

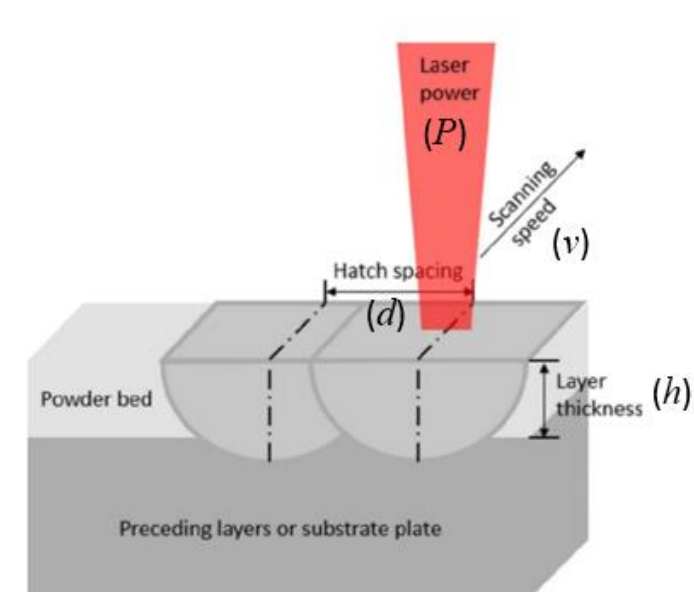
Abstract: An oxide dispersion strengthened (ODS) nickel-based superalloy (Alloy 718) was additively manufactured by direct metal laser sintering to investigate the viability of printing ODS superalloy systems. Two different weight percentages (0.5 wt% and 1.75 wt%) of chrome oxide (Cr_2O_3) particles were added to the Alloy 718 matrix. Their microstructure and mechanical properties were compared to control samples of pure Alloy 718. No visible cracks were observed in the ODS-Alloy 718 samples, and there was no significant decrease in mechanical properties.

This work is sponsored by Praxair Surface Technologies in Indianapolis, IN



Project Background

Direct metal laser sintering (DMLS) is a common additive manufacturing technique that is utilized to print many nickel-based superalloys, including Alloy 718. An equation for energy density has been developed based on the printing parameters.



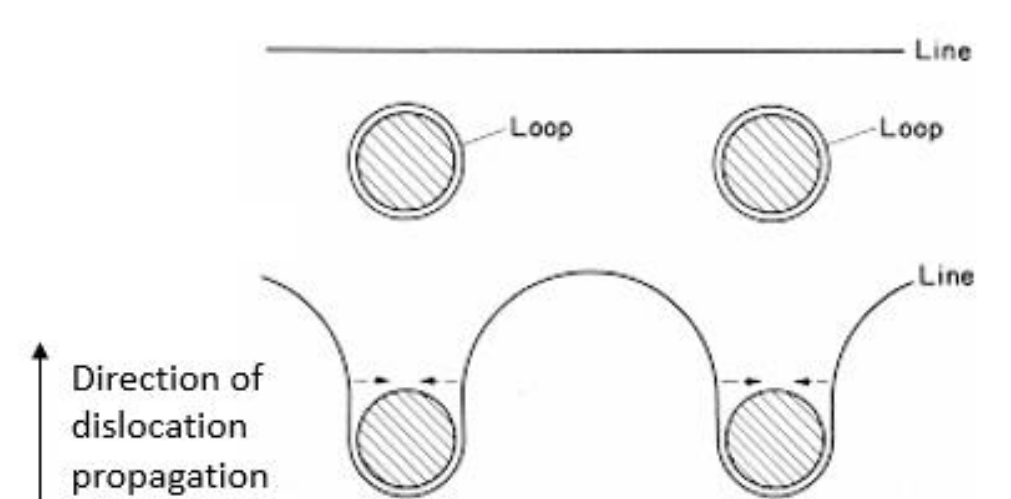
Schematic of DMLS process with the printing parameters labeled^[1].

