## High Throughput Characterization to Quantify Microstructural Heterogeneities in Additively Manufactured Haynes 282

Avantika Gupta<sup>1</sup>, Sriram Vijayan<sup>1</sup>, Olivia Schmid<sup>1</sup>, Joerg Jinschek<sup>1,2</sup>, Carolin Fink<sup>1</sup>

<sup>1.</sup> Materials Science and Engineering, The Ohio State University, Columbus, OH, United States.

Haynes 282 is a precipitation strengthened nickel (Ni) based superalloy that finds extensive application in industrial gas turbine engines used in Advanced Ultra Supercritical (A-USC) powerplants, primarily due to its excellent high temperature creep resistance and thermal stability. These properties in combination with good weldability make this alloy an ideal candidate for fabrication by Additive Manufacturing (AM). AM offers the advantage of near-net shape fabrication of complex geometries while optimizing the amount of material used.

Electron Beam Melting (EBM) is a powder bed fusion-based AM process which uses a high energy electron beam to melt metal powder particles spread over a bed, in a layer-by-layer fashion to generate a near-net shaped component. During EBM processing, the material experiences steep thermal gradients and rapid thermal cycling, which results in reduced partitioning of solute elements and formation of columnar grains with non-equilibrium phases. Recent reports have shown that spatio-temporal thermal transients during AM can be controlled by varying the beam scanning strategy (energy input) to produce a columnar to equiaxed transition [1-4]. Previous research on Ni-based superalloys has shown that  $\gamma$  precipitation is strongly influenced by solidification rates and thermal treatments. And precipitation distribution influences mechanical properties such as hardness and tensile strength [5-6]. A wider adoption of AM processes in industry is often challenged by a lack of significant datasets to generate coherent process-structure-property linkages. In order to enable industrial qualification of EBM deposited Haynes 282 components, the effects of varying thermal gradients and solidification rates on  $\gamma$  precipitation and mechanical properties needs to be systematically analyzed. Studies investigating evolution of  $\gamma$  as a function of varying EBM parameters are still very limited [7].

In this work, we use high throughput electron microscopy characterization to quantify  $\gamma$ ' size distribution, and micro hardness testing to evaluate the impact on mechanical properties. AM build and sample geometry, as well as sites for detailed microstructural characterization are shown in the schematics in Figure 1. Three columns with the same build height (10 mm) but with different thickness of 2, 3 and 4 mm, respectively, were investigated to understand the impact of build geometry. A 2-mm-high section at the base of the build was included in the study to investigate the impact of a change in scan velocity. In order to perform systematic site-specific characterization, the sample was classified into regions according to build height and column thickness (as shown in Figure 1b).

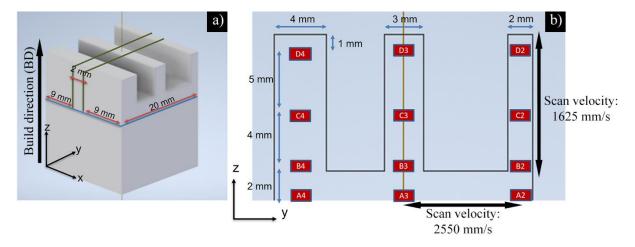
A low acceleration voltage (1 kV) and low working distance (5 mm) as well as in-column detectors were used in the scanning electron microscope (SEM) allowing for optimum resolution and contrast to resolve  $\gamma$ ' precipitates [8]. Both backscattered electron (Z contrast), and secondary electron (topological contrast) imaging was used to characterize  $\gamma$ ' precipitates at each location. MIPAR software was used for thresholding to obtain quantitative values for  $\gamma$ ' size. Automated image collection using FEI MAPS software was performed to generate a statistically significant dataset. As seen in the graph in Figure 2a,



