https://doi.org/10.2352/ISSN.2169-4451.2021.37.65

Hardness Testing Process for Enhanced Joining, Material Validation, and Mass Serialization

Katrina J Weinmann; Colorado State University; Fort Collins, CO
Steven J Simske; Colorado State University; Fort Collins, CO

Abstract

In this paper, a method for the simultaneous provision of material validation, mass serialization, and binding enhancement using a portable hardness tester is presented for 3D printed parts. The process described in this paper is intended for implementation using a robotic arm-mounted hardness tester for ease of integration into a manufacturing environment, and adaptability of the process for custom parts. Hardness testing can be used for material validation, but the process of hardness testing leaves an indent in the material where the test is performed. Thus, the indents must be placed where they do not affect desired aesthetics, or else coupled with another desired process. By administering the hardness tests in a specified pattern on the material, the indents created on the material can be used for two additional functions – increasing the surface area to enhance joining, and marking an item-specific serialization code on the part that can be used for later identification. The post-processing of 3D printed parts can be streamlined by completing these three objectives in a single process that is highly adaptable to customized manufactured parts through an implementation using a robotic arm.

Introduction

Additive manufacturing provides many advantages over traditional subtractive and molding-based manufacturing methods, such as the ability to produce parts with complex internal geometries and the ability to quickly produce custom parts without changes in manufacturing setup. However, additive manufactured parts face a unique set of post-processing challenges. One of these challenges is material validation. With subtractive or molding-based manufacturing, the material properties are usually validated prior to manufacturing. For additive manufacturing, the material properties of the manufactured part are affected by the 3D print settings, so they must be validated after the additive manufacturing process. Additionally, due to different surface properties, joining of additive manufactured parts can be challenging. Finally, while additive manufacturing greatly reduces cycle times for manufacturing custom parts, this presents a challenge for post-processing as it is difficult to use a static set-up for material validation and other post-processing operations for parts of a variety of shapes and sizes. This introduces the need for a flexible post-processing set-up that can accommodate a wide variety of parts with minimal changes in set-up between different parts.

As additive manufacturing enables custom manufacturing to become more mainstream, validation of multiple materials on multiple surfaces is important. Also, additive manufacturing can be used to produce specific-use accessories suitable for the custom manufactured goods [1]. These can even be single-use, since many additive manufacturing processes feature recycling of the materials. Additive manufacturing can also be used for de-customization of customized parts to allow them to fit into existing mass production lines. In this case, the accessories that are additively manufactured might be sprues or container elements allowing the custom parts to fit into the constraints of the existing production line. In each of these cases, being able to approach surfaces of different morphologies for the purpose of inspection, quality assurance (QA), and quality control (QC) is paramount.

Hardness testing, such as nanoindentation, is widely used for material validation [2] and surface characterization [3], but these indents have not been used for any purpose additional to material evaluation. As a consequence, these tests have generally been performed on nonvisible surfaces when possible. The method presented in this paper leverages the indents made by hardness testing for two additional functions. We describe a process by which nanoindentation appropriate to the materials used in manufacturing (including polymers and metals) is used for the simultaneous achievement of material validation, increasing the surface area between two joined elements to enhance bonding/adhesion, and material mass serialization labeling for item-level tagging. Combined, this process provides several important functionalities and also provides indentation marks that may be suitable for forensic-level authentication using high-resolution imaging [4], [5] among other approaches.

Method

The process described in this paper can be used on both plastic and metal 3D printed parts. Experiments were performed on two plastic materials – acrylic and polycarbonate – and one metal – copper. In the following sections, the four parts of the hardness testing procedure are described in detail. In the first section, the methodology for and implementation of a portable hardness tester for material validation is discussed. In the second section, the use of the indents for enhanced joining is presented. The third section discusses the use of the indents for mass serialization of parts, with potential applications in forensic evaluation. Finally, a brief description of the implementation of this process using a robotic arm is given.

