Investigation of 3D Printed Alloy 718 with Oxide Dispersion

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Abstract: To improve the high temperature mechanical properties of 3D printed nickel-based superalloy (Alloy 718), the use of oxide dispersion was investigated. A small amount of yttria particles were ball milled into the Alloy 718 matrix. Their microstructure and mechanical properties were examined and compared with the 3D printed Alloy 718 without oxide particles. The addition of yttria oxide did not result in cracking or a loss of ductility. A more significant strengthening effect is anticipated if the size of oxide particles can be reduced further.

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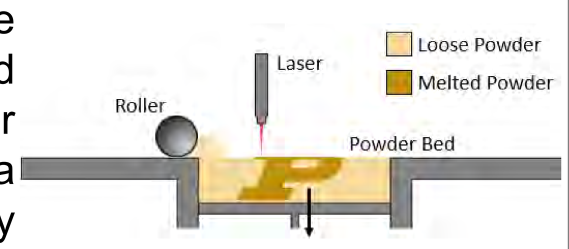
Project Background

Laser powder bed is an additive manufacturing technique used for 3D printing that uses a layer deposition process to melt a powder material. Laser energy density (ψ) is an empirical metric used to measure the optimal printer parameters for 3D printing.



Schematic of laser powder bed 3D printing.

Alloy 718 is a nickel based superalloy that is commonly used in the aerospace industry, highly weldable, and strong. At high temperatures, Alloy 718 needs increased creep resistance and mechanical strength. Incorporating oxide particles into the Alloy 718 matrix will fulfill these needs. There is a risk of losing ductility and creating cracks if the oxide particles are improperly introduced.

The project goal is to strengthen Alloy 718 with oxide dispersion strengthening (ODS). ODS consists of two possible strengthening mechanisms, dislocation pinning and deflecting crack propagation. [1]

Materials

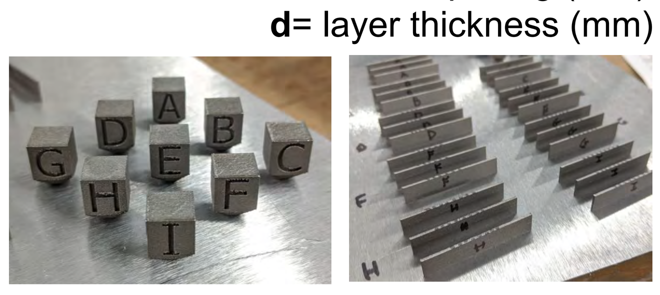
Alloy 718: Metal powder produced by gas atomization with a particle size of 30 – 50 µm was used for printing parts. Powders were produced by Praxair using their typical production parameters. [2]

Yttrium Oxide: Particles of yttria were ball milled into the Alloy 718 powder at 1 wt.%. Particle size ranges from approximately 1 – 5 µm.

Design of Experiments (DOE): The equation for calculating laser energy density (ψ) is shown to the right. The thickness (d) of each layer of particles is 0.11 mm and the hatch spacing (h) was held constant at 40 µm.

$$\psi = \frac{E}{v \cdot d \cdot h}$$

E = Laser Power (W)
v = Laser Velocity (mm/s)
h = hatch spacing (mm)
d= layer thickness (mm)



		Scan Speed (mm/s)		
		864	960	1056
Laser Power (W)	256.5	(A) ψ = 67.47	(B) ψ = 60.72	(C) ψ = 55.20
	285	(D) ψ = 74.97	(E) ψ = 67.47	(F) ψ = 61.34
	313	(G) ψ = 82.47	(H) ψ = 74.22	(I) ψ = 67.47

Specimens examined are highlighted in yellow.

Experimental Procedure

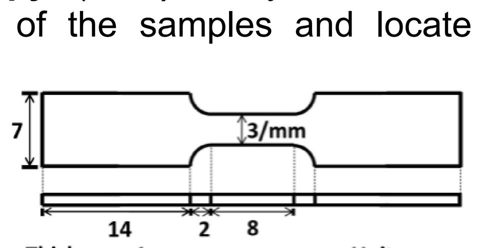
Laser Bed Printing: EOS 290 laser 3D printer used for production of samples is shown on the right.

XRD was performed with the Bunker D8 Focus diffractometer with a 2θ scan from 30° to 90° using a Cu-α wavelength emission of 1.54056 Å to prospect for general peak locations.

