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Experimental characterization and evaluation of additively manufactured PEEK dies for polymer profile extrusion

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Abstract

The challenge in prototyping of new extrusion profiles is the high development cost to develop and manufacture the corresponding extrusion dies. Thus, tooling process chains based on polymer additive manufacturing (AM) are of industrial interest to manufacture functional dies capable to produce a small batch of prototype extruded profile. The work presented here concerns the production and testing of a series of carbon fibre reinforced polyether-ether-ketone (PEEK) dies in polymer profile extrusion. The die geometry has been optimized by adopting a conical design to improve the die flow and the melt flow distribution. Therefore, the material selection and improved geometry design are implemented to withstand the combination of high temperature and pressure typical of the extrusion process. The dimensional and form measurement using a tactile coordinate measuring machine (CMM) was performed before experimental testing to investigate the accuracy and reproducibility of dies manufactured by the fused filament fabrication (FFF) process. From the measurement of 15 samples, the accuracy of dimensional measurement was identified by the absolute deviation between 39 µm and 435 µm depending on reference dimension. Relative standard deviation to the reference dimension of 0.2% and 2.35% indicates the reproducibility of FFF process. Furthermore, surface characterization of internal die was performed using replica technology in a focus variation 3D optical microscope. The surface roughness was evaluated using an average arithmetical mean height (Sa) value of 17.3 µm with standard deviation of $\pm$1.9 µm. PEEK die was experimentally tested with Polypropylene (PP) as material extruded. As the result, PEEK die was able to withstand the extrusion process conditions, yielding up to 330 meters of extruded product.

Additive Manufacturing, Polymer Extrusion, Fused Filament Fabrication, Tooling

1. Introduction

Tooling process chains based on polymer AM (soft tooling) are of industrial interest to manufacture small batches in prototyping of new extrusion profile with lower cost compared to metal AM (hard tooling). Hard tooling has shown the results comparable to conventional tooling in terms of dimensional accuracy and mechanical strength [1, 2]. However, studies in soft tooling have reported that the materials are prone to failure. The material properties of polymer AM cannot withstand the demanding process condition of profile extrusion. Earlier work indicates that conical die design can improve the melt flow distribution [3]. Die design aims for uniform melt flow, so that the flow avoids the abrupt pressure drop [4]. Thus, uniform melt flow reduces the mechanical impact to soft tooling during process extrusion.

Recent development in materials, carbon-fibre reinforced material has been increasingly applied due to its capability to improve the mechanical and thermal properties. Thus, the material selection as well as the improved die geometry are expected to mitigate the issues in soft tooling. FFF and FDM are the preferred process for printing composite material since the melted polymer with reinforcement material is uniformly extruded through the nozzle and deposited into the desired cross section of printed object [5]. However, FFF has the limitation of reduced surface quality because of the layering of filament [6].

Previous work has presented the dimensional and surface characterization of PEEK dies [7], this work is the continuation of dimensional and form measurement from different features, internal die surface characterization using a replica technology, and experimental testing of PEEK die.

2. Methodology

2.1. Measurement procedure

The dimensional measurement was performed in three different die designs each of them produced in five different samples as shown in Figure 1.

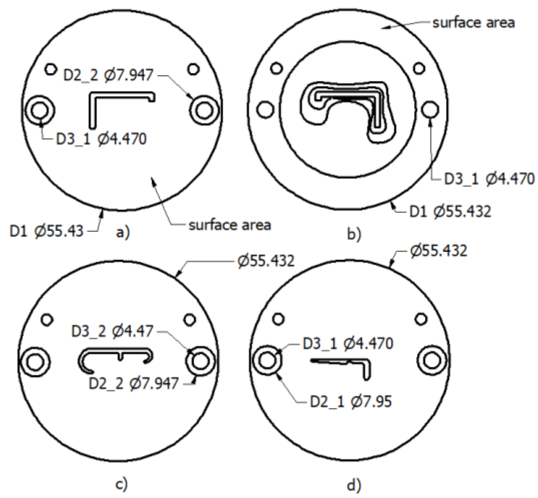
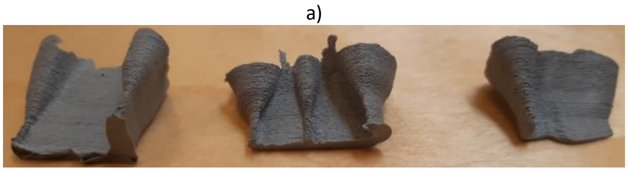