Hardness Testing

Material hardness is strongly correlated with elastic modulus, and can be used as a method of material validation. Material validation is especially important for additive manufactured parts, wherein the degree of QA and QC may vary considerably between the different parties involved in the manufacture of all the different elements of an item. Material characterization includes ensuring sufficient density, validating 3D print settings, and using sensors to ensure that the desired material properties are achieved. There are various methods of hardness testing in use, with the two most common being Rockwell hardness testing and Leeb rebound hardness testing. Rockwell hardness testing is performed by pressing an indenter tip into the material being tested with a known force. The size of the indent is then measured and used to calculate the hardness of the material. Leeb rebound testing is a dynamic test that uses an impact probe, and measures the initial velocity before impact and rebound velocity after impact with the material to calculate the material hardness. Both of these types of hardness testing leave an indent in the material where the test is performed. There are various other methods of hardness testing as well, and the preferred method of hardness testing depends on the material being tested and the geometry of the object (not to mention whether an indent is allowable on the surface for reasons including integrity and aesthetics). Generally, the two methods described above work better when applied to large, metal parts with large flat surfaces for testing.

Many 3D printed parts are small, plastic, or have complex geometries which lack the large, flat surfaces typically employed for hardness testing. For this experiment, we used two different types of portable hardness testers that are designed for use on small, thin objects. A Phase II PHT-980 Digital Shore D Durometer was used for the acrylic and polycarbonate, and a Phase II PHT-6005 UCI (ultrasonic contact impedance) hardness tester with a 5kgf probe was used for the copper. Each of these testers is suitable for material thicknesses as low as 2 mm, which means 20 layers at 100 microns/layer. The durometer measures the depth of an indent in a material from a known applied force to determine the hardness of a material. The ultrasonic contact impedance method of hardness testing measures the difference in ultrasonic frequencies generated from a vibrating diamond tip indenter being pressed into the material at a fixed load to measure material hardness. These portable hardness testers were chosen for their ability to be used on small objects and the relatively small size of the test actuator, giving the ability to administer the hardness tests using a robotic arm, and decreasing the flat surface area needed for the hardness tests.

Another concern with using hardness testing for material validation of additive manufactured parts is the lack of surface uniformity and relatively high surface roughness. Hardness testing is traditionally performed on flat, finished surfaces to ensure accuracy of the hardness measurement. However, it is undesirable to add a manufacturing operation to finish a surface for the sole purpose of preparing the surface for hardness testing. Here, we propose administering several hardness tests on the same surface to provide an average hardness value that is sufficiently accurate for material validation. This eliminates the need for surface finishing because any individual hardness reading variations due to surface roughness or anomalies will be averaged out by the Central Limit Theorem. Thus, this approach provides a material validation step that can be performed using the appropriate effector (human, robotic arm, etc.) for the process being performed.

Enhanced Joining Using Indents

Performing several hardness tests on the surface of a part also has the function of increasing the surface area available for bonding through the indents created by the hardness tests. The size, shape, and spatial density of the indents will determine how much increased surface area is available for joining. Using the material validation surface for bonding has the added benefit of either hiding the validation marks (if bonded to a solid material) or providing tamper protection for the serialization described in the following section (if bonded to a transparent material or forensically analyzed after failure or seizure of the object for investigatory purposes).

The spatial density of the indents is dependent on the capabilities of the robotic arm administering the hardness tests. The achievable spatial density for a given robotic arm will be discussed in more detail in the Process Implementation section below. The results and calculations presented in this section were achieved by manually administered hardness testing at a nominal grid spacing of 1mm. In order to also achieve the goal of serialization through marked patterns, indents were made in 50% of the grid locations. For example, a 4mm X 4mm grid therein contains a total of 8 indentations – 50% of the total 16 locations represented by the grid (Figure 1). The shape of the indent created by hardness testing is specific to the hardness tester used, and the size of the indent is affected by both the type of hardness tester and the hardness of the material being tested. Results are given for the three combinations of materials and hardness testers that were used in this experiment.

