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RPPR Final Report

as of 29-Dec-2021

Agency Code: 21XD

Proposal Number: 75634MSST2

Agreement Number: W911NF-20-C-0012

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Report Date: 22-Dec-2021

Date Received: 23-Dec-2021

Final Report for Period Beginning 21-Nov-2019 and Ending 22-Nov-2021

Title: Additive Manufacturing Feedstock Designed for Uniform Printing of Metallic Builds

Begin Performance Period: 21-Nov-2019

End Performance Period: 22-Nov-2021

Report Term: 0-Other

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Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees:

STEM Participants:

Major Goals: 1. Test components should be fabricated and characterized for both microstructural and mechanical properties, with adjustments to the composition and processing procedure being made as necessary

2. Twelve tensile specimens consistent with ASTM E8/E8M should be printed: six in the XY-direction (horizontal) and six in the Z-direction (vertical)

3. Visual, ultrasonic, and radiographic examination should reveal no exterior or interior flaws

4. Samples should have ultimate tensile strength (UTS) measurements with a coefficient of variation of < 3.5 % (parts printed in the same orientation)

5. Samples should have UTS measurements above 90% of commercial alloys of comparable composition

6. Samples printed in different orientations should show no statistically significant difference (to the 95% confidence level) in UTS

7. Twelve additional tensile samples should be printed on a separate machine

8. Samples printed on a separate machine should be characterized for any statistical differences in results between specimens printed on the previous machine

Accomplishments: Requirement 1 – MET: Dense, crack-free test components with equiaxed microstructures were successfully printed from the developed 3XX-TiAM stainless steel and A5083-RAM7 material feedstocks. Large numbers of compositions were investigated computationally with 19 stainless steel compositions printed for further characterization and down selection. Similarly, large numbers of aluminum compositions were computationally investigated with 5 alloys experimentally printed for further characterization and down selection. These experimental alloys as well as the final selections were characterized for microstructure and mechanical properties formulations. For stainless steel, several alloy formulations were tested to arrive at an optimal Ti content of 0.5-0.8% in the atomized feedstock. Additionally, oxide, fluorine, and amine coatings were applied to 316L powders using atomic layer deposition (ALD) to test their ability to confer resistance to clumping after humidification, but coated materials were observed to be no more free-flowing than the uncoated material. Furthermore, uncoated 316L had marginally higher yield strength (YS), ultimate tensile strength (UTS), and elongation at break than coated 316L.

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Requirement 2 – MET: For both 3XX-TiAM and A5083-RAM7, twelve or more tensile specimens were printed in an EOS M290 machine; six or more were printed horizontally and six or more were printed vertically in each case.

Requirement 3 – MET: Tensile specimens were machined to ASTM E8/E8M standards prior to testing and visually inspected for exterior flaws, which were not apparent. The specimens were evaluated for internal flaws and each found to be greater than 99.6% dense with no defects observed greater than about 20 μm in the 3XX-TiAM and none greater than about 30 μm in the A5083-RAM7 alloys.

Requirement 4 – MET: UTS measurements had coefficients of variation (CV) much less than 3.5% for both materials and in both print orientations (XY vs. Z-direction). The range of CV values was 0.33-0.94% for 3XX-TiAM and 0.7-1.8% for A5083-RAM7.

Requirement 5 – MET: UTS measurements for all printed 3XX-TiAM and A5083-RAM7 were well above 90% of the values listed for wrought 316L and A5083. 3XX-TiAM showed UTS 123% that of wrought 316L and A5083-RAM7 printed samples had UTS 157% that of strain hardened wrought 5083.

Requirement 6 – MET: UTS measurements for printed A5083-RAM7 showed no statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens for the specified sample size of 6 specimens per orientation thus meeting this objective.

UTS measurements for printed 3XX-TiAM did show a statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens however, this is due to the high repeatability and thus low variability in the tensile measurements for each orientation with CVs less than 1%. Importantly, the ratio of Z- to XY-direction UTS values is up to 97.3%, whereas data sheets for common 316L AM feedstocks give ratios of 91.5% (EOS) and 81-86% (Carpenter). Therefore, while statistically speaking the XY- and Z-direction UTS values do show a statistical difference, practically, for engineering purposes they are very close and show a much smaller difference than for commercial AM 316L feedstocks for EOS and Carpenter.

Requirement 7 – MET: Additional specimens were printed on two additional printers (Printer 2, an additional EOS M290 printer housed at Elementum 3D and Printer 3, an EOS M280 housed at Georgia Tech) for the 3XX-TiAM alloy. A second printer was also used for the A5083-RAM7 alloy.

Requirement 8 – MET: Additional specimens printed on additional printers using identical print parameters show the same mechanical properties trends for both 3XX-TiAM and A5083-RAM7 as those printed on Printer 1 (M290), with low variability, high UTS exceeding wrought and datasheet values for commercial AM materials, and higher isotropy than commercial AM materials.

Training Opportunities: Nothing to Report

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Results Dissemination: Advised Secretary of Commerce Gina Raimondo of the program and the results, including putting built parts in her hands from the new steel material, during her December 13, 2021 visit through the lab.

The Elementum 3D team presented the work on Phase II for the 3XX-TiAM to the Colorado School of Mines ADAPT 06/10/2021. It expanded on the partner ship and progress made to the Army STTR Phase II.

The work was discussed/presented at a Penn State "Additive Around the U.S." seminar on September 7, 2021

The work was presented to the Army Research Lab during a GT-ARL virtual materials and manufacturing summit on May 25, 2021

The work was presented to the SHAP3D IUCRC in a seminar called "Machine Learning in Metals Additive Manufacturing" on April 26, 2021

Findings were presented at TMS 2020

The first results on the CALPHAD methodology that were supported by this STTR were used to motivate follow-on funding from NSF Designing Materials to Revolutionize and Engineer our Future (DMREF) through the "GOALI: Physics-Informed Artificial Intelligence for Parallel Design of Metal Matrix Composites and their Additive Manufacturing", which was awarded September 2021. E3D is the GOALI Partner to GT, Utah, and University of Michigan, and the program is in collaboration with AFRL researchers as well.

Peer reviewed publications are in preparation and still expected in 2022.

Abstracts have been accepted to present the work at TMS 2022.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

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Person Months Worked: 8.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co-Investigator

Participant: Benjamin S Rafferty

Person Months Worked: 15.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co-Investigator

Participant: Branden Kappes

Person Months Worked: 15.00

Project Contribution:

National Academy Member: N

Funding Support:

RPPR Final Report
as of 29-Dec-2021

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Person Months Worked: 1.00
Project Contribution:
National Academy Member: N
Funding Support:

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Project Contribution:
National Academy Member: N
Funding Support:

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Funding Support:

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National Academy Member: N
Funding Support:

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Project Contribution:
National Academy Member: N
Funding Support:

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National Academy Member: N

Funding Support:

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Project Contribution:
National Academy Member: N

Funding Support:

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Project Contribution:
National Academy Member: N

Funding Support:

Participant Type: Technician
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National Academy Member: N

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Participant Type: Technician
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Project Contribution:
National Academy Member: N

Funding Support:

Participant Type: Other Professional
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Person Months Worked: 3.00
Project Contribution:
National Academy Member: N

Funding Support:

RPPR Final Report

as of 29-Dec-2021

Partners

Arizona State University
Tempe, AZ ALB

4

ASU provided knowledge into the stainless steel development for the second 12 months of Phase II.

Colorado School of Mines
Golden, CO USA

4

University partner for the first 12 months of the Phase II.

Kappes Materials Manufacturing Data
Castle Rock, CO USA

4

Kappes Materials Manufacturing Data was used as the leading integrated computational material engineering (ICME) consultant for 3XX-TiAM and A5083-RAM7 alloys.

Georgia Institute of Technology
Atlanta, GA USA

4

Georgia Institute of Technology was the primary university partner for the second 12 months of Phase II.

I certify that the information in the report is complete and accurate:

Signature: Benjamin S Rafferty

Signature Date: 12/23/21 1:56AM

Elementum 3D, Inc.

Contract #: W911NF20C0012

Design of Easy-to-Use Structural Alloy Feedstocks for Additive Manufacturing Topic: A18B-T003

Contract and Proposal Number:

W911NF20C0012 / A2-7889

Contractor's name and address:

Sinter Print, Inc. d.b.a. Elementum 3D, Inc., 400 Young Ct. Unit 1, Erie CO 80516

Title of the project:

Design of Easy-to-Use Structural Alloy Feedstocks for Additive Manufacturing

Contract performance period:

November 21, 2019– November 20, 2021

Total contract amount: \$999,983.18

Amount of funds paid by DFAS: \$999,983.18

Total amount expended/Invoiced to date: \$999,983.18

Number of employees working on the project: 8

Number of new employees placed on contract this month: 0

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1 Executive Summary

Elementum 3D (E3D), Georgia Institute of Technology (GT), Arizona State University (ASU), and Kappes Materials Manufacturing & Data (KMMD) have been working together on an Army Research Office (ARO) STTR to develop and utilize a computational and experimental research framework to create 'easy-to-print' iron and aluminum-based alloys with highly reliable properties. The alloys are also designed for minimal post-processing requirements and high environmental resiliency. The Phase II efforts build upon the research and integrated computational materials engineering (ICME) framework established in Phase I to identify favorable compositions for highly printable materials with functional and predictable properties. Phase I used calculated phase diagram (CALPHAD) and solidification prediction techniques to computationally investigate compositions centered around the 316L alloy range, while Phase II expands on these techniques coupled with experimental investigations to complete development of a highly reliable stainless-steel alloy as well as development of an aluminum alloy system based on a wider computational investigation. The team used theoretical and computational methods to establish candidate alloys and used laser powder bed fusion (LPBF) additive manufacturing (AM) to experimentally verify the printability and performance of these materials to fulfill the project goals.

During the execution of this Phase II STTR project, the support of the Army Research Office (ARO) enabled the team to develop, print, and test two new easy-to-use and highly reliable AM feedstock materials. An improved austenitic stainless steel alloy was chosen as the first target alloy due to a combination of useful characteristics including a primarily single phase (fcc) crystal structure, high fracture toughness, corrosion resistance, and good as-printed properties including strength without the requirement for post-build heat treatment. The computational work investigated the alloy space centered around the 316L specification. When printed by LPBF, the standard 316L composition, like most existing alloys, suffers from anisotropic mechanical properties with respect to part build orientation. A major goal of the project was to bring more isotropic materials with highly reliable properties to market.

With the help of the ICME framework, the team hypothesized that improvements to printability could be made by reducing the mushy zone temperature range, reducing the delta ferrite (BCC phase) formation, and avoiding slag formation. Integrated computational materials engineering (ICME) simulations designed 12 test compositions for 300-series stainless steels to experimentally validate the modeling and assumptions. The 12 compositions were selected to include specimens designed to accentuate aspects predicted to be detrimental as well as specimens designed to minimize these aspects. The printed experimental compositions showed that an increased mushy zone temperature range lead to the formation of microcracks and reduced fatigue life and slag forming compositions lead to increased defects compared to the baseline material. Based on the model validation results, a preferred composition was developed with controlled elemental ranges and the addition of titanium to the alloy. The titanium addition was designed to bind with and control interstitial carbon, nitrogen, and oxygen and form heterogeneous nucleation sites to produce highly consistent properties. The new stainless steel alloy (3XX-TiAM) was found to have a more equiaxed and finer grain structure as well as more consistent properties compared to unmodified 316L. The 3XX-TiAM had an ultimate tensile strength (UTS) 5% greater than wrought 316L and comparable YS and elongation. The UTS results showed coefficients of variation (CV) of less than 1% for each orientation for each of three printers tested. The 3XX-TiAM properties were also more isotropic between the horizontal (parallel to the build plate) and vertical (perpendicular to the build plate) directions, with samples demonstrating a vertical UTS within 95% of the horizontal UTS compared to the 316L base alloy having only 81-91% of the

Design of Easy-to-Use Structural Alloy Feedstocks for Additive Manufacturing Topic: A18B-T003

properties between orientations. The 3XX-TiAl also demonstrated greater corrosion resistance than standard wrought 316L.

