ASEE IL-IN Section Conference

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Wei Dai Vian
Purdue University

Nancy L. Denton
Purdue University

Vian, Wei Dai and Denton, Nancy L., "Hardness Comparison of Polymer Specimens Produced with Different Processes" (2018).
ASEE IL-IN Section Conference. 3.
https://docs.lib.purdue.edu/aseeil-insectionconference/2018/tech/3

Hardness Comparison of Polymer Specimens Produced with Different Processes

Wei Dai Vian, PhD and Nancy L. Denton, PE
School of Engineering Technology
Purdue University
Kokomo and West Lafayette, Indiana

Abstract

Development of new effective laboratory experiences to extend and reinforce knowledge from lectures plays an essential role in engineering technology education. This paper will address a set of labs based on hardness testing. A common mechanical property test, hardness testing measures the indent penetration or other surface characteristics of materials based on the hardness test type. The laboratory procedures and the testing specimens follow appropriate standards for materials testing. In an introductory materials course for the manufacturing and mechanical engineering technology degree programs at the campuses of Purdue University, polymer specimens are tested for their Shore Durometer hardness. The quality and manufacture of the specimens directly affects the final hardness test results. To help students understand this polymer property and the importance of the specimens' respective manufacturing processes, samples made of the same material and size, but by different production methods were evaluated. Student tasks spanned polymer specimen design, processing, testing, and analysis. Sample production approaches and comparison of hardness values corresponding to each process are discussed for several polymer materials. Changes in student understanding of variability and their interest in experimental research will be explored.

Background

The engineering material property of hardness can be determined numerous ways to show wear resistance, scratch resistance, impenetrability, energy absorption, and such[1]. In addition, hardness correlates directly with the tensile mechanical stiffness property, Young's modulus, a core concern for many mechanical design applications.[2] Some hardness tests can be conducted quickly with only limited instrumentation. ASTM D785-2015 defines the requirements and applicability of Shore Durometer hardness tests, a simple and easily implemented measurement.[3] This hardness test involves several scales based on the type of material being tested, where each scale sets the shape of the indentor. Numerous indentor shapes exist and are identified by letter(s). The durometer consists of a calibrated dial indicator with an indentor tip that is pressed into the test specimen, causing an indentation. The highest indicator reading is the hardness of the material, and is inversely related to the penetration distance. Durometer hardness tests of polymeric materials often use Shore A and Shore D indentors, where the type A has a flat-tipped cone point and the type D indentor cone point has a small radius.

Two campuses of Purdue University participated in the initial enhanced polymer hardness testing laboratory experience. PU-Kokomo is a commuter campus with a balanced population mix of traditional and non-traditional students and typical engineering technology class sizes of 10-20 students that are often taught in a studio format. PU-West Lafayette is a large residential campus populated by traditional students, transfer students, and a smattering of non-traditional students. Classes often run with 60-100 students per lecture division and 12-16 students per companion laboratory section. Each campus offers the same BS MET curriculum, as well as several complementary engineering technology majors, with distinctly different cultures and student expectations. At the West Lafayette campus, students are immersed in a research-oriented facility and a global population, with many opportunities to expand their personal and professional horizons. At the Kokomo campus, the campus culture tends to focus on efficient completion of educational tasks performed by students from central Indiana. To increase their awareness of experiment research work within the context of a required class, students in a sophomore-level strength of materials course at PU-Kokomo completed an expanded hardness testing laboratory regimen (plus a similar tensile testing experience that is beyond the scope of this paper). At PU-West Lafayette, a freshman student from a first-semester materials course conducted an abbreviated version of the hardness testing laboratory project as part of an undergraduate research experience. This provided access to additional hardness data from 3D printed polymer specimens.

Introduction

Hardness testing is a common category of mechanical property test for introductory materials courses, as can be seen via a simple web search. The indent penetration resistance, scratch resistance or energy rebound are found by these tests. For prior hardness testing by engineering technology students at Purdue University, prepared specimens were provided. The laboratory procedures and the specimens of interest generally follow appropriate ASTM standards. The focus of the laboratory has been simply learning the testing technique and identifying how closely the final hardness values match published hardness data.

To increase student awareness of scientific research practices and potentially both improve their critical thinking skills and their motivation to learn, a new materials testing laboratory project was designed for lower division manufacturing and mechanical engineering technology students, and implemented in the strength of materials course for the mechanical engineering technology degree programs at the Kokomo campus of Purdue University. The project was duplicated at West Lafayette by a freshman undergraduate researcher for comparison.