Scanning Electron Microscopy (SEM): Both the cubic and dog-bone samples were analyzed with a Quanta 650 scanning electron microscope.

Energy Dispersive X-Ray Spectroscopy (EDS) analysis is also performed, to investigate the chemistry of the samples and locate the yttria particles.

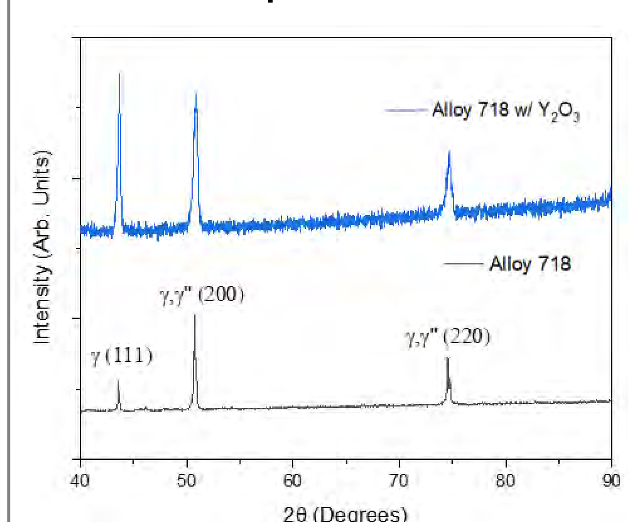
Uniaxial Tensile Testing: Tensile bars with the dimensions seen to the right were mechanically tested with a crosshead speed of 0.001mm/s.



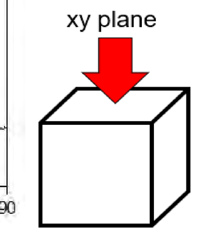
Vicker's Microhardness: An AMH55 Automatic Hardness Testing System indented nine measurements into each XY plane of cubes with varying printing parameters and a 200 g force. The average length between the diagonals of each indent was used to determine a Vickers Hardness Number.

Microstructure Analysis

XRD: XY planes scanned

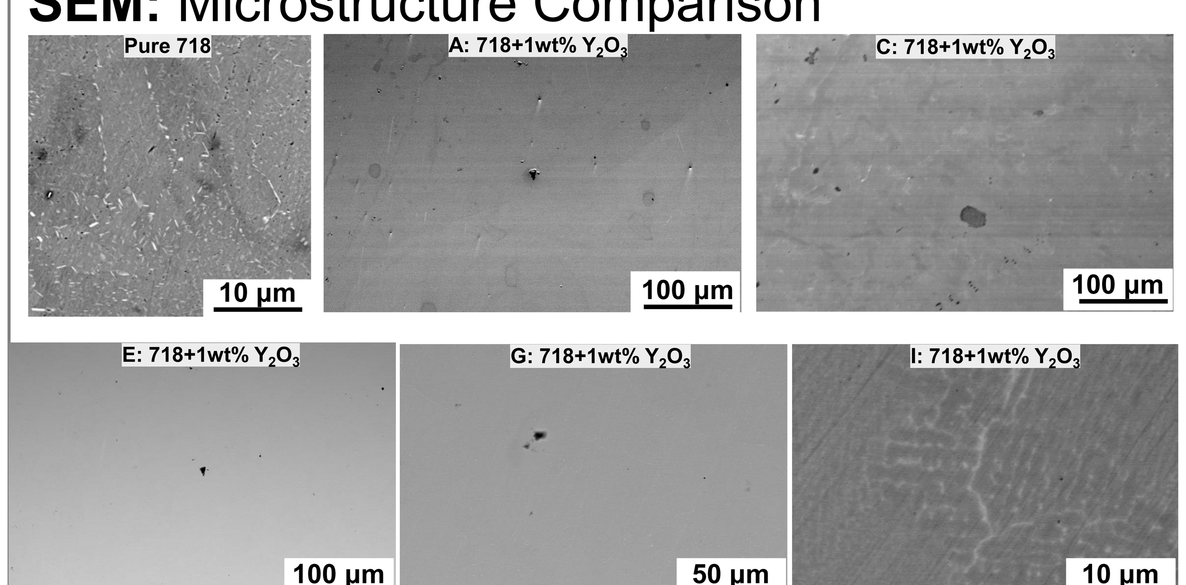


There were no drastic peak shifts when Y_2O_3 particles were added to the Alloy 718 matrix. The variation of peak intensity indicated the texture variation.



