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Hardness Variation of Microstructural Heterogeneities in Directed Energy Deposited 304L Stainless Steel

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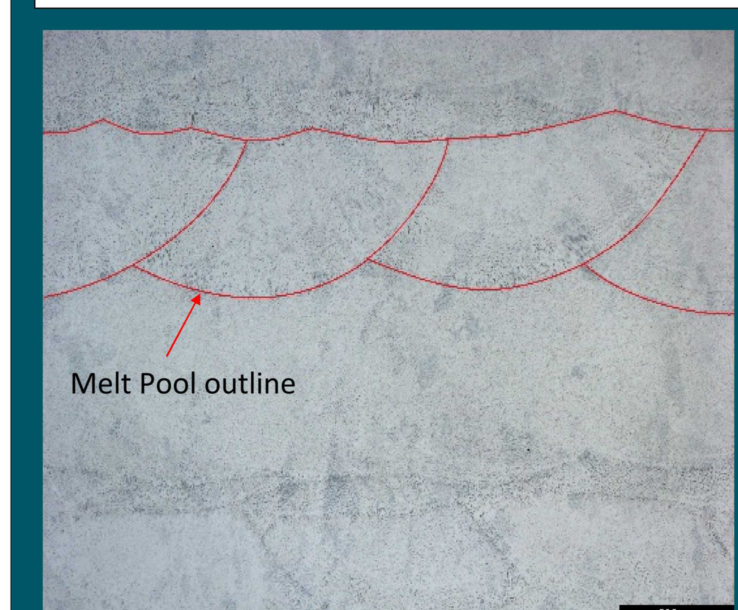
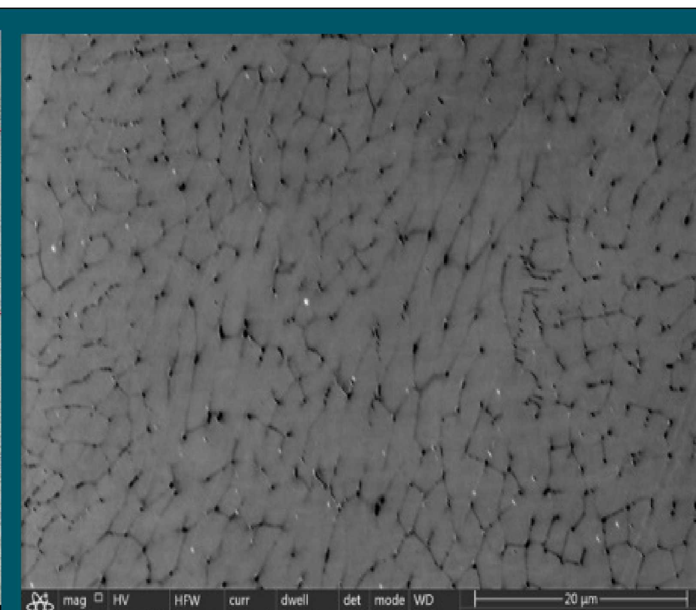
Introduction:

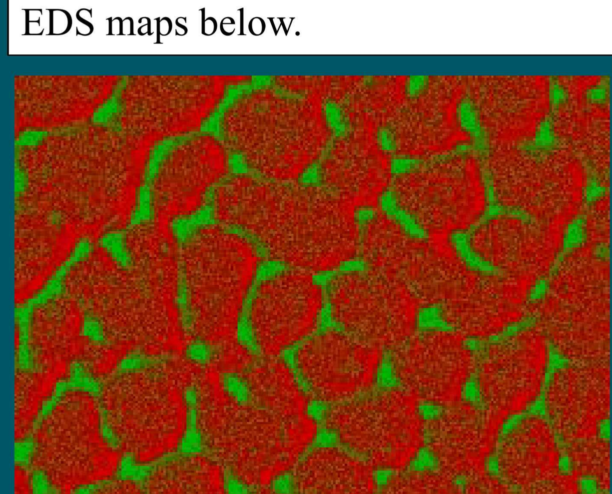
The rapid solidification that occurs during additive manufacturing processes results in a cellular structure with compositional variation across the cell boundaries. It has been shown that the dislocation interaction with the cell boundaries defined by this compositional micro segregation can impact the yield strength of the material [1]. While the Vicker's Hardness method can be used to measure the hardness of a bulk material, this technique cannot be used to measure the hardness of individual cell boundaries because the required indentation diagonal of greater than 17 ums in length [2] is much larger than the 1-2 um solidification cells. Nano-indentation, however, uses indent loads between 1 uN to 100 mN to acquire localized hardness of a material within a small area. The ability to measure hardness within a small volume of the material makes it a promising technique for isolating the mechanical properties of the distinct microstructural features in these materials that occur at the micron and submicron length scale.