New Laboratory Project

The newly designed materials testing laboratory project is highly student-centered. Students take responsibility for polymer specimen design, processing, testing, and analysis. All specimens in the project were made of thermoplastics, including polystyrene (PS), low-density polyethylene (LDPE), acrylonitrile butadiene styrene (ABS), and polylactic acid (PLA). Unlike thermosets, thermoplastics have secondary bonds. These are easily broken bonds between molecular chains which allow thermoplastics to be reshaped and/or recycled, often at relatively low temperatures. This means the Shore Durometer hardness test is compatible with all selected materials and applicable manufacturing processes were selected for making these thermoplastic specimens. Two popular and common polymer manufacturing processes, continuous filament deposition 3-D printing and injection molding, heat and reshape material. These two processes were applied to produce flat dogbone specimens from each of the project materials. In addition, different brands of 3-D printers were used in the processing to investigate potential effects on hardness, incorporating another aspect of variability into the project. Finally, machining of commercial sheeting rounded out the processing methods. All specimens were subjected to Shore A and Shore D Durometer hardness tests. All test results were recorded, plotted and compared to same-material specimens and to published hardness data. The quality of samples and the limitation of each process will be discussed in detail in the analyses based on the observation and testing results.

This paper will present the approaches taken to produce the samples and discuss the results of the comparison of hardness values that correspond to each process for several polymer materials. Students acquired experimental research experience by working through the hands-on design, processing and testing phases of the project, making assumptions and estimations before knowing their experimental results, analyzing the test data, and recommending future improvements. Their self-reported perception of the effects of the project on their research perspectives are presented.

Methodology

For this class-based research project, four-person student teams were formed from the sophomore-level strength of materials class at PU-Kokomo. For all group members, expected contributions were to help fabricate specimens by multiple methods, obtain dimensional measurements, complete hardness tests of each sample, and analyze the hardness data to determine the effects of manufacturing process and material quality. The discussion (improvement) of laboratory methods, manufacturing processes and material quality were required in their final project report to shed light on the project's efficacy as a means to increase student research awareness in addition to post-project survey responses.

Constraints

There were three manufacturing methods used to produce the test specimens in this project: 3D printing, cut-to-size plastic sheet, and injection molding. To facilitate subsequent tensile testing, the specimen dimensions are from ASTM D638— Standard Test Method for Tensile Properties of Plastics.[4] The Shore Durometer hardness test was conducted, as it does not limit the sample thickness to 6 mm minimum, a requirement of the often-used Rockwell Hardness test. The Shore Durometer hardness test requires the testing location to be 12.0 mm from each edge. For this laboratory project, the testing locations were actually 9.5 mm from two edges of the specimen tabs, a small deviation from ASTM D2240 requirements.

Process of producing specimens

To prepare for the 3-D printing process, students picked a computer aided design (CAD) software of their choosing to model a test specimen. Based on the geometry given from ASTM D638 shown in figure 1, the dogbone specimens had dimensions listed in Table 1, and were saved in the .stl format for the 3-D printer. The samples were printed from four different 3-D printers (see appendices for brands and types), using filament materials of ABS (silver grey), PLA (gold), and HIPS (yellow), with diameters of 1.75 mm or 3 mm. For each material, about 20 specimens were printed at PU-Kokomo. (See Table 2 for the number of specimens printed by each 3-D printers). Shore Durometer hardness tests were conducted on samples with no obvious defect. For PU-West Lafayette, sample lots were limited to five specimens per material.

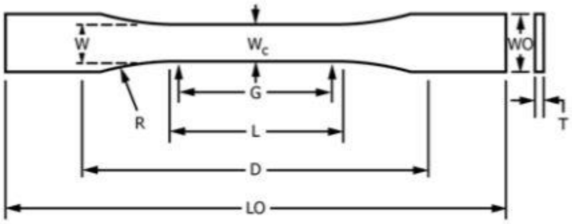

Figure 1 Specimen dimensions (ASTM D638[4])

Table 1 - ASTM D638 Standard Specimen Dimensions[4]

Type I	Dimensions: mm (in)
T-Thickness	7 (0.28) or under (3 mm for this lab)
W-Width of narrow section	13 (0.50)
L-Length of narrow section	57 (2.25)
WO-Width overall, min	19 (0.75)
LO-Length overall, min	165 (6.5)
G-Gage Length	50 (2.00)
D-Distance between grips	115 (4.5)
R-Radius of fillet	76 (3.0)

Table 2 - 3-D Printing Specimen Information

Printer	FlashForge	MBot Cube	Lulzbot Mini	Lulzbot TAZ	MBot Replicator 2X
Polymer (number)	HIPS (5)	PLA (6)	ABS (6)	HIPS (10)	ABS (15)
				PLA(15)	

The 1.75 mm and 3 mm polymer filament for 3-D printing was purchased online, with specifications listed in table 3.

Table 3 – Filament Specification for 3-D Printing

Polymer	Density (g/cm^3)	Extrusion Temperature (°C)
ABS	1.07	230-240
HIPS	--	220-230
PLA	1.25	205±15

The cut-to-size sheets for machined specimens, made of LDPE (clear), ABS (cream), and PS (white), were ordered directly from the supplier. Sheet size was 12 x 12 inch (305 x 305 mm). With assistance from the laboratory technician, students used CNC machining to cut approximately 12-14 specimens from each polymer sheet to dimensions matching those generated by 3D-printing. Ten cut specimens with no obvious defects were chosen for hardness testing.

