Dissolving Additive Manufacture Inconel 718 Support Structure Development

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Background

- Process
  - Sensitizing agent applied to AM part.
  - During stress relief heat treatment the sensitizing agent diffuses 100-200 μm into the part altering surface chemical composition.
  - Sensitized region dissolved in an electrochemical process.
  - Supports and surface material <100 μm is removed.

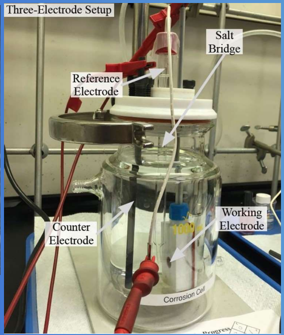
- Objectives
  - Demonstrate viability to dissolve AM IN718 support structures.

- Tasks
  - Print metallographic, tensile, HCF, & surface finish specimens, and full-scale part with inaccessible supports (MSFC).
  - Optimizes IN718 support structure etch process (CSM).
  - Evaluate microstructure, properties, and surface finish (MSFC).
  - Etch stator segment and stator (CSM).

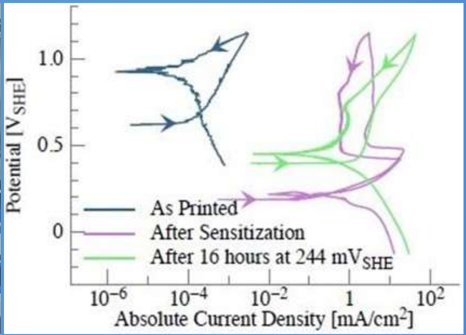
Dissolution Process Development

- Slurry: 3.3 to 1 (by mass) of $Na_4Fe(CN) \cdot 6H_2O$ (99%) & DI water.
- Coating: specimens submerged in slurry for 20 minutes.
- Specimens wrapped in 309 SS and packed with slurry.
- Sensitization heat treatment: 750 °C, 24 hrs (capture free Cr), N_2, water quenched.
- Ultrasonic cleaning: DI water, methanol, acetone, isopropyl alcohol for 10 minutes each.
- Electrolytic solution: HNO_3 (0.48 M, 70 wt%) and KCl (0.1M, 99 wt%) in DI water.
- Electrochemical etch: 16 hrs.

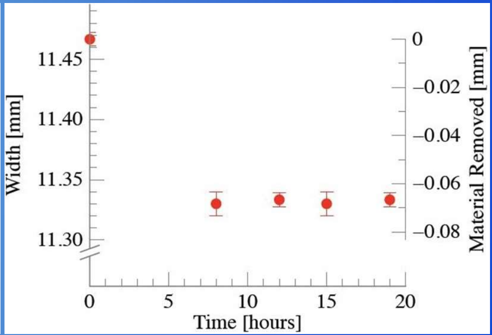
AM IN718 etch calibration disks (Ø16 x 5 mm).



Etch apparatus: IN718 electrode, graphite counter electrode.



Potentiodynamic polarization plots for IN718 disks after sensitization at 750 ˚C for 24 hours



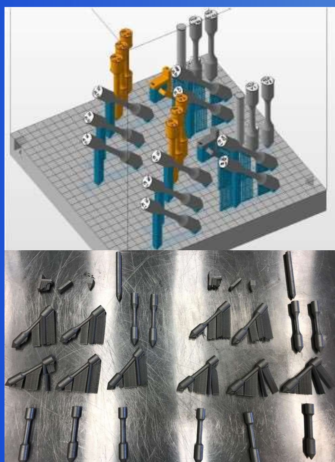
Change in diameter of sensitized AM IN718 during dissolution vs. time. Self-terminating reaction once sensitized region is dissolved.