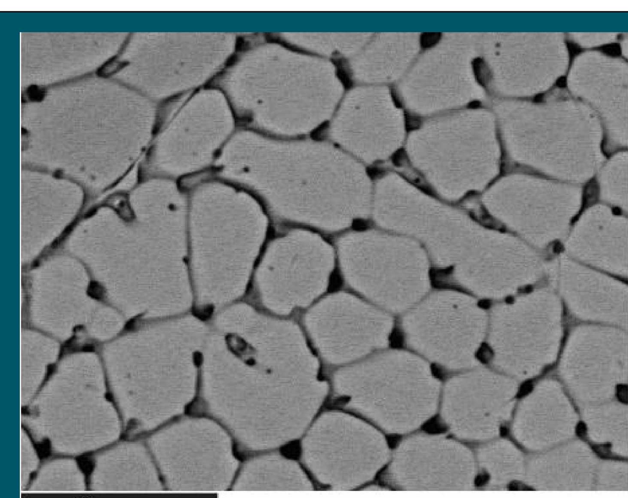
In the DED process considered here, powder is forced through nozzles into a laser energy source . A complex structure is built up one layer at a time by scanning the laser in a 2 D pattern for each layer.



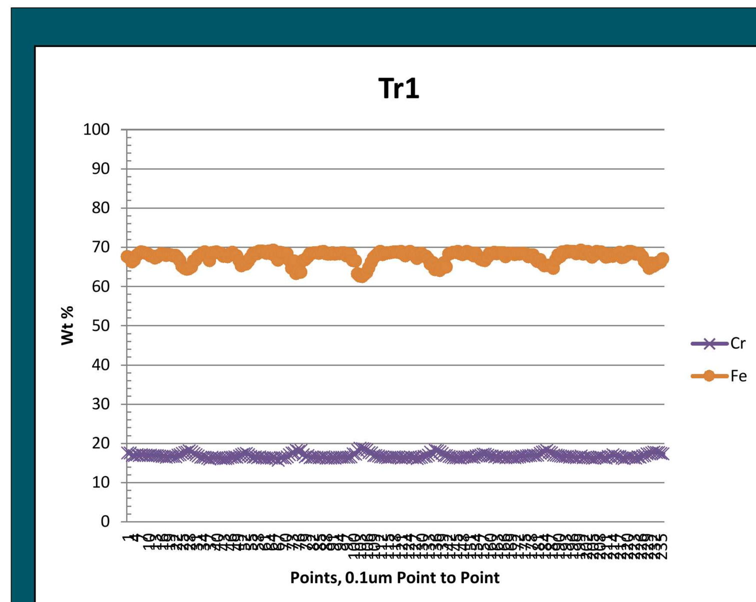


The etched optical micrograph (upper left) shows the array of adjacent melt pools formed by the laser scanning during deposition of 304L stainless steel. The red lines highlight the boundaries of the melt pools laser pass. The electron micrograph (upper right) of the same region shows that within the melt pools there exists cells that are defined by the chromium rich boundaries and a nickel rich interiors as shown in the EDS maps below.