For injection-molded specimens, an aluminum mold was CNC-machined. Its mold cavity has nominally the same dimensions shown in Table 1. Specimens were molded from polymer beads of LDPE (white), ABS (cream), and PS (clear). The temperature settings for processing these three materials were 230 ℃ (ABS), 110 ℃ (LDPE), and 210 ℃ (PS), respectively. Students ran the injection mold machine to make approximately 15 samples of each material. Flash was removed from the specimens, and ten apparently defect-free specimens were selected for hardness testing for each material.

Quality of products

The surfaces of the 3D printed samples were not as smooth as the cut sheet or injected samples. Conversely, some of the injected specimens were not very flat, and had a few defects. Defects included light brown spots on the LDPE and ABS specimens and multiple small visible bubbles inside the PS specimens ranging from 1-5 mm in diameter. Finally, the specimens cut from purchased LDPE sheets had some flash remaining from the cutting process.

Figure 2 Gold 3-D printed PLA specimens with measuring instruments and visible cross-sectional area measurement locations 1, 2, and 3

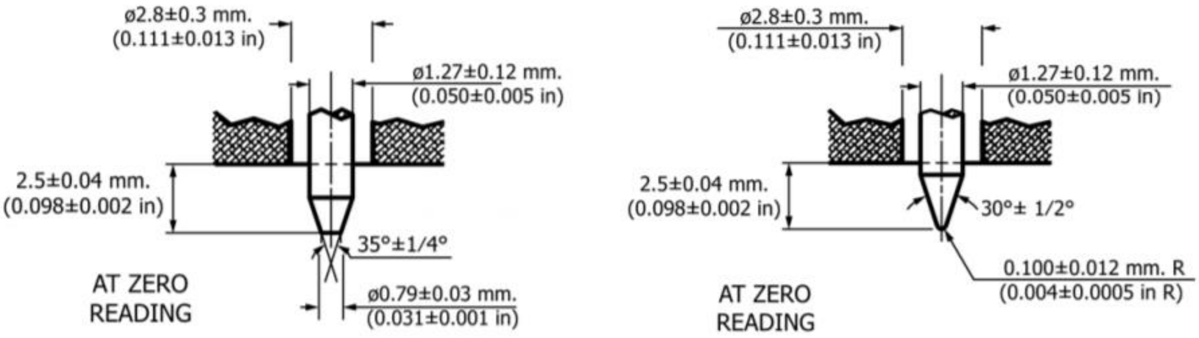

Figure 3 Type A Indentor (left) and Type D Indentor (right)[5]

Shore Durometer Hardness Testing procedure

Tools used for the hardness tests were the Shore Durometers, types A and D (shown in Figure 3), a 12-in scale (ruler); digital calipers and 0-1 inch micrometers. Each specimen was labeled by number, name, and indentation locations. The locations were referenced from one specimen end. Moving along the longitudinal axis, the first location was 0.50 in (12.7 mm) toward the center, on the longitudinal center axis. Subsequent points followed at 0.50 inch (12.7 mm) intervals from the first point toward the opposite end. Each sample had six test locations on one side for type A Shore Durometer hardness, and another six on the flipside for type D.

Figure 4 Shore Durometer is used to measure the hardness of a PS specimen

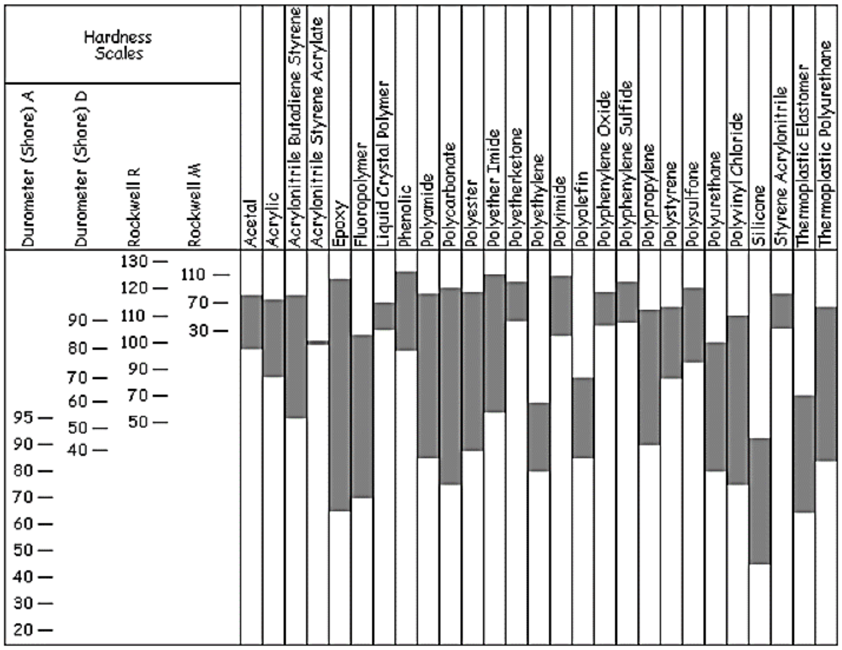

Figure 5 Hardness comparison chart[6]

Results

Figure 5 shows the chart presenting the hardness range by Shore Durometer indentor type supplied to students for reference. For ABS, the published data of shore A is above 95 while for Shore D it is between 55 and 100. For polyethylene, the published data of Shore A is between 80 and 98 and for shore D is between 30 and 60. For polystyrene, the published data of Shore A is above 95 and for Shore D is between 70 and slightly above 90 The students' hardness data generally exceeded minimum Shore Durometer values for all three materials.

