

Additive Manufacturing of Advanced Ni-Based Superalloys

Matthew Hope, Matthew Carr, David Starr, Owen Wellborn

Faculty Advisor: Dr. Xinghang Zhang

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Abstract: The use of a wider particle size distribution (10-63 μm) of Inconel 718 powder was investigated as an alternative to the more expensive particle size distribution of 15-45 μm currently used in the additive manufacturing industry. 1.5 wt.% yttria was also added to wide distribution powder to see if oxide dispersion strengthening would occur. While microstructure had only a substantial change in the yttria group, the mechanical properties significantly varied between all three groups. The results show that yttria has the potential to be used in additive manufacturing.

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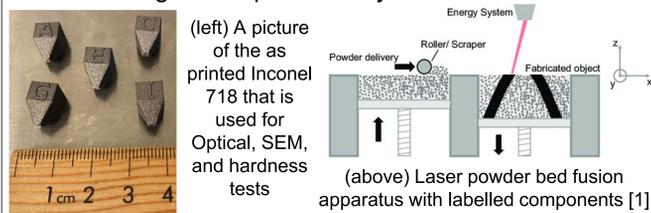
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Project Background

Inconel 718 is a widely used Ni-based superalloy that is favorable for harsh environments due to its corrosion resistance, strength, weldability, and ductility. It is ideal for use in additive manufacturing projects in applications such as gas turbine components, combustors, turbocharger rotors, etc.

Laser powder bed fusion is often used as an additive manufacturing technique for Alloy 718.



Oxide dispersion strengthening increases the mechanical properties of a material through the dispersion of small oxide particles [2]

Project goal: investigate the effects of nano yttrium oxide particles on microstructure and mechanical behavior of AM Alloy 718 and compare the properties of AM Alloy 718 with a control particle size distribution (PSD) (15-45 μm), wide particle size distribution (10-63 μm), and a wide particle size distribution alloyed with 1.5 wt% yttrium oxide particles.

Materials

Alloy 718: Samples were printed using a control PSD powder, a wide PSD powder, and a wide PSD powder with 1.5 wt% yttria added. All powders produced via gas atomization by Praxair.

Yttria: Nanoparticles of yttrium oxide were milled into 718 powder using YSZ cylindrical 10mm media for 1hr.

Design of Experiments (DOE): Laser power and scanning speed used in printing can be seen in the table below. Layer thickness (d) was 0.11 mm and the hatch spacing (h) was 40 μm . Examined specimens highlighted in yellow. Laser energy density (Ψ) equation is below on the right.

Laser Power (W)	Scan Speed (mm/s)			$\Psi = E/(v \cdot d \cdot h)$
	864	960	1056	
256.5	(A) $\Psi = 67.47$	(B) $\Psi = 60.72$	(C) $\Psi = 55.20$	E = laser intensity v = laser velocity d = layer thickness h = hatch spacing
285	(D) $\Psi = 74.97$	(E) $\Psi = 67.47$	(F) $\Psi = 61.34$	
313	(G) $\Psi = 82.47$	(H) $\Psi = 74.22$	(I) $\Psi = 67.47$	

Experimental Procedure

Powder Preparation: For the yttria added samples, 1.5 wt% of 30-40 nm yttria oxide powder was added to the 10-63 μm 718 powder. It was then milled using a Vibratom vibratory milling machine and cylindrical YSZ media.

Optical Microscopy: An Olympus BX41M optical microscope was used to examine the XY faces of the polished samples. Porosity was determined using grayscale thresholding on ImageJ with a threshold value of 100.

Scanning Electron Microscopy (SEM): Backscatter detection and energy dispersive x-ray spectroscopy (EDS) were carried out by using a Quanta 650 SEM at 15 KV and a spot size of 5.5.

Uniaxial Tensile Testing: Samples were printed and then cut to a dog bone shape. Polishing was done to 2000 grit to remove any surface imperfections. Tensile testing crosshead speed was 0.75 $\mu\text{m/s}$.

Vickers Microhardness: Each sample was tested 10 times with a 0.200 kg-f pyramidal diamond indent with a 13 second dwell time using a Wilson Hardness Tukon 1202. The average of the diagonal lengths were used to calculate the Vickers hardness value.

Microstructure Analysis

Optical Microscopy



Optical Micrograph of Sample E with 1.5 wt% yttria taken at 200x magnification.

Optical Micrographs revealed two types of porosity in 718: ceramic inclusions (red) and natural porosity in 718 matrix (blue). A table of the % porosity for each sample (XY plane) is below.

	1.5 wt% Yttria (%)	Control PSD (%)	Wide PSD (%)
A	2.3 \pm 1.3	0.010 \pm 0.003	0.014 \pm 0.006
C	1.8 \pm 0.7	0.037 \pm 0.030	0.044 \pm 0.018
E	1.3 \pm 0.3	0.010*	0.014 \pm 0.009
G	1.6 \pm 0.7	0.015 \pm 0.017	0.010 \pm 0.005
I	2.8 \pm 1.5	0.020 \pm 0.022	0.018 \pm 0.012

Ceramic inclusions were not seen in the wide or control group samples. Inclusions are likely agglomerated yttria or contamination from milling at Praxair.