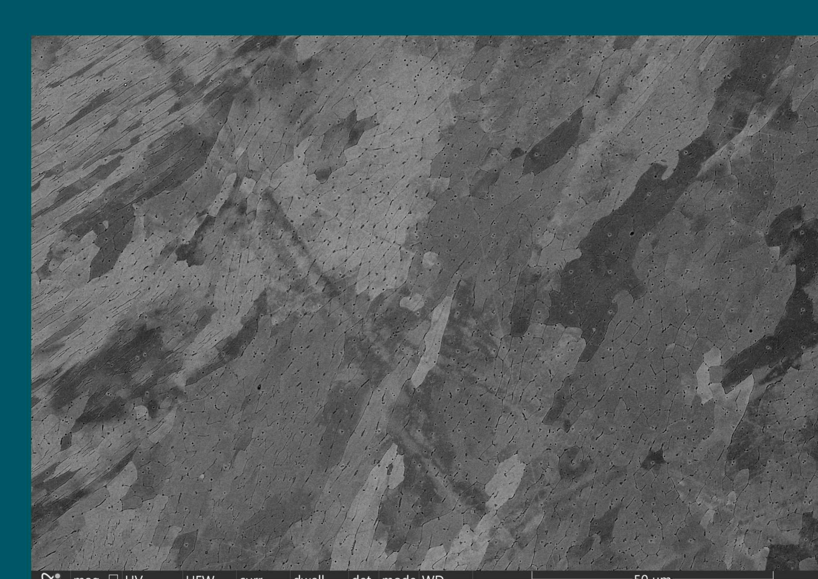
Cr-rich
Ni-rich

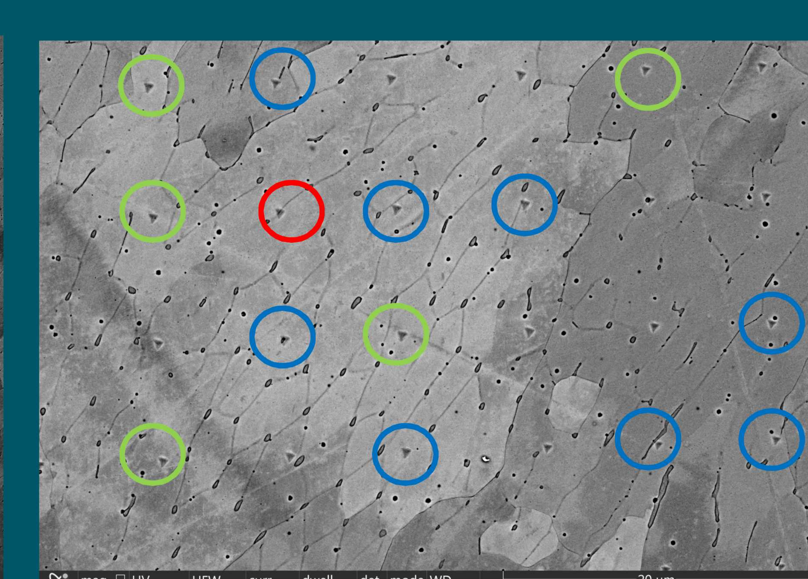


Microprobe data shows the quantitative compositional variance of Ni and Fe along the cell structures [3] as shown in the micrograph on the right.

Using modern software automation, it is possible to perform many indents without intervention from an operator. In this way, it is possible to obtain hundreds of measurements regularly spaced on the heterogeneous microstructure of these materials. A large number of total indents ensures that the large variety of microstructural features get measured multiple times. Then, statistical distributions can be used to understand how the different microstructural features contribute to the overall harness of these additively manufactured stainless steels.