A key aspect of this project was to consider the variability in Shore Durometer hardness values based on manufacturing method. For individual specimens, the variability of their hardness across locations was assumed to reflect the specimen quality. Hardness testing results from this project focused on exploration of these two elements of variability.

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Comparisons of hardness value by processing method appear in figures 6-15. (Test points are connected on the plots for visual purposes only, to clearly indicate each material). Although Figures 6 and 7 show ABS Shore Durometer hardness results. Both indentor shapes produced hardness values within a range of 4-6. The type A results nearly merge at point 6 (geometrically mirror of point 1).

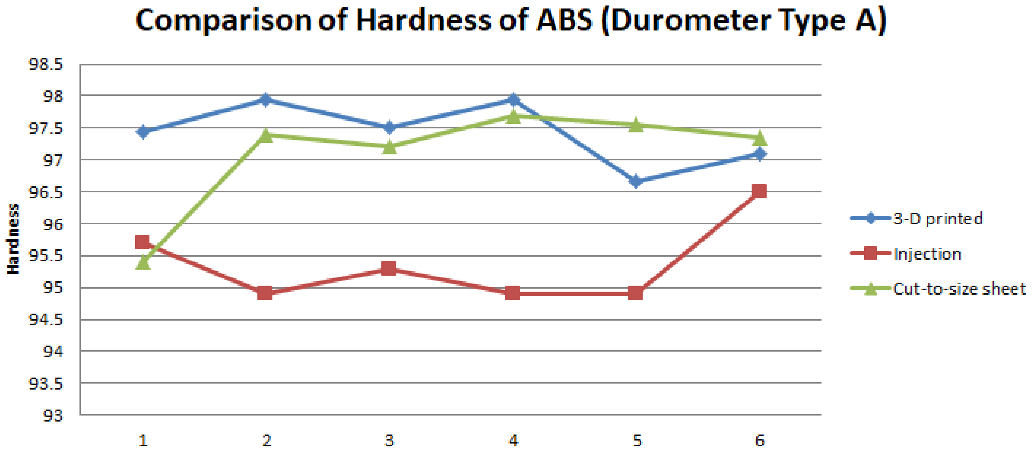

Figure 6 Comparison of ABS average shore A Durometer hardness (with six testing locations)

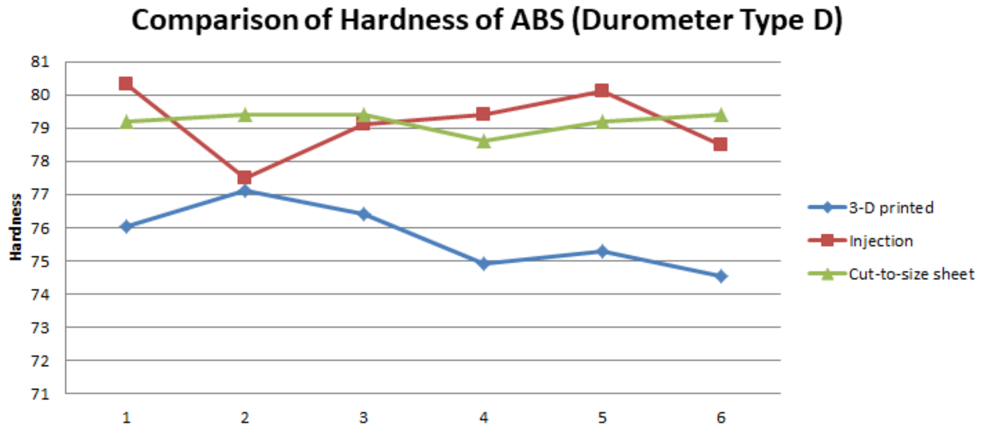

Figure 7 Comparison of ABS average hardness in type D (based on six testing spots)

For the LDPE, where machined from sheet specimens were omitted, Figures 8 and 9 illustrate much closer correlation between processing methods than was seen for ABS for the flat-tipped indentor testing with Shore Durometer A, but an increasing spread for the conical, nearly pointed type D indenter.