The next alloy target was development of an aluminum alloy with highly-reliable high-strength properties without the need for post process heat treatment such as aluminum alloy 5083 which is used extensively in the military in applications ranging from armor to fuel storage. High strength wrought alloys fitting these criteria, such as aluminum alloy 5083, suffer from severe solidification cracking (hot tearing) during fusion-based AM processes. ICME calculations evaluated the effects of alloy composition centered around aluminum alloy 5083 to reduce susceptibility to solidification cracking and produce consistent and high tensile strength. The newly developed alloy termed A5083-RAM7 utilized reactive additions to form dispersion strengthening nucleation sites in situ in the melt pool to promote solidification of a fine-grained equiaxed microstructure and solve the solidification cracking problems while also increasing strength. The equiaxed and dispersion strengthened microstructure promotes isotropic properties and the alloy had extremely uniform properties with specimens printed in different orientations showing no statistically significant difference to the 95% confidence level in UTS. The alloy also exhibited CV's of under 2% in UTS each orientation for each of two printers tested. The alloy also showed statistically indistinguishable yield and ultimate strengths as-printed and after an anneal (O condition) heat treatment at 650 °F (343 °C) for 2 hours. Perhaps most impressively, the alloy exhibited as-printed yield strengths approximately 80% greater than strain hardened (H321) wrought 5083 aluminum and 186% greater (nearly triple) that of annealed (O condition) wrought 5083. The ultimate tensile strengths were also extremely impressive with approximately 57% greater strength than strain hardened (H321) wrought 5083 and 71% greater strength than annealed (O condition) 5083. These results represent a true breakthrough; to our knowledge, no previous study has demonstrated successful, crack-free LPBF printing of Al5083. [1] [2] [3] The newly developed alloy is also believed to have demonstrated the highest as-printed strength (no heat treatment) of any LPBF produced aluminum alloy in addition to demonstrating extremely consistent properties that met all project objectives.

Our newly designed stainless steel and aluminum alloys with increased isotropy and printability represent a breakthrough in AM alloy design that can be exploited in robust parts manufacturing systems to shorten lead times, enable improved performance, and reduce total lifecycle costs. These improvements will increase readiness, benefit the individual warfighter, and help modernize the US Army logistics and supply chain.

2 Project Objectives & Deliverables Summary

The main objective of this Phase II effort was to develop two easy-to-use, highly printable powder feedstocks – one based on 316L stainless steel, and the other from an aluminum alloy – for L-PBF additive manufacturing (AM). These feedstock materials should be processed into components with crack-free, equiaxed microstructure and isotropic mechanical properties. According to the program solicitation, the following specific requirements were to be met for the newly developed alloys. The relevant achieved results are described with further details on the achievements presented in the Technical Results section of this report.

Requirement	Met?
1. Test components should be fabricated and characterized for both microstructural and mechanical properties, with adjustments to the composition and processing procedure being made as necessary	Yes
2. Twelve tensile specimens consistent with ASTM E8/E8M should be printed: six in the XY-direction (horizontal) and six in the Z-direction (vertical)	Yes
3. Visual, ultrasonic, and radiographic examination should reveal no exterior or interior flaws	Yes
4. Samples should have ultimate tensile strength (UTS) measurements with a coefficient of variation of < 3.5 % (parts printed in the same orientation)	Yes
5. Samples should have UTS measurements above 90% of commercial alloys of comparable composition	Yes
6. Samples printed in different orientations should show no statistically significant difference (to the 95% confidence level) in UTS	Yes
7. Twelve additional tensile samples should be printed on a separate machine	Yes
8. Samples printed on a separate machine should be characterized for any statistical differences in results between specimens printed on the previous machine	Yes

Requirement 1 – MET: Dense, crack-free test components with equiaxed microstructures were successfully printed from the developed 3XX-TiAM stainless steel and A5083-RAM7 material feedstocks. Large numbers of compositions were investigated computationally with 19 stainless steel compositions printed for further characterization and down selection. Similarly, large numbers of aluminum compositions were computationally investigated with 5 alloys experimentally printed for further characterization and down selection. These experimental alloys as well as the final selections were characterized for microstructure and mechanical properties formulations. For stainless steel, several alloy formulations were tested to arrive at an optimal Ti content of 0.5-0.8% in the atomized feedstock. Additionally, oxide, fluorine, and amine coatings were applied to 316L powders using atomic layer deposition (ALD) to test their ability to confer resistance to clumping after humidification, but coated materials were observed to be no more free-flowing than the uncoated material. Furthermore, uncoated 316L had marginally higher yield strength (YS), ultimate tensile strength (UTS), and elongation at break than coated 316L.

Design of Easy-to-Use Structural Alloy Feedstocks for Additive Manufacturing Topic: A18B-T003

Requirement 2 – MET: For both 3XX-TiAM and A5083-RAM7, twelve or more tensile specimens were printed in an EOS M290 machine; six or more were printed horizontally and six or more were printed vertically in each case.

Requirement 3 – MET: Tensile specimens were machined to ASTM E8/E8M standards prior to testing and visually inspected for exterior flaws, which were not apparent. The specimens were evaluated for internal flaws and each found to be greater than 99.6% dense with no defects observed greater than about 20 μm in the 3XX-TiAM and none greater than about 30 μm in the A5083-RAM7 alloys.

Requirement 4 – MET: UTS measurements had coefficients of variation (CV) much less than 3.5% for both materials and in both print orientations (XY vs. Z-direction). The range of CV values was 0.33-0.94% for 3XX-TiAM and 0.7-1.8% for A5083-RAM7.

Requirement 5 – MET: UTS measurements for all printed 3XX-TiAM and A5083-RAM7 were well above 90% of the values listed for wrought 316L and A5083. 3XX-TiAM showed UTS 123% that of wrought 316L and A5083-RAM7 printed samples had UTS 157% that of strain hardened wrought 5083.

Requirement 6 – MET: UTS measurements for printed A5083-RAM7 showed no statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens for the specified sample size of 6 specimens per orientation thus meeting this objective.

UTS measurements for printed 3XX-TiAM did show a statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens however, this is due to the high repeatability and thus low variability in the tensile measurements for each orientation with CVs less than 1%. Importantly, the ratio of Z- to XY-direction UTS values is up to 97.3%, whereas data sheets for common 316L AM feedstocks give ratios of 91.5% (EOS) and 81-86% (Carpenter). Therefore, while statistically speaking the XY- and Z-direction UTS values do show a statistical difference, practically, for engineering purposes they are very close and show a much smaller difference than for commercial AM 316L feedstocks for EOS and Carpenter.

Requirement 7 – MET: Additional specimens were printed on two additional printers (Printer 2, an additional EOS M290 printer housed at Elementum 3D and Printer 3, an EOS M280 housed at Georgia Tech) for the 3XX-TiAM alloy. A second printer was also used for the A5083-RAM7 alloy.

Requirement 8 – MET: Additional specimens printed on additional printers using identical print parameters show the same mechanical properties trends for both 3XX-TiAM and A5083-RAM7 as those printed on Printer 1 (M290), with low variability, high UTS exceeding wrought and datasheet values for commercial AM materials, and higher isotropy than commercial AM materials.

3 Work Summary

	STTR Milestones	Subject	Research
Y E A R 1	Custom Feedstock II selection	Determine Chemistry	Literature/CALPHAD/Machine learning
	Custom Feedstock I selection	Continued development	Finalization of Composition
	CALPHAD Machine Learning Finalization	Computational evaluation	ICME Computational development of the Alloy through Machine learning and CALPHAD

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	Custom Feedstock I Parameter Development	Printing Parameters	Laser Powder Bed Fusion Parameter development
	Custom Feedstock II Parameter Development	Printing Parameters	Laser Powder Bed Fusion Parameter development
	Atomized Custom Powder	Feedstock I	Order from Supplier
		Feedstock II	
Y	Minimum of 12 tensile bars produced/tested	Feedstock I	UTS should have a coefficient of variation 3.5 % or less between parts prints and be within 90% of UTS Commercial alloys
		Feedstock II	
E	Environmental testing	Feedstock I	Feedstock resistance to changing environments
		Feedstock II	
A	Nitrogen printer Environment testing	Feedstock I	Microstructure and Mechanical properties
		Feedstock II	
R	Titanium addition	Feedstock I	Microstructure and Mechanical properties
		Feedstock II	
2	Mechanical Properties	Feedstock I	Minimum 12 Tensile Bars X and Y Direction Final material single powder or feedstock
		Feedstock II	
	Powder Analysis	Feedstock I	SEM/XRD/Particle analysis
		Feedstock II	
	Microstructure Analysis	Feedstock I	Optical/SEM Microscopy Analysis/Chemical Analysis
		Feedstock II	
	Commercialization feasibility	Feedstock I	Supply Chain logistics/research powder suppliers/Feedstock Manufacturing
		Feedstock II	
	Custom Feedstock I Parameter Development	Printing Parameters	Finalization of key process parameters on EOS M290
	Custom Feedstock II Parameter Development	Printing Parameters	Finalization of key process parameters on EOS M290
	Second 3D printer evaluation	Feedstock I	Process and physical properties
		Feedstock II	Statistical property evaluation
	Third 3D printer evaluation	Feedstock I	Process and physical properties
	Phase II Final report		

4 Technical Results

4.1 New Austenitic Stainless Steel Development (3XX-TiAM)

An improved austenitic stainless steel alloy was chosen as the first target alloy due to a combination of useful characteristics including a primarily single phase (fcc) crystal structure, high fracture toughness, corrosion resistance, and good as-printed properties including strength without the requirement for post-build heat treatment. The computational work investigated the alloy space centered around the 316L specification. When printed by LPBF, the standard 316L composition, like most existing alloys, suffers from anisotropic mechanical properties with respect to part build orientation. A major goal of the project was to bring more isotropic materials with highly reliable properties to market.

4.1.1 Integrated Computational Materials Engineering (Stainless Steel Alloy)

CALPHAD methods were used to understand the effects of compositional changes on solidification and phases within the 316L system. The study focused on the calculation of the minimum and maximum width of the mushy zone, amount of low temperature slag phase, and the presence of body centered cubic (BCC) phases in 316L. The simulation was adjusted to explore the full compositional space around the 316L alloy specifications to generate the data needed for application of computational and machine learning analysis techniques. Figure 1 displays an example of the 316L alloy ranges used for these calculations in CALPHAD. The results found that the mushy zone temperature range can vary from a low of only 12°C to a maximum of 107°C within the evaluated composition space.

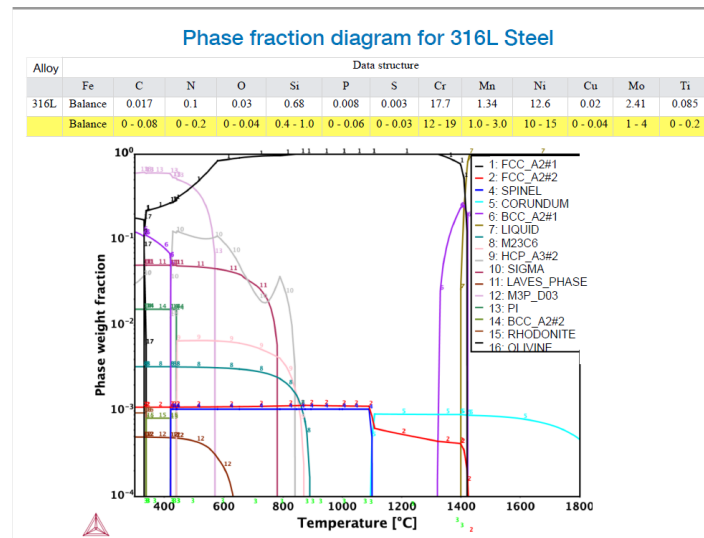


Figure 1: Displays the CALPHAD simulation for the 316L chemistries observed.

Figure 2 displays the low temperature slag phase that appeared during the simulation. It was found that 20% of the data generated during simulation has a low temperature slag phase. This is suspected to be a critical source of hot cracking.

Low Temperature SLAG phase

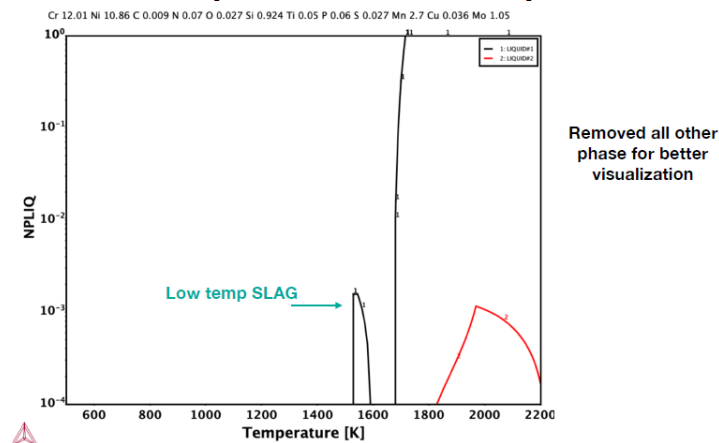


Figure 2: The chart displays the slag phase in the 316L with all the other phase omitted.