Figure 1: *Hardness Indents in Copper from PHT-6005, with eight indents being made in a 4 x 4 grid area.*

PHT-980 Shore D Durometer

The Shore D Durometer has a conical-shaped indenter tip, with a 30° tip. The increased surface area that each indent creates can be calculated using equation 1, where r is the radius of the indent, and θ is the tip angle of the indenter.

$$A_i = \pi r^2 \left(\sqrt{1 + cot^2\left(\frac{\theta}{2}\right)} - 1 \right) \quad (1)$$

The percent increase in surface area can then be calculated as a function of grid spacing using equation 2, where d is the nominal grid spacing, and f is the percentage of grid locations with an indent. In the case presented in this paper, 50% of the grid locations have indents, so $f = 0.50$.

$$p_{SA} = \frac{f A_i}{d^2} \, X \, 100 \quad (2)$$

Results showing the increase in surface area created from the indents for acrylic and polycarbonate, using the Shore D Durometer and 1mm grid spacing with 50% of grid locations with indents are given in Table 1. These are relatively hard plastics, so an even greater increase in surface area would be expected in softer plastics that would have larger indents from the hardness testing.

Table 1: *PHT-980 Indents in Plastics*

Material	Hardness (Shore D)	Indent Radius	Percent Increase in Surface Area
Acrylic	90.6	0.16 mm	11.5%

Polycarbonate	83.4	0.18mm	14.6%

PHT-6005 UCI

The PHT-6005 UCI hardness tester has a diamond indenter tip with an angle of 136°. The increased surface area created by an indent with this intender shape can be calculated using equation 3, where w is the width of the of the indent measured perpendicular to the edges, and θ is the tip angle.

$$A_{i,t} = w^2 \left(csc\left(\frac{\theta}{2}\right) - 1\right) \quad (3)$$

The percent increase in surface area as a function of grid spacing and fill percentage can be calculated using equation 2 and equation 3 to calculate A_i. The increase in surface area for 1mm grid spacing and 50% of grid locations with indents for copper is given in Table 2.

Table 2: *PHT-6005 Indents in Copper*

Material	Hardness	Indent Width	Percent Increase in Surface Area
Copper	110 HV	0.31 mm	0.38%

Hardness Tester Selection

As seen in the results presented in the previous sections, the geometry of the indenter tip of a portable hardness tester has a large impact on the increase in surface area generated by the hardness testing indents. The main factor affecting the increase in surface area per indent is the tip angle of the indenter – the PHT-980 has a much smaller tip angle than the PHT-6005, resulting in deeper indents and a larger increase in surface area. Because of this, the geometry of the indenter tip should be considered when selecting a portable hardness tester to use for this process. However, the increase in surface area is not the only way that the indents improve joining. Indents of any shape serve to rough up the surface, creating better shear behavior, and thus enhancing the joining properties of the surface. Therefore, other factors should also be considered when selecting a portable hardness tester, such as indent size for the material that is being tested, and any minimum size or thickness requirements of the material being tested for the hardness tester.

Mass Serialization

In order to provide security for a physical item, there are three necessary elements: (1) mass serialization, in which a unique ID is placed on each item; (2) copy prevention, in which a physical mark used for authentication deteriorates in a measurable way when an attempt to replicate it occurs; and (3) tamper-evidence, wherein administering hardness tests in a specified pattern of locations allows the indents to be used as a barcode (data bearing patterned mark). Using a specified grid spacing, indents are placed at locations in the grid corresponding to the desired serialization code. The barcodes can be represented by a binary string, where a '1' is represented by a grid location with an indent, and a '0' is represented by a grid location without an indent. When the code is read line by line, top to bottom, it can be represented by a 1D binary string that matches the serialization code for the part. For example, the code shown in Figure 1 would be read as '1110010000101101.'