Energy density (ψ)

- P : laser power
- v : scan speed
- d : hatch spacing
- h : layer thickness

$$\psi = \frac{P}{vdh}$$

The addition of oxide particles to a metal matrix theoretically strengthens the system through the mechanism of Orowan strengthening. The oxide particles act as obstacles to dislocation motion and aid in deflecting crack propagation, leading to oxide dispersion strengthening (ODS).



Schematic of the Orowan strengthening mechanism^[2]. The oxide particles inhibit dislocation motion, causing the dislocation to bow around the particle.

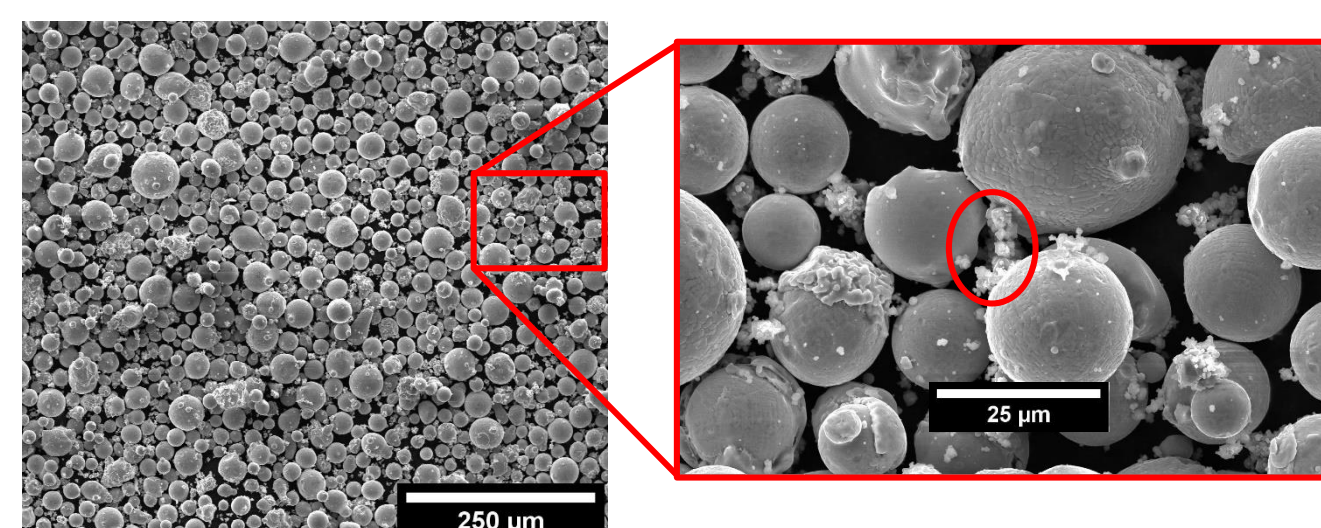
Goal: The goal of this project is to determine the viability of printing ODS-Alloy 718 via DMLS by characterizing the microstructure and mechanical properties of the printed parts.

Materials

Alloy 718 is a nickel-based superalloy with good creep resistance and weldability. The following chart is the chemical composition of the Alloy 718 powder in weight percentage. The average powder diameter was 28.9 μm .

Ni	Cr	Fe	Nb	Mo	Ti	Al
53.3	18.8	balance	5.20	3.00	0.9	0.4

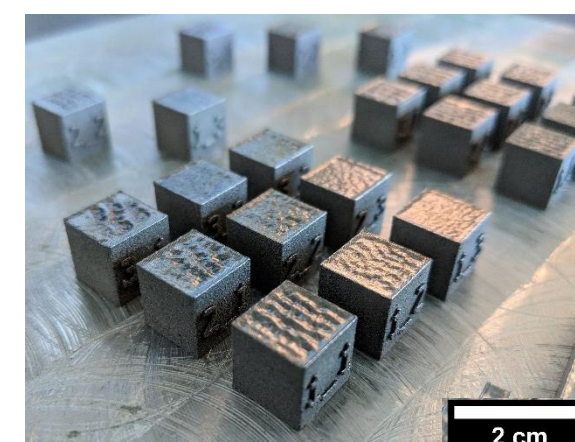
Chrome oxide (Cr_2O_3) was chosen as the strengthening oxide because there was a greater chance of matrix cohesion. 0.5 wt% and 1.75 wt% of Cr_2O_3 were utilized. The average particle diameter was 1 μm .