Figure 1. Extrusion die profile designs with main dimensions shown in mm: a) MEK01 Front, b) MEK01 Back c) MEK02, and d) MEK03

The measurements were conducted in each die using a tactile coordinate measuring machine (CMM) with stylus size of diameter Ø 3mm. The uncertainty contributed from maximum permissible error of CMM Duramax ($MPE_{Duramax}$) is 2.4 +L/300 µm where L is the measured length in mm. The objective of the measurement is to investigate the accuracy and reproducibility of FFF process for both dimensional and form measurements.

Internal die surface characterization was performed using replica technology. Thus, the acquisitions setting was set for negative or replica mode in Alicona Infinitefocus 3D optical microscope with magnification of 20x. Figure 2 a) and b) show the preparation of replicas using RepliSetGF1 Dispensing System, a rapid curing silicone for 3D high resolution replicas and the replicas after removing from the dies, respectively

a)



b)

Figure 2. a) The preparation of replica inside the dies. b) Replicas of profile MEK01, MEK02 and MEK03 from the left to the right.

2.2. Experimental testing procedure

The experimental testing was performed with die design of MEK-01. Experimental testing was aimed to test whether the die can withstand the maximum screw speed of extruder and to extrude around 300 meters. The material extruded was PP with process temperature as shown in Table 1. Zone E1, E2, and E3 correspond to the process inside single screw extruder: feeding, melting, and melt conveying zone, respectively.

Table 1. Process Temperature of PP

Zone E1 [°C]	Zone E2 [°C]	Zone E3 [°C]	D2 [°C]	D1 [°C]
190	200	205	215	215

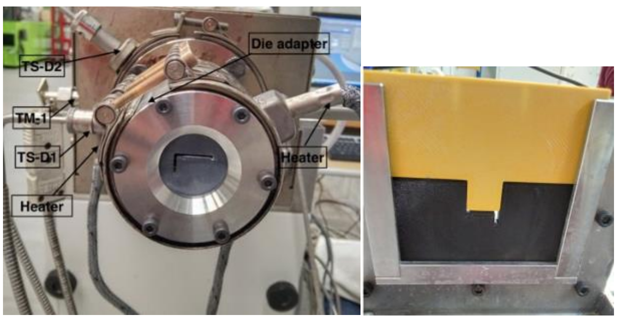


Figure 3. a) Experimental set up of PEEK MEK-01 Die assembled inside Die adapter b) Calibration die

D2 and D1 are heat ring upstream the die and heat ring on the die, respectively. Temperature and pressure sensors and heaters were mounted on the heat ring as shown in Figure 3 a). TS and TM stand for temperature sensor and temperature of melt, respectively. Furthermore, die was assembled inside the die adapter and the heat ring. Die adapter was required as the adapter connection from the die to the extruder. Extrudates was calibrated using calibration die which was designed oversized by 1mm from the original die design profile dimensions. Calibration die was manufactured using rapid prototyping Ender 3 printer with materials of PLA 0.4 mm for bottom part and PLA 0.8 mm for top part, the setup of calibration die is shown in Figure 3 b).

