Surface Design of 3D-printed PEEK by Controlling Slicing Parameters

Carlo S. Emolaga[1], Shaun Angelo C. Arañez[1], Persia Ada N. de Yro[1,2], Jocelyn P. Reyes[1], Brigida A. Visaya[1], Blessie A. Basilia [1,2], and Rigoberto C. Advincula[3]
[1]Industrial Technology Development Institute, Department of Science and Technology, Bicutan, Taguig City, Philippines
[2]School of Chemical, Biological, and Materials Engineering and Sciences (CBMES), Mapua University, Muralla St., Intramuros, Manila
[3] Case Western Reserve University, Cleveland, Ohio, USA
Email: csemolaga@itdi.dost.gov.ph

***Abstract*—Additive manufacturing of high-performance materials such as polyether ether ketone (PEEK) is constantly gaining attention because of its applications in diverse fields. PEEK is a semicrystalline thermoplastic that exhibits superior mechanical properties, biocompatibility, and wear resistance that makes it suitable for biomedical and other industrial applications. Most of these applications call for surface designs where roughness and porosity are a major consideration. In this study, PEEK samples with different surface designs were prepared by modifying slicing parameters such as wall line, infill density, and raster angle. The samples were printed using fused deposition modelling and were characterized using a non-destructive method, X-ray micro computed tomography (X-ray micro-CT). X-ray tomograms and void content analysis show that voids usually occur at the junction between the walls and the infill for all three designs. Reducing the infill travel path by adding inner walls resulted to higher defect volume ratio. Defect volume ratio increased from 0.06% to 0.36% after the addition of inner walls. Reduction in infill density further increased the defect volume ratio. These results show that different surface and internal designs can be prepared by modifying slicing parameters and its defects/void content can be readily evaluated by X-ray micro-CT.**

***Index Terms*—PEEK, FDM, 3D printing, slicing, X-ray computed tomography**

I. Introduction

Polyether ether ketone (PEEK) is a semicrystalline thermoplastic that is used for high performance engineering applications due to its desirable characteristics such as high melting and glass transition temperatures (Tm=340ºC, Tg=143 ºC) [1, 2, 3]. PEEK and its composites are also reported to have widespread applications in biomedical industry [4, 5]. The development of PEEK-based medical devices and other components however can be very difficult using the traditional manufacturing techniques because of the existence of patient specific parameters that generally demands specificity [4]. Additive Manufacturing is currently being explored to overcome these existing manufacturing hurdles as it is capable of building physical 3D geometries layer by layer [4]. Compared to conventional manufacturing, 3D printing also offers other advantages including manufacturing of complex microstructured geometries [6].

One of the additive manufacturing techniques is fused deposition modeling (FDM). In this process, a coil of feedstock thermoplastic filament is initially stacked into the printer and then deposited in form of a thin layer onto a heated plate, following the pattern prescribed by the G-code instruction generated by the software controlling the machine. After the completion of one layer, the bed or nozzle head moves one step down, equal to layer height, and prints the successive layer until the object is completely printed [7,8].

The final effect of the manufactured part is influenced by a number of factors such as method, material, and process parameters. The process parameters are directly connected to the accuracy of 3D-printed parts [9]. These FDM printing parameters can be geometry-based (e.g. nozzle size and filament size), process-based (e.g. melting temperature, printing speed, and bed heat) or structural-based (e.g. layer thickness, infill geometry, infill density, number of layers, and raster angle) [6,10,11]. While Le Duigu et al [6] showed the effects of slicing parameters on the properties of continuous flax/PLA composites, others [10,11] only give a general picture of how these parameters can affect the properties of commonly used polymers such as PLA, ABS, TPU, and nylon. The effect of slicing parameters on the properties of 3D-printed PEEK is therefore worth investigating.