SEM micrographs of the 1.75 wt% Cr_2O_3 - Alloy 718 powder. The Cr_2O_3 particles are circled in red.

Design of Experiment (DOE): Scan speed and laser power were varied while the layer thickness and hatch spacing were kept constant at 0.04 mm and 0.11 mm, respectively. Analysis was focused on the middle energy density samples, which is highlighted in the table below.

		Scan Speed (mm/s)			
		864	960	1056	
Laser Power (W)	256.5	67.5	60.7	55.2	
	285	75	67.5	61.3	
	313.5	82.5	74.2	67.5	



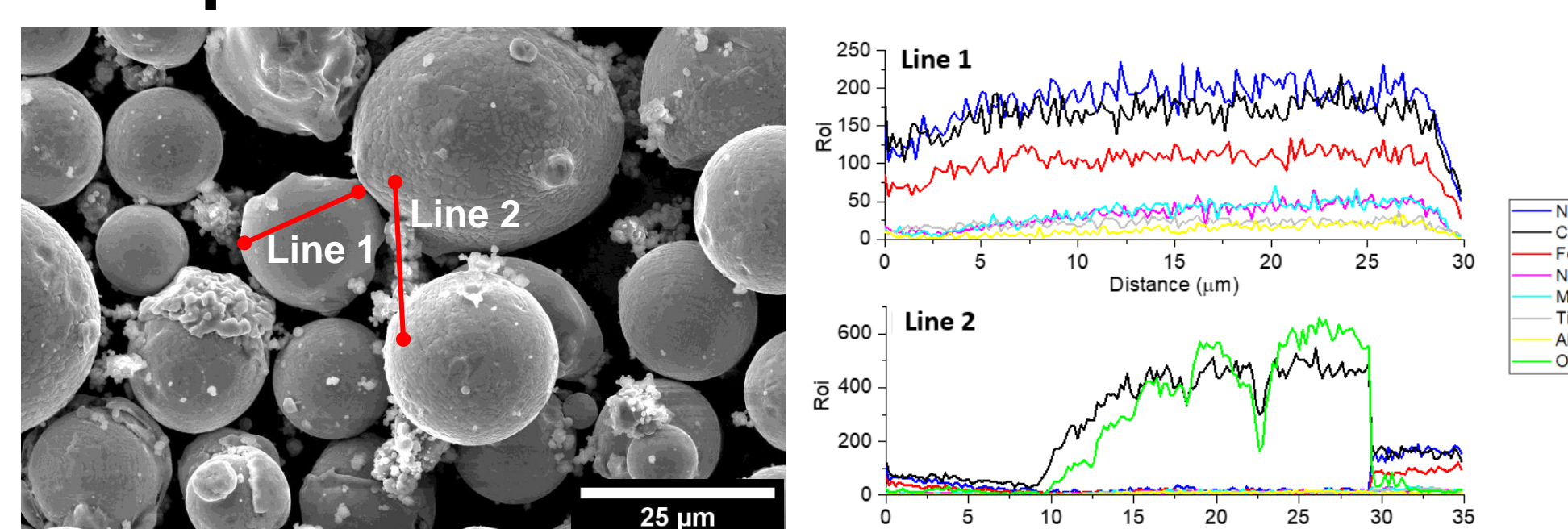
The 1.75 wt% Cr_2O_3 -Alloy 718 samples produced with the EOS M290 printer.

Experimental Procedure

- **SEM** was performed with the FEI Quanta 650. SE and BSE micrographs were obtained as well as EDS and EBSD data.
- **XRD** was performed with a Bruker D8 Focus diffractometer with a 2θ scan range from 30° to 100° .
- **Tensile tests** were completed with the MTS Insight with a jog rate of 0.006 mm/s and 30 kN load cell.
- **Vickers microhardness** utilized a Leco LV 100 system with a 1 kg-f load cell and 10 second dwell time.