3. Measurement and Data Analysis

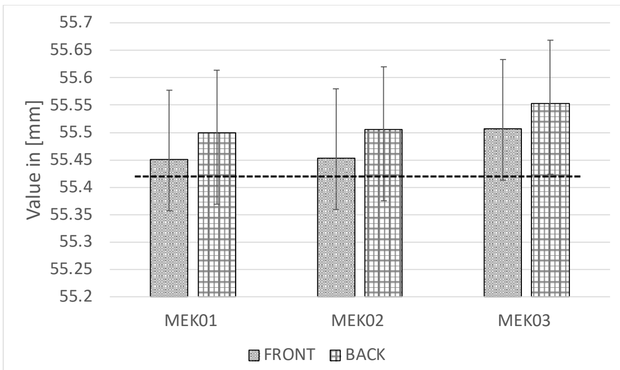
3.1. Dimensional Measurement

With reference to figure 1 the measurement campaign quantified and measured diameters D1, D2, D3 and T (Thickness) for front side, and D1 and D3 for back side. The measurement was performed once across 15 samples. Thus, the measured average is the average measurement from 15 samples. The deviation is the difference between the reference value and the average measured value. The reference value is based on the designed dimensions in CAD file. The deviation between 39 µm and 435 µm implies the accuracy of FFF process, as indicated in Table 2. Furthermore, FFF process achieves the reproducibility with relative standard deviation of 0.2% and 2.3% subject to features size.

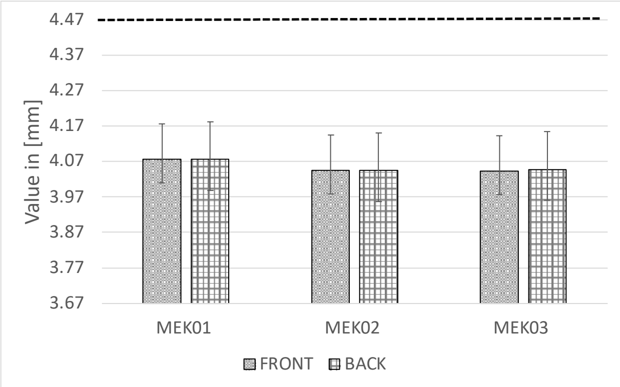
Figure 4 indicates the average measurement of D1 and D3 for both front and back side with standard deviation in error bar. D3 has the highest deviation to reference value but it is still within dimensional tolerance of ±500µm. High deviation was contributed by the stylus size diameter of Ø 3mm, while the measured diameter of D3 is 4.470 mm. Measurement of D3 is quite consistent for both front and back side implied by lower standard deviation, whereas the measurement of D1 is higher in back side compared to front side.

Table 2. Dimensional measurement of Front and Back Side

Description	Ref. value [mm]	Meas. Average [mm]	Deviation [mm]	STD. Dev [mm]	Relative STD. Dev [%]
Front side					
D1	55.432	55.471	0.039	0.111	0.2
D2_1	7.947	7.626	0.322	0.072	0.9
D3_1	4.470	4.050	0.421	0.078	1.8
D2_2	7.947	7.616	0.332	0.100	1.3
D3_2	4.470	4.061	0.410	0.083	1.9
T	33.300	33.228	0.072	0.238	0.7
Back Side					
D1	55.432	55.519	0.088	0.127	0.2
D3_1	4.470	4.077	0.393	0.096	2.2
D3_2	4.470	4.035	0.435	0.101	2.3



a)



b)

Figure 4. Bar chart of average dimensional measurement with standard deviation in error bar for both front and back side of a) Diameter 1. b) Diameter 3.

The forms measurement was conducted for flatness and cylindricity. Flatness indicates the unevenness in the surface area of D1 as indicated in figure 1 a) and b). The flatness measurement in CMM was conducted by putting stylus in several probing probe points or following polylines path defined across the surface area of D1. The flatness and cylindricity have standard deviation between 26 µm and 203 µm indicated as the error bar in line chart as shown in Figure 5. As observed, Flatness2 (F2) has higher deviation compared to Flatness1 (F1). F2 was measured using both probing points and polyline whereas F1 was measured using probing points only. Therefore, F2 is more precise than F1 because it has more points or surface area coverage. In addition, back side flatness has lower deviation due to smaller surface area. Cylindricity verifies the roundness of cylindrical form of D1, D3_1 and D3_2. CMM checked the cylindricity by putting several probing points in the cylindrical form. The cylindricity of D1 has higher deviation compared to the cylindricity of D3_1 and D3_2. Moreover, the cylindricity of D3_1 and D3_2 across all samples has similar value and low deviation.