One non-destructive inspection technique that provides high-resolution, high-quality, structural 3D information of different types of materials is X-ray micro computed tomography (micro-CT). This method can be used to characterize and quantify internal structures, dimensions, density, voids, and other manufacturing defects that can help better understand the expected performance of the materials [12,13]. In terms of surface measurements, the values obtained from CT scans are in a good agreement

Manuscript received July 21, 2021; revised November 1, 2021.

doi: 10.18178/ijmerr.11.3.181-186

with evaluation of the same surfaces using traditional surface measurement techniques [13].

In this study, PEEK samples with different surface designs were printed using an FDM printer and evaluated using X-ray micro computed tomography. This is to assess the effects of the slicing modifications on the morphology of the 3D printed specimens using a nondestructive method. This is also to confirm the resolution and fidelity of the printed samples to the original CAD design.

II. Materials and Methods

The samples were printed using Intamsys Funmat HT FDM Printer (Intamsys Technology, Ltd., Shanghai, China). This printer has a build volume of 260x260x260 mm^3 with nozzle temperature up to 450ºC, build plate temperature up to 160ºC, and chamber temperature up to 90ºC. A commercial PEEK filament (ThermaX^{TM}, 3DXTECH, Michigan, USA) with a diameter of 1.75 mm was used for the printing of PEEK samples. This filament can be used for printing samples under the following printing conditions: extruder temperature of 375-410ºC, Bed temperature of 130-145ºC, and heated chamber temperature of 70-140ºC.

A. FDM Printing

The Surface Tessellation Language (STL) file of a 16.0 x 16.0 x 3.0 mm^3 sample was created using Solidworks 2018 (Dassault Systemes, Waltham, Massachusetts, USA) while the print path code and other parameters were generated using the slicing software IntamSuite (version 3.5.3, Intamsys). The printing parameters used for the printing of PEEK samples are shown in Table I.

TABLE I. Printing Parameters for PEEK

Parameter	Value
Nozzle diameter (mm)	0.4
Nozzle Temperature (ºC)	380
Buildplate Temperature (ºC)	130
Chamber Temperature (ºC)	65
Print Speed (mm/s)	15
Material Flow (%)	100

B. Designs

The different surface designs were created by modifying some of the slicing parameters of the same STL file. The summary of differences in the slicing parameters are shown in Table II while the different designs and their corresponding printing patterns are shown in Fig. 1. The design was based on a sample used for cell tests. These three designs represent some of the slicing modifications that are applicable to this application.

TABLE II. Slicing Parameters of the three Designs

	Design 1	Design 2	Design 3
Layer height (mm)	0.2	0.2	0.2
Wall line count	1	5	1
Infill density (%)	100	100	60
Raster angle (deg)	+45/-45	+45/-45	+90/-90

C. Characterization

The different 3D-printed PEEK samples were characterized using 3D X-ray Computed Tomography (North Star Imaging, Inc. USA). The system is equipped with X-ray source of microfocus tube type with a focal spot size of 16 μm. The energy beam used was 40kV and the distance from the specimen and the detector was set to 56.744 mm. Data acquisition was carried out with exposure duration of 30m 23s and no pre-filtration. The number of projections was set to 2000 projections. The NSI efX-CT software was used for data visualization and reconstruction of the samples. VGStudio software was used to obtain the porosity/void content of the different samples.

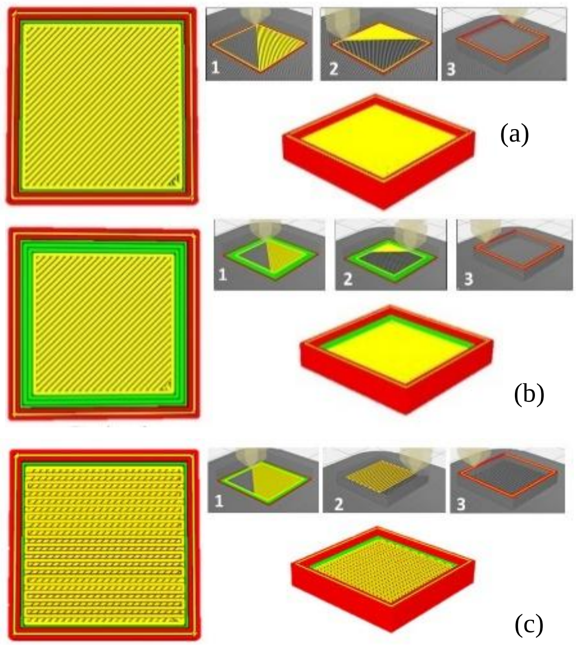


Figure 1. Printing patterns for the different surface designs (a) Design 1, (b) Design 2 and (c) Design 3

