performed to correct the shear rate for non-parabolic velocity profile.

$$\dot{\gamma}_{true} = \left(\frac{3n+1}{4n}\right)\dot{\gamma}_{app} \quad (7)$$

Therefore, the true viscosity is given by:

$$\eta_{true} = \frac{\tau_{true}}{\dot{\gamma}_{true}} \quad (8)$$

Dimensional accuracy

A cube with a dimension of 15 mm × 30 mm × 3 mm was printed at 5 printing speeds between 20-100 mm/s at 240°C. The dimensional accuracy of printed samples (n=2) was evaluated by measuring the dimension of test

sample using a digital caliper (Mitutoyo, Japan). From selected speed specimen, the same measurement was repeated at different temperatures (250°C and 260°C). To note, the printing temperatures were chosen based on the melting temperature of polyamide 6 which was in the range of 220°C. In this study, only the thickness of the sample was taken and compared with the thickness defined in the CAD software. The mean $(\overline{x}_t)$ and standard deviation (σ_t) was calculated based on the 12 measurements for each specimen. In order to increase the reability of the dimensional accuracy measurement, standard deviation of the mean $(\sigma_{\bar{t}})$ and also total errors (δt_{tot}) were subsequently calculated as follows [12]:

$$\sigma_{\bar{t}} = \frac{\sigma_t}{\sqrt{N}} \tag{9}$$

$$\delta t_{tot} = \sqrt{(\delta t_{ran})^2 + (\delta t_{sys})^2} \tag{10}$$

Where ***N*** is the number of measurements, dt_{ran} is the random component of the uncertainty, which is equal to $\sigma_{\bar{t}}$ and δt_{sys} = ±0.127 is the systematic component of the uncertainty, which is set based on the accuracy of printed part manufactured using production-grade FDM 3D printer Fortus 450mc by Statasys Ltd [13]. By combining both speed and temperature parameters, the optimum parameters were achieved from this range.

Surface Finishing

The surface finishing of the printed parts was also studied under a stereo zoom microscope (Kunoh Robo, Japan) 0.7 × magnification. The photographs of their top and edge surfaces were captured and qualitatively compared at different speeds and temperatures.

Results and discussion

Basic requirements for fused deposition modeling process

Fused deposition modeling (FDM) technique deals with polymer-based materials with operating temperatures below than 300°C. The feedstock which is in a filament form of 1.75±0.1mm is pushed through a system using a pinch roller mechanism, illustrated in Fig. 1. A stepper motor is connected

to one of the rollers to provide energy to move the filament through the system. One or both of the rollers may have a grooved or toothed surface like a gear to create sufficient friction for the roller to grab the filament and feed it to the liquefier without any slippage. The material is then melted in a heated liquefier zone and deposited on a platform surface layer by layer, with a layer thickness between 0.1 and 0.4 mm.

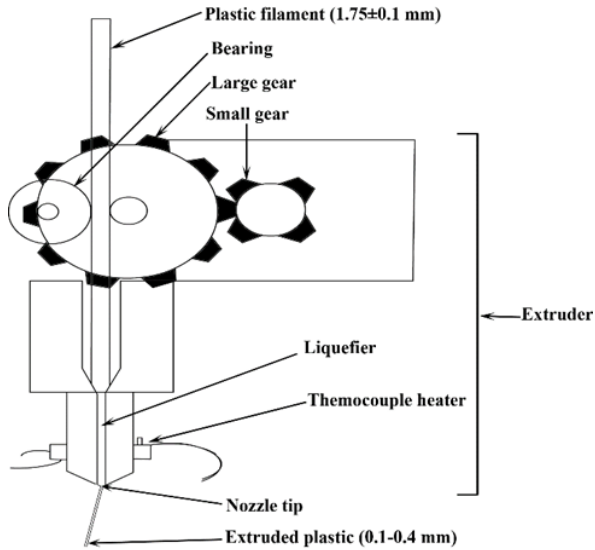

Figure 1: Schematic diagram of fused deposition modeling

The success of FDM process is highly dependent on two major factors: the selection of suitable materials and the right setting of FDM parameters during printing. Basically, amorphous polymer material is suitable to be used for FDM 3D printing. However, if semi crystalline polymer is selected, its behavior should be resembled to the amorphous polymer, particularly during cold crystallization from the polymer melt. Amorphous polymer molecules are randomly orientated which do not exhibit any crystalline structure in both molten and solid phases. Due to the inability to crystallize, amorphous melt is easily solidified in a relatively short time upon cooling process. This behavior is really crucial in order to achieve a good piece of build print with the successive addition of thin layers. Semi crystalline polymers, in turn normally can crystallize typically ranges between 15-80% [14,15] depending on their molecular structure and the cooling rate applied. In this study, polyamide 6 was selected due to the low degree of crystallinity, which is varied from 22-45% [16] thereby exhibits a

high tendency for polymer molecules to be quickly cooled or quenched from the melt.