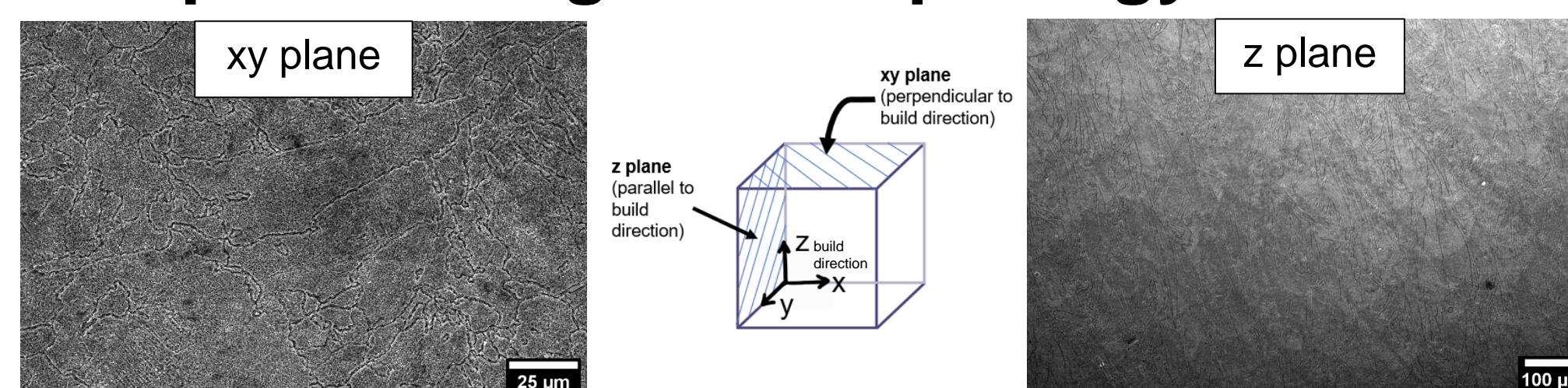
Microstructural Analysis

Oxide particle detection



The second EDS line scan of the 1.75 wt% Cr_2O_3 powder mixture displays a sharp increase in oxygen and chromium content, indicating the presence of the oxide particles.

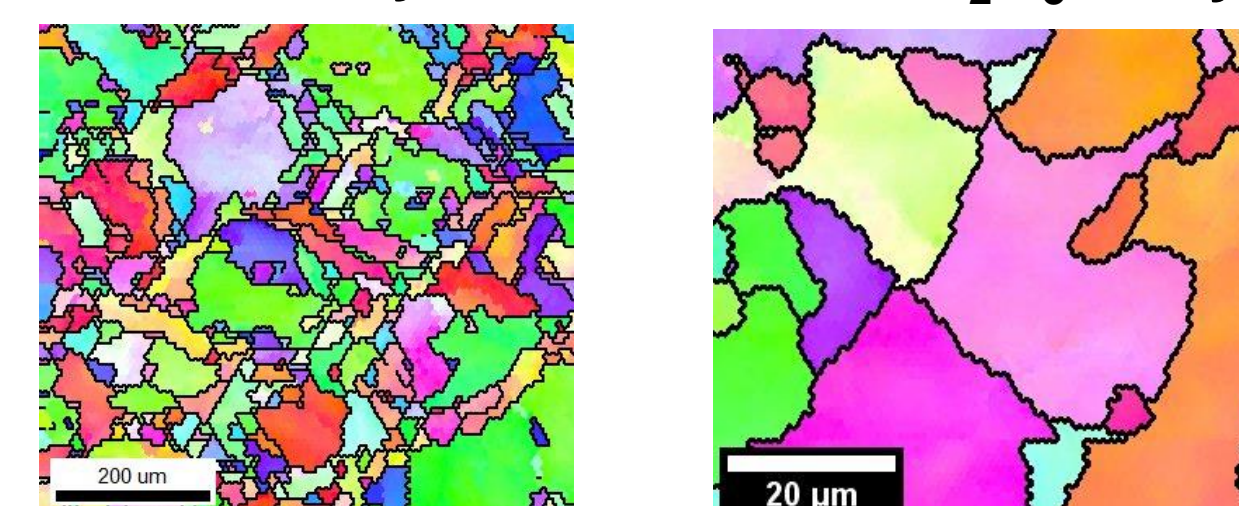
Comparison of grain morphology



Due to the anisotropic cooling rate inherent to the DMLS process, the grains are columnar when parallel to the build direction (z plane). However, the grains have a more equiaxed morphology when perpendicular to the build direction (xy plane).