Figure 3 shows the area where BCC phases appear in the solidification process for the 316L candidate. The simulation revealed that 48% of the data has a δ -ferrite (2nd-BCC) phase during solidification, which will transform to austenite with a decrease in temperature.

Three types of BCC_A2 phase

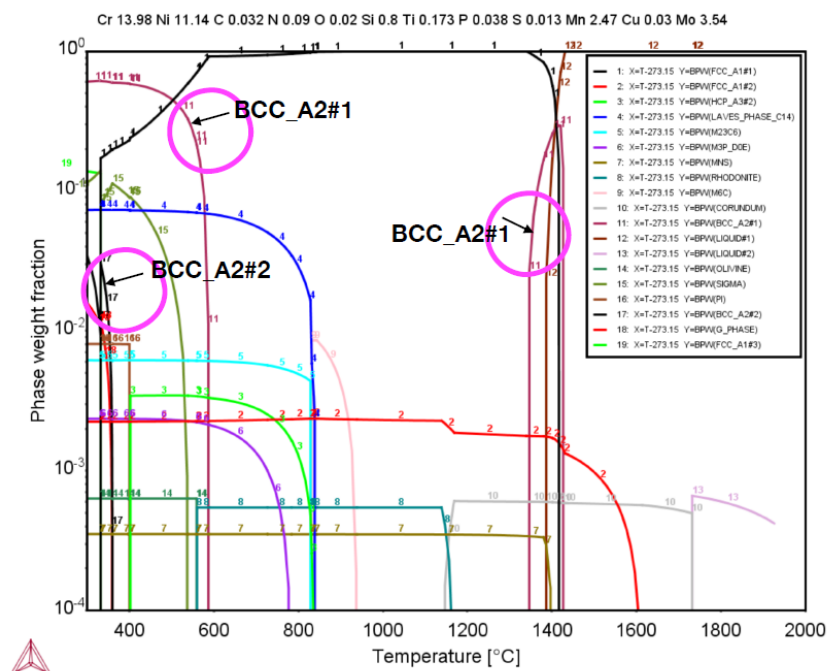


Figure 3: Displays the area of BCC phase in the 316L compositions observed during CALPHAD simulations.

The CALPHAD predictions continued as the team researched titanium's influence on the development of 316L stainless steel microstructures. The titanium is being used as a deoxidizer, to bind soluble oxygen, reducing the formation of a deleterious slag phase that may increase hot cracking.

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Additionally, the resulting oxides and nitrides could serve as nucleation sites during solidification, leading to a more equiaxed, refined microstructure. This would improve printability in nitrogen and increase strength in the 316L-centric system.

The team experimentally observed that titanium alloying additions increase ultimate tensile strength, UTS, by an average of 8 ksi from the baseline 316L. This led to various compositions in the 321 stainless steel range to compare with the current candidates. The 321 stainless steel composition includes titanium, as seen in the ASTM standard chemistry in Table 1.

Table 1: The compositions used for CALPHAD diagrams presented in Figure 1 and Figure 5 for the phase weight fraction highlighting the differences between 321 and 316L compositions.

Alloy Base	Image	Fe	C	Mn	Si	Cr	Ni	P	S	N	O	Cu	Mo	Ti
321	Fig. 1	Bal	0.08	2	1	18	10.5	0.045	0.03	N/A	N/A	N/A	N/A	0.4
316Ti	Fig. 2	Bal	0.018	2.83	0.97	18.12	12.94	0.038	0.005	0.2	0.03	0.03	2.73	0.123

A database was created using the CALPHAD simulations containing more than 12,000 compositions that captured thermodynamically stable liquid and solid phases and their stable temperature ranges between room temperature and 1600 °C. This database was used to train a neural network model, which helped predict correlations between composition and temperature dependent phases and select experimental test alloys for model validation.

The first alloy system selected was based on 316L and was used as a basis for creating suitable chemistries to begin practical test printing. However, because a complex interplay between compositional elements controls the formation or absence of specific phases, the neural network was used to identify compositions within and around the 316L specification unlikely to form the slag phase hypothesized to promote hot cracking.

Based on the data generated by CALPHAD simulations, including the Phase fraction diagrams shown in Figure 4 and Figure 5, the team identified two phases which might be critical for the microstructure and mechanical properties of the printed parts. Phases of interest include a low-temperature slag, composed of liquid oxides of Mn, Si, and Ti, and delta ferrite which can negatively affect the mushy zone of the alloy. One or both of these phases may appear around the solidus temperature and play a significant role on the properties of the alloy.

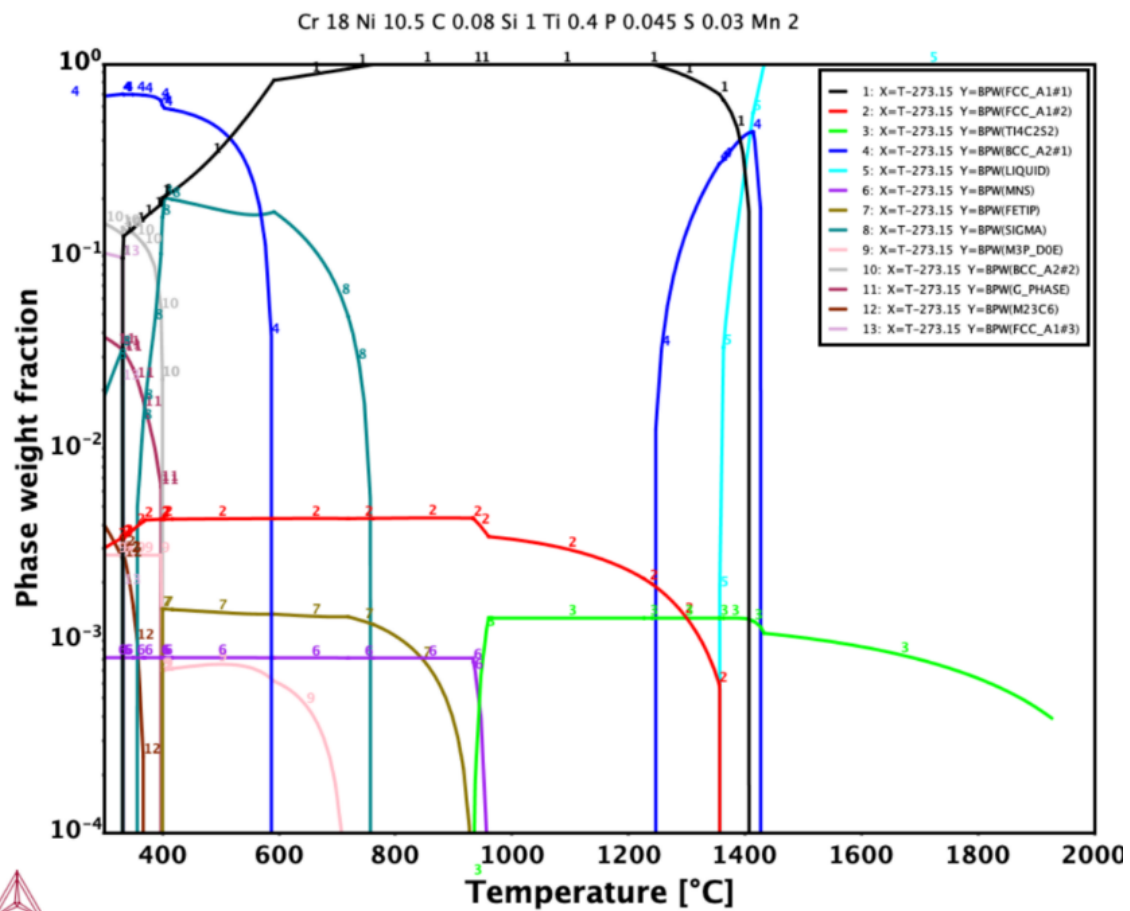


Figure 4: The phase weight fraction for an ASTM standard 321 stainless steel for comparison to a 316L-Ti based alloy.

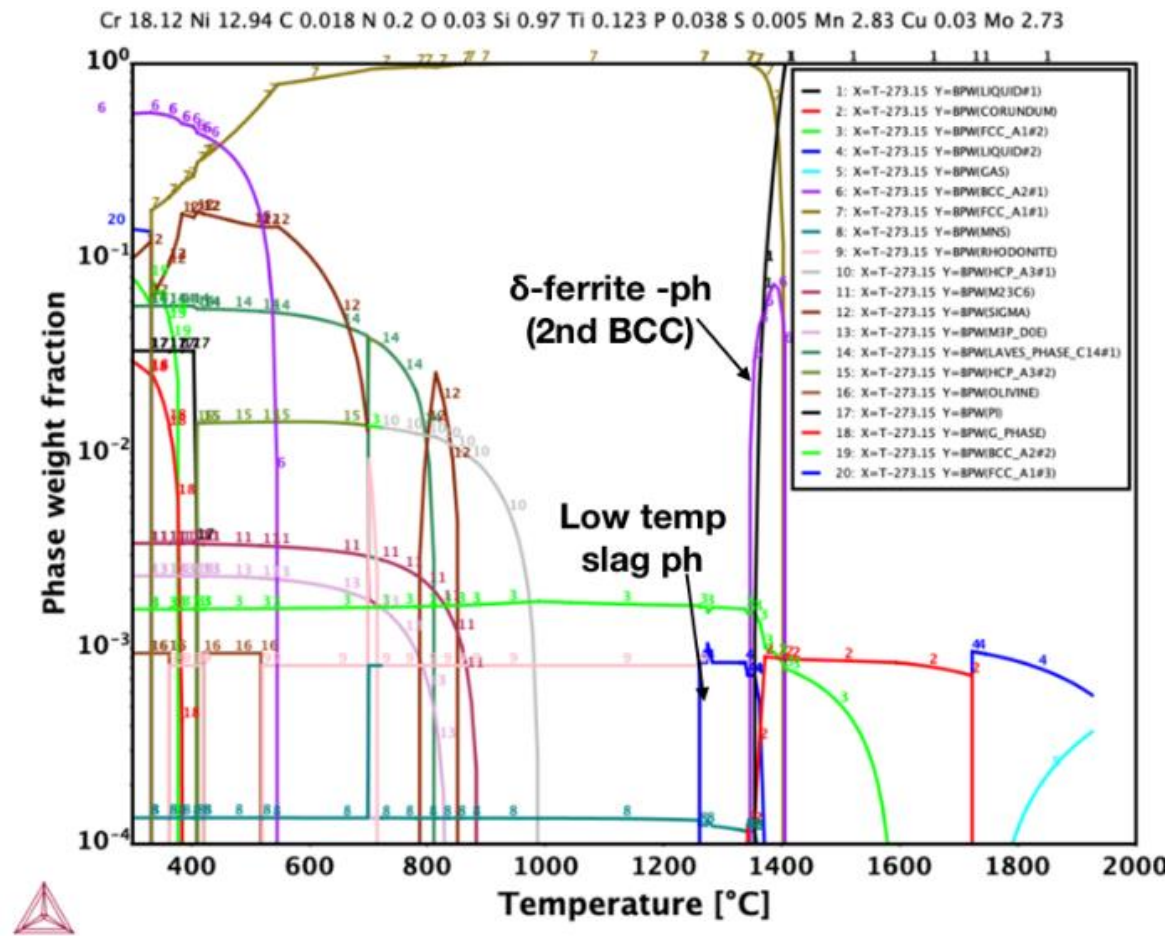


Figure 5 : Phase fraction diagram of a 316-L-Ti based composition showing the presence of both low-temperature slag phase and delta ferrite phase around solidus temperature.

The presence of these phases can negatively impact the properties of the printed material. The low-temperature slag phase would most likely appear at the grain boundaries, leading to hot cracking. The presence of delta ferrite around the solidus temperature would make the alloy solidify into two phases: austenite and delta ferrite. These phases have different coefficients of thermal expansion, and so as the alloy solidifies, would introduce residual stresses into the system. Residual stresses are known to form due to nonequilibrium conditions present during additive manufacturing, but the presence of delta ferrite would make the system more susceptible to residual stress.

These phases are highly dependent on the composition of the alloy, which is confirmed from the distribution of CALPHAD-generated data. The composition space of the 316L steel can be divided into four parts based on the presence of these two phases, as shown in Figure 6

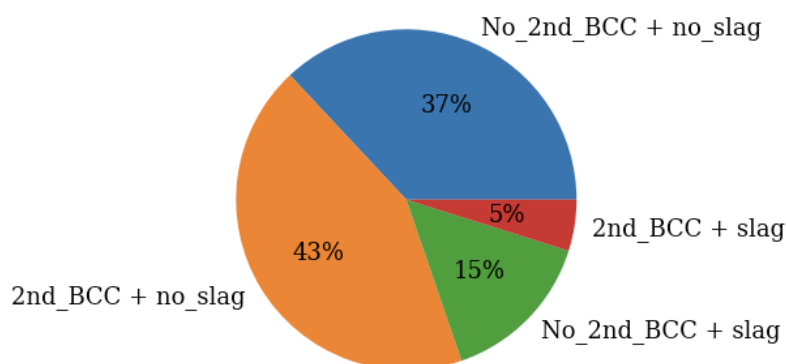


Figure 6: The pie chart displays the distribution of database into four classes based on the presence of delta-ferrite and low temperature slag phase. 2nd BCC is used as the designator for the delta ferrite phase.