In addition to the selection of materials, the right process parameters is crucial to ensure the material can print correctly with good surface finishing and high precision. The process parameters of FDM technique are including layer thickness, orientation, infill density, printing orientation, extruded temperature and printing speed [17]. Each parameter may have different value for different material, which merely dependent on its flow properties. In this study, we only evaluate the effect of printing speed and temperature on the dimensional accuracy and surface finishing of printed part. Basically all printers apply 2 setting for speeds which is known as a printing or extruding speed and the other as a traveling speed. The printing speed defines the rate at which the print head moves around, together with coming out of the plastic from the nozzle. Meanwhile, traveling speed describes how fast the print head is moving when it's not printing, usually is set higher than the printing speed. The trade-off with printing speed is between the high resolution build and the fast build times. If the speed settings are not configured properly, the printer may extrudes too much plastic at low speeds or not enough plastics at high print speeds. Therefore, selection of both speed and temperature are very crucial and may need lots of work tuning the parameters because of the difference of flow behavior between polymers.

Understanding the role of temperature is another important factor in achieving good print quality. The selection of temperature also strongly dependent on its viscosity and should be adjusted with right printing speed. The setting of too high temperature may cause reduction in polymer viscosity, and the melt become too fluidly and highly flow, causing a lot of plastic leaks out from the hot end (nozzle) during printing, reducing both dimensional accuracy and surface finishing. Meanwhile, if the temperature is too low, the layer obtained is not simply stick to the previous layer and the surface of a thread could be a bit rough. The optimized temperature for printing is the temperature that can extrudes the plastic with a good quality of print and possesses a sufficient strength of thread bonding between layers. The solidification process should also result in minimal internal stress within the part.

Therefore, capillary rheometer measurement was carried out to understand the flow behavior through FDM process. In this study, the shear rates of FDM was assumed to be close to the typical shear rates of extrusion process because both processes show almost the same behavior. The obtained FDM shear rates might falls at 2-3 s^{-1} (log scale) [18] meaning that the printing process is achieved at relatively high shear rate of deformation. Meanwhile, the actual shear stress of FDM cannot be measured due to the unknown pressure at the nozzle. Therefore, the shear stress in FDM system

was estimated based on capillary rheometer measurement which to be coincide with shear rates of 2.0-2.3 s^{-1} on log scale plot.

Capillary rheometer analysis

Fig. 2 shows the relationship between $\log \eta_{true}$ vs $\log \dot{\gamma}_{true}$ for PA 6 and PA6/10HA composite at the temperatures ranging from 240°C to 260°C after performing the Bagley and Rabinowitsch corrections on the $\log \tau_{app}$ vs $\log \dot{\gamma}_{app}$ plot. It can be seen the shear viscosity of all samples decreases with increasing shear rates, showing a typical property of pseudoplastic non-Newtonian melts. Increase in shear rate would exalt the chain mobility of polyamide molecules, therefore the molecules can be easily moved and aligned towards the flow direction. In addition, the shear viscosity was also decreased with increasing the temperature. By applying more heat to the materials, the free volumes between molecules was also increased, providing more space for polymer chains to move in the direction of flow, eventually lowers the shear viscosity.

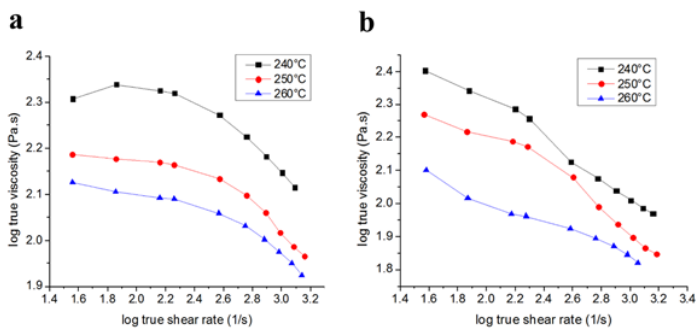

Figure 2: Temperature dependent viscosity of a) PA6 pure and b) PA6/10HA at 240°C-260°C