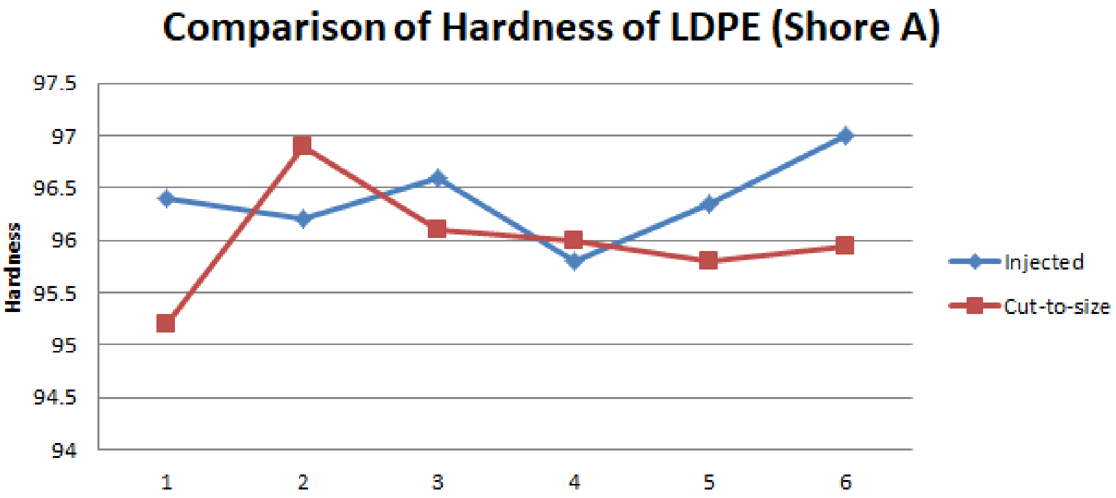

Figure 8 Comparison of average hardness of LDPE in type A (based on six testing spots)

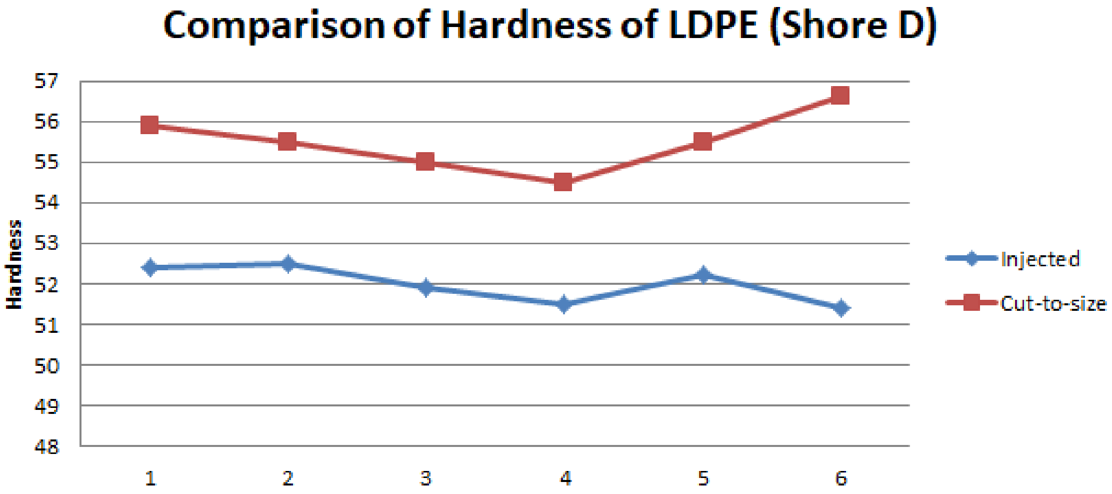

Figure 9 Comparison of average hardness of LDPE in type D (based on six testing spots)

Polystyrene hardness plots shown in figures 10 and 11 have trends similar to those for LDPE, with a much broader difference in Shore Durometer hardness Type D values that again spread out as they go to point 6.

Figures 12 through 15 show hardness testing results for 3D printed PLA and HIPS, from different printers and at different campuses, working from a common .stl file. Type A Shore Durometer hardness results generally are again more similar in value than those from the Type D testing. The reason(s) for the differences by type have not yet been explored, but are presumed to be based on how the indentor tip geometry interacts with the variation in molecular bonds generated by each processing method and machine.

