Characterization of Direct Metal Laser Melted Yttrium Oxide Dispersion Strengthened Ni-Cr Superalloys Parts for Launch System Applications

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Direct Metal Laser Melted Inconel MA 754 was microstructurally and mechanically characterized. EDS and EBSD show the presence of $Cr_{23}C_6$ carbides, a Ni_5Y phase uncharacteristic of MA 754 and preferential grain growth in the build direction. An annealing trial showed progressive recrystallization at a range of temperatures. Creep and tensile trials indicate the material obeys three-phase creep behavior and meets expected performance. Fracture surface examination showed brittle fracture and lack of fusion within the build. Future work includes improving powder development and developing hot isostatic pressing parameters to improve build fusion.

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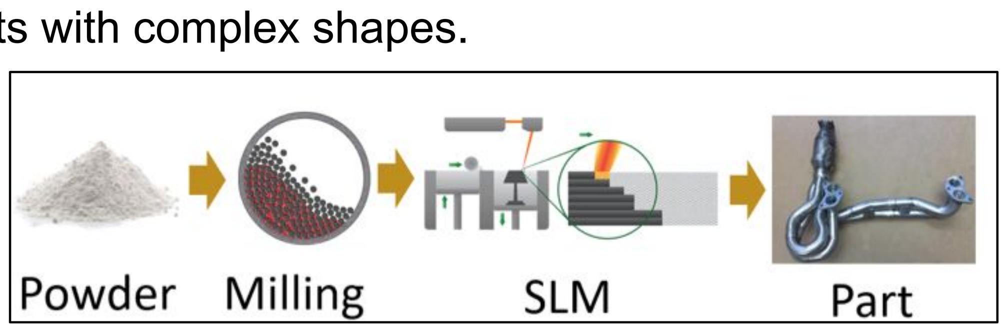
Project Background

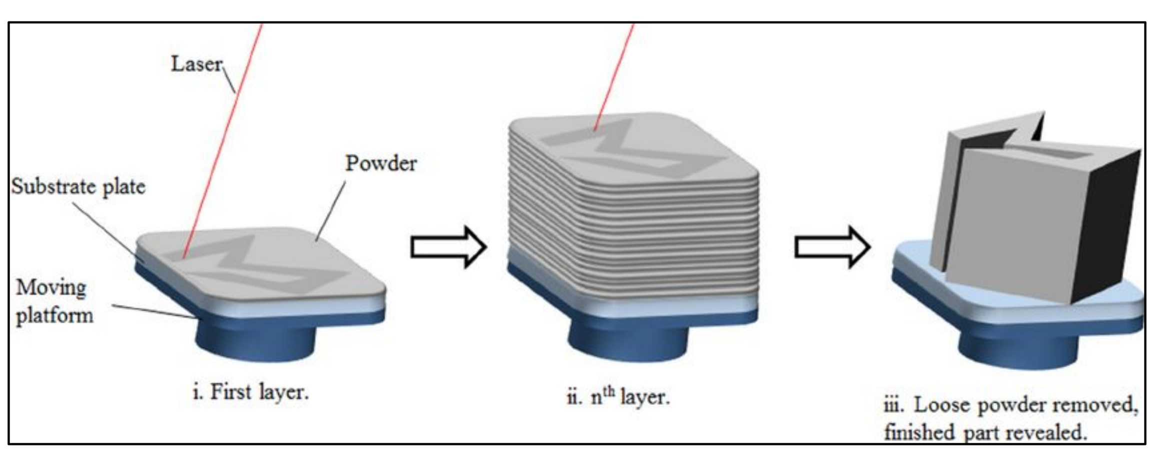
Direct Metal Laser Melting (DMLM) Inconel MA 754 parts are being explored for aerospace applications.

Inconel MA 754 is an Oxide Dispersion Strengthened superalloy known for its exceptional mechanical properties and corrosion resistance at high temperatures due to the presence of Yttria (Y_2O_3).

	Ni	Cr	Fe	C	Al	Ti	Y_2O_3
Wt. %	78	20	1	0.05	0.3	0.5	0.6

DMLM selectively melts powder in layers to create bulk parts with complex shapes.





DMLM parts solidify directionally due to thermal gradients. Annealing can relieve resultant residual stresses.