A variety of codes were marked on acrylic, polycarbonate, and copper surfaces. These codes were then imaged and analyzed to verify accurate identification of the indents marked by the hardness testers. The purpose of the experiment was verification of the ability to identify the indents through imaging. Overall, 10 different codes were marked in 4 x 4 grids on each of the tested materials. Each code contained 8 indents, for a total of 80 indents in each material. Indent recognition accuracy was 100% for all three tested materials, with no false positive indent identifications, for the initial set of indents, demonstrating reliable identification for the process (240 total indents).

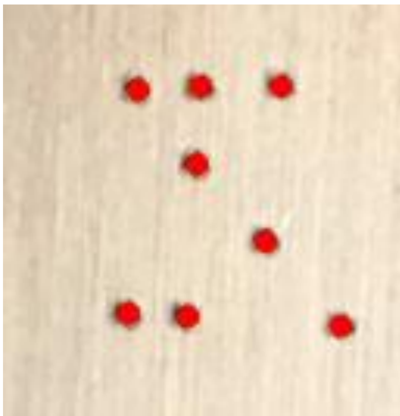

Figure 2*: Indent identification of pattern shown in Fig. 1. Red marks correspond to calculated indent centers.*

In addition to the use of the indents for mass serialization, images of the indents can also be used for forensic identification of parts. Variations in indent placement with respect to centered grid locations, indent orientation, indent size, and edge variations all provide identifying features unique to each indent. Every serialization code will contain several indents, each with unique identifying features, which when combined together will provide a very high level of forensic security. Forensic security provides copy prevention and tamper evidence simultaneously, since the marks cannot be copied, and will not be authenticated if they have been tampered with.

Process Implementation Using Robotic Arm

One of the reasons to implement this process using a robotic arm is in order to make the process flexible, making marking on surfaces in different orientations and in harder-to-reach locations possible. The use of a programmable robotic arm means that no change of set-up is required when switching between parts. Instead, one simply needs to modify the program for the appropriate surface location, angle, and grid size. This caters especially to additive manufactured parts, which often have complex geometry and are produced in small batch sizes. It saves considerable manufacturing time to be able to use the same setup when switching between parts. It is also possible to mount multiple sensors to the robotic arm so that imaging of the indents can be performed in the same manufacturing step.

Process Considerations with Robotic Arm

There are certain parameters that need to be considered when implementing the process described in this paper with a robotic arm. The first consideration is the accuracy (not the repeatability, which is more important when the same point in space is accessed many times, as in mass production scenarios) of the robotic arm.

This will affect the achievable spatial grid density in the hardness testing process. The grid spacing must be a minimum of double the positional accuracy of the robotic arm. This means that if the robotic arm can move to within a radius of 1mm of given position, then the minimum grid spacing that can be used with that robotic arm is 2mm. This is at the very limit of the robotic arm, and a better constraint would be a minimum grid spacing of 2.5 times the accuracy of the robotic arm in order to prevent overlap of indents and ensure suitable readability of the serialization code marked. However, it should be noted that increased grid spacing will decrease the percent increase in surface area from the indents for enhanced joining. Thus, it is desirable to use the minimum grid spacing achievable with the robotic arm.

Another consideration is the ability of the robotic arm to provide sufficient force to generate accurate hardness readings. The portable hardness testers described in this paper are actuated by applying force into the material until the tip is sufficiently depressed in the material. If the force is inadequate to achieve this, the hardness readings will be inaccurate and inappropriate for material validation. The required force is dependent on the hardness tester that is being used, the angle of the material surface with respect to gravity, and the material that is being validated.