EBSD: xy planes

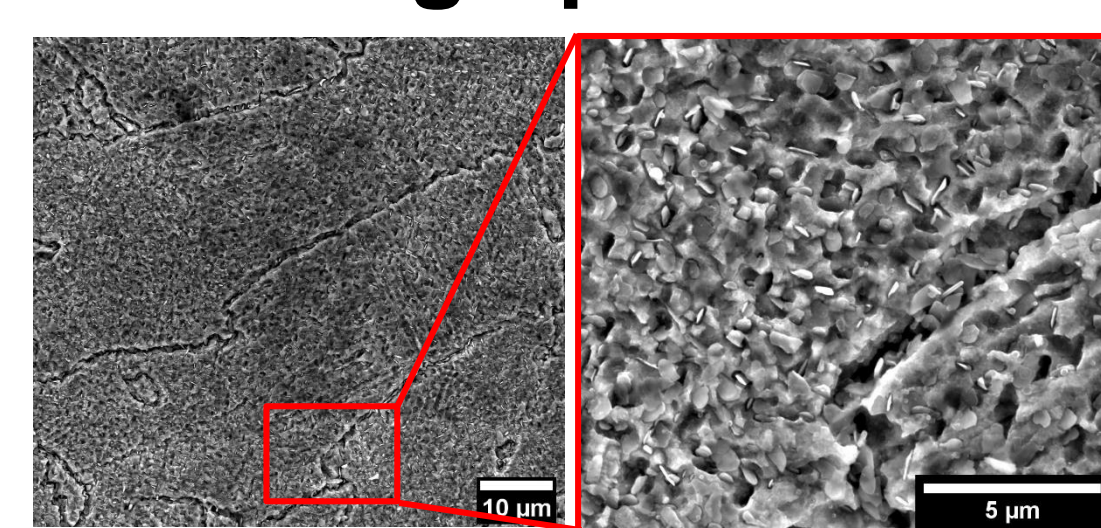
Control Alloy 718 1.75 wt% Cr_2O_3 -Alloy 718



Average grain diameter: 33.2 \pm 21.3 μm

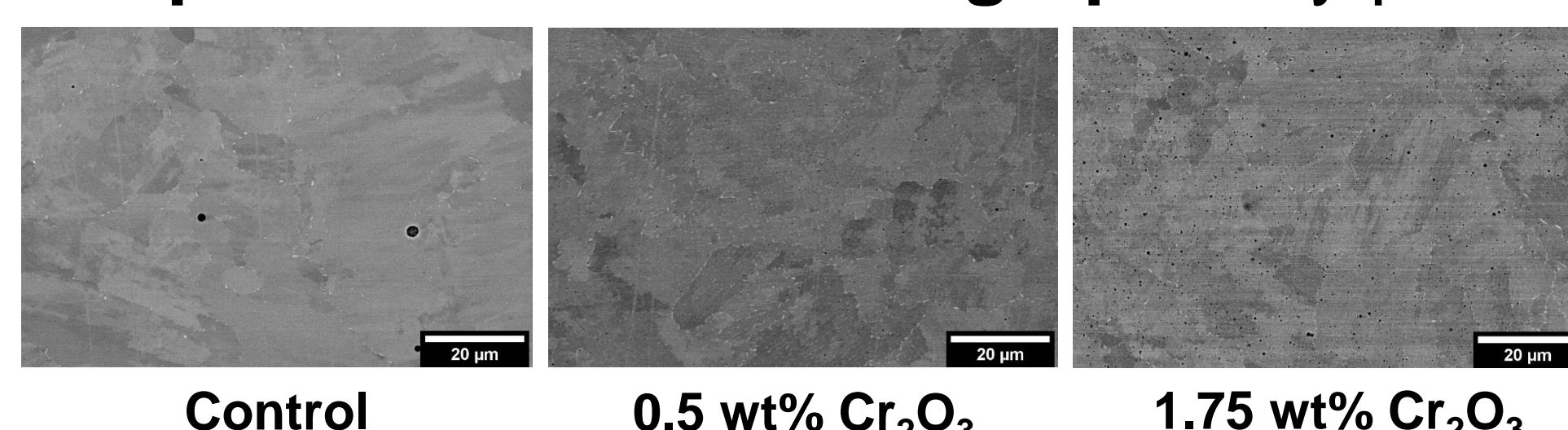
Inverse pole figures comparing the grain size of the control Alloy 718 to the 1.75 wt% Cr_2O_3 -Alloy 718.

