

IMECE2022-88301

CHARACTERIZATION OF ADDITIVELY MANUFACTURED BETA MATERIALS

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Milwaukee, WI, United States**ABSTRACT**

Additive manufacturing (AM) is transforming industrial production. AM can produce parts with complex geometries and functionality. However, one of the biggest challenges in the AM world is limited material options. The purpose of this research is to develop new material mixtures and determine their mechanical properties for use at the MSOE Rapid Prototyping Center and provide valuable insight into beta materials for use in AM industry. Elastomeric polyurethane (EPU 40) and Rigid polyurethane (RPU 70), resins developed by Carbon3D, are employed for this research. Initially, EPU 40 (100%) and RPU 70 (100%) were used to print tensile and hardness test specimens so that their mechanical properties could be compared to the standard values presented by Carbon3D and used as benchmarks for newly developed material. Mixtures of the two materials, EPU 40 and RPU 70, in multiple ratios were then created and used to print tensile and hardness test specimens. Data collected from tensile and hardness tests show that EPU 40 and RPU 70 can be combined in various ratios to obtain material properties that lie between the two individual components. In addition to developing these new materials, the effect of printing orientation on mechanical properties was also studied in this paper.

INTRODUCTION

Unlike the traditional manufacturing process that involves subtracting (removing) material to achieve the desired shape, Additive manufacturing (AM) builds by adding materials layer upon layer [1] into complex three-dimensional (3D) shapes. 3D topographical mapping methods, which laid the concept of creating 3D printed items in layer upon layer, predate the 1970s. However, the documented evolution of 3D printing technology

started in the 1980s [2]. In its early days, the applications of 3D printing were extremely narrow and limited mainly to prototypes. Nonetheless, most modern AM can produce functional end-use products, and many of them are made of more advanced materials such as ceramics, composites, or even metals [2,3]. As a result, 3D printing has been rapidly adopted for prototyping and production purposes. Today, there are many additive manufacturing processes; they differ in the way layers are deposited to create parts, in the operating principle, and in the materials that can be used. In general, there are five steps in most 3D printing technologies. It starts with design ideation, after which computer-aided designs (CAD) can be created. CAD designs can then be converted to a commonly used boundary representation model, such as Standard Tessellation Language (STL) files, describing the surface geometry of the three-dimensional components. In this process, the drawing made in the CAD software is typically approximated by triangles and sliced, containing the information for each layer that is going to be printed. By using those principles, AM delivers parts of very intricate and complex geometries [4]. Every step that is explained above regarding the general build flow process of 3D printers is shown in Figure 1.

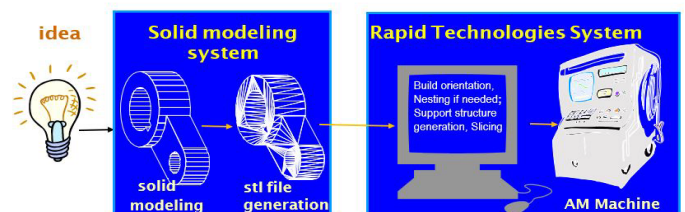


Figure 1. The general build flow process of 3D printers.

The main advantages of AM are associated with the ability to manufacture complex geometries, lighter structures, the ability to allow customization, and the ability to integrate multiple parts into a single component [3].

Through close collaboration with AM industry original equipment manufacturers (OEMs), the MSOE Rapid Prototyping Center (RPC) (and the consortium it supports) obtains early access to new materials and processes through its participation as a beta test site. The RPC produces benchmark parts in the beta material/process for the consortium members and solicits feedback on the performance of the benchmark to share with the OEM. Benchmark parts are typically built in parallel with existing or known solutions. It is also valuable for the RPC to run internal benchmark parts to test and compare physical material characteristics to existing or known solutions. MSOE team involved in earliest efforts with Carbon3D published the challenges and opportunities at IMECE 2017 [5]. The purpose of this research is to develop new materials and establish their mechanical properties as part of the beta testing at the Rapid Prototyping Center.