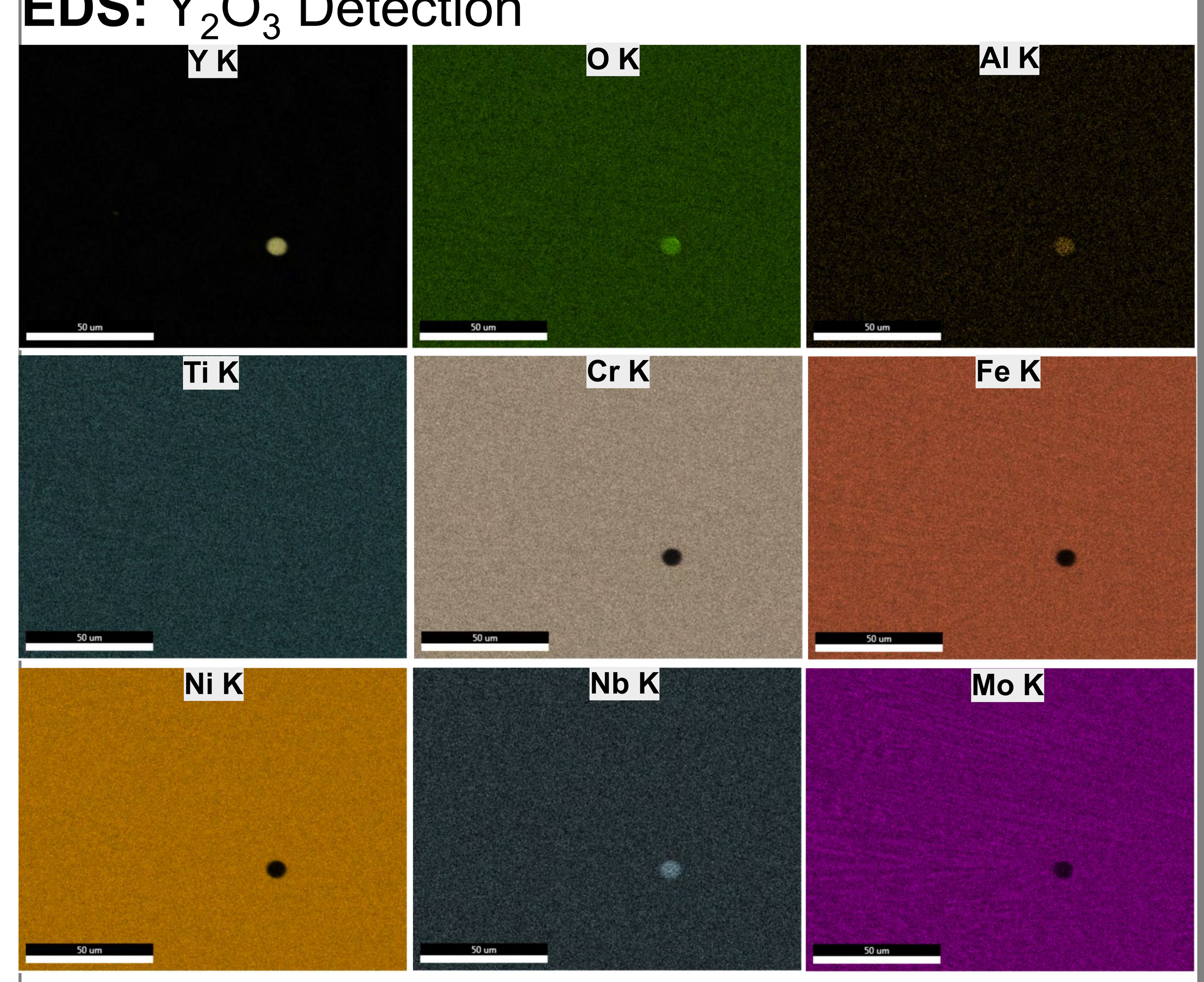
Both samples are printed with a scan speed of 960 mm/s and laser power of 285 W.

