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Using Microscopy and Image Analysis to Show Density and Property Variations in Additive Manufactured Ti-6Al-4V

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Additive manufacturing of metals can be achieved by several methods, the most common of which fully melts the metal powder together selectively to form the desired shape. Alternatively, the powder can be bonded together and then post-processed to achieve densification. This method of binding powder selectively is used in powder bed binder jet printing: (1) a layer of powder is spread and flattened in the print bed, (2) a printhead selectively deposits binder for the layer, (3) the bed is dried with a heater, (4) the bed moves down one layer thickness and the process repeats. Parts are then cured and densified using post-processing techniques, which can include sintering and/or hot isostatic pressing (HIPing).

This study investigates density and microstructural variations in sintered and HIPed Ti-6Al-4V (Ti-64) large parts, in order to visually demonstrate that densification rates during post-processing vary within a part, leading to property variations within solid parts. Details on the first sample from the sintered part can be found in the publication [1]. Two samples, shown in Figure 1a, were printed in an ExOne M-Flex binder jetting 3D printer with gas-atomized Ti-64 powder sieved to -100/+325 (-147 µm /+44 µm), layer thickness of 150 µm, and binder saturation at 70%. Sintering as a precursor for HIPing was performed in vacuum with a ~100 h ramp to 1000 °C and a holding time of 2 h. HIPing was conducted at 954.4 °C and 101.7 MPa, for 8 h. Samples were cut with a bandsaw along a samples radius and the resulting wedges, shown in Figure 1b, were cut into eight pieces with a metallographic saw and hot mounted.

A Keyence VHX-600 optical microscope was used to image the cut sample pieces, which were then reassembled digitally to recreate the entire slice. After reassembly, the digital slices were split into a 7 × 2 grid for ImageJ analysis [2]. Each of larger grid block was then sub-divided by an ImageJ macro into 400 (20 × 20) images that were each analyzed for solid sample area fraction. In most images, this was equal to sample density for that block, except at edges. To solve the false density values at the sample edges, a manually solidified sample was created by using (fill holes + dilate) operations *i* times until the part was solid, then (erode) *i* times to return edges to their initial state, followed by a repeat of the solid sample area fraction calculation for each small sub-divided image. Sample density for each sub-divided image was then found using *density = (area of solid sample in original image)/(area of solid sample in manually solidified sample)* and mapped to visualize variations, results for which are in Figure 1c.

As extensively described in [1], density variations within the green part that are a product of the printing process will affect sintering and densification, including binder and powder effects [3,4]. This primarily explains the less dense edges of the part, but the flats vs. curve discrepancy required more investigation. In the current work, slice 2 is presented for additional data, and flat areas are not significantly less dense than the curve, as shown by Table 1. For slice 2, the average of the flat sections was 87%, versus 89% for the curve. That 2% difference is narrowed to 1% by calculating the median, which is less sensitive to outliers that were more prevalent at the edges of flat sections. Therefore, the difference between the flat sections and the curve in slice 1 was not a result that is consistent throughout the entire part.

The net shape of the HIPed sample was examined computationally to establish potential trends relating post-HIP shape distortion with density variation in the sintered preform. Regions of greater preform density will reach a state of compete densification during the HIP processing before adjacent initially less dense regions, potentially affecting property and microstructure distribution as well as shape distortion. A Zeiss Sigma500 FESEM was used to examine HIPed parts for microstructure variation for comparison against Vickers microhardness averages in different areas. Preliminary results suggest that there is a relationship between hardness and a combination of primary-α grain size and shape, though the effect of inhomogeneous densification rates on grain morphology has not yet been explored. [5]

[1] E Stevens et al., Additive Manufacturing **22** (2018), p. 746-752.
[2] CA Schneider, WS Rasband, KW Eliceiri, Nature Methods **9** (2012), p.671-675.
[3] H Miyanaji, S Zhang and L Yang, Int J Mach Tool Manu **24** (2018), p. 1-11.
[4] T DebRoy et al., Progress in Materials Science **92** (2018), p 112-224.
[5] The authors acknowledge Fred Yolton and Adam Byrd from Carpenter Technology Corporation. E. Stevens was partially supported by the Department of Defense (DoD) through the National Defense Science & Engineering Graduate Fellowship (NDSEG) Program. S. Schloder was funded by the PPG foundation, and would like to thank the Swanson School of Engineering for supporting undergraduate research. University of Pittsburgh MMCL facilities were used in this research.

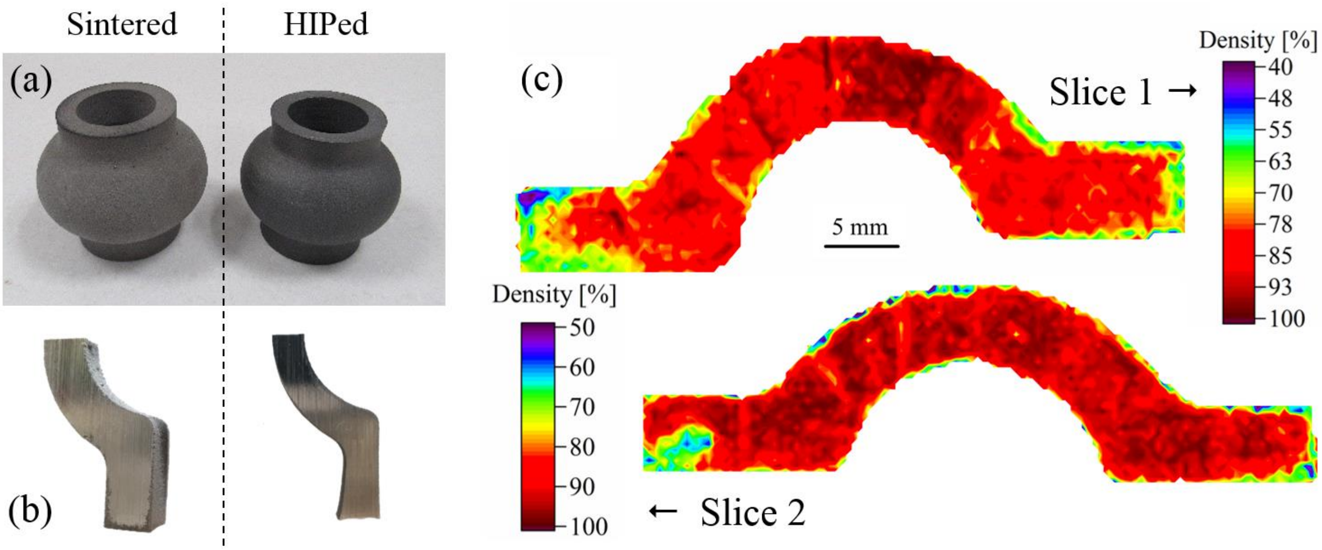

Figure 1. (a) Ti-64 parts before cutting, sintered and HIPed. (b) Bottom section of one slice, sintered and HIPed. (c) Sintered part density plots for slices 1 and 2, note color scale differences.

Slice	Flats Average	Flats Median	Curve Average	Curve Median
1	78%	81%	87%	89%
2	87%	92%	89%	93%

Table 1. Sintered part slice average and median densities, calculated from flat sections (bottom and top combined) and the curved sections.