SE micrographs: 0.5 wt% Cr_2O_3 -Alloy 718



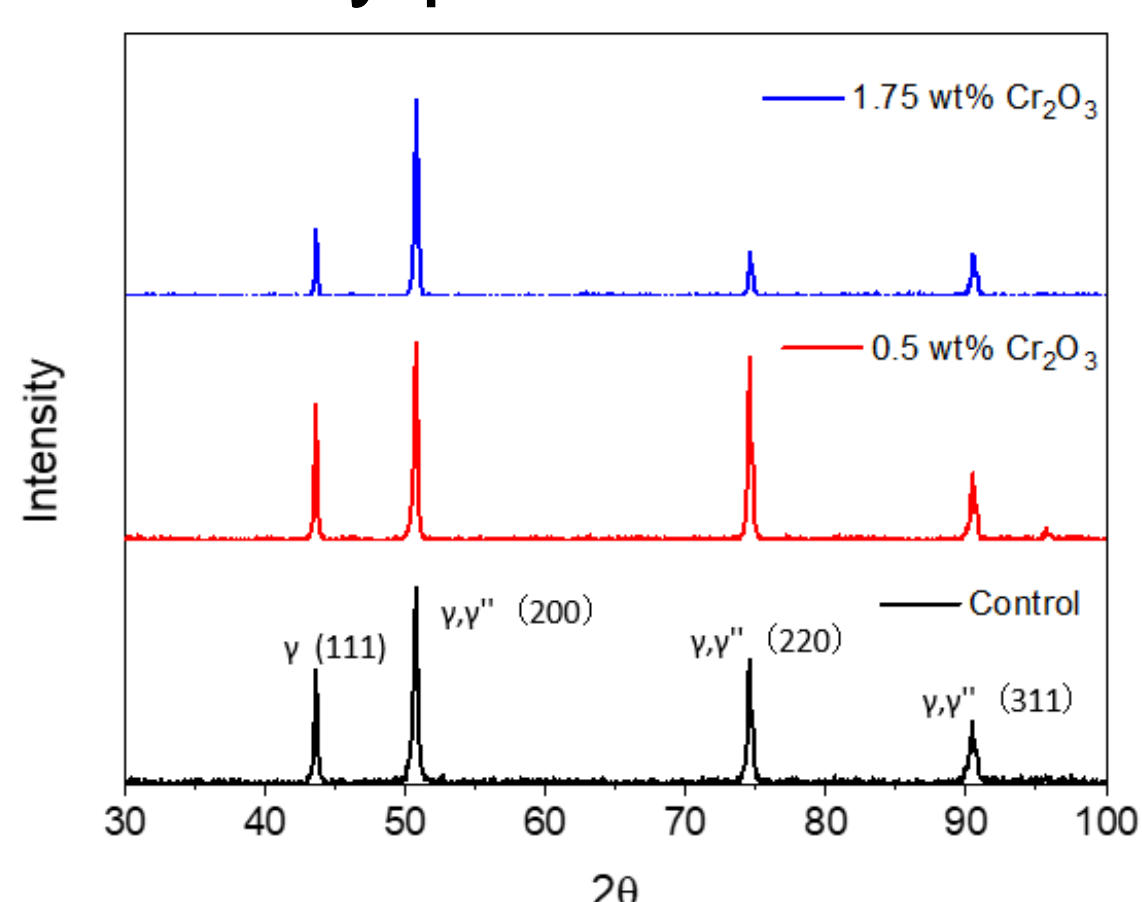
SE micrographs of the 0.5 wt% Cr_2O_3 -Alloy 718 in the xy plane. The sample was electro-etched with 10 wt% chromic acid. The intragranular precipitates are clearly visible.