SEM: Microstructure Comparison



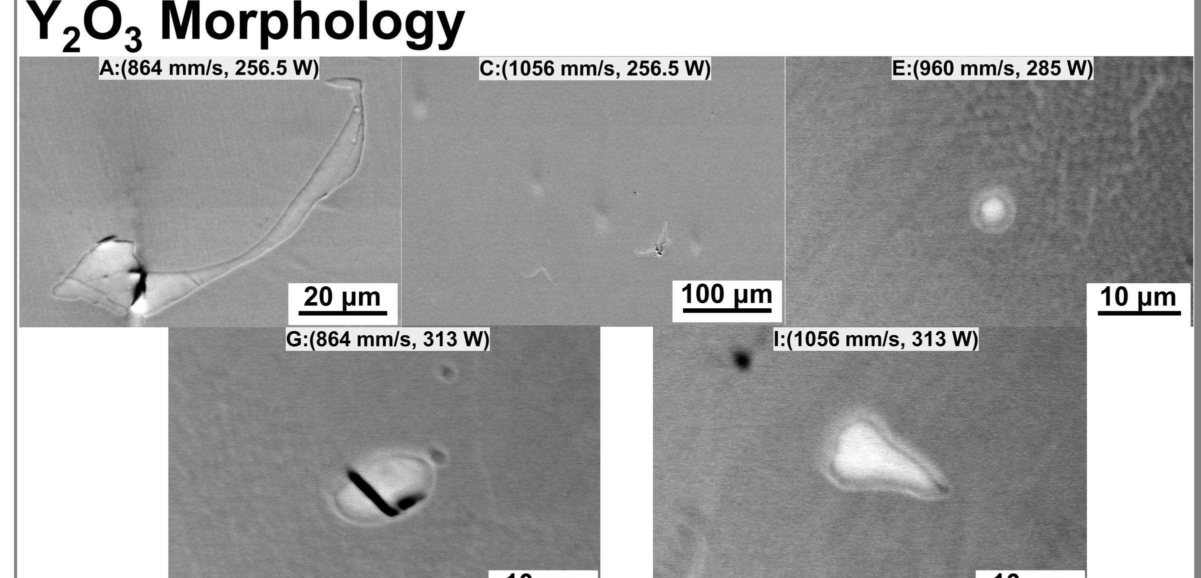
The SEM imaged revealed that after adding Y_2O_3 no significant cracking was introduced to the system. Cellular structure was also observed in these samples.

EDS: Y_2O_3 Detection



The EDS analysis confirmed the presence of the Y_2O_3 , indicating that Y_2O_3 particles can survive the high processing temperature.

Y_2O_3 Morphology



The morphology of the oxide particles depends on the printing parameters.The printing parameter E (960 mm/s, 285 W) leads to the smallest and spherical oxide particles. Whereas, agglomeration of yttria occurs when applying other printing parameters.

Acknowledgements

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Mechanical Properties

Uniaxial Tensile Testing

Figure (right): Engineering stress vs engineering strain for tensile bars with 1 wt.% yttria. The 718 label indicates pure Alloy 718 printed at position E.

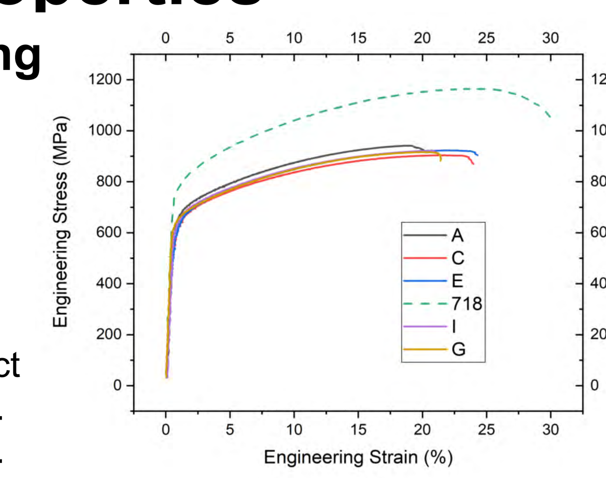


Figure (below): plot of 0.2% offset yield strength with respect to scan speed and laser power. The red arrow shows the trend.