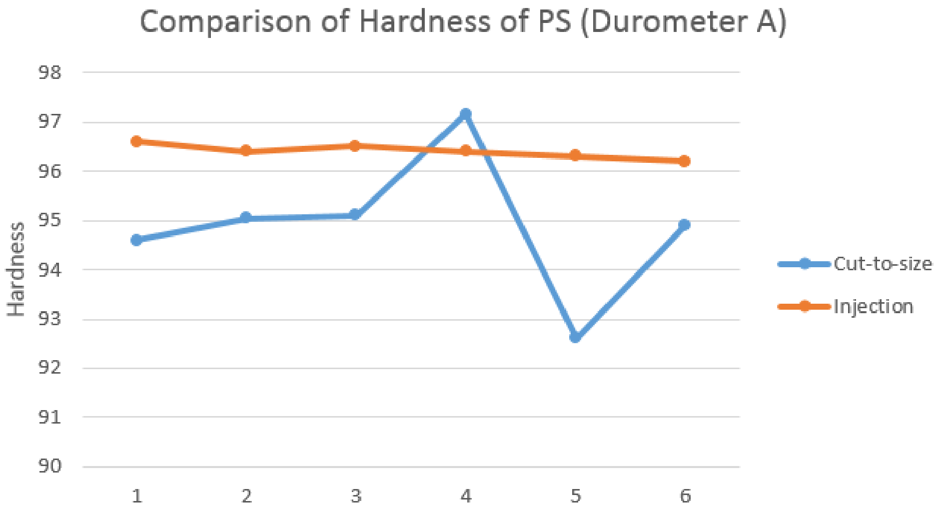

Figure 10 Comparison of average hardness of PS in type A (based on six testing spots)

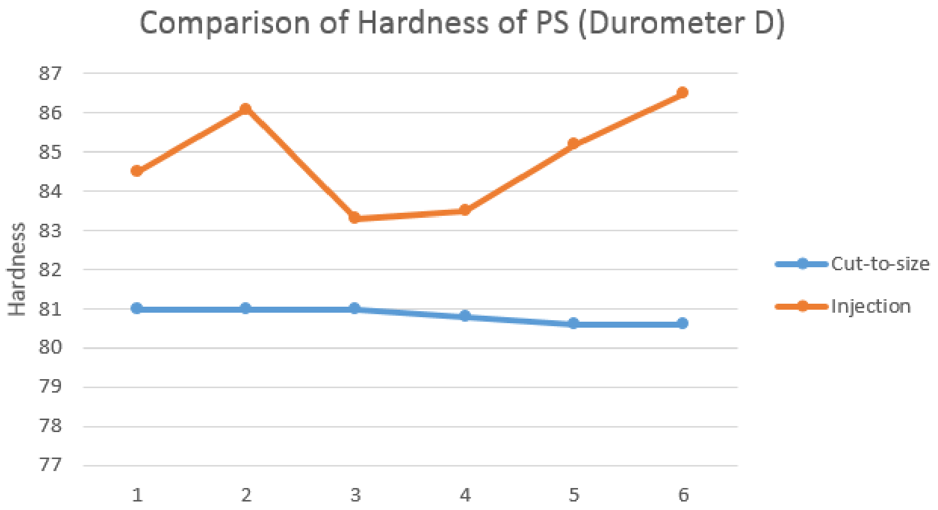

Figure 11 Comparison of average hardness of PS in type D (based on six testing spots)

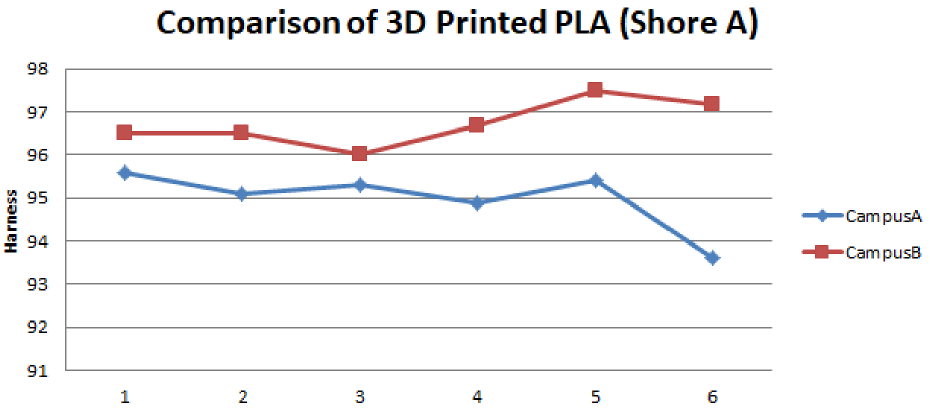

Figure 12 Comparison of hardness of PLA in type A (printed by two printers at two campuses)

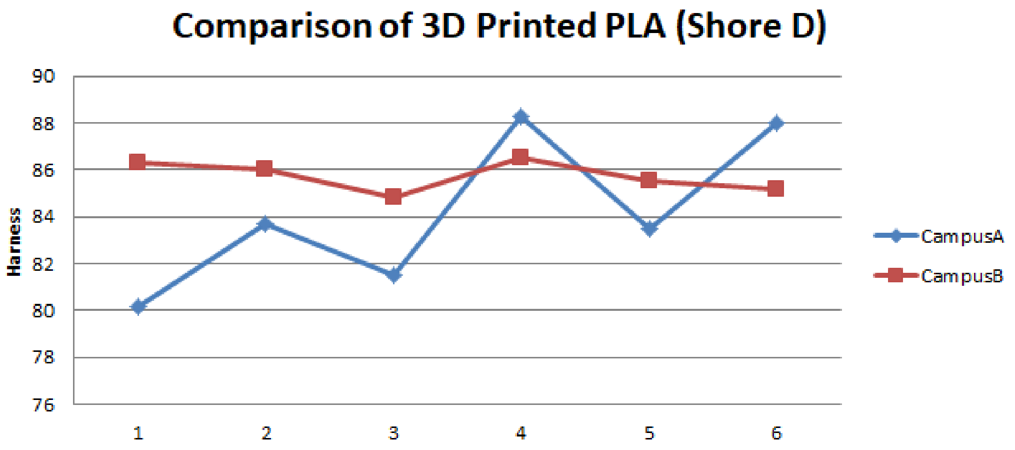

Figure 13 Comparison of hardness of PLA in type D (printed by two printers at two campuses)