Comparison of BSE micrographs: xy planes



As the oxide percentage increased, the porosity and number of intragranular precipitates increased as well.

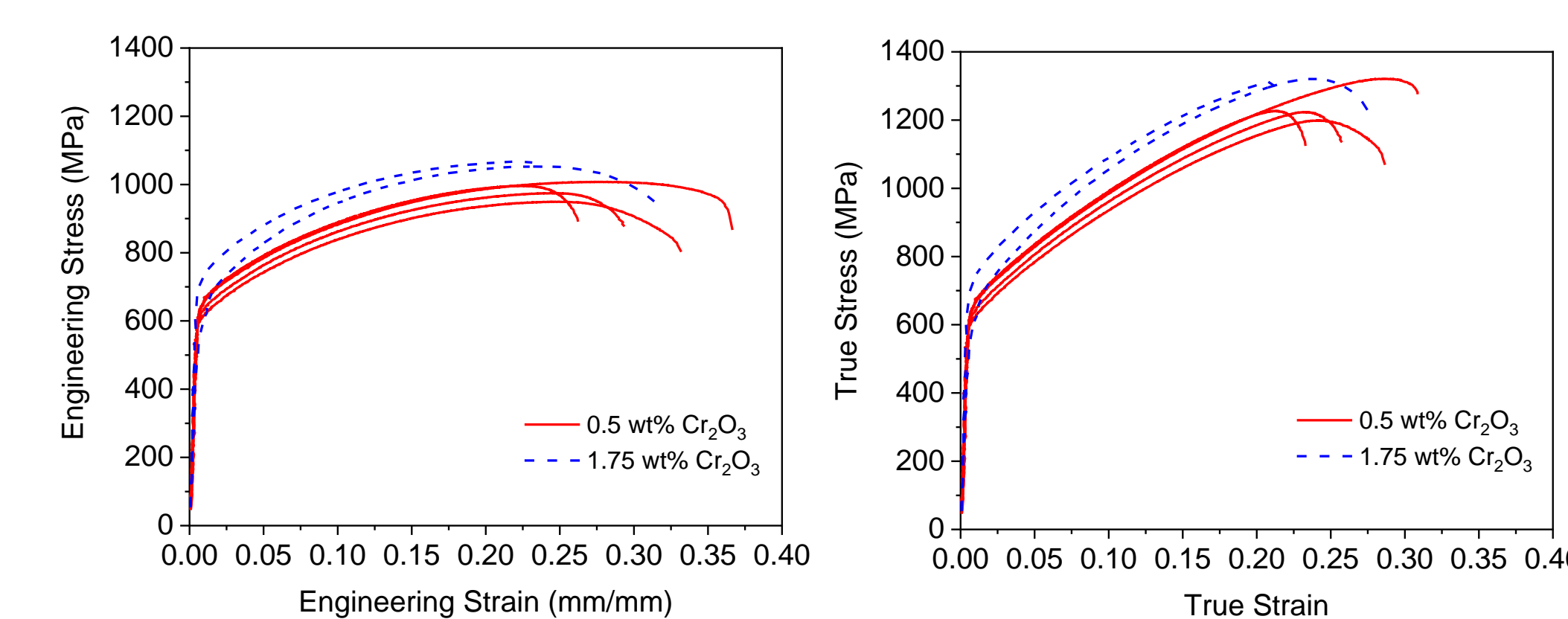
XRD: xy planes



There are no peak shifts as oxide concentration increases, indicating no variation in interplanar spacing. However, peak intensity fluctuates, signaling a texture change.