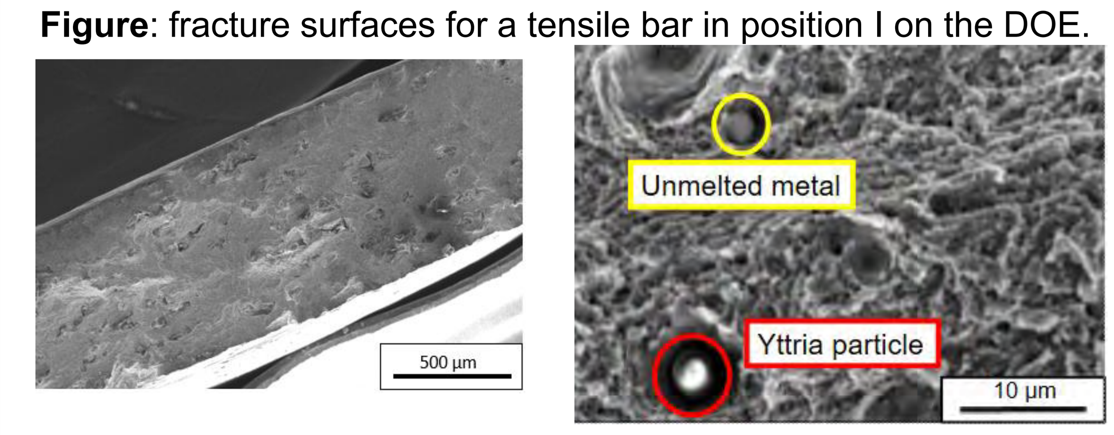
Yield Strength (MPa)		Scan Speed (mm/s)		
		864	960	1056
Laser Power (W)	256.5	597		583
	285		609	
	313	637		608

The pure Alloy 718 sample was stress relieved while the samples with yttria were tested as printed. The stress relief was performed at 982°C for one hour. This stress relief likely precipitated γ' and increased overall strength of the pure 718 sample with deformable particles to pin dislocations.

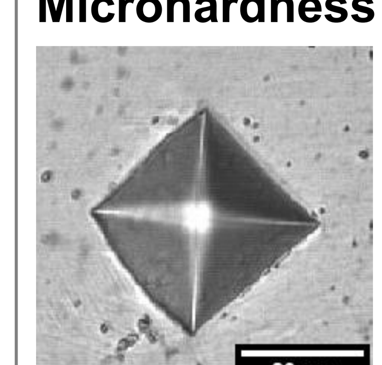
Table: Calculated mechanical properties for tensile specimens with 1 wt.% yttria (see DOE) and pure 718 with position E print parameters.

Property	A	C	E	I	G	718
Modulus (GPa)	90	89	86	111	114	130
0.2% YS (MPa)	597	587	609	608	637	744
Tensile stress (MPa)	942	904	924	921	917	1166
Strain Hardening Coef. (n)	0.128	0.120	0.126	0.128	0.126	0.128
Elongation (%)	21	24	25	22	22	30

Figure: fracture surfaces for a tensile bar in position I on the DOE.



Microhardness Tests



An indentation (right) on the surface of a cube sample describes the hardness of the material. A greater average diagonal length is indicative of a softer material. The microhardness is typically three times that of the flow stress, which is depicted in the table below

	A	C	E	G	I	Pure 718
Avg Vickers Microhardness	276.4 ±5.7	276.5 ±8.2	256.1 ±17.2	253.5 ±7.2	283.0 ±10.7	317.4 ±7.2
Flow Stress (MPa)	92.1	92.1	85.3	84.3	94.3	105.6

For the E position on the DOE, a two tail t-test with 5% significance level found that there was a significant decrease in strength from pure 718 when 1 wt.% yttria was added. The statistical analysis also showed that positions A, C, and I on the DOE had significantly higher Vicker's microhardness than G and E, however within these groups there is no statistical significance.

Conclusions

- The addition of oxide inclusions was successful and did not compromise the mechanical properties of laser printed Alloy 718.
- The optimal laser parameters for printing Alloy 718 with yttria particles is a scan speed of 960 mm/s and laser power of 285 W, which can limit the presence of unmelted powders and agglomerated yttria particles.

Recommendations

- The team advises future studies to add nanosized yttria particles to Alloy 718 to improve the mechanical properties
- Apply a scan speed of 960 mm/s and laser power of 285 W for future 3D printing parameters to minimize defects

Reference

1. W.C. Oliver, W.D. Nix. High temperature deformation of oxide dispersion strengthened al and Al-Mg solid solutions, Acta Metallurgica, Volume 30, Issue 7, 1982, Pages 1335-1347.
2. Praxair Surface Technologies. (2018). TruForm 718 Metal Powder. *Praxair S.T. Technology, Inc.*