Experimental Procedure

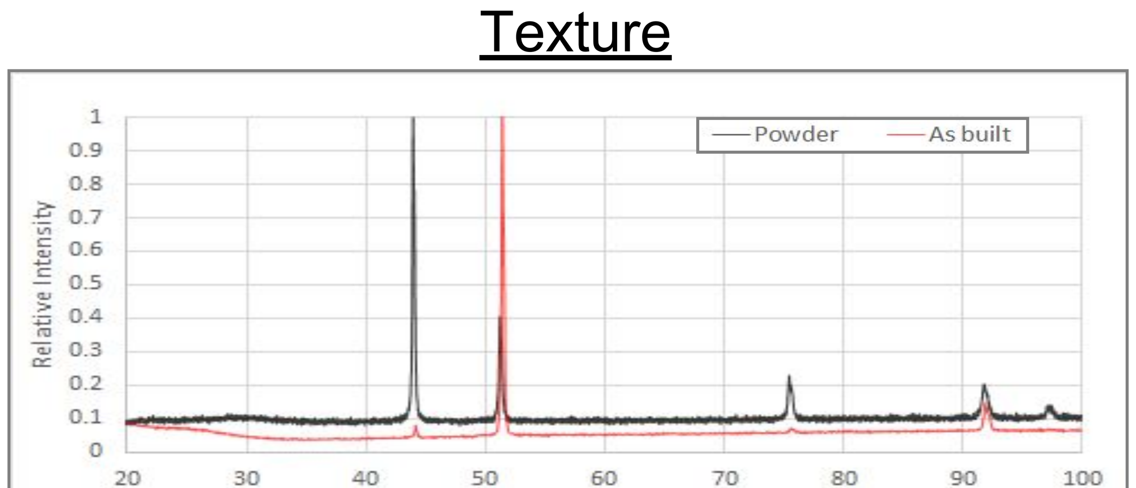
Experiments were designed to characterize the annealing behavior, tensile properties and creep resistance of additively-manufactured (AM) samples. Effectiveness was gauged using hardness and grain size measurements. Microstructure and composition of all samples were characterized using scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). Fractography was conducted using SEM on tensile samples.

Work Distribution

NASA	Purdue
Annealing Trials	Annealing Trials
SEM/EDS/EBSD	SEM/EDS/EBSD
Mechanical Testing	XRD

Results & Discussion

Texture

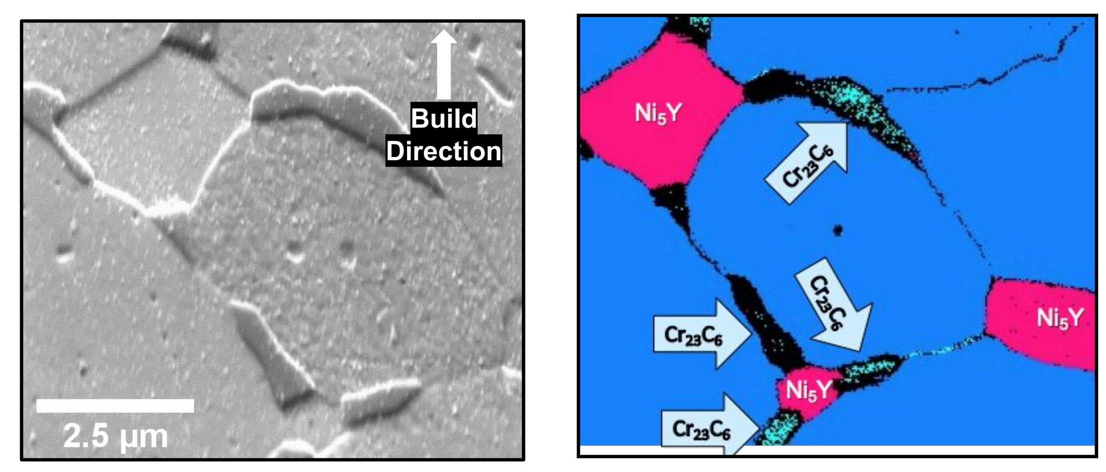


Analyses show higher relative peak intensity of the as-built sample in the (200) plane, indicating a strong crystallographic texture in the observed columnar grains.

Results & Discussion (cont.)