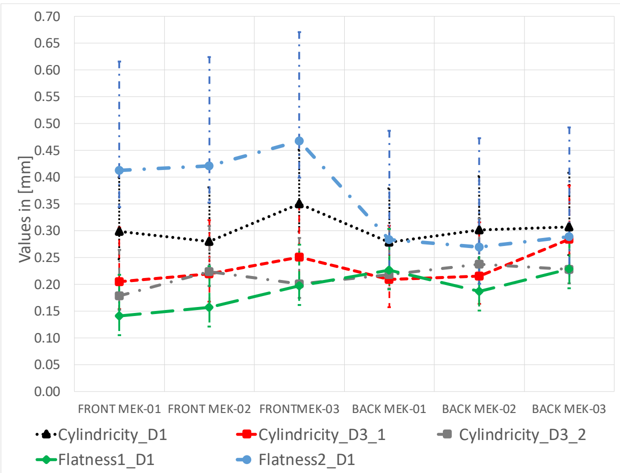


Figure 5. Flatness and cylindricity line chart with error bar of standard deviation

3.2. Surface characterization

Surface topography of internal die was characterized from the image of replica by setting negative mode in Alicona 3D optical microscope. Furthermore, the acquired image from 3D optical microscope were all equally levelled and filtered using S-filter with cut-off values of λs = 0.25 µm using SPIP image metrology software. As shown in Figure 6, surface topography of internal die consists of sharp peaks and sharp valleys which resembles the deposition of filament layers from FFF process.

The measurement was conducted repeatedly in three points in the y-direction, 3mm, 6mm and 9mm from die outlet for each sample. Each die design has two samples of replica, thus from eighteen measurements an average Sa value of 17.326 µm with standard deviation of ±1.978 µm was obtained. It was quite high, because the arithmetical mean roughness (Ra) of extrudates with smooth surface is around 1–2 µm. Figure 7 shows the bar chart of Sa value from six samples with standard deviation in error bar. Based on Sa value, the smoothest surface is from MEK01-samples1 with average Sa value of 16.022 µm, whereas the roughest surface is from MEK02-samples2 with average Sa value of 18.024 µm.

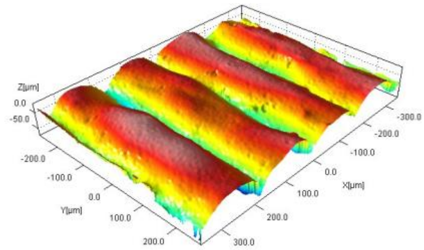


Figure 6. Surface topography of internal die with cross section of 700 µm x 500 µm in y-direction of 3 mm from the die outlet.

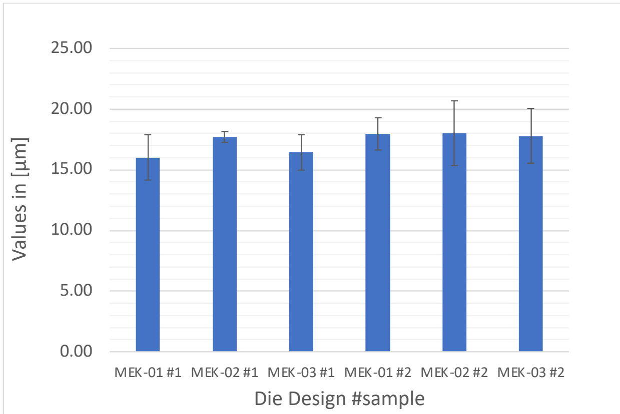


Figure 7. Arithmetical Mean Height (Sa) of each die designs.

4. Experimental Testing Result

The experimental testing was performed starting from low screw speed of $0.52s^{-1}$. The screw speed was slowly increased and timed to extrude 30 meters as shown in Table 3. Increasing screw speed resulted in a linear increase of die head pressure. Process temperature at maximum screw speed of $3.09s^{-1}$ is shown in Table 4. The temperature at each zone achieved the process temperature as set in Table 1. Melt temperature of 219.4°C was observed higher compared to die temperature of 215°C. PEEK die sustained the demanding process condition of profile extrusion without tool breaking until the maximum screw speed of $3.09s^{-1}$. It continued to extrude for another 150 meters for 21 minutes at the maximum screw speed. In total PEEK die yielded 330 meters of extrudates with total duration of 82 minutes 15 seconds.