To develop these new materials, two currently commercially available and industrially used liquid resins called Elastomeric polyurethane (EPU 40) and Rigid polyurethane (RPU 70) were used. Both materials were developed by Carbon3D for their Digital Light Synthesis (DLS) technology. The DLS process is driven by Continuous Liquid Interface Production™ (CLIP™) and programmable liquid resins. CLIP is a photochemical process and uses digital light projection in combination with oxygen-permeable optics. By projecting light through an oxygen-permeable window into a reservoir of UV-curable resin, CLIP cures liquid plastic resin into solid parts using ultraviolet light. As a sequence of UV images is projected, the part solidifies, and the build platform rises [6-9]. EPU 40 is a family of rubber-like materials and is a highly elastic material, and it has low stiffness, which provides a compliant structure. It can replace thermoplastic Polyurethane (TPU) which is used as a soft engineering plastic or as a replacement for hard rubber. EPU 40 can be used for applications that require dampening, energy dissipation, shock absorption, and recovers from very large deformations [10]. RPU 70 was created to replace PC-ABS /& ABS, which is typically used for injection molding applications. RPU 70 can be used for applications that require rigidity, toughness, and moderate heat resistance [11]. After studying the applications and characterization of each material, the process of developing new material started with planning how the two materials can be mixed, as shown in Figure 2.

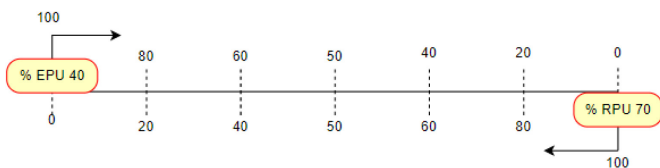


Figure 2. First stage combination ratios for EPU 40 and RPU 70.

The tensile bar for all these combinations was printed using an RPC test specimen, as shown in Figure 3a. To compare the mechanical properties of the two materials to the standard values presented by Carbon3D, we 3D printed both EPU 40 (100%) and RPU 70 (100%) following ASTM standards as shown in Figure 3b & 3c.

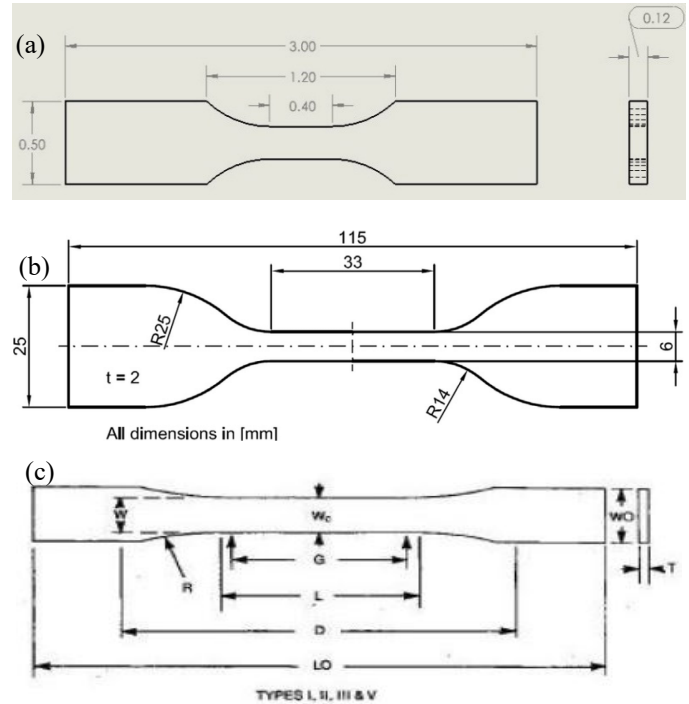


Figure 3. Dimensions of the tensile bar for the RPC test specimen used for all combinations, (b). shows the dimensions of the tensile bar for ASTM D 412 die C, and (c). shows the general shape of ASTM D 638 type V. All dimensions are in [mm]

Concurrently, by blending in 50% each of EPU 40 and RPU 70, the tensile test specimen was 3D printed in both ASTM standards. Then, the tensile tests of the three materials were conducted. Shore (Durometer) test or Rockwell hardness test are commonly used to measure the hardness of plastics. For this research, the hardness of the samples was measured by employing a durometer (Shore A and Shore D scales). The Shore A scale is used for softer rubber-like material, and the Shore D scale is used for harder material [12].

MATERIALS AND METHODS

Following the ASTM standards for EPU 100% and RPU 100%, the 3D models of the tensile specimen were created using SolidWorks. Then to 3D print the specimen, it was necessary to determine the total volume of resin needed for each design. Finally, the determined volume of resin was poured through a resin dispenser gun into the Carbon M2 printer cassette, which was placed in the optical deck. According to RPC, it is essential to use a dispenser gun to get the correct mix of resin photo-initiators to ensure that the intended 3D part prints properly. For

any EPU 40 and RPU 70 combinations, the process is a little different.