Mechanical Testing

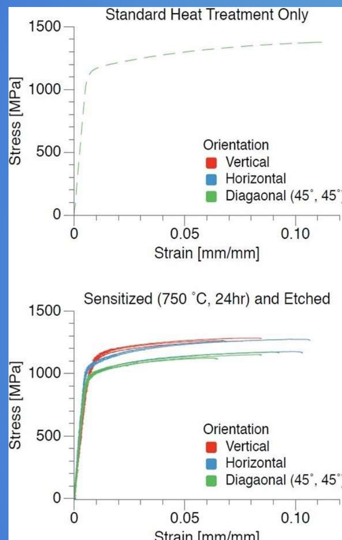
AM IN718 Concept feasibility specimens.

Specimen	Condition	Orientation	YS_{med} (MPa)			UTS_{med} (MPa)			ϵ_{med} (%)			E_{med} (GPa)		
Reference-Z	Control	Vertical	1154.73	±	3.38	1379.3	±	1.17	24.23	±	0.27	184.37	±	5.52
Tensile-Z	Control	Vertical	1144.53	±	8.69	1377.43	±	7.20	23.32	±	0.84	193.88	±	6.67
Tensile-45°	Etch	45°	1176.38	±	15.45	1412.12	±	6.75	19.68	±	0.64	222.01	±	2.02

Feasibility study of AM IN718 control and etch condition tensile property comparison .



EOS M290 build layout and resulting specimens.



Stress-Strain Curves. UTS variation attributed to different heat treatments.

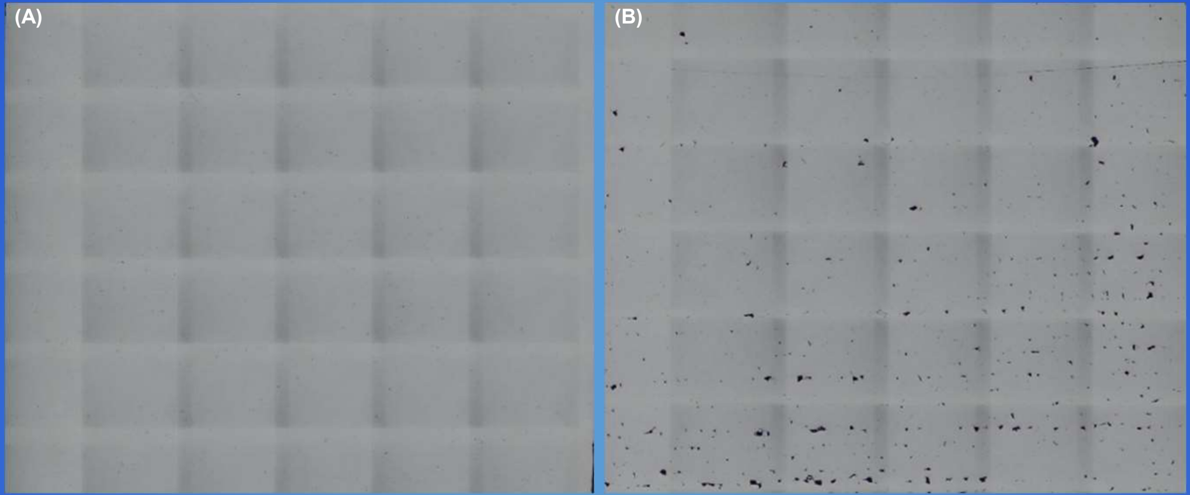
Name	Condition	Orientation	YS_{med} (MPa)			UTS_{med} (MPa)			ϵ_{med} (%)			E_{med} (GPa)		
Reference	Normal	Vertical	1154.73	±	3.38	1379.3	±	1.17	24.23	±	0.27	184.37	±	5.52
TN-V	Normal	Vertical	1169.28	±	7.90	1378.61	±	12.03	10.91	±	2.72	206.77	±	3.88
TN-V-E	Etch	Vertical	1193.34	±	7.22	1426.59	±	98.28	17.39	±	9.89	214.63	±	11.69
TN-45	Normal	45°	1179.83	±	6.14	1330.10	±	73.28	5.22	±	4.99	216.39	±	2.49
TN-45-E	Etch	45°	1199.96	±	9.76	1368.40	±	86.07	3.26	±	2.56	219.25	±	3.78

Expanded AM IN718 control and etch condition tensile property comparison.