Machine learning (ML) algorithms in general, and neural networks in particular, capture correlations between inputs and outputs, but not necessarily causal relationships. Additionally, ML algorithms tend to overfit; that is, they fit well the training data, but do not generalize well. To address each of these, the data used to train an ML algorithm was divided into a test/train set and a validation set. Validation data is set aside as the best approximation to the out-of-sample performance of the trained model. The model was trained and the performance tested repeatedly using the test/train data set. This division ensures the model architecture is complex enough to capture the relationships that exist between inputs and outputs, but does not overfit to the training data.

The dataset of approximately 12,000 compositions was randomly divided into two datasets. 80% of the total dataset was chosen as the test/train dataset, while the remaining 20% was taken as a validation dataset. The 80% of the test/train dataset was used to train the model; 20% used to evaluate model error. Figure 7 shows the division of dataset into training, validation and test sets.

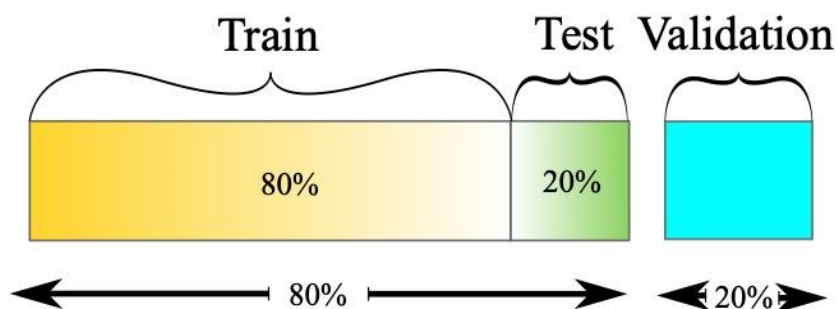


Figure 7. Demonstration of the division of dataset into training, validation, and test sets.

The results in Figure 8 show the training and validation losses for our model over training epochs. (One epoch is one pass through the randomized test/train data set.) One can observe that both training and validation loss is decreasing with the number of epochs, and then become saturated at a larger number of epochs. Around 300 epochs, we found that there is not much of a decrease in the validation losses. The validation accuracy at 300 epochs was found to be around 92.7%. Based on our trained model we evaluated the accuracy of the model on the test set and found that our model predicts the presence of slag and delta ferrite phase with an accuracy of 92.4%.

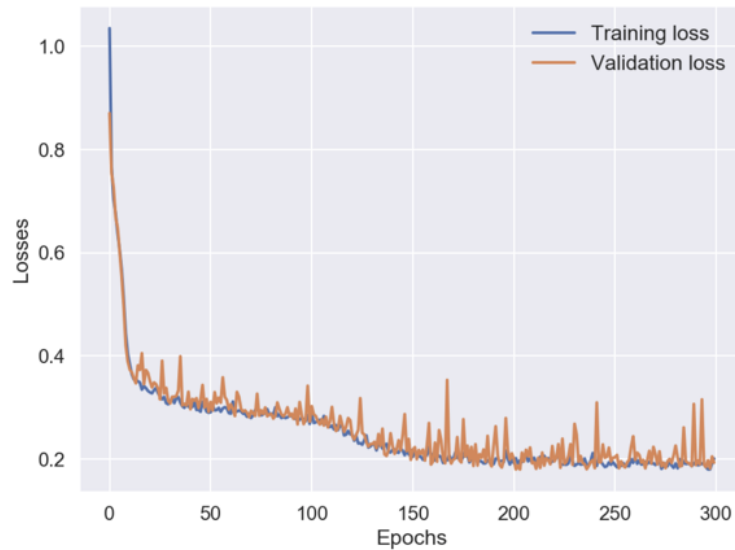


Figure 8. The training and validation losses displaying the optimization of the trained neural network model and indicating that the model is not being overfitted.

The predictions were made from our well-trained data and four compositions were selected for model validation. The candidates were chosen based on their mushy zone width. The experiments on these test alloys will help us to test our hypothesis on the presence of slag and delta ferrite phase.

The results in Figure 9 show the neural network architecture of our model. The input layer of the neural network has 12 features to account for the 13 elements in our alloy composition. The 13th element, iron, is always in balance and can be calculated from the sum of the weight percentages of the other elements. Therefore, only 12 degrees of freedom are present in a 13-element alloy, so iron was not included as a feature in the input layer. The next three layers are the hidden layers with 10, 8, and 6 nodes, respectively. We optimized the number of hidden layers based on the losses we found in our model and found that three hidden layers give us better accuracy. The output layer of this model has four nodes which are associated with the four categories shown in Figure 6.

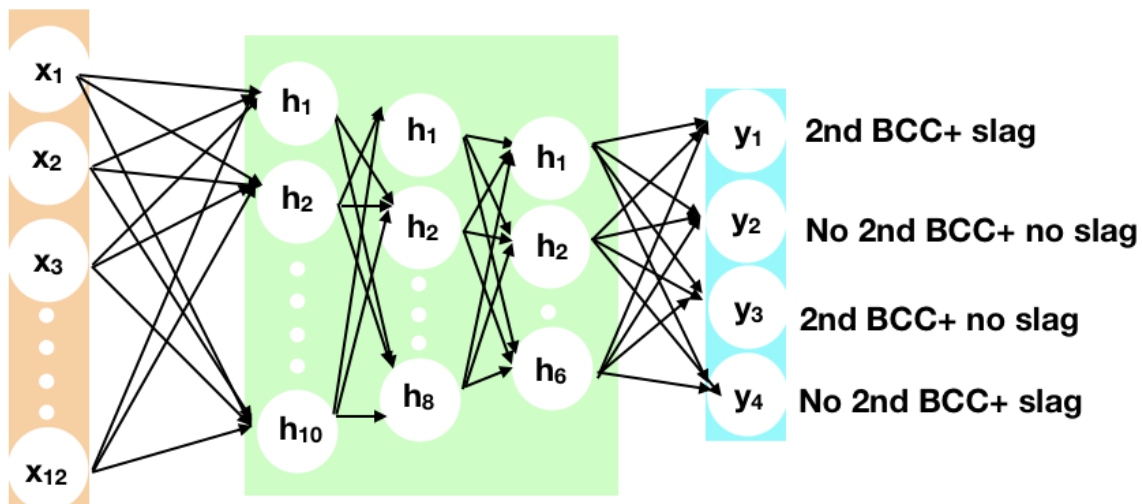


Figure 9. The neural network architecture with one input layer three hidden layers and an output layer. The input layer has 12 nodes that describe the 12 elements in our base alloy. The hidden layers have 10, 8, and 6 nodes respectively. The output layer has four nodes, which will give the probability of a particular composition of being in the classes.

The losses from the model have been calculated using cross-entropy, which is defined as

$$\text{cross-entropy loss} = - \sum_{c=1}^M y_{o,c} \log(p_{o,c})$$

where M is the number of categories, or classes, y_{ij} is the indicator if class label c is the correct classification for observation o , and $p_{o,c}$ is the predicted probability observation o is of class c .

From the CALPHAD simulations, 12 alloys were designed and recommended to test the effects of δ -ferrite and slag formation on the mushy zone and printability. The slag phase was identified as a multi-cation oxide. Alloys predicted to form this oxide slag phase were found to have higher nitrogen weight fractions, higher concentrations of silicon and oxygen, or lower concentrations of titanium than the baseline 316L. In the presence of oxygen, silicon and manganese preferentially form their native oxides, which together form the low melting temperature slag phase.

The mushy zone is believed to be a key influence on the printability of stainless steel. CALPHAD calculations revealed the presence of a low-temperature oxide slag phase and a high-temperature delta ferrite phase. Three hypotheses are put forward that correlate composition controlled microstructural features to properties that, we propose, impact printability.

- A narrow mushy zone reduces the propensity for hot tearing.
- The presence of a low temperature oxide slag, even in small volume fractions, may segregate to the grain boundaries and lead to hot cracking.
- Delta ferrite can form for some compositions and can also lead to hot cracking.

To test the accuracy of one, two, or all three of these hypotheses, we identified twelve (12) compositions: compositions that form small, medium and large mushy zone widths and, for each of these, compositions that form (1) both slag and ferrite phases; (2) slag, but no ferrite; (3) ferrite, but no slag; and (4) neither slag nor ferrite.

The team evaluated the test alloys used to understand the mushy zone, BCC phase, and slag phases created during the 316L solidification. We found that an increase in phosphorus can result in an increased mushy zone width and a slag phase, but because a suitable phosphorus source was not readily available, alternative test compositions were designed. The ML model was used to select compositions for experimental verification based on modification of the carbon and sulfur concentration. This emphasizes the strength of correlative ML models—they can identify multiple solutions in highly overdetermined problems.

Table 2 displays the chemistries chosen for this experiment. It was found that the elemental addition of carbon using spherical graphite was found to be the most accessible method to verify the effects of the mushy zone size on printability.

Table 2: Results from testing the chemical compositions picked from machine learning for testing the mushy zone, BCC phases, and slag phases present in 316L plus titanium additions. All changes to the 316L baseline composition are highlighted green.

Test Alloy	Cr	Ni	C	N	O	Si	Ti	P	S	Mn	Cu	Mo	Phase	Slag Phase	M.Z. width °C
Alloy 1	17.8	12.7	0.017	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31	BCC	No slag	27
Alloy 2	20.0	12.7	0.12	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31	BCC	No slag	64
Alloy 3	20.0	12.7	0.25	0.09	0.03	0.62	0.200	0.007	0.01	0.83	0.02	4.0	BCC	No slag	112
Alloy 4	17.8	15.0	0.017	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31	FCC	No slag	27
Alloy 5	17.8	14.0	0.12	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31	FCC	No slag	52
Alloy 6	17.8	12.7	0.30	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31	FCC	No slag	103
Alloy 7	17.8	15.0	0.017	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	2.31	FCC	slag	27
Alloy 8	17.8	14.0	0.12	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	2.31	FCC	slag	63
Alloy 9	17.8	12.7	0.30	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	2.31	FCC	slag	103
Alloy 10	17.8	12.7	0.017	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	2.31	BCC	slag	31
Alloy 11	20.0	12.7	0.12	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	2.31	BCC	slag	60
Alloy 12	20.0	12.7	0.25	0.09	0.05	1.00	0.05	0.007	0.01	2.0	0.02	4.0	BCC	slag	110

The baseline 316L composition powder, the powder humidified for 7days, and 316L + 0.535 wt% titanium powder were analyzed for non-metallic inclusions using an automated steel cleanliness analysis tool (ASCAT). Inclusions were identified and analyzed for composition with most of the inclusions found to be oxide or sulfide based. The inclusion types, numbers, and average sizes are shown in Figure 10.

The average concentrations of these impurities were overlaid on the experimentally known slag ternary phase diagrams, as shown in Figure 11. It was found that many of these oxide inclusions lie near the eutectic regions. This indicates that these oxides must have been present in the steel as a slag phase during the printing as they have a low melting temperature. The inclusions that are in the high-temperature regions can be present as particles or slag during the printing as they have melting temperatures much higher than the base alloy. This observation will help us to understand which oxides form low melting temperature slag and may be most detrimental for the printability.