Because the correct amount of resin must be well mixed before pouring it into the Carbon M2 printer cassette, the disposable hot cup was placed in the balance, and the balance was tared to read zero. Then, first, the required amount of EPU 40 was dispensed to the disposable hot cup using a dispenser gun. While the disposable hot cup is still in the balance, the balance was tared again to add the required amount of RPU 70. After measuring and obtaining a reasonable total volume of resin, the combined resin was steered for 5 minutes to ensure the two resins are well mixed. Finally, the resin was poured into the Carbon M2 printer cassette. When mixing in and pouring from a disposable hot cup to the M2 printer cassette, the volume might not be the same as the total volume required. Thus, a reasonable amount of volume was added in advance and divided according to the required ratios. For example, if the total required volume of 50% EPU and 50% RPU combinations was 190 ml, then 100 ml of each resin was added, making the total volume 200 ml.

The printing process of all the combinations was the same; to start 3D printing the specimen, the build platform is installed, and the STL file was uploaded to the Carbon3D build site to initiate the print process. The required parameters to run the build, which include the resin type, print orientation, support constructions, and then the print initiation, were entered. For this research, all copies of the test bars (specimens) were printed in the x-y (flat) and z (vertical) orientation, which can also be seen in Figure 4. For each combination, eight specimens were printed (4 horizontally and four vertically). Thus, 3 of each print orientation specimen were used for the tensile test, and one of each specimen was used for the hardness test.



Figure 4. A build platform and ASTM D 412 die C tensile test bars (specimens) in a Carbon M.

Post-processing (RPC protocol)

Once the printing is done, the part is removed gently from the build platform and washed with Filmcol 190. They were then placed in an orbital shaker which has 99% Isopropyl alcohol (IPA) for 1 min at 30s interval in 120RPM for EPU 40 (100%) and 5min at 30s interval in 120RPM for RPU 70 (100%). For

any other combinations depending on their ratio, 1-5 min was used. To remove excess resin from the specimen, the specimen was air pressure dried. After the parts were cleaned and all the supports were removed, the parts were thermally cured by placing them on a non-stick ceramic tray or aluminum foil, then placed in a convection oven (Constant temperature oven (120 ° C) 4 hours.

Tensile Testing of the Specimen

To perform tensile testing, an MTS tensile test machine and TestWorks4 software were used. Throughout this test, a wedge with a maximum load was 5kN was used to hold the specimen. First, the specimen's width and thickness were measured by using the caliper each time before testing the new specimen. After placing the specimen MTS tensile test machine, the grips were fastened, and its grip separation was measured for each test bar. Then all the measured dimensions were added to the TestWorks4 software. Then by applying different loading rates (follow the ASTM standard) for different specimen designs, the specimen was stretched until its breakpoint.

Hardness testing of the specimen

The ASTM D2240 specification was used to conduct the hardness of the newly developed and 100 % of the original materials. The hardness was measured by employing Rex Operating Stand, which has a built-in load weight that applies the proper force, that helps by reducing or even eliminating human error. Durometer has a spring-loaded indenter which applies an indentation load to the specimen, thus sensing the hardness meaning the Durometer relates the penetration of an indenter into a specimen. A sample of each combination was tested three times to obtain an average hardness value in both shore A and shore D scales by employing Rex Durometer model OS-2H. A setup and testing procedure were followed to get the hardness results for each sample.

RESULTS AND DISCUSSION

It is fascinating to use two different materials with known mechanical properties to create/develop more materials and study their mechanical properties to use these newly developed materials in the AM industry. For this research purpose, two liquid resins were used to create five more materials with different mechanical properties by blending them in the various volume ratios. The hypothesis was that new materials that hold mechanical properties within the two initially used materials could be developed by mixing two industrially used materials with known mechanical properties.

After deciding the ratios used to mix the two materials and 3D printing the tensile test bar, their mechanical properties were obtained through tensile and hardness testing. In addition, MSOE RPC wanted to know how the printing orientation may affect mechanical properties; thus, all the combinations were printed in XY and Z orientations, as shown above in Figure 4. Each combination has eight pieces of test bars, so three of each

orientation were used for tensile testing, and one of each orientation was used for the hardness test.