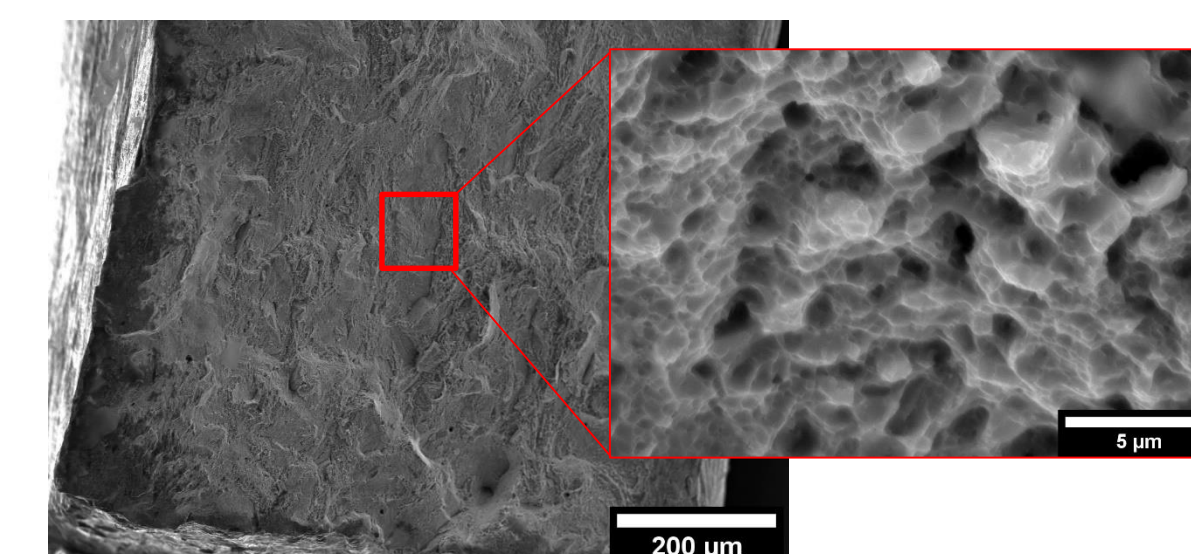
Mechanical Properties

Tensile Tests



	Young's Modulus (GPa)	Yield Stress (MPa)	Elongation at Fracture (%)	ϵ_{neck} (%)	Strain Hardening Exponent (n)
Industry Alloy 718 ^[3]	160 \pm 20	780 \pm 50	27 \pm 5	n/a	n/a
0.5 wt% Cr_2O_3	123 \pm 3	626 \pm 18	31 \pm 5	25 \pm 2	0.25 \pm 0.01
1.75 wt% Cr_2O_3	118 \pm 32	555 \pm 132	28 \pm 6	25 \pm 3	0.25 \pm 0.03

Table of mechanical properties obtained from the tensile curves. The industry Alloy 718 data is from literature^[3]. The addition of oxide particles decreases the modulus and yield strength of Alloy 718 but has no significant effect on ductility.



The fracture surface of the 0.5 wt% Cr_2O_3 -Alloy 718 sample displays characteristic signs of ductile fracture.

Microhardness

Sample	Average Hardness (HV)
Control	366 \pm 16
0.5 wt% Cr_2O_3	370 \pm 11
1.75 wt% Cr_2O_3	365 \pm 13

Comparison of the average Vickers hardness values in the xy plane as oxide concentration increases.

No conclusion can be drawn from the hardness differences between samples because these differences did not prove to be statistically significant.

Conclusions

- It is possible to produce crack-free oxide dispersion strengthened Alloy 718 parts by direct metal laser sintering.
- The addition of chrome oxide particles does not increase the mechanical properties of Alloy 718 as expected.
- An increase in the oxide particle concentration seems to indicate an increase in porosity.

Recommendations

- Investigate the effects of varying printing parameters on the microstructure and mechanical properties.
- Utilize oxide particles with a chemical composition different from that of the matrix to aid in their detection and identification.

References

- [1] C.Y. Yap, et al., "Review of selective laser melting: Materials and applications", *Applied Physics Reviews*, 2015, **2(4)**, 041101.
- [2] O. Wouters, "Plasticity in Aluminum Alloys at Various Length Scales", 2006, 40.
- [3] EOS NickelAlloy IN718 Material Data Sheet, EOS GmbH - Electro Optical Systems, May 2014.