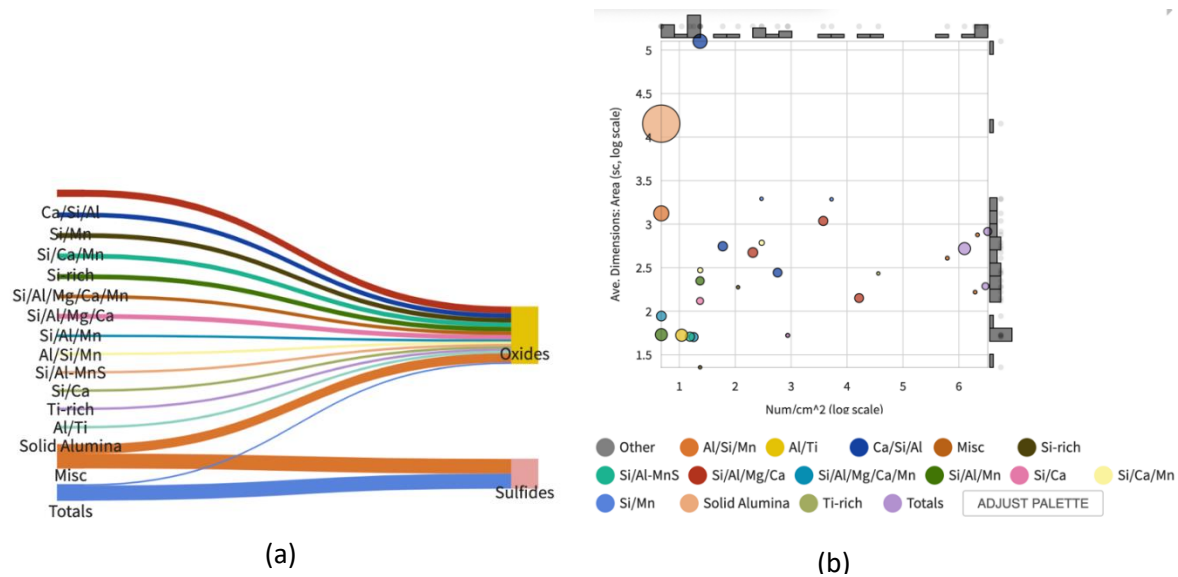


Figure 10. (a) Type of inclusions present in the samples. (b) Average dimension vs. number density of these inclusions.

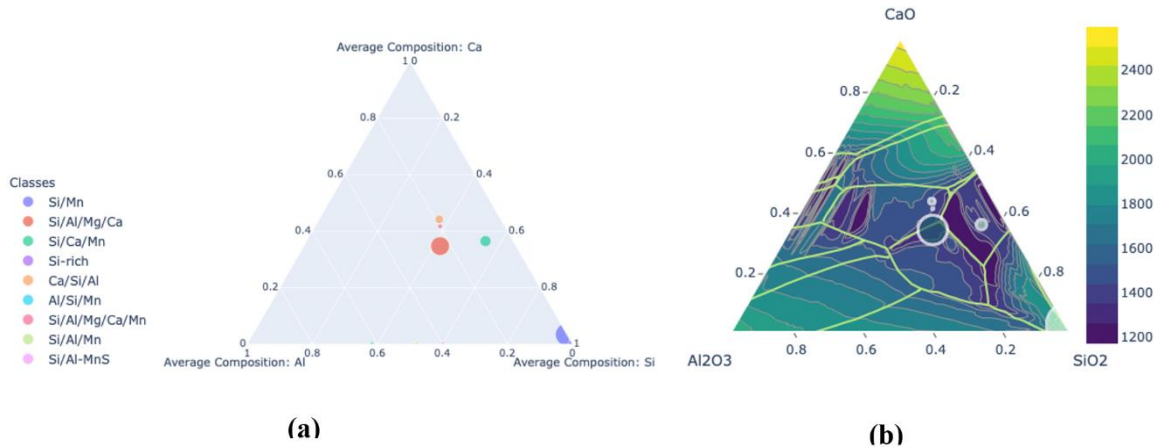


Figure 11. (a) Average composition of Al, Si and Ca in the oxide inclusions indicated by the colored circles. The radius of the color circles expresses the number density of the inclusions. (b) Experimental ternary phase diagram of slag CaO-Al₂O₃-SiO₂. The colormap indicates the melting temperature, while green continuous line indicates the liquidus line. The composition of the inclusions has been mapped on to the ternary diagram. The sizes of the particles have been doubled for better visualization.

As was reported during year 1, an extensive dataset of stainless-steel compositions surrounding the 316, 316L, and 316Ti standard compositions were explored as the basis for the 3XX-TiAM. The resulting phase diagrams were analyzed for distinctive characteristics that would allow the various chemistries to be categorized on their potential response to laser powder bed fusion (L-PBF) processing based on three key indicators: the temperature range between the liquidus and solidus (mushy zone width), the existence of a high-temperature delta (δ)-ferrite, and the presence or absence of a low temperature (sub-solidus) oxide slag phase. These were selected based on the hypotheses that a wide mushy zone

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width would lead to interior void and crack formation, the presence of a δ -ferrite at the solidification front, could impact crack formation, and that a sub-solidus oxide slag could encourage intergranular fracture under the strain induced by the residual stresses common to AM. Twelve chemistries were chosen to explore each of the four combinations formed:

- δ -ferrite
 - Present
 - Absent
- Low-temperature oxide slag
 - Present
 - Absent
- Mushy zone temperature range
 - Low (approximately 10K)
 - Medium (approximately 50K)
 - High (approximately 100K)

The chemistries require the independent modification of 9 elements including Cr, Ni, C, N, Si, Ti, S, Mn, and Mo, which creates an 11-dimensional space formed from these 9 elements and the two dependent variables, a response value and its associated error. Locally Linear Embedding represent a collection of methods to project high-dimensional spaces, such as this 9-dimensional compositional space, onto a two-dimensional plot, as shown in Figure 12.¹ This projection attempts to keep as neighbors in 2-dimensions points that are neighbors in 9-dimensions.² The response variable in Figure 12, UTS, is colored by magnitude; yellow-to-brown for the measured values, green-to-blue for values predicted by a Gaussian Process Regression (GPR) model³ trained on the measured values. In addition to being colored blue-to-green based on UTS, the modeled values are also sized based on their coefficient of variation. These predicted responses, which are supported by their respective compositions, are consistent with the measured values. With a goal of maintaining a coefficient of variation less than 5%, which extends the 3% CV project goal to account for model inaccuracy, this model suggests that such a target could be met at a specific chemistry but based on point-to-point difference in its magnitude, the UTS is likely to change significantly, even with “adjacent” chemistry changes.

¹ Locally Linear Embedding projects an N-dimensional space onto any M-dimensions space where $M < N$; it is not limited to $M = 2$. However, the projection to two dimensions is the most common to enable visualizations, such as Figure 1.

² There is a temptation to ascribe to the lower-dimensional axes a comparable metric to the higher-dimensional space—to use the distance between two points as a measure of similarity or to interpolate between points, assuming two points close in the lower-dimensional space will also be close in the higher-dimensional space. However, this does not hold. While the map is locally linear, it is globally non-linear; that is, the line connecting any two points is not co-planar with the axis but extends into-and-out-of the plane of the page.

³ The Gaussian Process Regression model was trained using an RBF (radial basis function) kernel after testing the performance of both stationary (RBF, Matérn) and non-stationary (dot product) kernels. The out-of-sample error was estimated using 5-fold cross validation. The wide variability in the results of each fold ($-2.68 < R^2 < 0.97$) indicates more data is necessary to reliably model the composition space spanned by these twelve samples.

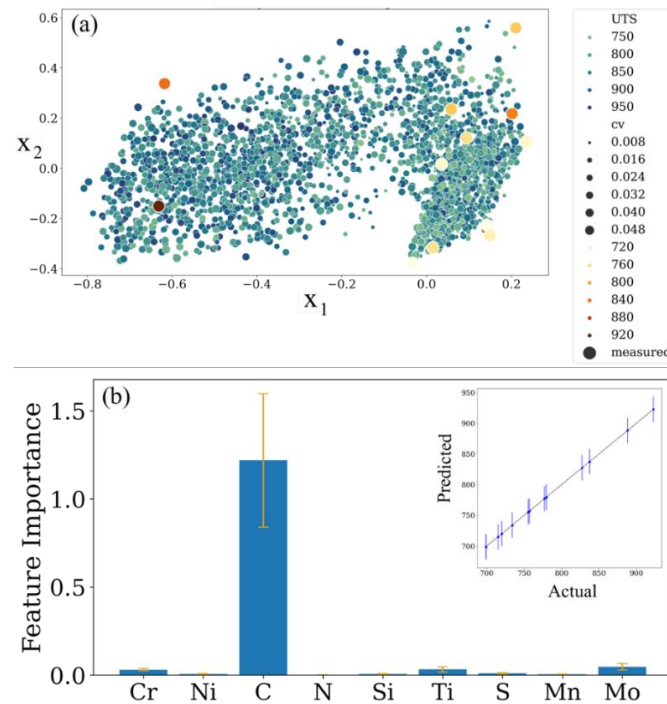


Figure 12: Exploring the composition space using (a) local linear embedding to project the 9-dimensional composition space onto a 2D plot. Interpretation points close together in this 2D plot are also close together in the 9D plot. Green-to-blue are estimated values, yellow-to-brown are measured values. (b) Evaluation of the Gaussian Process Regression model used to predict the blue-green points in (a). The parity plot (inset) shows excellent agreement between the predicted and actual UTS ($R^2 = 0.97$).

UTS was predicted using this GPR model over the ASTM accepted composition ranges for 316, 316L, and 316Ti. As in Figure 12, an investigation into the predicted performance of several thousand candidate materials was made using the GPR model, starting with a search for a range of compositions with a minimum coefficient of variance as estimated by the model within the 5% target window. These are shown in Figure 13. Since none of these compositions met the 5% CV target, further estimation of the UTS is unnecessary. The model does not accurately extrapolate to these composition ranges.

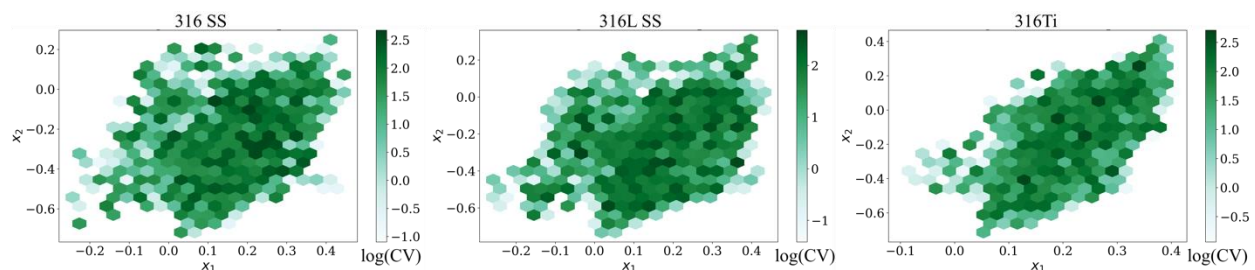


Figure 13: Each hexagon is colored according to the minimum coefficient of variation (CV) found in the enclosing section of the plot. The axes maintain the same locally linear embedding of the compositional volume projection described in Figure 12. Note the logarithmic color scale. No compositions explored achieved the 5% target CV.

4.1.2 Experimental Development (Stainless Steel Alloy)

The 3XX-TiAM development began by testing the 12 test alloys discussed in pages above. The test alloys were created through elemental additions of titanium, chromium, nickel, and manganese.

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The development would find an addition of titanium printed under a nitrogen environment would lead to grain refinement and eventually the as-printed isotropic properties desired after printing, as discussed above in the 3XX-TiAM final results.

Buils were designed to be conducted in a nitrogen shielding gas environment. The purpose was to investigate how the nitrogen environment affects the physical and chemical properties of the 316L alloy candidates. If the nitrogen process gas does not negatively impact properties, nitrogen use would enable lower print costs and improve field readiness. The builds consisted of different 316L alloys, for example, a 316L centric composition with titanium additions, and include tensile and density specimens for testing.

Nitrogen is a strong austenite stabilizer in steel, and therefore, is expected to suppress the formation of delta ferrite, but is also known to impact hot cracking. The use of nitrogen as a shielding gas can also significantly simplify the supply chain since nitrogen can be concentrated directly from the atmosphere. Both the argon and nitrogen builds used the same 316L base powder as well as the 0.535wt% added titanium powder. The parts consisted of 15mm cubes and 100mm tensile bars printed horizontally. Figure 14 displays the nitrogen build after printing.

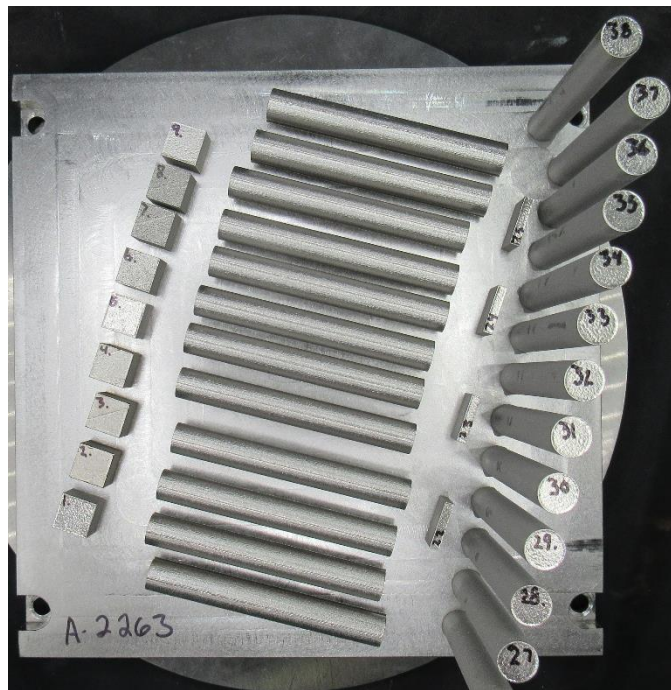


Figure 14: A completed nitrogen environment build for testing the titanium addition powder and printer shielding gas.