RPC test design

The RPC test specimen design shown in Figure 3a was used to 3D print all the combinations shown in Figure 2. The tensile test was conducted with the loading speed rate between 0.2 to 4 in/min.

Figures 5 through 7 show the tensile properties of EPU 40 and RPU 70 in different combination ratios (as shown in Figure 2) with the RPC tester specimen design, which is shown in Figure 3a.

As expected, the newly developed materials' tensile strength, tensile modulus, and hardness values are in the range between EPU 40 (100%) and RPU 70 (100%), as shown in Figures 6, 7, and 8, respectively. However, some of the combinations showed interesting results especially for the elongation at break. As shown in Figure 5, elongation at break for EPU 40/RPU 70 of (80/20, 60/40, 50/50, and 40/60) was higher than EPU 40 (100%). For EPU 40/RPU 70 of 80/20, elongation at break was about two times EPU 40 (100%) elongation at break. Therefore, to see if this was caused by the RPC test design, it was decided to conduct these combinations in ASTM standard designs. That is, for the case with higher percentage of EPU 40, ASTM D 412 die C was used and for the combination with higher percentage of RPU 70, ASTM D 638 type V was employed. The results are shown in Table 5 and 6. More will be discussed later.

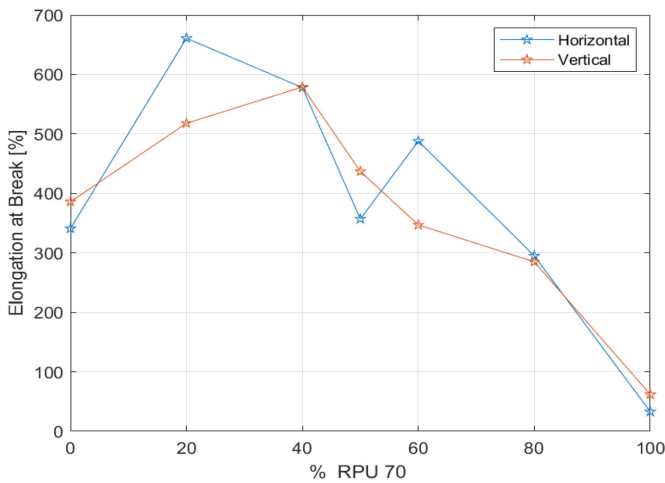


Figure 5. Elongation Vs % (RPU 70/EPU 40)

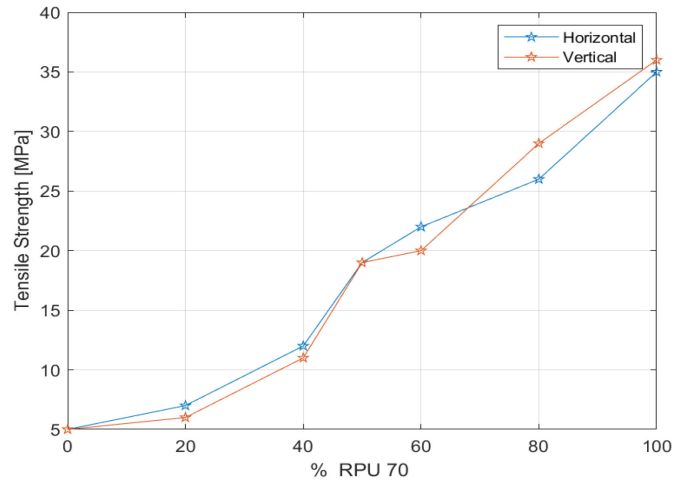


Figure 6. Tensile strength Vs. % (RPU 70/EPU 40)

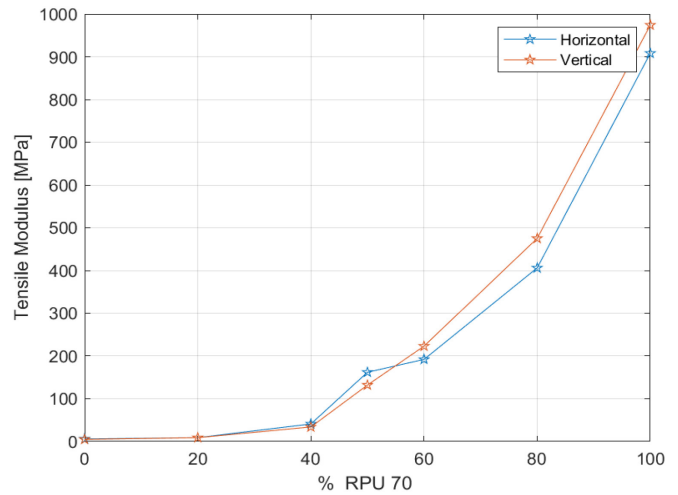


Figure 7. Tensile Modulus Vs. % (RPU 70/EPU 40)