Name	Condition	Orientation	σ_{max} (MPa)	Cycles (#)
HCF-V-1	Normal	Vertical	517.11	140,190
HCF-V-2	Normal	Vertical	517.11	150,965
HCF-V-E-1	Etch	Vertical	517.11	157,524
HCF-V-E-2	Etch	Vertical	517.11	214,814
HCF-45-1	Normal	45°	517.11	97,594
HCF-45-2	Normal	45°	517.11	124,262
HCF-45-E-1	Etch	45°	517.11	132,878
HCF-45-E-2	Etch	45°	517.11	129,872

High cycle fatigue (HCF) comparison of control & etch conditions.

Microstructure



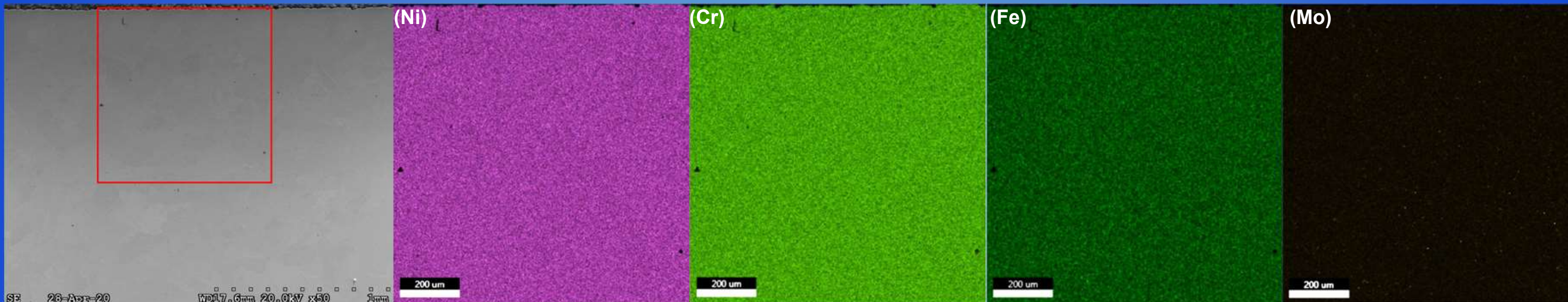
SEM micrographs of IN718 as-built (A) and etched condition (100x).

- As-built condition show minimal porosity and dendritic grains typical for AM.
- Etch condition shows higher porosity, which is unexpected since HIP decreases porosity, and the evaluation region was not exposed to sensitization/etchant chemicals.
- Etched mechanical properties do not decrease relative to control data.
- Porosity variation still under investigation.