The team has had chemical analysis conducted on the iron used for alloy modification of the 316L base powder. The nitrogen chemical analysis to evaluate the amount of nitrogen absorbed during printing. Baseline 316L and 316L+0.535wt% Ti printed in argon and nitrogen are being sent to the IMR test lab for chemical analysis for nitrogen and oxygen. The chemical analysis was performed to understand the feasibility and potential side effects of printing in a nitrogen environment.

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Table 3 outlines the materials used and the results found after performing a LECO test to quantify nitrogen and oxygen content of as printed samples. The results show no significant differences in nitrogen or oxygen content when shielding build platforms with Argon versus Nitrogen. These results also suggest that nitrogen was not consumed by the additional titanium within the 316L-centric composition when printing in nitrogen or argon environments. An automated SEM and EDAX testing was performed to see if more can be revealed in alloy system 1 that LECO did not. It is suspected that titanium is reacting with nitrogen within the alloy system but said phenomenon was not detected by the LECO analysis.

Table 3: Nitrogen and oxygen concentration across four builds in two different shielding atmospheres.

Specimen	1	2	3	4
Material	Baseline 316L	Baseline 316L	316L+0.535Ti	316L+0.535Ti
Atmosphere	Argon	Nitrogen	Argon	Nitrogen
Nitrogen	0.09	0.09	0.09	0.09
Oxygen	0.03	0.03	0.03	0.03

The team was asked in the Phase II callout to demonstrate that the UTS coefficient of variation is less than or equal to 3.5% for 12 specimens and has a microstructure free of flaws such as pores or microcracking. Figure 15 displays the standard deviation seen in the UTS results compared to the perimeter of 3.5%. The results show that all the samples tested are within the perimeter percentage. From Table 4 and Figure 15, it can be observed that the tensile specimens show lower standard deviations than specified in the army callout for UTS.

Table 4: Overall tensile results for alloy system one.

Experiment	Test alloy	YS (KSI)	UTS (KSI)	Elongation (%)
Baseline Phase II	Carpenter US#81196 (Vertical)	76 +/- 0.23	91 +/- 0.47	67.1% +/- 1.2%
	Carpenter US#81196 (Horizontal)	88 +/- 0.42	101 +/- 0.65	54.6% +/- 0.6%
Environmental exposure	316L humidified 7 days	88 +/- 0.33	101 +/- 0.17	55.2% +/- 0.1%
	316L humidified 14 days	88 +/- 0.37	100 +/- 0.18	54.6% +/- 0.4%
	316L humidified 30 days	87 +/- 0.59	101 +/- 0.59	52.2% +/- 0.8%
Printing Atmosphere Nitrogen vs. Argon	0.535wt%Ti 99.465_316L Argon	83 +/- 0.39	106 +/- 0.57	47.6% +/- 1.0%
	0.535wt%Ti 99.465_316L Nitrogen (Vertical)	73 +/- 0.6	100 +/- 0.9	37.75 +/- 7.0
	0.535wt%Ti 99.465_316L Nitrogen (Horizontal)	83 +/- 0.21	108 +/- 1.10	48.6% +/- 1.5%
Model Verification Alloys	Test Alloy 2 (Horizontal)	97 +/- 0.56	113 +/- 0.3	42.0% +/- 2.3%
	Test Alloy 3 (Horizontal)	109 +/- 0.27	134 +/- 0.2	36.0% +/- 1.0%
	Test Alloy 4 (Horizontal)	89 +/- 0.53	101 +/- 0.45	48.3% +/- 0.5%
	Test Alloy 5 (Horizontal)	95 +/- 0.6	110 +/- 0.4	41.2% +/- 0.8%
	Test Alloy 6 (Horizontal)	107 +/- 0.27	129 +/- 0.66	29.0% +/- 1.8%
	Test Alloy 7 (Horizontal)	90 +/- 0.58	104 +/- 0.49	46.3% +/- 0.2%
	Test Alloy 8 (Horizontal)	95 +/- 0.61	110 +/- 0.4	43.1% +/- 0.7%
	Test Alloy 9 (Horizontal)	102 +/- 0.01	120	34.0% +/- 0.2%
	Test Alloy 10 (Horizontal)	90 +/- 0.03	104 +/- 0.09	50.9% +/- 0.1%
	Test Alloy 11 (Horizontal)	95.9 +/- 0.75	113 +/- 0.45	40.4% +/- 0.4%
	Test Alloy 12 (Horizontal)	102 +/- 0.35	121 +/- 0.38	29.0% +/- 1.4%

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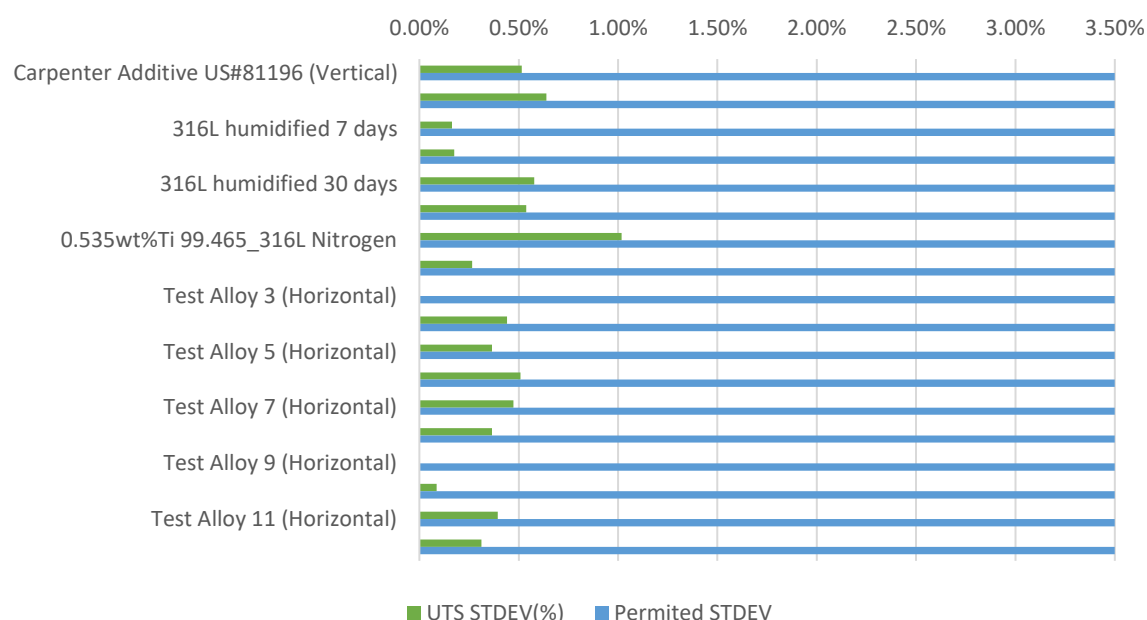


Figure 15: The UTS standard deviations for the verification alloy systems compared to the permitted standard deviation of 3.5% maximum outlined by the phase II callout.

The addition of elemental titanium to the 316L feedstock led to an increase in ultimate tensile strength. Table 4 shows an overview of the printed 316L and 316L plus Ti. In conjunction with the titanium additions, the printing atmosphere was changed to nitrogen. Nitrogen and titanium are believed to create TiN within the stainless-steel microstructure to free up chromium from sensitization. The added effect that the titanium and nitrogen atmosphere led to grain refinement within the 316L SS. Figure 16 compares two EBSD images of 316L and 316L plus Ti printed under nitrogen. The 316L plus Ti increased low cycle fatigue (LCF) life compare to as-printed 316L. Table 5 compares as-printed 316L and 316L plus Ti printed under N LCF. The development of this material has resulted in improved mechanical properties and microstructure.

Table 5: The Low Cycle fatigue result for as-printed 316L and 316L plus Ti printed under Nitrogen. Six specimens were used for each material printed in the horizontal direction. The LCF follows ASTM E606 and us the a Strain equal to 0.5%, R-value equal to -1, failure defined as 50% load drop, and Room temperature.

Material	Atmosphere	Mean Cycles	STDEV
316L baseline	Argon	4419	268
316L plus Ti	Nitrogen	4677	191

The average grain size for the 30-day humidified sample decreased in comparison to the other specimens examined. The microstructures examined are primary FCC with the percentage of BCC ranging from 0.2wt% to 1.2wt%. Table 7 shows outlines all the grain size evaluation taken during the phase II to see how the composition changes effected the printability/weldability of 316L.

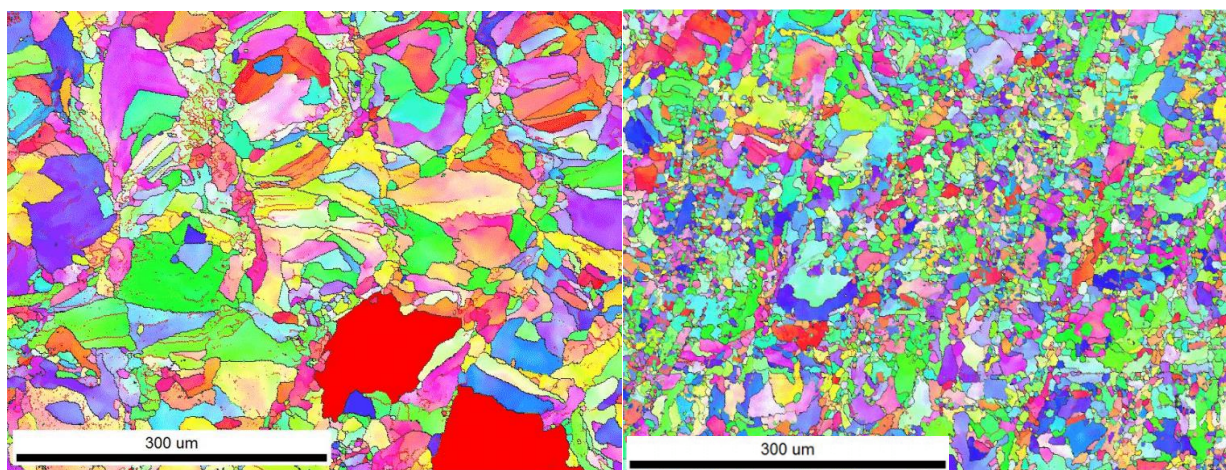


Figure 16: (Left) EBSD image of 316L printed under a nitrogen atmosphere showing an average grain size of 27.5μm. (Right) EBSD image of 316L plus Ti specimen printed under the same conditions showing an average grain size of 14.7μm.

Table 6: Results obtained for the ratio of BCC phase (δ -ferrite) to FCC phase (austenite) for selected alloys based on the 316L system. The table also displays the calculated mushy zone width, slag phase, carbon concentration, observed cracking, and observed porosity that occurred in these alloys.

Alloy	Ferrite/ Martensite	Austenite	grain size (μm)	STDEV (μm)	Mushy- Zone width (°C)	Expected slag phase (CALPHAD)	Carbon wt %	Observed cracks	Observed porosity
316L baseline	1.20%	98.80%	27.83	26.58	30	Yes	0.017	No	No
Humidified 30 days	0.20%	99.80%	20.01	21.25	30	Yes	0.017	Yes	Yes
316L +nitrogen	0.20%	99.80%	27.48	31.57	30	Yes	0.017	No	No
Test Alloy 1	0.30%	99.70%	28.21	32.60	27	No	0.017	No	Yes
Test Alloy 6	0.80%	99.20%	27.30	22.32	103	No	0.3	Yes	Yes
Test Alloy 7	1.30%	98.70%	23.10	25.46	27	No	0.017	No	Yes

Table 7: The grain size comparison for the stainless steel development in phase II

Sample name	Atmosphere	Class	Chemistry	Mushy zone (°C)	Phase map	Grain Size Average (μm)	Grain Size STDEV (μm)
Base 316L	Argon	BCC, No slag	-	30	0.012	27.8	26.6
Base 316L Humidified for 30 days	Argon	BCC, No slag	-	30	0.002	20.0	21.3
Base 316L	Nitrogen	BCC, No slag	-	30	0.002	27.5	31.6

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Test Alloy 1	Argon	BCC, No slag	0.085Ti	27	0.000	28.2	32.6
Test alloy 6	Argon	FCC, No slag	0.3C, 0.085Ti	103	0.008	27.3	22.3
Test alloy 7	Argon	FCC, No slag	15Ni, 0.05O, 1.0Si, 0.05Ti, 2Mn	27	0.013	23.1	25.5
316L plus Ti	Argon	-	0.535Ti		0.018	20.2	17.3
316L plus Ti	Nitrogen	-	0.535Ti		0.007	15.0	14.7

The mechanical test results have shown that an increase in carbon used to produce the increased mushy zone width resulted in micro cracking within the microstructure. The microcracking demonstrates that the printability of 316L has decreased. Figure 17 shows test alloys 4, 5, and 6 and illustrates that as the carbon content increases, porosity and microcracking are introduced.