III. Results and Discussion

Fig. 2 shows the images of the 3D-printed PEEK disks. All samples show the characteristic beige color of PEEK and indicates that the nozzle temperature used for printing did not cause any degradation of the sample. PEEK that reach the thermal degradation temperature usually starts to form darker degraded polymer at the outlet of the 3D printing nozzle [14].

Figure 2. Images of the 3D-printed PEEK coupons showing the different surface designs

In the printing of PEEK specimen using various FDM 3D printers, Wang et al [3] reported that the 3D-printed objects appear to be within reasonable resolution and achieve near fidelity over the original CAD design but noted that higher scrutiny must be observed with the printing of much smaller objects. Since this study involves much smaller objects than those investigated by Wang et al [3], measurements of the dimensions of the printed PEEK samples were also determined and summarized in Table III. All designs show more than 99% conformity to the designed dimension in terms of printing in the x and y directions (length and width) but exhibited a higher deviation when printing in the z direction (thickness). Wang et al [3] also observed this in their samples printed using Hyrel and Intamsys. This noticeable deviation in the z direction is because of the set layer height (0.2 mm) that caused the nozzle to compress the filament on the bed and the previous layer [6].

TABLE III. AVERAGE MEASUREMENTS OF 3D-PRINTED SAMPLES

	Designed Dimension (mm)	3D printed measurements/designed dimension x 100		
		Design 1	***Design 2***	***Design 3***
Length (mm)	16.0	99.6±0.47	99.8±0.22	99.1±0.55
Width (mm)	16.0	99.4±0.42	99.3±0.22	99.5±0.85
Thickness (mm)	3.0	95.3±4.36	92.1±1.39	95.6±4.62

The surface of the different samples shown in Fig. 3 reveal some dents between the outer wall and the infill for Design 1 (see arrow). Design 2 showed a more uniform surface from the outer wall up to the infill due to the addition of several layers of inner wall or contours. The grooves on the surface of Design 3 were generated by reducing the infill density and choosing the zigzag pattern during the slicing phase. The layer below the grooves however shows visible gaps between the layer lines (encircled).

The surface topography of the different 3D-printed PEEK samples was also analysed using X-ray computed tomography. This non-destructive method is a powerful tool not only in defect detection, but also in dimensional analysis, density measurements, and surface roughness analysis. Fig. 4 shows that PEEK samples printed using Designs 1 and 2 have fine lines that are 540 to 550 µm apart and are around 70 µm high. These fine lines were actually created using the ironing option in the slicing software. It can also be noticed that in Design 2, these fine lines only appear a few millimetres from the wall due to the additional contours/inner wall. The dimension of the grooves on Design 3 was also measured using X-ray CT. Unlike the fine lines in Designs 1 and 2, the grooves in Design 3 are more defined with lines around 300 µm thick, 490 µm high, and around 600 µm apart. The 2D images were taken from the planes in the middle of the x, y, z directions, as shown by the accompanying cut images of each sample. The images of Design 1 in both the xz (a) and yz (c) planes show good layer adhesion along the z-axis but showed some large gaps between the outer wall and the infill (encircled). The good adhesion along the z axis is due to the fact that during printing, the nozzle compresses the filament on the bed or the previous composite layer [6]. This shows that the deposited PEEK lines during infill printing did not fuse effectively to the previously printed walls. This was also observed in the work of Rinaldi et al [7]. The orientation of the printed filaments (±45º) may also have contributed to this. Design 2 reduced the gap between the outer wall and the subsequent layers, producing a more solid cross-section along the xz and yz planes. This however resulted to the creation of smaller voids between the inner wall lines (see arrow). The voids observed in Design 3 are a consequence of the reduction in infill density. The representative layers along the xy plane show that Design 1 has a more uniform appearance while Design 2 shows voids especially between inner wall lines. Other studies noted that the bottom layers (i.e. the layers closer to the heated plate) appear more compact with very few pores of small dimensions and increases with increasing thickness of the printed part. They ascribed the formation of the pores to factors such as the thermal mismatch during filament deposition and surface roughness of the printed part. Although this is not observed in the printed samples (probably due to the thin samples), the same phenomenon could be ascribed to the formation of voids between extruded lines. Due to the low thermal conductivity of the solid PEEK, the newly deposited line is adjacent to a previously deposited line that is cooler than the heated plate. Surface roughness of the printed PEEK also plays a role leading to less contact areas and weaker adhesion [7].