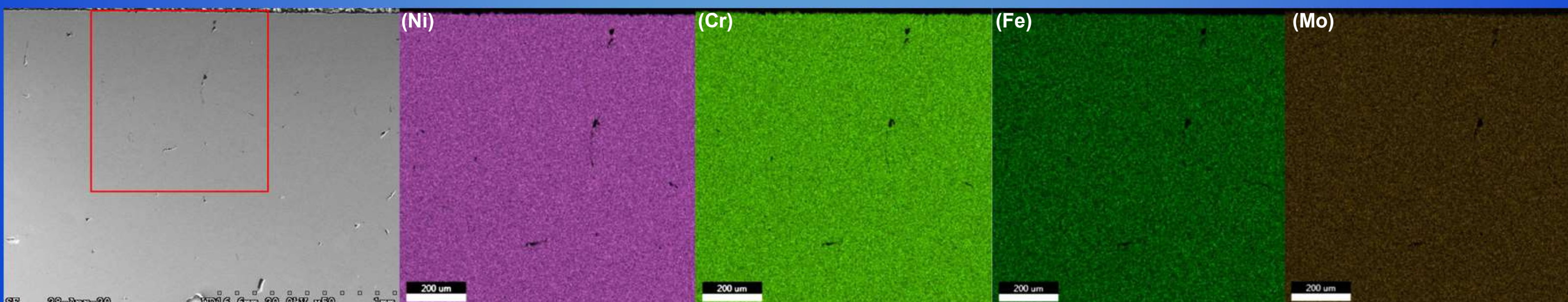
Microstructure

Generalized IN718 composition

Elemental Composition (wt%)			
Ni	Cr	Fe	Mo
Bal	20.5-23	17-20	8-10



SEM micrograph and EDS maps of IN718 As-built (50x).



SEM micrograph and EDS maps of IN718, stress-relived, HIP'd, sensitized at 750 °C for 24 hours, and etched (50x).

Microstructure

Generalized IN718 composition

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Ni	Cr	Fe	Mo
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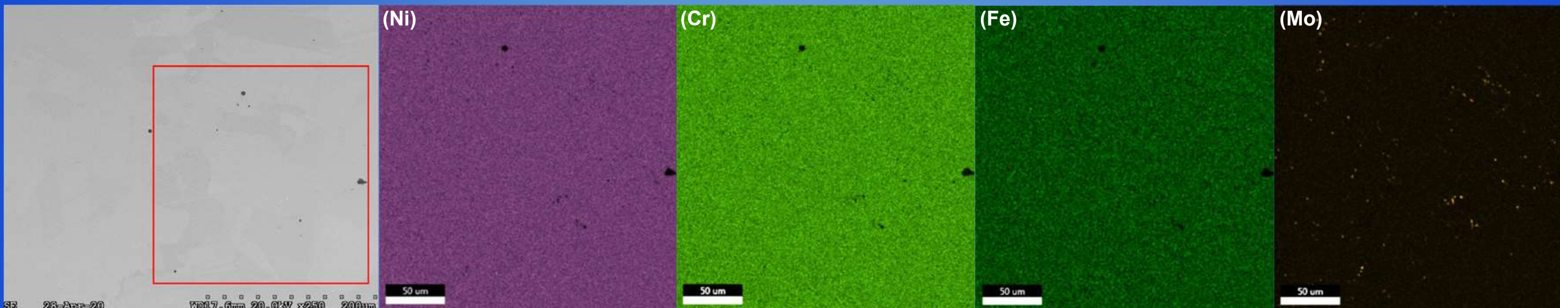
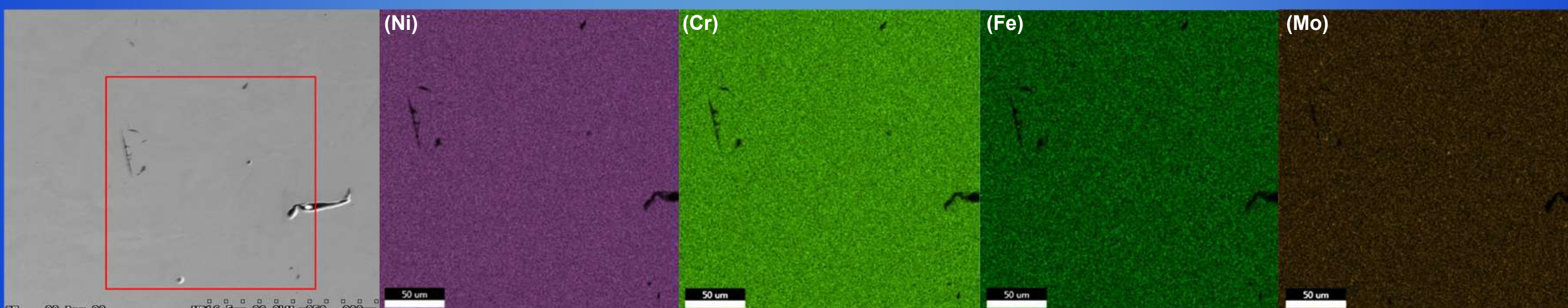