In order to evaluate the effect of hydroxyapatite filler on the viscosity of PA6, the plot of $\log \eta_{true}$ as a function of $\log \dot{\gamma}_{true}$ for the pure PA6 and PA6/10HA is represented at 240°C, as shown in Fig. 3. Interestingly, PA6/10HA was only found to be more viscous than neat PA6 at low shear rates until the two curves cross and afterwards the shear viscosity of PA/10HA became lower than that of neat PA6. The results obtained was different compared with common polymer which showed an increment of

shear viscosity after incorporation of fillers. In turn, increasing in shear rates facilitates the alignment of filled polymer chains to the direction of shear compared with the neat PA6 alone. This might be ascribed to the hydrogen bonding between PA6 chains which restrain the movement of polymer chains, causes a slow decrement in viscosity for neat PA6 than filled PA6. As can be seen, PA6 pure exhibits more Newtonian behavior at low shear rate (< 2.2 s^{-1}) and only displayed shear thinning behavior at the higher shear rates. The presence of hydroxyapatite induces an enhancement of shear thinning characteristics, lowering the shear viscosity over the higher shear rates. In agreement with our finding, a study by Baldi et al. [19] also found the decrease in viscosity for the filled PA6 compared with pure PA6. Based on these results, it can concluded that within the shear rates of 2-3 s^{-1}, the shear viscosity of PA6 can reduced when increases the printing speed and temperature in the FDM system. Incorporation of fillers also lowers the viscosity and changes the behavior of polyamide printing.

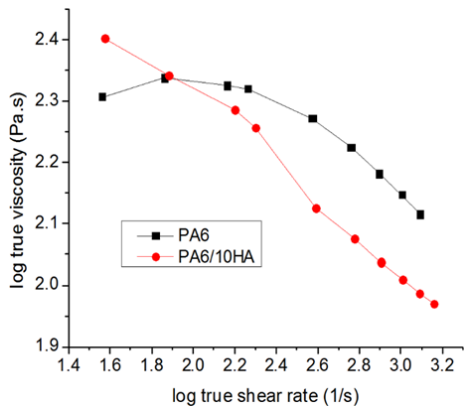

Figure 3: Log η_{true} of PA6 pure and PA6/HA composite as a function of log $\dot{\gamma}_{true}$ at 240°C.

Dimensional accuracy

The aim of dimensional accuracy measurement was to optimize the speed and the temperature used in FDM 3D printer. Basically, the dimension of printed part should be closely resembled to the nominal solid model dimension. In this study, only the thickness of the printed part was accounted for the dimensional analysis with CAD model as a reference. Previously, Melenka et al. [10] has reported the percentage error on thickness was significantly higher compared with the length and the width of the sample. The Makerbot Replicator 2X 3D printer can achieve a print resolution from 0.1 to 0.4 mm

layer height. However, the achievable accuracy of the part was not stated by the manufacturer. Many factors contribute to the accuracy including geometric design, temperature, printing speed, material type etc. In this study, we evaluate the effect of printing speed and temperature on the dimensional accuracy of printed part by using a simple model of 15 mm × 30 mm × 3 mm. Comparing the dimension of printed part with CAD model was still inadequate without including the theory of systematic errors. This is because the printed part may vary from the specified dimension and to obtain a part with a very high precision is often not attainable. Therefore, the part should be considered to have some definite systematic uncertainties. In this study, the accuracy of printed part manufactured using production-grade FDM 3D printer Fortus 450mc by Statasys Ltd. was used as a reference. The value of ±0.127 mm was taken as a systematic error (δt_{sys}) in the calculation to obtain total uncertainty (δt) as shown in Equation 10.

As shown in Table 3, only samples printed at the speed of 80 and 100 mm/s were found to have high dimensional accuracy compared with samples printed at lower speeds. As shown, the thickness of both samples were reduced as the speed increased. This is because, printing at lower speeds results in excess material which ruin the outer dimension and the blobs also emerges on the surface texture. This possibly due to the melt viscosity of PA6 still does not tailored with the speeds of the print head. The same results was obtained for PA6/10HA (Table 4) but the samples meet the dimensional precision from the speed of 60 mm/s. The lowered viscosity shown by PA/10HA might eases the process of printing and reduces the blobs on printed surface, which usually caused from the viscous material.

Table 3: Effect of printing speed on dimensional accuracy for PA6 pure

Speed (mm/s)	PA6 pure				
	mean	SD	SDOM	Accepted error	Accepted value
20	3.423	0.108	0.031	±0.131	2.869-3.131
40	3.288	0.096	0.028	±0.130	2.870-3.130
60	3.188	0.098	0.028	±0.130	2.870-3.130
80	3.095	0.083	0.024	±0.129	2.871-3.129
100	3.017	0.072	0.021	±0.129	2.871-3.129

Table 4. Effect of printing speed on dimensional accuracy for PA6/10HA composite.