ASTM Standard Vs. Carbon technical datasheet

Because it was important to see how close the four mechanical properties are to the standard data presented by Carbon, ASTM standard tensile test bar (ASTM D 412 die C for EPU 40 and ASTM D 638 type V for RPU 70) were designed, and 3D printed. In addition to EPU 40 and RPU 70 (100%), EPU 40/ RPU 70 (50/50%) was designed and printed in both ASTM standards, as shown in Figures 3b and 3c. Since a 50/50% combination of EPU 40 and RPU 70 is a beta material, designing and testing in both ASTM standards helps to compare and see how the different designs may have affected the outcomes. Both the tensile and hardness test was conducted following ASTM conditions and a Carbon technical datasheet for both materials for both horizontally and vertically printed specimens. Like the RPC test bar, the elongation at break was higher than EPU 40 (100%), but tensile strength and tensile modulus were in the range of the two materials. Tables 1 through 3 show the tensile properties of EPU 40 100%, RPU 70 100%, and 50/50 % EPU/RPU with the ASTM

standard specimen design. Table 4 includes the experimental variability.

Table 1. Elongation Vs. % (RPU 70/EPU 40)

	Elongation [%]			
	ASTM 412		ASTM D 638	
	Horizontally printed	Vertically Printed	Horizontally printed	Vertically Printed
EPU 40 (100%)	204	275	-	-
EPU 40/RPU 70(50%/50%)	244	268	401	402
RPU 70 (100%)	-	-	64	51

Table 2. Tensile strength Vs. % (RPU 70/EPU 40)

	Tensile Strength [MPa]			
	ASTM 412		ASTM D 638	
	Horizontally printed	Vertically Printed	Horizontally printed	Vertically Printed
EPU 40 (100%)	4	6	-	-
EPU 40/RPU 70(50%/50%)	17	19	15	16
RPU 70 (100%)	-	-	35	36

Table 3. Tensile modulus Vs. % (RPU 70/EPU 40)

	Tensile Modulus [MPa]			
	ASTM 412		ASTM D 638	
	Horizontally printed	Vertically Printed	Horizontally printed	Vertically Printed
EPU 40 (100%)	6	7	-	-
EPU 40/RPU 70(50%/50%)	143	154	125	133
RPU 70 (100%)	-	-	1262	1333

When the data was compared to the commercially available technical data, as shown in Table 4, the measured tensile properties of EPU 40 and RPU 70 (100%) were either near or less than what is given in the Carbon3D technical datasheet. For 100% EPU 40, tensile strength and modulus are near 8 MPa range. Elongation at break is lower, about 8% for vertical build. For 100% RPU, tensile strength is 36 MPa (10% lower than datasheet) and elongation at break is much lower (~40%).

Table 4: Tensile properties for EPU 40 and RPU 70 (100%) ASTM Standard Vs. Carbon3D datasheet.

100% EPU 40			
Standards	Elongation at break (%)	Tensile Strength [MPa]	Tensile Modulus [MPa]
ASTM D412 (Horizontal)	204 ± 47.97	4 ± 0.84	6 ± 0.12
Carbon Datasheet	300	9	8
ASTM D412 (Vertical)	275 ± 6	6 ± 0.22	7 ± 0.79
100% RPU70			
Standards	Elongation at break (%)	Tensile Strength [MPa]	Tensile Modulus [MPa]
ASTM D638 (Horizontal)	64 ± 18.98	35 ± 0.97	1262 ± 101.31
Carbon Datasheet	100	40	1700
ASTM D638 (Vertical)	51 ± 24.59	36 ± 2.26	1333 ± 110.8

Thus, to learn more about the difference between the experimental results and the values listed in Carbon3D technical datasheet, RPC contacted Carbon3D Inc. and discovered that this difference might be because of tensile testing machines used to test the samples. In this research, all the materials were tested by using MTS tensile tester and Carbon3D used Instron. Note: Mechanical properties for two tests can be compared and expected to have the same results only if they were tested under similar conditions. These conditions include test speed, temperature, testing device, etc. It was believed that the difference between the testing device might have caused the difference in the tensile properties.