Fig. 5 shows the void distribution in the different PEEK samples. It is easy to see that the voids in all designs are mostly concentrated in the areas where the outer wall and the infill meets. Design 2 also shows voids on the corners of the disk/coupon. This is probably where the starting and endpoint of the lines meet. Table 4 shows the summary of the void content of the samples. Design 1, despite the observed gaps between the wall and the infill showed the lowest defect volume while Design 3 showed the highest defect volume because of the intentional decrease in infill density.

These results show that good and uniform adhesion between the layers or deposited material relies on a well-controlled thermal environment in the printing process. Thermal gradient across layers must therefore be minimized to promote good adhesion. This is actually more challenging for PEEK because of its high crystallization speed and melting point. One way to overcome this is to decrease the cooling time for each layer to prevent thermal gradient and poor layer adhesion [15]. The printing of additional contours in Design 2 must have allowed the walls to cool before the deposition of the next layer causing the observed gaps between the adjacent lines.

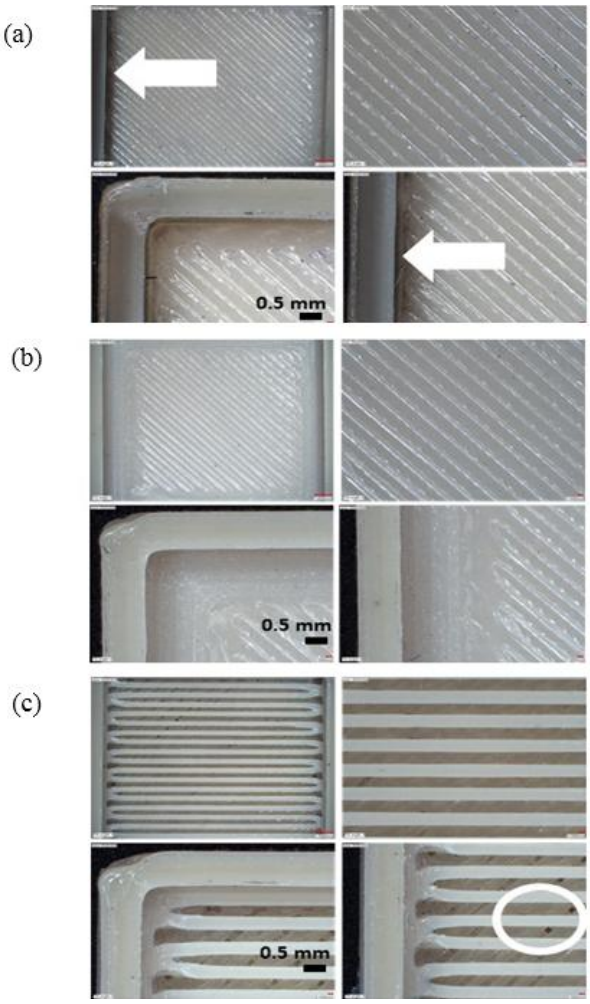
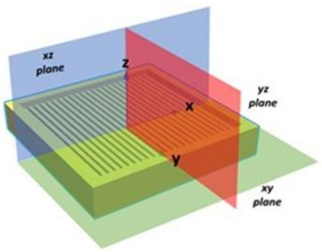
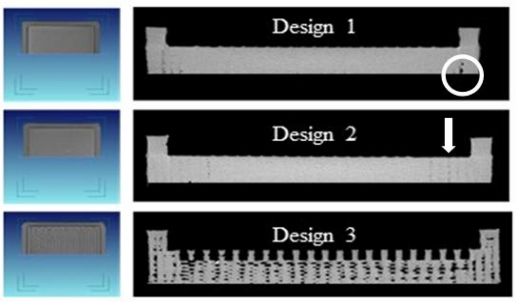
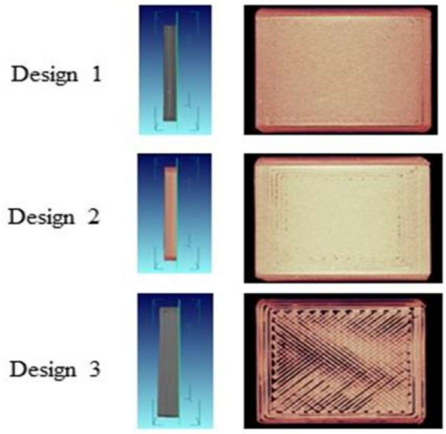