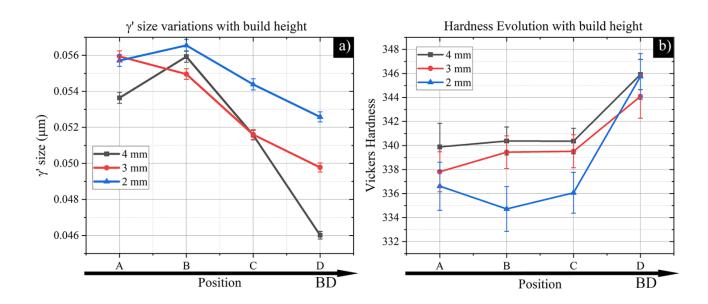
<sup>&</sup>lt;sup>2.</sup> National Centre for Nano Fabrication and Characterization, Technical University of Denmark, Kgs. Lyngby, Denmark.

in all three columns the size of  $\gamma$ ' precipitates decreases with increasing build height. This is a direct result of the increased thermal cycling that occurs in the bottom regions. This evolution is analogous to  $\gamma$ ' response in ageing treatments in wrought alloys [5]. Additionally,  $\gamma$ ' size increases on transition from location A to B for the 2 mm and 4 mm column, as lower scan velocity in region B enhances thermal cycling further. In the 3 mm column, this effect may be mitigated due to higher cooling rates.

Finally, Vickers micro hardness testing (Figure 2b) was performed to identify a correlation between location specific microstructural and mechanical properties. Micro hardness values increase with increasing build height, relating inversely to the size of  $\gamma$ ' precipitates which decreases with increasing build height. This validates the argument that as-deposited EBM Haynes 282 alloy exhibits  $\gamma$ ' precipitation strengthening [9].



**Figure 1.** (a)Schematic of EBM Haynes 282 build geometry indicating sample sectioning. (b) Schematic indicating 12 locations (in z-y plane), varying in build height and column thickness, used for site specific microstructural characterization.



**Figure 2.** a)  $\gamma$ ' size variations; precipitate size decreases with increase in build height for all three columns. b) Vickers hardness variations; hardness values increase with increasing build height and indicate an inverse relationship to  $\gamma$ ' size

## References:

- Shao, Meiyue et al (2020). The effect of beam scan strategies on microstructural variations in Ti-6Al-4V fabricated by electron beam powder bed fusion. Materials and Design. 196. 109165. 10.1016/j.matdes.2020.109165
- Sabina Kumar et al (2020). Dynamic phase transformations in additively manufactured Ti-6Al-4V during thermo-mechanical gyrations, Materialia, Volume 14,2020,100883, ISSN 2589-1529, DOI:10.1016/j.mtla.2020.100883.
- 3. Sames W. J. (2016). The metallurgy and processing science of metal additive manufacturing, Pages 315-360, DOI: 10.1080/09506608.2015.1116649
- 4. Vijayan, S. et al (2021). Quantification of extreme thermal gradients during in situ transmission electron microscope heating experiments. Microscopy Research and Technique, 1–11. https://doi.org/10.1002/jemt.24015
- 5. Ceena Joseph et al (2021). Precipitation of γ' during cooling of nickel-base superalloy Haynes 282, Philosophical Magazine Letters, 101:1, 30-39, DOI: 10.1080/09500839.2020.1841314
- 6. Blackwell, C. et al (2020). The Effect of Beam Scan Strategies on the Microstructure of EBM Additively Manufactured Inconel 738. Microscopy and Microanalysis, 26(S2), 2940-2941
- 7. Unocic, Kinga et al (2019). Evaluation of additive electron beam melting of haynes 282 alloy. Materials Science and Engineering: A. 772. 138607. 10.1016/j.msea.2019.138607.
- 8. Benjamin M. Georgin et al (2021). Optimizing image contrast of second phases in metal alloys, Ultramicroscopy, Volume 228, 2021,113346.
- 9. The authors acknowledge funding from Office of Naval Research, MURI grant 102359.