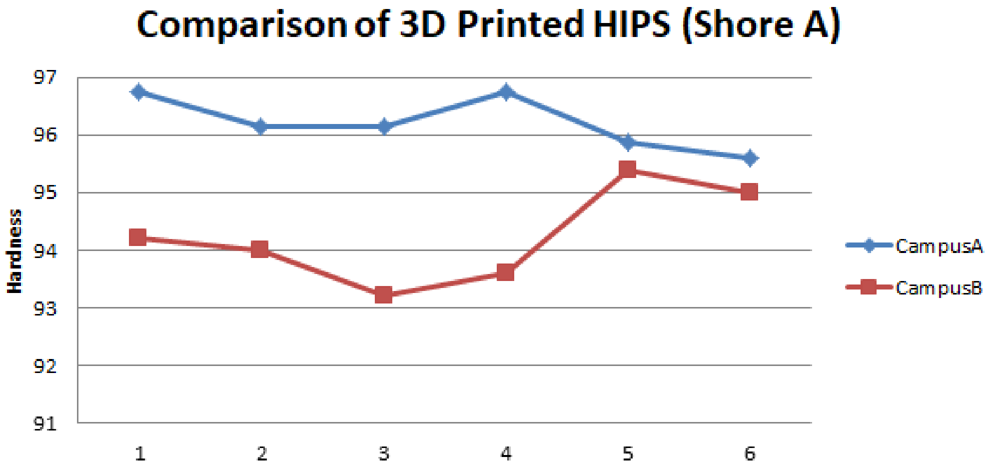

Figure 14 Comparison of hardness of HIPS in type A (printed by two printers at two campuses)

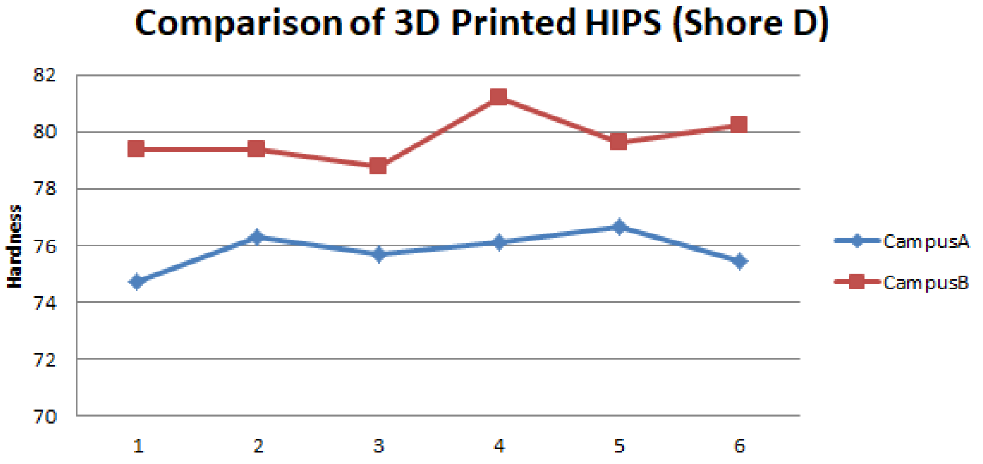

Figure 15 Comparison of hardness of HIPS in type D (printed by two printers at two campuses)

In the conclusion and summary section of their laboratory reports, students stated this new laboratory project clearly showed how hardness differs among specimens of the same material specimens made by different manufacturing methods. This lab allowed them to determine reasonably accurately which material's hardness was modified through a change in manufacturing method. Some students wanted to continue the testing on more materials. Students also were aware of and discussed the defects and possible errors caused by the respective processes and testing approach, such as the bubbles in injection molded parts, inaccuracy in size measuring, and limited contamination of the material.

Discussion

Participating PU-Kokomo students were mainly MET sophomores taking their required strength of materials course. Through this project, their understanding of experimental variation improved. For example, students used injection molding to produce sample specimens. They observed bubbles in their PS samples and ABS specimens lacking flat surfaces. There was an immediate concern and related discussion among students regarding whether bubbles and the corresponding density reduction should cause any significant difference in hardness value since this situation occurred with the PS specimens. There were also questions regarding the hardness testing effects of curved surface will affect the hardness testing. Certainly, there was no single simple solution; the students had to pursue answers for themselves. Moreover, the students needed to analyze the results to determine if they differ with from assumptions. Students' learning interest was increased through the step-by-step working and testing procedure and the follow-up consideration of variability and its effects.