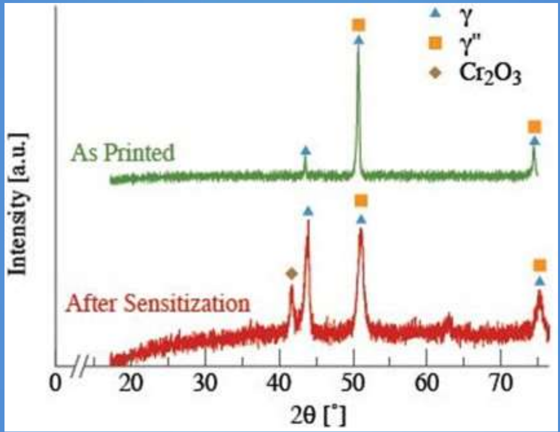
SEM micrograph and EDS maps of IN718 As-built (250x).



SEM micrograph and EDS maps of IN718, stress-relived, HIP'd, sensitized at 750 °C for 24 hours, and etched (250x).

Microstructure

- X-ray diffraction (XRD) to investigate potential phase change evolution vs. condition.
  - Fully heat treated condition: stress relief, HIP, solution, age.
  - Dissolution condition: stress relief, HIP, sensitization, etch, solution, age.
- γ Ni-Cr solid-solution matrix phase (FCC) and γ'' Ni_3Nb precipitate phase (BCT) peaks evident.
- γ and γ'' phases are typical for IN718 in the fully heat treated condition.
- No deleterious δ, carbide or laves phase peaks evident.
- Phase type, size, & distribution responsible for IN718 mechanical properties (precipitation strengthened).

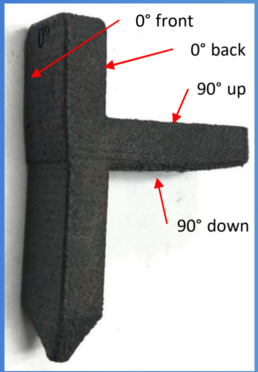


IN718 XRD diffractograms.

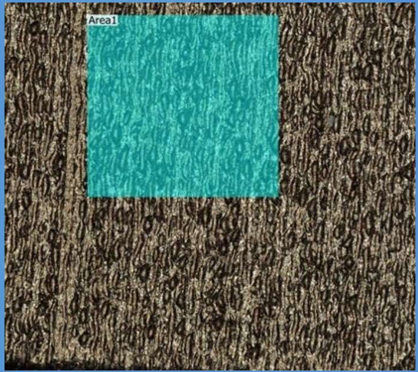
Surface Roughness (Sa)

- Sensitization Heat Treatment: 750 °C for 24 hours.
- Etch time: 47 hours.
- Material removed: approximately 50 µm.
- As-built and etched Sa vs. angle measured using a Keyence VR-3200.

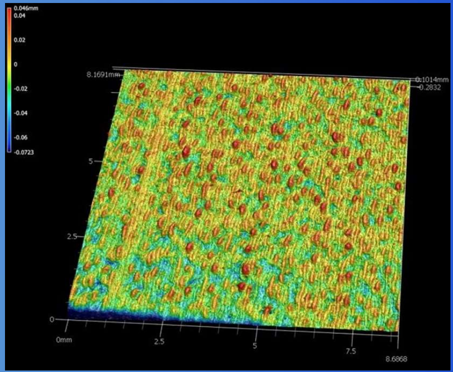
Sa specimen (pre-etch).



Sa specimen (post-etch).



Pre-etch optical image (80x). Courtesy Rengifo (EM22).

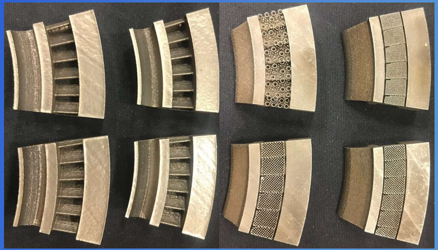


Scale limited image (80x). Courtesy Rengifo (EM22).