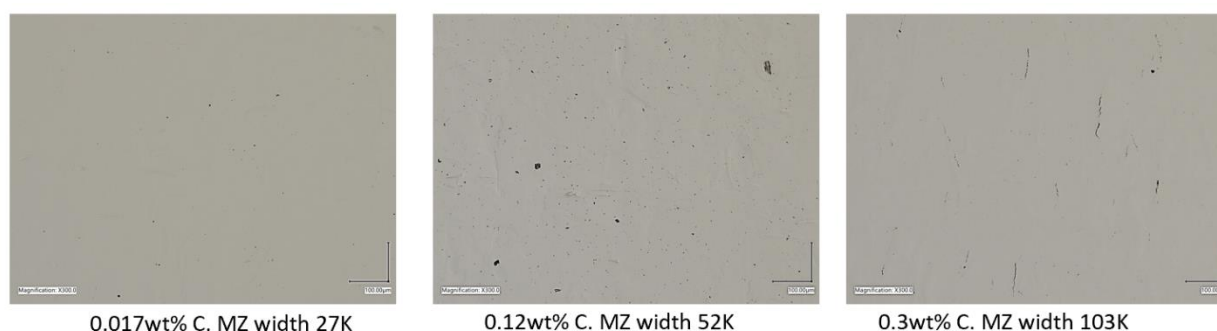


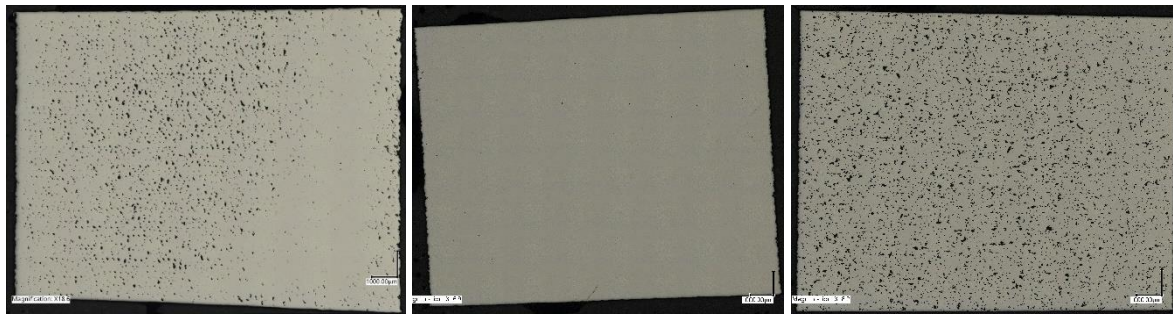
Figure 17: From left to right displays test alloy 4, 5, and 6. The test alloys isolated the change in the mushy zone width. The difference in mushy zone was created from carbon additions.

The ferrite content analyzed by EBSD, the average cross-sectional grain size, and the tensile properties for 316L baseline, and with 0.535 wt% titanium addition in argon and nitrogen process gas are presented in Table 8. The baseline specimen had an average grain size of approximately 28 μm while the Ti addition specimen printed in argon has an average of 20 μm and the specimen with added Ti printed in nitrogen had an average of 15 μm . This indicates that the titanium promoted grain refinement and the titanium addition with nitrogen atmosphere resulting in the most grain refinement. The specimens with the added titanium also had higher ultimate strengths than the baseline but lower yield strength and slightly lower elongations. The lower yield strengths may be in part due to the titanium binding interstitial strengthening elements like carbon and nitrogen counteracting the expected Hall Petch strengthening from the finer grain size.

Table 8: Microstructure and tensile results for 316L baseline and added titanium with argon and nitrogen process gas.

Alloy	Gas	orientation	Ferrite (wt%)	Ave. Grain Size (μm)	YS (ksi)	UTS (ksi)	Elongation (%)
316L Baseline	Argon	XY	0.012%	27.83	88 $\pm$ 0.42	101 $\pm$ 0.65	54.6 $\pm$ 3.5
316L +0.535wt% Ti	Argon	XY	0.018%	20.23	83 $\pm$ 0.39	106 $\pm$ 0.57	47.6 $\pm$ 1.0
316L +0.535wt% Ti	Nitrogen	XY	0.007%	15.02	83 $\pm$ 0.21	108 $\pm$ 1.1	48.6 $\pm$ 1.5

To understand the robustness of baseline 316L to processing conditions, the team printed a build with varying laser powers. The laser powers ranged from 370 W to 100 W with every other printing parameter held constant. Figure 18 displays the 300 W, 200 W, and 100 W results showing how changing laser power affects the printability of 316L. The 300 W sample shows an increase in porosity due to keyholing; a result indicative of too much laser power. The 100 W shows high porosity due to incomplete fusion. Figure 19 displays the laser power versus optically measured density for the sample set of test cubes. The results show that laser power between 250 W and 150 W produces the highest density specimens with a wide range of laser powder able to produce 99% or greater relative density.

**Figure 18: The images show the level porosity created with varying laser power. From left to right 300W, 200W, and 100W.**

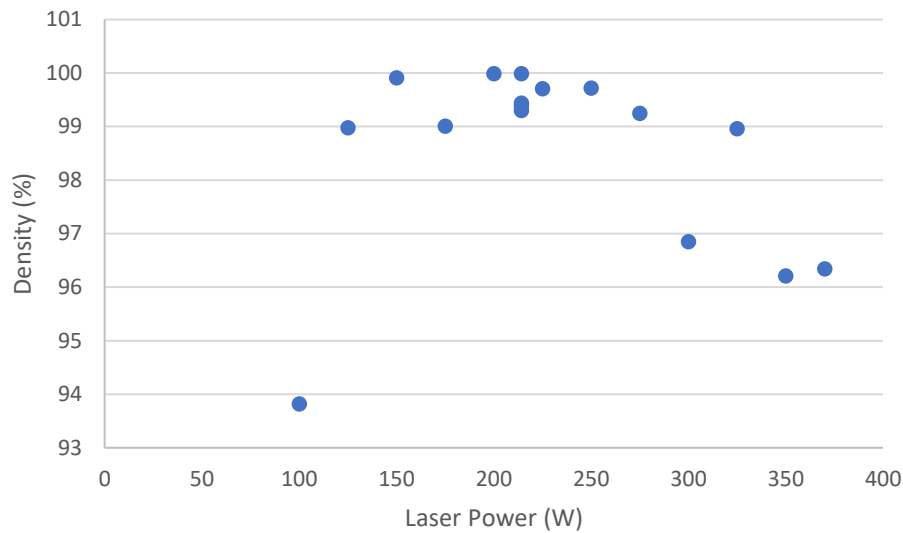


Figure 19: Optically measured density vs laser power for printed 316L with other parameters held constant.

Four test alloys were selected for low cycle fatigue testing as an addition to the planned project scope. The fatigue test will use an R value of -1, and comprise seven ASTM E606 flat sheet fatigue specimens with 12 by 6 mm gage length.

The selected alloys are the 316L baseline, test alloy 6, test alloy 10, and 316L plus 0.535wt%Ti printed in nitrogen. These materials were down selected from the 17 test alloy prints for low cycle fatigue to help further differentiate their performance. Test alloy 10 was chosen based on the increase in YS and UTS mechanical properties compared to 316L baseline. The 316L base plus 0.535wt% titanium is expected to form titanium nitride precipitates that may act as heterogeneous nucleation sites to produce a finer sized and less directional grain structure that may improve mechanical property consistency. Test alloy 6 had increased strength from its carbon addition, but the microstructure suffered from microcracking as seen in Figure 27 and was selected to examine if fatigue testing could highlight the mechanical effects of the microcracking better than tensile testing alone. The 316L baseline was selected as a control for comparison to the three other selected alloys.

Table 9 shows the data collected from fatigue testing. The results indicate that the change in chemical composition has greatly affected the LCF of 300-grade stainless steel. The 316L plus Ti samples demonstrated an improvement in fatigue life compared to the 316L baseline and test alloys 6 and 10. The LCF results were utilized to help select the composition specification range for the pre-alloyed powder production. The improved fatigue life in the 316L plus Ti is believed to be due in part to a reduction in grain size due to heterogeneous nucleation on TiN precipitates. Figure 16 shows the 316L baseline printed under nitrogen compared to the 316L plus Ti printed under nitrogen. The average cross-sectional grain diameter was reduced from 27.5 μm to 14.7 μm in the modified composition.

Table 9: Overview of fatigue testing progress.

Material	Atmosphere	Mean Cycles	STDEV	BCC Phase Predicted?	Slag Phase Predicted?	Mushy Zone ΔT°
316L baseline	Argon	4419	268	Yes	No	30.5
Test Alloy #6	Argon	1542	345	Yes	No	112
Test Alloy #10	Argon	2147	415	Yes	Yes	31
316L plus Ti	Nitrogen	4677	191	Yes	No	30.5
Test Alloy #4	Argon	4282	319	No	No	27

GT also examined test alloy 10 using SEM to characterize the slag phases that may be present in this alloy. Test alloy 10 was one of the alloys created by ICME to see the effects of slag phases on 316L stainless steel. Figure 20 displays image scans of a titanium-rich portion of test alloy 10. The team thought it might be a slag inclusion but found the area to contain a high concentration of titanium. It has been inferred that this area of concentrated titanium resulted from incomplete dissolution of the added titanium powder during the laser printing process.

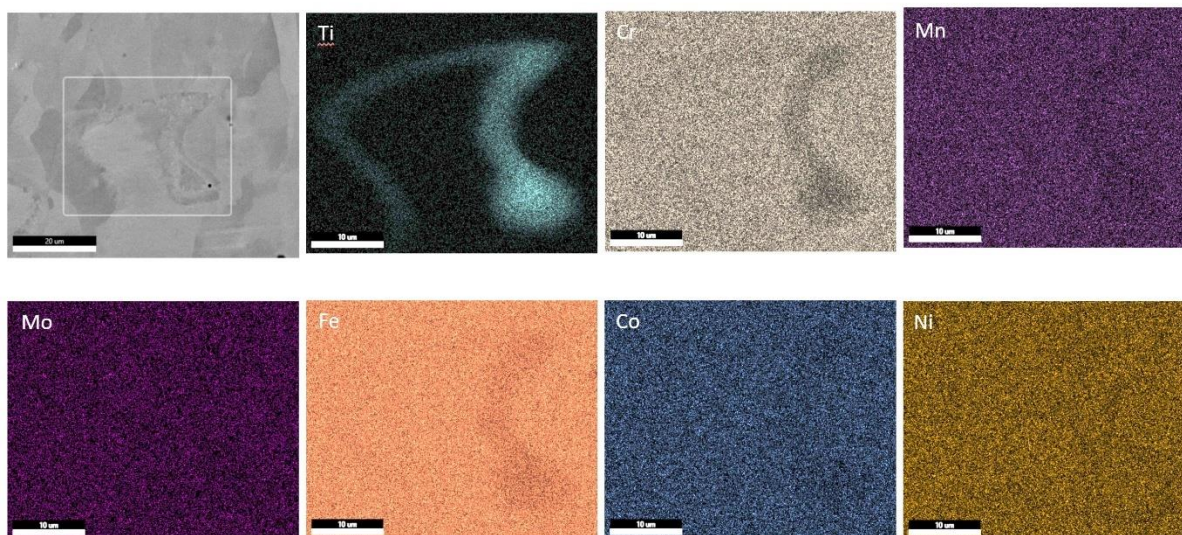


Figure 20: Image scan conducted by GT of a titanium-rich spot found in test alloy. (Top left image scale bar = 2 μ m. All other scale bars = 10 μ m).

A valuable critique of our Phase II Sequential proposal was a recommendation to improve materials development through integrated computational materials engineering (ICME). A multi-step approach was planned based on that suggestion, beginning with the calculation of stable precipitate phases. Figure 21 displays the results of Scheil simulations of the baseline austenitic stainless steel used in Phase I and Phase II.