As-Built Composition and Phases

	Ni	Cr	Fe	C	Al	Si	Ti	O	Y
Wt. %	Bal.	20.19	1.23	5.19	0.06	0.64	0	0.29	3.31

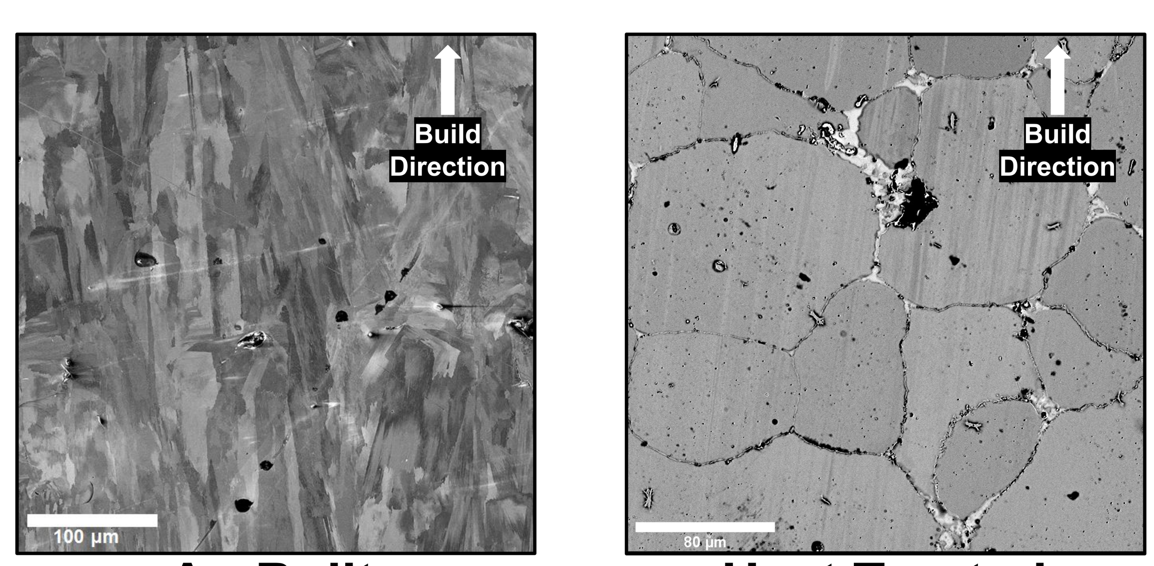


EDS and EBSD indicate the presence of $M_{23}C_6$ carbides, a Ni_5Y phase, and a Ni-Cr matrix. Y and O were detected but not present in fine Y_2O_3 dispersoids. The presence of Si and decreased amount of Ti and Al indicate inadequate processing.

Heat Treatment

Trial	As-Built	1300ºC 1 hr	1300ºC 2 hr	1300ºC 4 hr	1350ºC 1 hr	1350ºC 2 hr	1350ºC 4 hr
Avg. Hardness (HV)	269.4	161.3	160.6	164.4	176.4	160.9	159.3

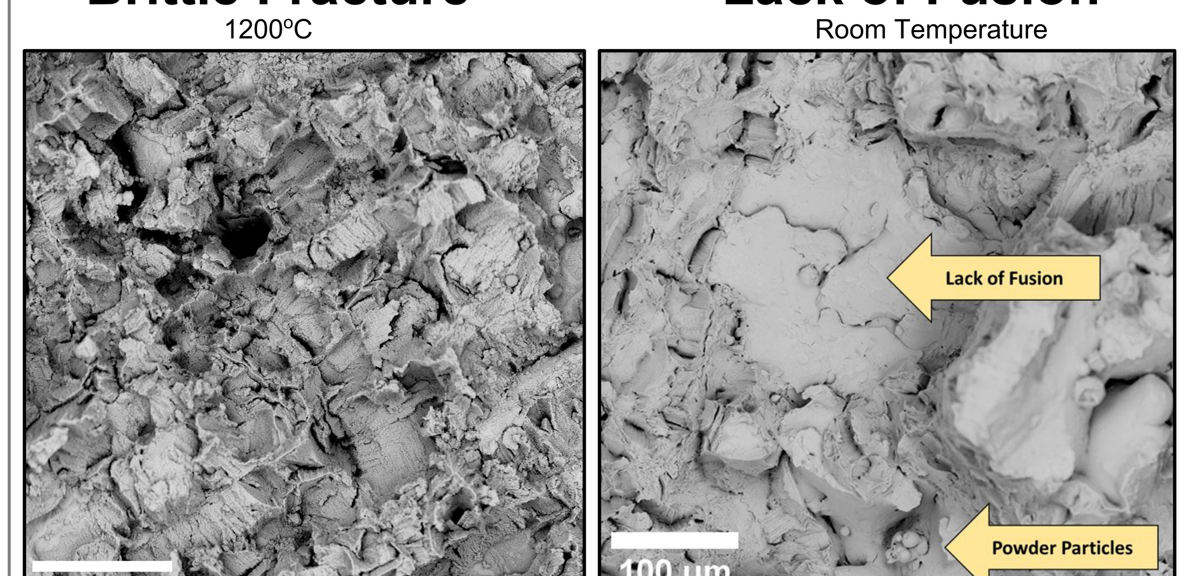
Heat treatment trials resulted in full recrystallization of grains leaving equiaxed annealed structure without an as-built elongated structure. Microhardness measurements show reduction of the residual stresses between the as-built and post-heat treatment samples.