The important learning from this research is regarding the combination materials. As seen in Tables 1-3, both ASTM 412 and D 638 yielded similar results on tensile strength and tensile modulus. The elongation at break is markedly higher with D 638 specimen. In all these cases, the printing direction did not have much influence. The question is raised for the standards. Should there be updates on how to deal with rigid and elastomeric combination materials.

Hardness

A durometer was used to measure the hardness of different combinations. Although there are many shore scales to measure hardness, shore A and D scales were used for this research purpose. Typically, shore A is used for softer materials, and shore D is for rigid materials [12]. Even though the Carbon datasheet shows that they used shore A for EPU 40 and shore D for RPU 70, both shore A and D scales were being employed to measure the hardness for all the combinations in this research. Especially for the new materials, there is no specification on which type of shore scale should be used for the mixture of elastic (EPU 40) and rigid (RPU 70) polyurethane. Thus, using both scales gives a complete data set, meaning the data can be translated between the two scales by obtaining both shore A and D scales. For example, as shown in Figure 8, the hardness of 71, A is about 21, D.

Based on the data obtained through shore A, it was discovered that shore A could only be used for the ratio of EPU 40/RPU 70 (100/0, 80/20, 60/40, and 50/50). Because the scale can only measure the maximum of 100 and the hardness of the 50/50 mixture was already 95, A, past 50/50 mixtures, the hardness measurement for the shore A scale shows no/negligible change, which is not valid. Since RPU 70 percentage was increasing, the new material's hardness should also need to show an increment. So, the results for any combinations beyond EPU 40/RPU 70 50/50 are invalid and can be disregarded (for Shore A). On the other hand, the shore D scale can be used for all mixtures, as shown in Figures 8 and 9. The hardness of EPU 40 (100%) and RPU 70 (100%) are quite close to the data found in the technical data sheet that Carbon3D provided, and all the values obtained for other combinations are in a reasonable range. Thus, RPC can confidently use the data to print any desired parts with hardness in the range of 100% EPU 40 and 100% RPU 70.

Figure 8 and 9 shows the relation between hardness and percentage combination of RPU/EPU.

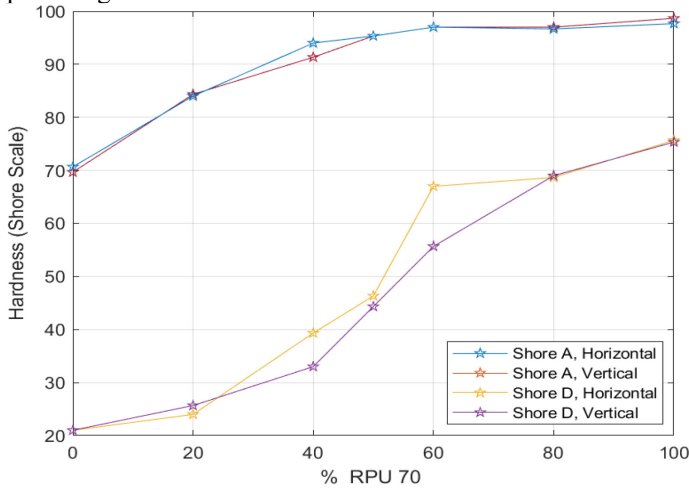


Figure 8. Shore (A & D) Vs. % RPU

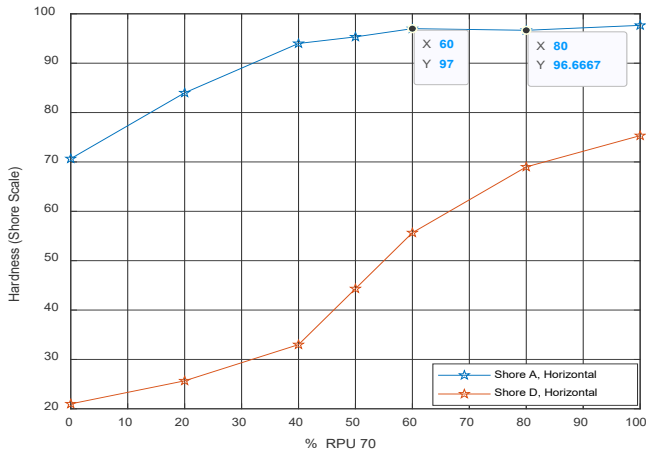


Figure 9. Shore (A & D) Vs. % RPU for just horizontally printed sample.