Process Prototyping with Robotic Arm

As part of the research described in this paper, the grid marking procedure is being prototyped with a WidowX250-6DOF robotic arm. This is a six-degree-of-freedom (6DOF) robotic arm composed of six revolute joints. Currently, the grid marking procedure is being prototyped without the use of a hardness tester to validate the ability of the robotic arm to mark grids with sufficient accuracy. The robotic arm is being controlled through the use of Python (NumPy and other standard libraries) using analytic solutions to the inverse kinematics to determine joint positions for any given position and orientation. Each motor is controlled by an individual PID controller. The locations for a grid are calculated by defining a starting location and a plane on which to mark the grid. Current testing has been in the XY plane and the YZ plane with the grid oriented along these respective axes. When more complex planes are tested, the grid orientation will also be required to define the mark locations. Once this phase of prototyping is complete and the placement accuracy of our robotic arm has been experimentally established, the hardness testing process will be prototyped to determine if this robotic arm can generate sufficient force to obtain accurate hardness values. The payload capabilities of our robotic arm are relatively low, so we may be unable to prototype this step of the process with our current equipment. If so, we will determine an appropriate robotic arm for more advanced prototyping and testing.

Conclusion

This paper describes the process by which nanoindentation is used for validation of materials and for unique marking of materials in an additive manufacturing-relevant scenario. Preliminary results show its value for simultaneously validating a material, preparing a surface for adhesion/binding, and labeling the surface with a unique ID. The additional value of this approach for forensic identification is also introduced.

A potential application of this process is in the binding of parts manufactured using dissimilar processes, such as bonding of an injection molded part to an AM part. In many cases, AM can be used to design more streamlined and complex parts. However, size constraints and other limitations in the AM process can be limiting to the parts that can be manufactured. This can be solved through joining of AM parts to parts manufactured through other means to maintain the complexity afforded through AM, while avoiding the AM process limitations. All four functionalities (material validation, serialization, forensic identification, and enhanced joining) are relevant to this use case. Additionally, in the case of part failure, this process would be helpful in determining the forensics of the failure. For example, the indentations which are no longer readable may be indicative of stress concentration during failure, which information would help identify vulnerabilities in the manufacturing processes taken. Finally, this process could be used for asserting copyright, similar to a watermark, with the only feasible means of removal cost-prohibitive for would-be fraudulent agents. The indentations are small enough to be nearly invisible to the casual observer, but under high-resolution imaging can be used to verify the origins of a part.

Future work will include prototyping the entire process described in this paper with a robotic arm. This will include further testing to determine achievable grid spacing, marking and identifying serialization patterns, and administering hardness tests with a robotic arm. Additionally, forensic analysis of the hardness testing indents will be explored to determine the level of identification (e.g. the number of independent bits of validation) that can be achieved using the indents.

References

[1] E. Hintsala, U. Hangen, D. Stauffer, “High-Throughput Nanoindentation for Statistical and Spatial Property Determination”, JOM 70, 494-503 (2018).

[2] S. Klein, G. Adams, F. Dickin, S. Simske, “3D Printing: When and where does it make sense?”, NIP & Digital Fabrication Conference, 2013 Int. Conf. on Digital Printing Technologies, 5-8 (2013).

[3] C. Sanchez-Camargo, A. Hor, M. Salem, C. Mabru, “A Robust Method for Mechanical Characterization of Heterogeneous Materials by Nanoindentation Grid Analysis”, Materials & Design, 194 (2020).

[4] G. Adams, S. Pollard, S. Simske, “High Resolution Imaging for Forensics and Security,” 2010 International Conference on Digital Printing Technologies, 372-376 (2010).

[5] A. Goddard, A. Hillman, J. Bond, “High Resolution Imaging of Latent Fingerprints by Localized Corrosion on Brass Surfaces”, Journal of Forensic Sciences, 55, 58-65 (2010).

Author Biography

Katrina Weinmann received her BS in Mechanical Engineering from the University of Pittsburgh (2018). She worked as a Mechanical Development Engineer in the Mechanical R&D division of Aerotech, Inc in Pittsburgh, PA until 2020. She is currently pursuing an MS in Mechanical Engineering at Colorado State University.

Steve Simske received his PhD from the University of Colorado in Electrical Engineering (1990). He worked for a NASA CCDS until 1994, then at HP and HP Labs until 2018. He is a Professor of Systems Engineering at Colorado State University, and an affiliate of the Mechanical and Biomedical Engineering Departments.