As-Built NASA **Heat Treated** (1350ºC 4hr) Purdue

Fractography

Brittle Fracture 1200ºC **Lack of Fusion** Room Temperature



The fracture surfaces consistently show evidence of brittle fracture, such as planar surfaces. The brittle fracture surfaces examined feature perpendicular cracks, which may be due to the hatching pattern used during printing. The presence of larger smooth surfaces and unmelted powder particles on the fracture surface indicate a lack of fusion in the build, which contribute to the lower than expected mechanical properties.

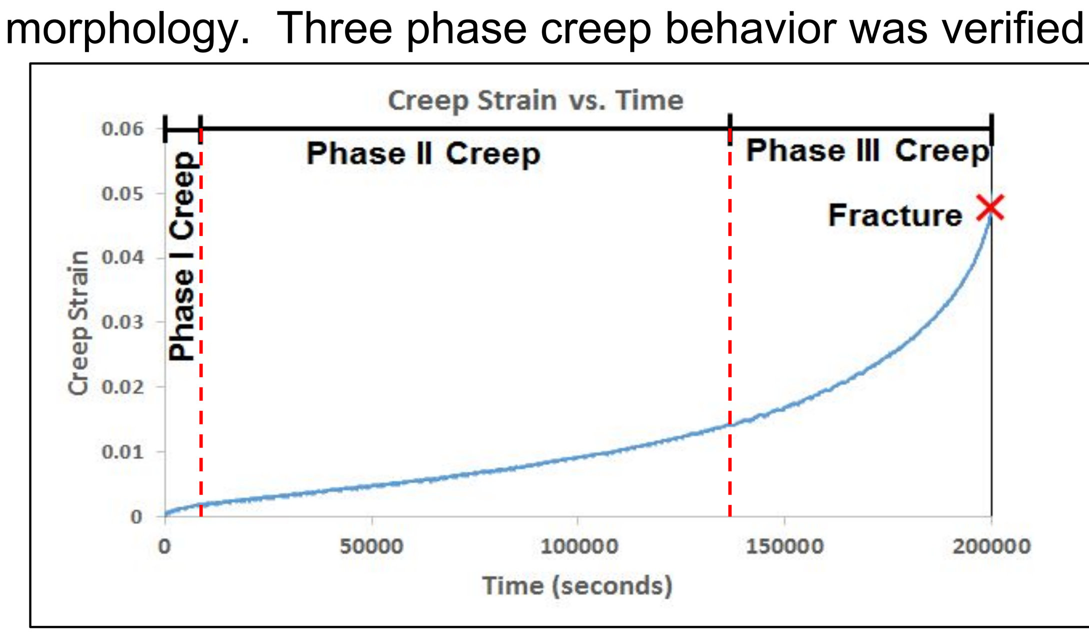
Results & Discussion (cont.)