More study on RPC test design

After seeing results for elongation, especially for EPU 40/RPU 70 of 80/20 combination, it was decided to study a little further with appropriate ASTM standard design as discussed above to see if the RPC test design has something to do with these outcomes. Table 5 shows a comparison of tensile properties for the RPC test design and ASTM D412 die C design which was also used for 100% of EPU 40. ASTM D412 die C was used for this combination because EPU 40 holds more percentage than RPU 70 (80/20). Similarly, Table 6 shows a comparison of tensile properties for the RPC test design and ASTM D638 type V design which was also used for 100% of RPU 70. In both cases, the results for the RPC test design showed higher values, especially for elongation at break. This could be because of the test design itself or the way loading rate applied to the sample [13]. Or maybe it is possible to develop material(s) with elongation at a break higher than EPU 40 by combining EPU 40 with RPU 70. Therefore, elongation at the break for the new materials needs more studying. It would be a good idea to

consider studying the chemical bonding of the two materials. For now, it is recommended to use the ASTM standard depending on the percentage of the combinations.

Table 5: Tensile properties for EPU 40 and RPU 70 (80/20%) ASTM Standard Vs. RPC test design.

Standards	Elongation at Break [%]		Tensile Strength [MPa]		Tensile Modulus [MPa]	
	Sample print orntation		Sample print orntation		Sample print orntation	
	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal
ASTM D412 die C	269 ± 9.6	244 ± 24	7.6 ± 0.3	7 ± 0.5	7 ± 1	6 ± 0.8
RPC test design	518 ± 61	661 ± 105	6 ± 0.3	7 ± 0.4	9 ± 1	9 ± 1

Table 6: Tensile properties for EPU 40 and RPU 70 (20/80%) ASTM Standard Vs. RPC test design.

Standards	Elongation at Break [%]		Tensile Strength [MPa]		Tensile Modulus [MPa]	
	Sample print orntation		Sample print orntation		Sample print orntation	
	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal
ASTM D638 type V	189 ± 5	166 ± 4	23 ± 0.3	19 ± 0.6	622 ± 13	550 ± 46
RPC test design	285 ± 2	295 ± 23	29 ± 1	26 ± 0.4	475 ± 31	406 ± 31

All RPC test designs had eight samples, four printed vertically and four printed horizontally and one piece of each was used for hardness test and three of both vertically and horizontally printed used for tensile properties. To make sure the results for the hardness were consistent, it was decided to measure the sample three times; then the average was obtained to represent the results. And in the case of the ASTM standard designs, four samples were tested to determine their tensile properties.

Improvement that could be made

Some improvements that could be made to all the combinations to reduce these property variations are using the same testing equipment, using a part washer (VF-1 solvent) during processing conditions, keeping the same temperature all-time for tensile bars, and enhanced mixing. Mixing the two materials by hand may not give a good mix; thus, it is good to look at other options.

CONCLUSION

There are many 3D printers, and each of them has unique compatibility with different materials. The mechanical properties and surface finish are highly dependent on the type of 3D printer and material used. Thus, material selection is critical, and there are not enough material options. Therefore, this research aimed to explore new material mixtures from EPU 40 and RPU 70 resins currently used in the AM industry.

The newly developed materials were expected to obtain all mechanical properties (elongation at break, tensile strength, tensile modulus, and hardness) within the range of the two initially used material mechanical properties. The hardness of EPU 40 (100%) and RPU 70 (100%) are quite close to the data found in the technical data sheet, and all the values obtained for other combinations are in a reasonable range. The slight difference in the tensile properties may have been caused due to the use of different tensile testing machines. However, the data is still valid, and RPC can confidently use the data to print any desired parts with hardness in the range of 100% EPU 40 and 100% RPU 70. In all cases, vertically printed specimens showed

better mechanical properties compared to horizontally printed specimens. The idea of blending/combining two materials could be implemented in other materials to increase the option of 3D printing materials. Finally, studying the shrinkage rate of newly developed materials is recommended.

ACKNOWLEDGEMENTS

The material is based upon work supported by the National Science Foundation under Grant No. EEC-2045738. Any opinions, findings, and conclusions, or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation. The first author would also like to acknowledge Dr. Subha Kumpaty and Mr. Jordan Weston (advisors), Luke Ponga, Mr. Liam Coen, RPC staff, and the rest of the REU staff and REU peers.

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