Condition	S_a (µm)					
	0°		45°		90°	
	Front	Back	Up-Facing	Down-Facing	Up-Facing	Down-Facing
As-built	12.5	11.1	20.4	41.3	21.1	44.6
Etched	13.7	12	-	-	14.6	40.12

Surface roughness vs. etch condition. Courtesy Rengifo (EM22).

Full Size Component Demonstration

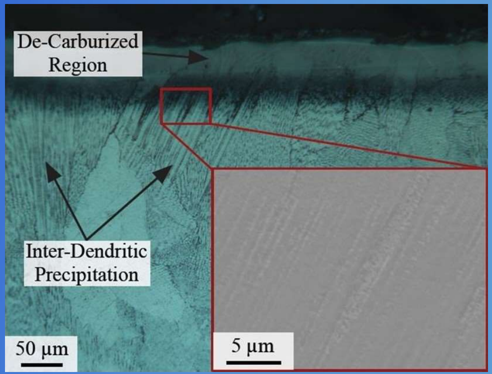


AMDE TOEP IN718 stator segments (pre-etch).

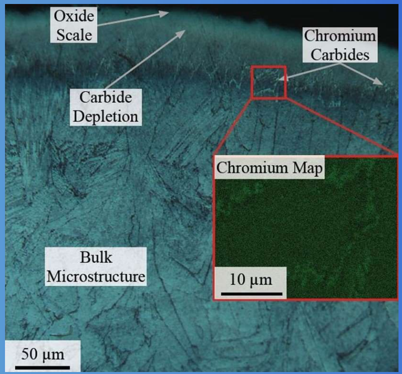
IN718 stator segments pre-etch (left) and post-etch (right). Supports were not fully dissolved but sufficiently weakened to remove via grit blasting.

AMDE OTP IN718 stator - supported blades.

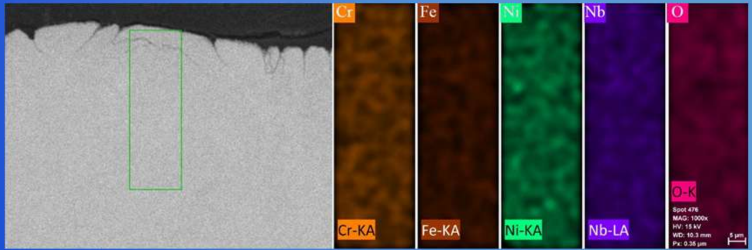
Microstructure



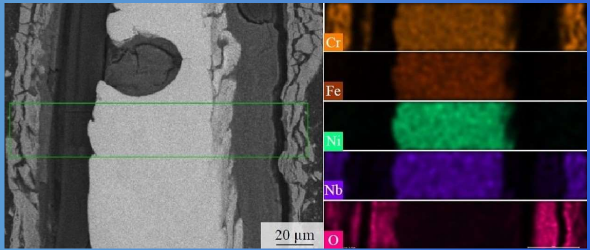
Optical micrograph of IN718 sensitized at 750 °C for 24 hr then etched (20x).



Optical (20x) with EDS map: oxide formation near surface and carbide precipitation dominates 20-70 µm from the surface.



EDS map - Stator surface post-sensitization



EDS map - stator support post-sensitization

Conclusions

- Conclusions
  - Electrochemical dissolution of IN718 supports shown feasible with optimization initiated.
  - Support structure etching should be limited as a measure of last resort due to increased cost and schedule implications.
  - Appropriate applications include components with geometry that cannot be optimized for AM.
  - Geometry examples: aerodynamic surfaces (fins and blades), thin features, and inaccessible passages where supports cannot be mechanically removed.

- Recommendations for future work
  - Higher fidelity characterization of AM IN718 mechanical (particularly HCF), microstructural, and surface finish as a function of etch condition should be conducted.
  - Formulations for support structure etching of standard and emerging AM allows should be developed and optimized for broad-scale industrial adoption.

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