Mechanical Testing

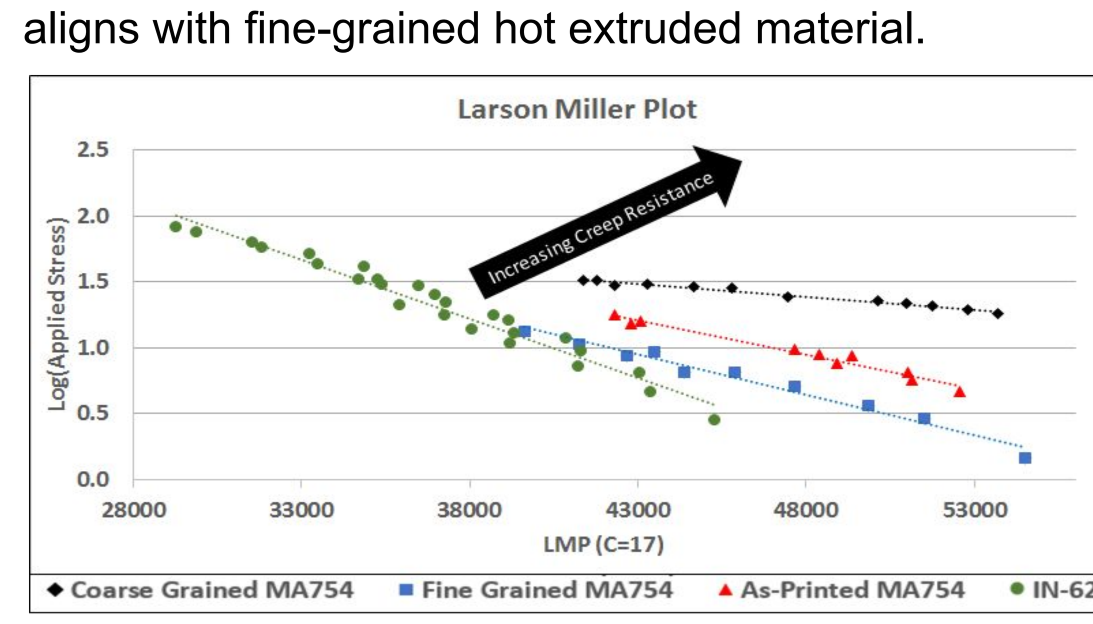
Yield Stress (MPa) | UTS (MPa)

	Room Temp	800ºC	1000ºC
Coarse Grained [1]	600 | 1000	340 | 380	200 | 210
Fine Grained [2]	1175 | 1275	120 | 150	60 | 60
Experimental	572 | 773	208 | 215	102 | 106

Tensile strength of as-built MA 754 aligns with that of hot-extruded material, particularly with fine grain morphology. Three phase creep behavior was verified.



The Larson-Miller plot indicates that AM MA 754 outperforms incumbent IN 625, validating use in aerospace application. Creep behavior generally aligns with fine-grained hot extruded material.



Comparison to literature suggests initial creep testing occurred in low creep exponent region where fracture by cavitation would be expected.

Creep Stress Exponent (n)	800ºC	900ºC	1000ºC	1100ºC
As-Built AM MA754	10.4	N/A	6.2	4.2
Fine Grained [2]	10.0	7.9	3.9	N/A

Conclusions & Future Work

Analysis showed the presence of $M_{23}C_6$ carbides, a Ni_5Y phase, and poorly dispersed Y_2O_3. A combination of lowered hardness values and change in grain morphology indicate recrystallization. As-built tensile fracture surfaces showed brittle fracture, lack of fusion, and presence of unmelted powder. Tensile testing showed lower strength at room temperature (though higher than fine-grained MA 754 at elevated temperature) than expected, indicating printing defects are present but do not affect elevated temperature mechanical properties as much as expected. Creep resistance and as-built texture matched expectations, encouraging that continued development is viable.

Future work includes improving powder processing, namely adjusting the oxidation process to ensure optimal Y_2O_3 content. This analysis encourages future use of hot isostatic pressing of the as-built MA 754 parts to encourage a higher degree of fusion and reduce porosity.

Acknowledgements

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Sources

[1] Totemeier, T., Lillo, T. Effect of Orientation on the Tensile and Creep Properties of Coarse-Grained INCONEL Alloy MA754. *Metallurgical and Materials Transactions.*

[2] Totemeier, T., Lillo, T., Sampson, J.A. Elevated Temperature Strength of Fine-Grained INCONEL Alloy MA754. *Metallurgical and Materials Transactions.*