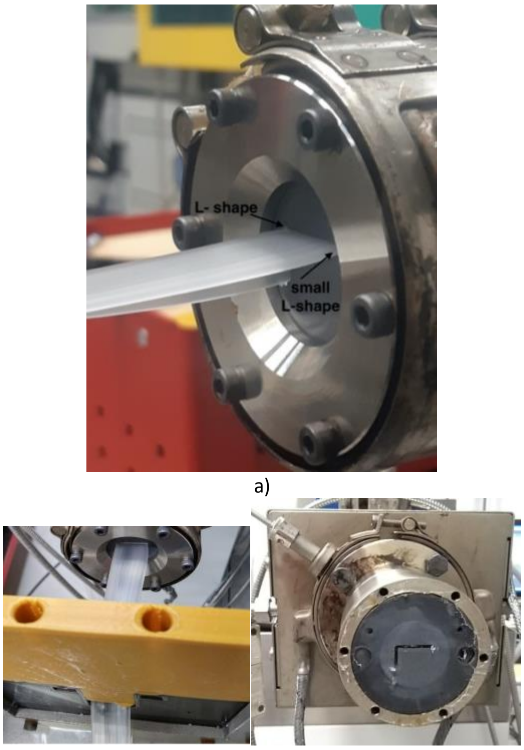
As observed in Figure 8 a), extrudates followed L-shape of profile MEK01. However, the small L-shape in the right end was quite difficult to achieve. Calibration die did not sustain the process temperature of extrusion due to limitation of PLA as the material, as shown in Figure 8 b). Consequently, the L-shape was not achieved as the extrudate was calibrated using other calibration dies. As observed in Figure 8 c), there is no sign of tool breaking from PEEK die even though PEEK die was expanded after experimental testing due to the combination of high process temperature and pressure of extrusion.

Table 3. Experimental testing parameters and results

Screw speed [1/s]	Line speed [m/min]	Time [min]	Pressure [MPa]	Meter extruded
0.52	1.2	25	1.23	0-30
1.03	2.4	12.5	1.74	30-60
1.56	3.6	8.33	2.06	60-90
2.09	4.8	6.25	2.29	90-120
2.61	6	5	2.39	120-150
3.09	7.2	4.17	2.62	150-180
3.09	7.2	21	2.58	180-330

Table 4. Experimental testing process condition at max. screw speed

Des cription	Zone E1 [°C]	Zone E2 [°C]	Zone E3 [°C]	D2 [°C]	D1 [°C]	Melt (TM) [°C]	P [MPa]
Setting	190	200	205	215	215		
Actual	190	200.1	207.5	215.8	215.1	219	2.62



a)

b) c)

Figure 8. Experimental testing results a) Extrudates from PEEK die during extrusion. b) Extrudates passing through calibration die. c) PEEK dies after experimental testing.

5. Conclusion

Dimensional and form measurements were performed to evaluate the reproducibility and the accuracy of FFF technique. The reproducibility was achieved by FFF process within 0.2% and 2.35% standard deviation relative to reference dimension and subjected to its features size. The accuracy was indicated by the absolute deviation between 39 µm and 435 µm. Although, dimensional deviation is still observed in small features, FFF process meets the product dimensional specification. Furthermore, surface roughness was evaluated using an average Sa value of 17.3 µm with standard deviation of ±1.9 µm. The value was considered quite high compared to smooth surface extrudates with an average Ra value of 1-2 µm. PEEK die was experimentally tested with Polypropylene (PP) as the material of extrudates. As the result, PEEK die sustained the extrusion process conditions, yielded up to 330 meters of extruded product with total duration of 82 minutes. Future work is to investigate the surface roughness of extrudates and the influence of screw speed to the surface roughness. It is expected that higher screw speed yields into a smoother extrudates. In addition, the calibration die should be manufactured with suitable material and manufacturing techniques so that it can sustain the process temperature of extrusion.

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