Figure 3. Optical images of the 3D-printed PEEK coupons showing (a) the dents between the outer wall and infill in Design 1 (white arrows), (b) the uniform surface of Design 2, and (c) the gaps between the layer lines in Design 3 (encircled).

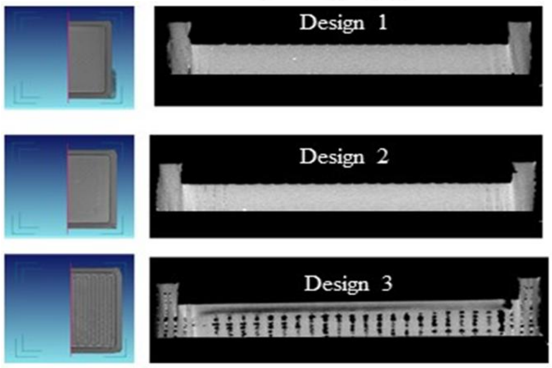




a) Along xz plane



b) Along xy plane



c) Along yz plane

Figure 4. 2D images of the different 3D-printed PEEK samples taken at different planes

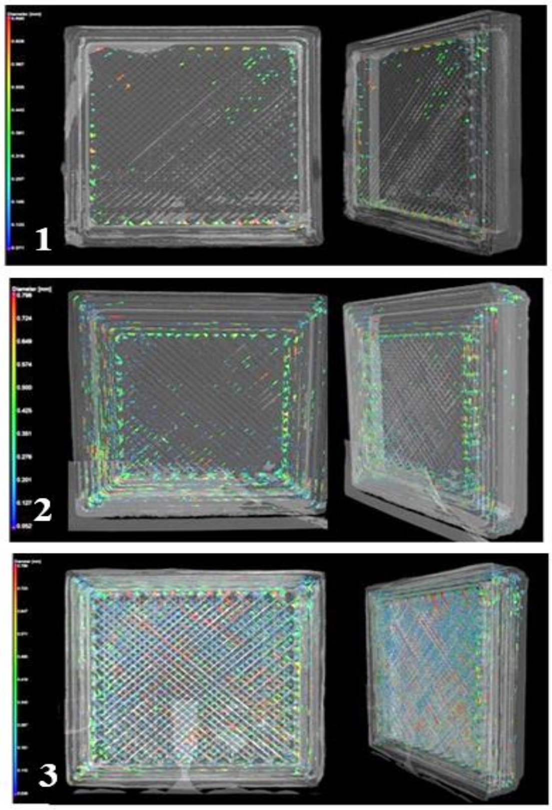


Figure 5. Void distribution in the PEEK samples with different surface designs. The number on each image signify the design number.