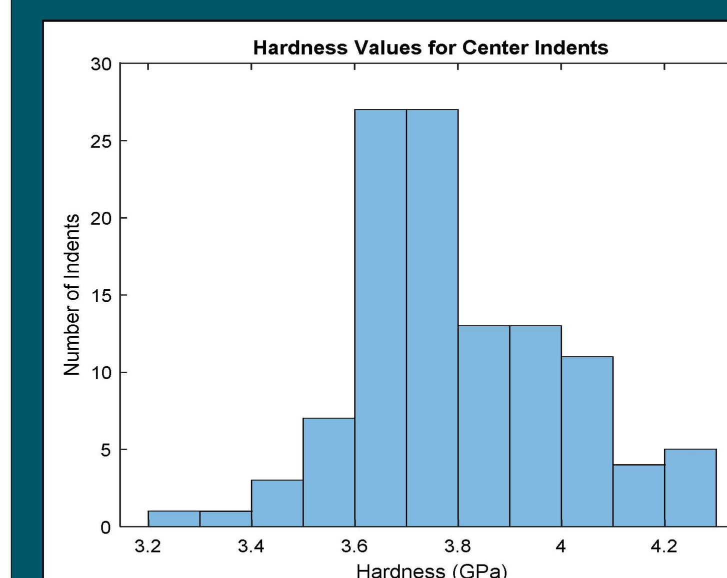
Nanoindentation was performed on directed energy deposited 304L austenitic stainless steel using a TI-950 Triboscan. The indentation was performed with a Berkovich tip with a tip radius of 100 nm. The indentation load was 900 uN to obtain indent radius of approximately 1 um and with an indent spacing of approximately 8 um on a polished surface. The sample was etched with an electrolytic etch that consisted of 55ml of H20, 15 ml of HCl and 60 ml of Nitric Acid at 1.15 Volts for approximately 5 minutes. An array of 19x14 indents covering an area 8 um x 8 um was analyzed.

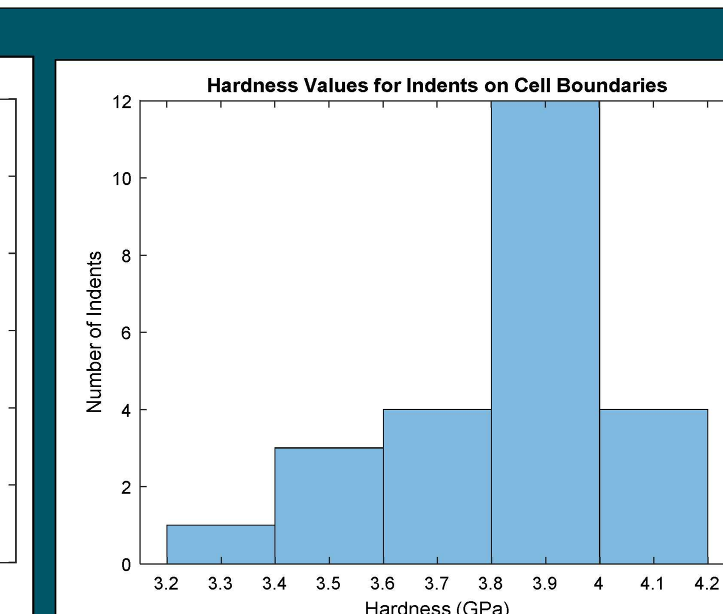




The array of indents was located in the SEM so that their location with respect to microstructural features could be identified. The electron micrographs above show where the indents were made in respect to the cells and other microstructural features on the etched DED 304L . The indent circled in red is considered to be on a cell boundary and the indents circled in green are considered to be in the center of the cell. Indents circled in blue were discarded because the indent was either on ferrite, grain boundaries, an oxide particle or the indent is partially on a cell boundary.

Out of 124 indents located within the center of a cell, 43.5% of the indents had hardness values that ranged from 3.6 GPa to 3.8 GPa. Out of the 24 indents that were on cell boundaries 50% of those indents ranged from 3.8 GPa to 4 GPa. There is an 8% difference in hardness between the cell boundaries and the interior of the cell with the boundaries being slightly higher on average.





Conclusion:

The results suggest that a difference exists indents located on cell boundaries and within the center. The higher hardness of cell boundaries is consistent with annealing behavior of DED 304L [1]. The difference in hardness, however, is quite small, and the statisitics for cell boundaries are still quite low for 24 out of 266 total measurements. More confidence could be gained with a larger array and smaller indents. These types of measurements elucidate the microstructure property relationships in DED stainless steels, and ultimately will guide processing steps that produce high quality material with reliable and predictable properties.

References:

[1] Thale R. Smith, Joshua D. Sugar, and Chris San Marchi, Julie M. Schoenung, Acta Materialia, **164** (2019) Pages 728-740.
[2] ASTM E384-11, Standard Test Method for Knoop and Vickers Hardness of Materials, ASTM International, West Conshohocken, PA, 2011, www.astm.org
[3] Richard Grant, Sandia National Laboratories New Mexico
[4] Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

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