SEM: EDS

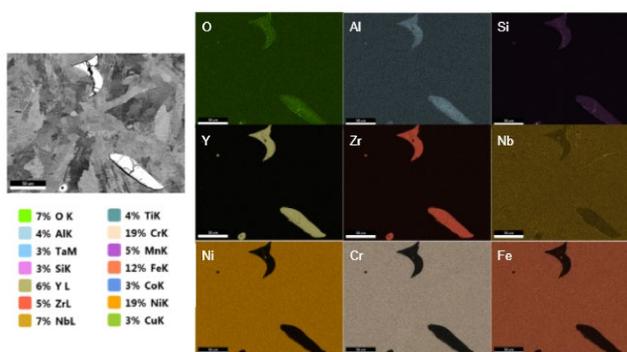


Figure shows backscatter image of yttria added sample E on the left with the color map of elements, and the right shows the elemental EDS area scans of the backscatter image.

EDS revealed that some of the porosity shown in optical were actually inclusions of yttria, oxygen, and zirconia which can be seen from the area scan above. These inclusions could be sourced from the YSZ milling media used in the high energy vibratory powder milling process which could have led to a degradation of the milling media. The scan also shows that the Ni, Cr, Fe, and Nb are well distributed throughout the sample.

SEM: Backscatter

Backscatter imaging revealed a cellular structure to the sample typical with additively manufactured metals. Closer inspection shows nanoparticles on the grain boundaries of the cellular structure. These lead to ODS strengthening.

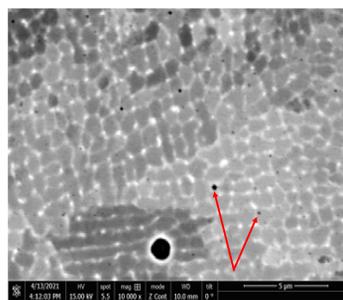
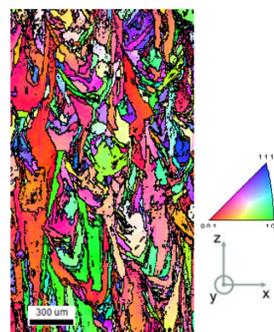


Figure shows backscatter image of yttria added sample E. Red arrows point to suspected nano yttria oxide particles.

SEM: EBSD

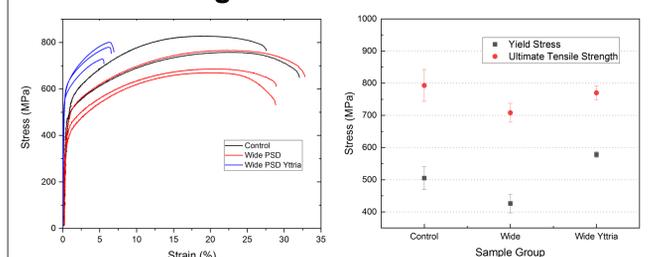
EBSD imaging of all control and wide samples revealed elongated grain structure in the build (z) direction. Grain size measurements revealed no clear grain size difference between the control and wide samples. Yttria added samples showed a more equiaxed grain structure.



EBSD image of wide sample E (x-z plane) showing texture and elongated grains in the z direction.

Mechanical Properties

Tensile Testing



(left) Two samples were tested for the control group and three samples for wide particle size distribution and wide particle size distribution with 1.5 wt.% yttria. (right) The calculated mean values with standard deviations from tensile testing for each group.

The tensile testing revealed a difference in mechanical properties of each sample group. The wide particle size distribution of the Inconel 718 reduced the yield and ultimate tensile strength compared to the control. The addition of 1.5 wt.% of yttria improved the values and put the mean value within the standard deviation of the control group. Despite showing an increase in porosity, the added Yttria showed signs that the Inconel 718 was oxide dispersion strengthened to result in the best yield stress. The reduction in ductility compared to the control and wide groups is likely due to the increase in porosity.

Vickers Microhardness

Sample	Vickers Hardness (HV0.2)				
	A	C	E	G	I
Control PSD	264 \pm 10	260 \pm 7	263 \pm 19	268 \pm 7	266 \pm 11
Wide PSD	270 \pm 7	274 \pm 14	279 \pm 14	265 \pm 12	271 \pm 18
Wide PSD Yttria	263 \pm 8	260 \pm 9	265 \pm 6	264 \pm 17	267 \pm 11

The hardness for the control PSD and wide PSD yttria have similar results and have an overlap of standard deviation with the Wide PSD. There is not a significant difference between the three sample groups.

Conclusion

- Wide group samples have reduced mechanical properties than control group
- ODS system was likely observed in 1.5 wt% yttria samples as it had better mechanical performance
- While the microstructure of the control and wide PSD was similar, the Yttria group had significant changes

Possible Future Work

- Analysis and alteration of ball-milling techniques to reduce porosity in Yttria sample group
- TEM study of 1.5 wt% yttria to verify ODS system
- Use control's narrow PSD with 1.5 wt.% yttria
- Tensile tests at elevated temperatures

References

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2. Oliver, W.C. and Nix, W.D. High temperature deformation of oxide dispersion strengthened Al and Al-Mg solid solutions, Acta Metallurgica, Volume 30, Issue 7, 1982, pp. 1335-1347.