TABLE IV. VOID CONTENT OF THE PRINTED PEEK SAMPLES

	Design 1	Design 2	Design 3
Total Volume (Voids) mm ^{3}	0.304	1.635	2.578
Total Surface Area (Voids) mm ^{2}	34.092	187.046	457.140
Defect Volume Ratio	0.06%	0.36%	0.57%

The results also show the great potential of creating PEEK designs with various surface topography and porosities. Porosity and pore size, for example, are important in developing scaffolds for biomedical applications. The minimum requirement for pore size is usually 100 µm due to cell size, migration requirements, and transport while pore size higher than 300 µm is recommended for the enhancement of new bone formation and the formation of capillaries [9].

IV. CONCLUSION

This study shows that modifications in the slicing parameters could produce PEEK samples with different surface designs. Surface designs are important in many applications of PEEK, especially in the medical field where roughness and porosity are often a major consideration. Even for smaller samples, FDM printing using PEEK displays high fidelity to the designed dimensions. X-ray tomograms and void content analysis show that voids usually occur at the junction between the walls and the infill for all three designs. Reducing the infill travel path by adding inner walls or contours however resulted to higher defect volume ratio, although samples with more inner walls appear to be more compact by visual inspection. This is illustrated by the higher defect volume ratio of PEEK samples printed using Design 2 (defect volume ratio of 0.36%) than the samples printed using Design 1 (defect volume ratio of 0.06%). Reduction in infill density also further increased the defect volume ratio. These results also highlighted the use of X-ray micro-CT as a powerful non-destructive tool in evaluating the properties of the 3D-printed PEEK samples.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Authors contributed to the planning, testing, analysis, and the writing of the manuscript.

ACKNOWLEDGMENT

Authors would like to acknowledge Mr. John Kenneth Valerio of the Advanced Device and Materials Testing Laboratory (ADMATEL) of DOST-ITDI, Philippines for the 3D CT Xray Analysis; Engr. Cyron Custodio of MATDEV for the optical images of the printed samples; and Engr. Denise Florante of the DOST-Metals Industry Research and Development Center (DOST-MIRDC) for the preparation of the STL file.

REFERENCES

[1] M. Thiruchitrambalam, D. B. Kumar, D. Shanmugam, and M. Jawaid, “A review on PEEK composites–manufacturing methods, properties and applications,” *Materials Today: Proceedings*, vol. 33, pp. 1085–1092, 2020.

[2] D. Kumar, T. Rajmohan, and S. Venkatachalapathi, “Wear behavior of PEEK matrix composites: A review,” *Materials Today: Proceedings*, vol. 5, pp. 14583-14589, 2018.

[3] R. Wang, K. J. Cheng, R. C. Advincula, and Q. Chen, “On the thermal processing and mechanical properties of 3D-printed polyether ether ketone,” *MRS Communications*, 2019.

[4] S. Singh et al, “3D printing of polyether-ether-ketone for biomedical applications,” *European Polymer Journal*, vol. 114 pp. 234-248, 2019.

[5] H. Spece, et al, “3D printed porous PEEK created via fused filament fabrication for osteoconductive orthopaedic surfaces,” *Journal of the Mechanical Behavior of Biomedical Materials*, vol. 109, 103850, 2020.

[6] A. L. Duigou, G. Chabaud, R. Matsizaki, and M. Castro, “Tailoring the mechanical properties of 3D-printed continuous flax/PLA biocomposites by controlling the slicing parameters,” *Composites Part B*, vol. 203, 108474, 2020.

[7] M. Rinaldi, et al, “Additive layer manufacturing of poly (ether ether ketone) via FDM,” *Composites Part B*, vol. 145, pp. 162–172, 2018.

[8] M. Subramaniyan, P. Eswaran, A. Appousamy, S. Raju, V. Rahini, T. R. Madhumitha, and R. Thisha, “A survey on applications of additive manufacturing techniques in tissue engineering,” *Materials Today: Proceedings*, vol. 45, pp. 8036-8040, 2021.