Speed	PA6/10HA

(mm/s)	mean	SD	SDOM	Accepted error	Accepted value
20	3.407	0.09	0.026	±0.130	2.870-3.130
40	3.216	0.109	0.032	±0.131	2.869-3.131
60	3.113	0.045	0.013	±0.128	2.872-3.128
80	3.108	0.07	0.02	±0.129	2.871-3.129
100	2.98	0.077	0.022	±0.129	2.871-3.129

With the aforementioned results, the experimental examination is further designed to obtain the optimum temperature between 240-260°C. As shown in Table 5 and 6, the thickness of both samples printed at 250°C and 260°C lies far outside from the measured accepted range values. Increasing the temperature above 250°C may significantly drop the viscosity which causes the polymer can flow too easily and spreading out more, resulting a reduced dimensional accuracy of the samples. It can be concluded that within such a small temperature window for FDM process, it is generally desirable to set the printing temperature just above the melting temperature of PA6, and therefore the polymer would have a small enough viscosity to flow out through the nozzle. In agreement with the rheological measurement, the viscosity of polyamide was significantly reduced with the increase of 10-20°C.

Table 5: Effect of temperature on dimensional accuracy for PA6 pure.

Temperature (°C)	PA6 pure				
	mean	SD	SDOM	Accepted error	Accepted value
240	3.095	0.083	0.024	±0.129	2.871-3.129
250	3.170	0.101	0.029	±0.130	2.870-3.130
260	3.261	0.076	0.022	±0.129	2.871-3.129

Table 6: Effect of temperature on dimensional accuracy for PA6/10HA composite.

Temperature (°C)	PA6/10HA				
	mean	SD	SDOM	Accepted error	Accepted value
240	3.108	0.070	0.020	±0.129	2.869-3.131
250	3.278	0.107	0.031	±0.131	2.870-3.130
260	3.348	0.073	0.021	±0.127	2.871-3.129

Surface finishing

The final step of the experiment is to evaluate the surface finishing of the selected printed samples. Fig. 4 shows the printed surfaces of polyamides at different print speeds at T=240°C. For both samples, printing at very low shear rate results in excess plastic due to the over-extrusion that can ruin the outer dimension of the printed part and also affecting the thread lines on the top and the edge of surfaces which were not exactly straight. From the results, the best printing were found at the speeds between 60-80 mm/s. At these ranges, the thread lines were the most distinct and straightly aligned. At the fastest printing speed which is 100 mm/s (Fig. 5), although the thread lines were found straight for both samples, however there were some gaps or hollows between the extruded threads at certain areas. In other word, printing at 100 mm/s was not an effective speed for extruding polyamide 6. After these steps, it was concluded that the chosen optimum condition for PA6 printed at 240°C and higher speeds 80 mm/s while for PA/10HA composite, it shows the best print at the temperature of 240°C and speeds between 60-80 mm/s.

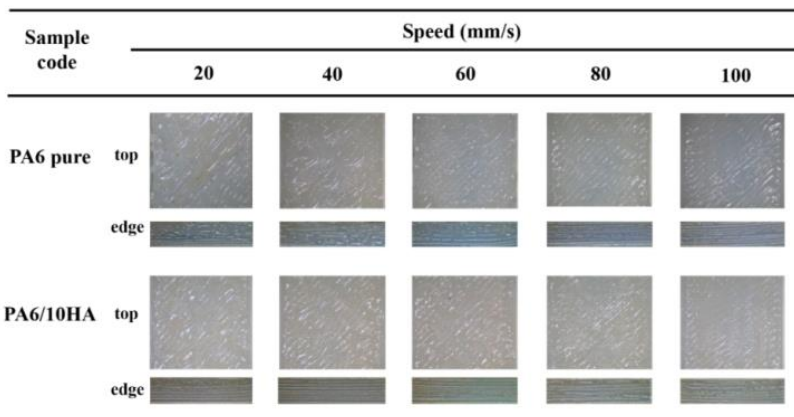

Figure 4: Stereomicroscope photographs of printed PA6 and PA6/10HA composite at constant temperature (240°C).

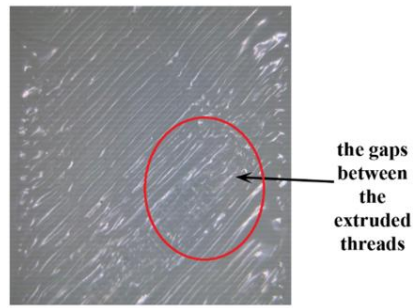

Figure 5: The surface of PA6 part printed at 100 mm/s.

Conclusion

The evaluation of dimensional accuracy and surface finishing of new polyamide 6 and 10wt% filled PA6 composite have been evaluated with variation of speeds and temperatures. It has been found that the temperature, speed and filler loading has a substantial influence on their rheological behaviors. It can be concluded that a balance of these parameters is necessary for the successful of FDM process with good print quality and high dimensional accuracy. The pure PA6 displays an optimum condition when it is set at lower temperature (T=240°C) and higher print speed (80 mm/s). Meanwhile, 10wt% filled PA6 shows the best quality when it is printed at the temperature of 240°C and speeds between 60-80 mm/s. The aforementioned method can also be used for optimizing of other existing or new materials where good surface finishing and high dimensional accuracy is required.

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