The PU-West Lafayette student came into this project seeking an initial undergraduate research experience. As a first-semester freshman, he was eager to become involved at any level. His contributions afforded him a sampling of several roles. Unlike the PU-Kokomo students, he worked from a CAD file supplied by the PU-Kokomo lab technician. This meant exposure to some of the unexpected challenges a research project. A few examples include mix-ups when the supplied CAD file does not have an established naming convention that is known to all parties and tracking down 3D printers where the intended filament materials were allowed. PU-West Lafayette has multiple 3D printing laboratories, three of which were utilized for this project. Critically thinking about how to compare the hardness testing results, exposure to designing an experiment and executing analysis of the resulting data all occurred as part of the student's research project involvement.

To give the faculty insight insight into the students' views of how the project may have altered their perceptions of experimental methods and research, PU-Kokomo students were surveyed The survey results were very encouraging in terms of student perceptions of experimental work. 100% of the students indicated they now understand that the manufacturing process can affect a material's hardness, and 89% agree that testing multiple identical specimens is appropriate for property determination. All students believe that consistent test locations are important and that specimen quality affects material properties. Their views of experimental research, unfortunately, were not as positive. While 78% noted that working on a team-based research project produced a better experience than doing similar work individually, only 44% responded favorably to the item, "Experimental research intrigues me."

Conclusions/summary:

The initial offering of this hardness testing laboratory project met its goals of increasing student awareness of the variability in hardness properties based on manufacturing and enhancing their observation and understanding of experimental research. This project was coupled with a similar tensile testing effort to reinforce student recognition of process-based effects on material properties and provide more experimental research practice. The instructors plan to revisit the project with more guided analysis of the process-based and location-based variability in hopes that this slight increase in research focus will produce more intriguing results for future students.

References:

1. Black, J.T., and Kohser, R. (2012). *DeGarmo's Materials and Processes in Manufacturing*, 11th edition, John Wiliey & Sons, Incorporated.
2. Cheng, Yang-Tse, and Chang, Che-Min (1998). Relationships between hardness, elastic modulus, and the work of indentation, *Applied Physics Letters, 73(5),* 3 August 1998.
3. ASTM D785-08(2015) Standard Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials
4. ASTM D638 – 14 Standard Test Method for Tensile Properties of Plastics
5. ASTM D2240-15 Standard Test Method for Rubber Property – Durometer Hardness
6. https://plastics.ulprospector.com/properties/hardness-conversion-chart, retrieved November 28, 2017.

Appendix A: Polymer Suppliers, Equipment, and Property references

1) 3-D printers applied in this project
    i. Lulzbot TAZ 5
    ii. MakerBot Replicator 2X
    iii. MakerBot Cube
    iv. Lulzbox Mini
    v. FlashForge Creator

2) Filament material supplier webpages
    - Gold PLA: https://www.matterhackers.com/store/l/pro-series-gold-pla-filament-3.00mm/sk/MYW2EHZX
    - HIPS (yellow is no longer available; this appears to be without pigmented): https://www.matterhackers.com/store/l/hips-300mm-1kg/sk/MFAM5YE9
    - Silver ABS page: https://www.matterhackers.com/store/l/175mm-abs-filament-silver-1-kg/sk/M5A2YT7Z

3) Cut-to-size Plastic Sheeting
https://www.tapplastics.com/product/plastics/cut_to_size_plastic

4) The following references are generic properties for molded plastic:
ABS:

http://matweb.com/search/DataSheet.aspx?MatGUID=eb7a78f5948d481c9493a67f0d089646
LDPE:
http://matweb.com/search/DataSheet.aspx?MatGUID=557b96c10e0843dbb1e830ceedeb35b0
Polystyrene:
http://matweb.com/search/DataSheet.aspx?MatGUID=df6b1ef50ce84e7995bdd1f6fd1b04c9

5) The following links show strength of generic filament materials:
https://eu.makerbot.com/fileadmin/Inhalte/Support/Datenblatt/MakerBot_R__PLA_and_ABS_Strength_Data.pdf
https://3dprint.com/42417/3d-printing-material-strengths/
https://www.lifewire.com/3d-printed-material-strength-2230
http://www.sciencedirect.com/science/article/pii/S2214860416300859

Appendix B: Project survey items

(Likert scale, 5 choices from strongly agree to strongly disagree).

1. I now understand the manufacturing process may affect the hardness of a material.
2. I now understand the manufacturing process may affect the tensile strength and stiffness of a material.
3. The ASTM requirement for testing multiple identical specimens is appropriate when determining material properties.
4. Establishing consistent test locations across specimens is important.
5. Specimen quality affects material properties.
6. Experimental research intrigues me.
7. Working with other students on a team improved my research project experience (when compared to doing an individual research project).
8. Comments