[9] P. Szymczyk-Ziolkowska, M. B. Labowska, J. Detyna, I. Michalak, and P. Gruber, “A review of fabrication polymer scaffolds for biomedical applications using additive manufacturing techniques,” *Biocybernetics and Biomedical Engineering*, vol. 40, pp. 624-638, 2020.

[10] M. M. Prabhakar, A. K. Saravanan, A. H. Lenin, I. J. Leno, K. Mayandi, and P. S. Ramalingam, “A short review on 3D printing methods, process parameters and materials,” *Materials Today: Proceedings*, vol. 45, pp. 6108-6114, 2021.

[11] B. S. Rupal, K. G. Mostafa, Y. Wang, A. J. Qureshi, “A reverse CAD approach for estimating geometric and mechanical behavior of FDM printed parts,” *Precedia Manufacturing*, vol. 34 pp. 535-544, 2019.

[12] S. Sommacal, A. Matschinski, K. Drechsler, and P. Compston, “Characterization of void and fiber distribution in 3D printed carbon-fiber/PEEK using X-ray Computed tomography,” *Composites Part A.*, vol. 149, 106487, 2021.

[13] M. R. Khosravani and T. Reinicke, *Journal of Nondestructive Evaluation*, 39 75, 2020.

[14] T. Jayaraghul et al, “On the use of x-ray computed tomography in assessment of 3D-printed components,” *Materials Today: Proceedings*, vol. 44, pp. 2963-2967, 2021.

[15] C. Basgul, D. MacDonald, R. Siskey, and S. Kurtz, “Thermal localization improves the interlayer adhesion and structural integrity of 3D printed PEEK lumbar spinal cages,” *Materiala*, vol. 10, 100650, 2020.

Carlo S. Emolaga is a Senior Science Research Specialist at the Materials Science Division, Industrial Technology Development Institute, Department of Science and Technology (DOST-ITDI). He holds a BS and MS in Chemistry degree from the University of the Philippines. His research interests include natural fiber-thermoplastic composites, nanoclay production, plastic recycling, and additive manufacturing.

Shaun Angelo C. Arañez is a Licensed Mechanical Engineer and currently a Science Research Specialist of the Materials Development Laboratory of the Industrial Technology Development Institute, Department of Science and Technology (DOST-ITDI). He currently works on the use of various materials for additive manufacturing.

Persia Ada N. de Yro, PhD, is a Senior Science Research Specialist at the Materials Science Division, Industrial Technology Development Institute, Department of Science and Technology (DOST-ITDI). She holds a BS in Metallurgical Engineering; and MS and PhD in Materials Science and Engineering from the University of the Philippines, Diliman. She is currently an Associate Professor at the MAPUA University.

Jocelyn Paz Reyes is a licensed Chemical Engineer and holds a Master of Science Degree in Chemistry. She is a Science Research Specialist at the Materials Science Division, DOST-ITDI. In addition to additive manufacturing, she also has strong technical background on nanomaterial synthesis, characterization, composites, and nanosafety.

Engr. Brigida A. Visaya is a Senior Science Research Specialist of the Industrial Technology Development Institute - Department of Science and Technology (ITDI-DOST), Philippines. She is a licensed Chemical Engineer and has completed her Masters in Public Management with emphasis on Technology Enterprise Development at the Ateneo de Manila University, Philippines. Her research areas include nanomaterial production, polymer nanocomposites, plastic recycling, advanced materials characterization, and nanosafety.

Blessie A. Basilia, PhD, is a Research and Development Leader of the Materials Development Laboratory of the Industrial Technology Development Institute, Department of Science and Technology (DOST-ITDI). She is currently a professor of the School of Graduate Studies, MAPUA University.

Rigoberto C. Advincula, PhD, is Research Professor at Case Western Reserve University, Professor at University of Tennessee and Governor Chair at Oak Ridge National Laboratory. He is an expert in additive manufacturing, polymers, advanced materials, and nanomaterials. His research areas include additive manufacturing, polymers, nanoscience, organic materials, hybrid materials, ultrathin films, polymers and additives for oil and gas, and corrosion.