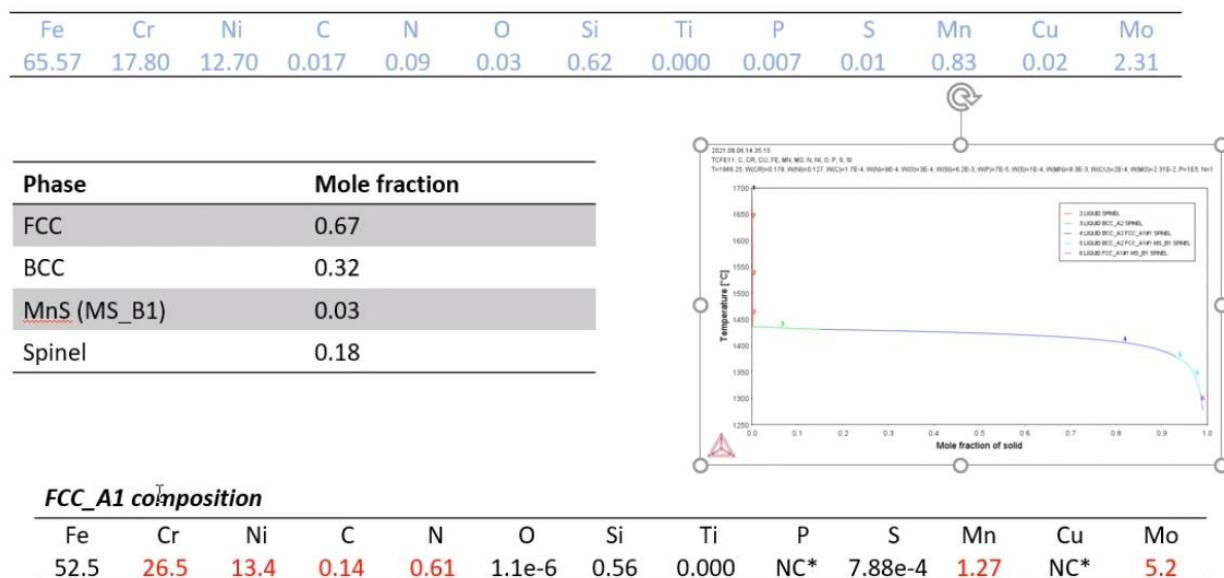


Figure 21: Thermocalc Scheil solidification simulation of austenitic stainless-steel baseline. Red values were varied for the analysis.

The composition that has been chosen to move forward for the AM 3XX stainless steel is seen in Table 10. The selected composition showed that the material has a finer microstructure after printing, increased ultimate tensile strength (UTS) and fatigue life, and better utilization of nitrogen printing environments from the addition of elemental titanium.

Table 10: AM 3XX stainless steel composition

AM 3XX SS	Fe	Cr	Ni	C	N	O	Si	Ti	P	S	Mn	Cu	Mo
WT%	Bal	17-18	11-14	0.03	0.09	0.03	1	0.5-0.8	0.045	0.03	1	0.01	2.0-3.0
Single Numbers are given as Maximums													

The titanium-containing stainless steel was printed with the standard EOS 316L laser parameters, and the resulting build produced highly dense parts. Table 11 shows the Archimedes density results for titanium-containing stainless steel. Figure 22 shows the first completed deliverable build from printer 1. Cube one from deliverable build one showed little porosity after printing, as shown in Figure 23. After printing the titanium-containing stainless-steel, it appears that the observed printability matches that of the 316L baseline material.

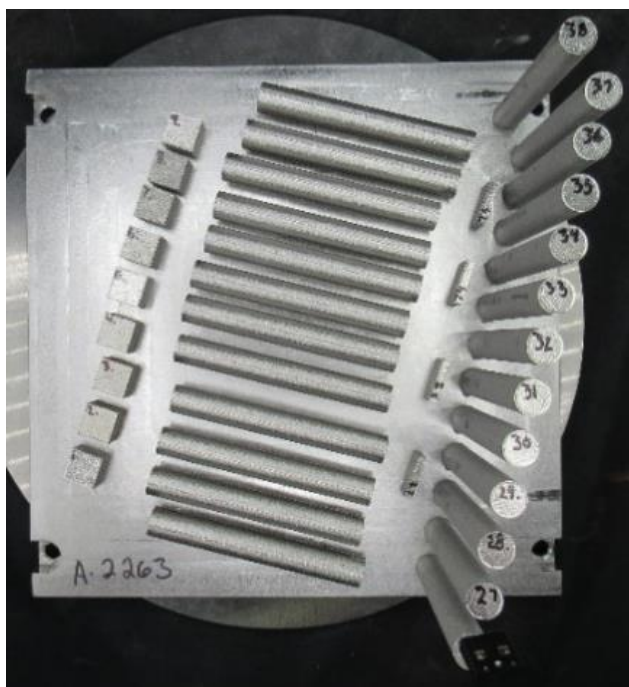


Figure 22: Custom atomized stainless steel first build utilizing printer 1.

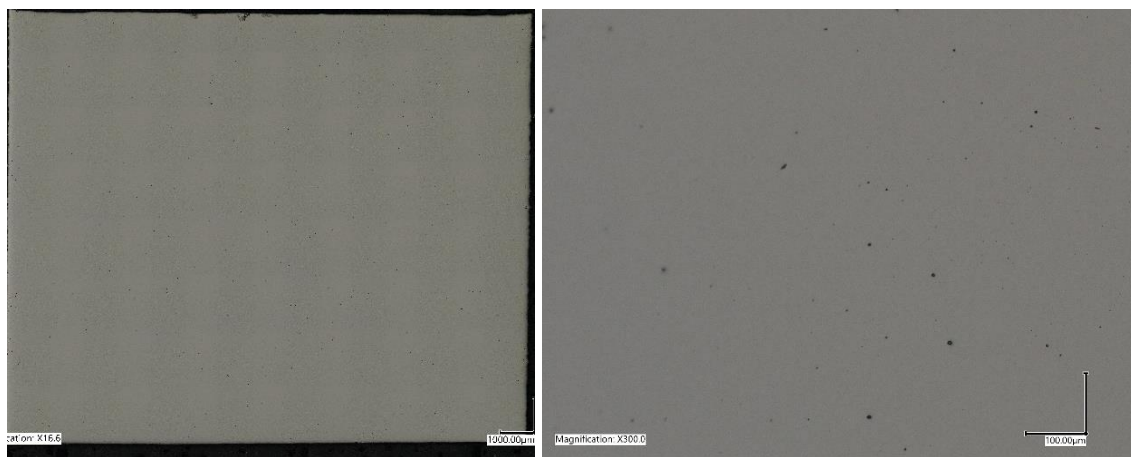


Figure 23: Cube 1 from the first print of the titanium-containing stainless steel from L-PBF printer 1. The micrograph shows a dense cube with small pores throughout the surface. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube

Table 11: Archimedes density results for deliverable build one, printer 1.

Material	Delivery Build Number	Printer	Cubes	Theoretical Density (g/cc)	Apparent Density %	Standard Deviation of the AD
3XX Stainless steel	1	1	6	7.99	99.77%	0.02%

The custom stainless steel powder was evaluated to see the powder quality before printing. In addition, the team performed SEM imaging to understand the morphology of the powder. The custom atomized titanium-containing stainless steel appeared to have irregular particles, but after printing, has not affected the observed printability. Figure 24 shows the custom atomized titanium-containing stainless steel powder. Compared to the 316L baseline, the custom atomized titanium-containing stainless steel powder could be more spherical Figure 25.

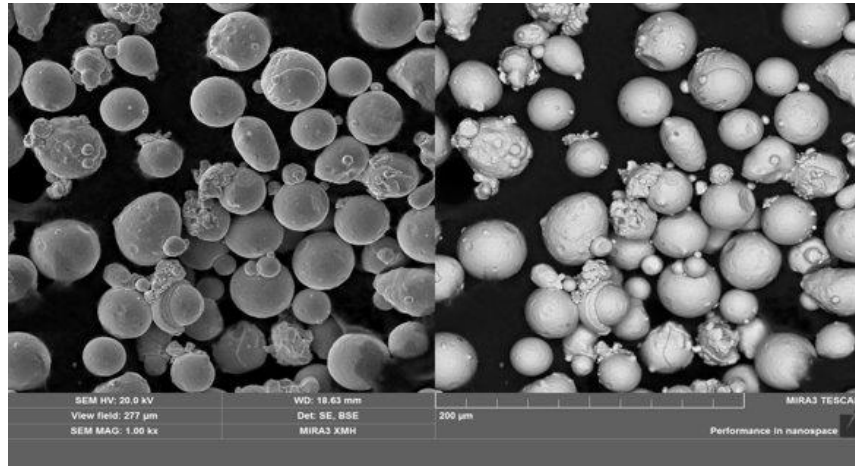


Figure 24: Custom atomized titanium-containing stainless steel atomized by Praxair. The image shows a spherical powder with irregular particles scattered throughout.

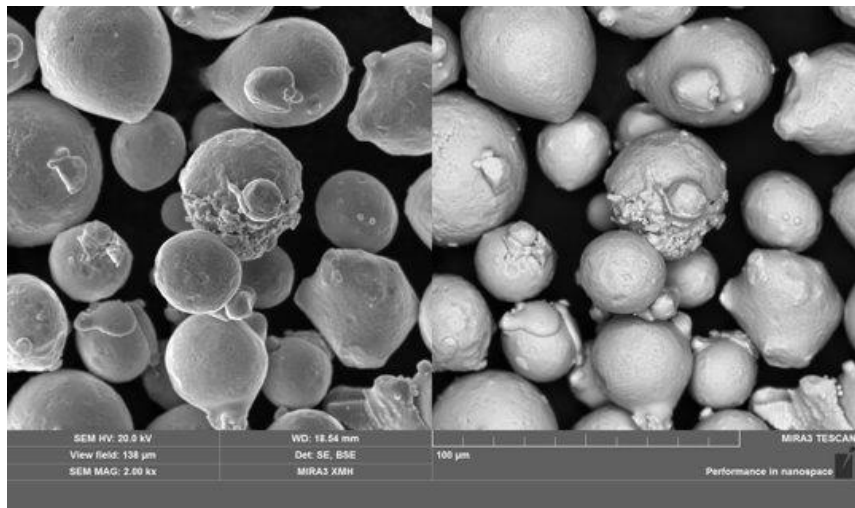


Figure 25: The 316L stainless steel baseline atomized by Carpenter Additive. The gas atomized powder appears to have fewer irregular particles.

The Phase II 316L baseline tensile results for vertical (printed samples in the z-direction) and horizontal samples (printed parallel to the XY plane) were used to compare tested alloys. GT tested these samples following the ASTM E8 procedure. We expected that 3XX SS plus Ti would improve isotropic properties after printing, as seen in the horizontal printed tensile results and refinement of the microstructure from the Ti additions. Compared to the 316L baseline, results from vertically and

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horizontally printed 3XX SS plus Ti samples showed similar trends of reduced yield strength and increased ultimate tensile strength, but these samples had more sporadic elongation than any others. Table 12 and Figure 26 show the average results.

Elongation results for vertically printed samples of 3XX SS plus Ti did not show improved isotropic properties; there was a large standard deviation between the six specimens pulled. Next, the team will review the extensometer data and confirm the elongation results with the fit-back method to understand the standard deviation compared to the horizontal tensile samples and the vertical baseline.

The microstructures of the 3XX SS plus Ti samples were reviewed to see if the elemental Ti additions were equally distributed throughout the samples. If this evaluation shows that the samples did not have Ti equally distributed throughout the microstructure—specifically in the fracture area—this could indicate why the elongations were not consistent. The elongation is also believed to be improved by the pre-alloyed 3XX SS plus Ti because the Ti would be contained in the powder rather than as an elemental addition and therefore the even distribution of Ti would not rely on good mixing and blending.

Table 12: List of the average mechanical properties measured for the 316L baseline and 3XX SS plus Ti samples.

Material	YS (KSI)	YS STRDev. (KSI)	UTS (KSI)	UTS STRDev. (KSI)	Elongation %	Elongation % STRDev.
Base 316L (VTB)	76.4	0.2	91	0.5	67.07	1.17
3XX SS plus Ti Nitrogen (VTB)	73.4	0.6	100	0.9	37.75	7.17
316L Baseline (HTB)	88.0	0.4	101	0.6	54.55	0.57
3XX SS plus Ti Nitrogen (HTB)	83.5	0.2	108	0.2	48.6	1.42

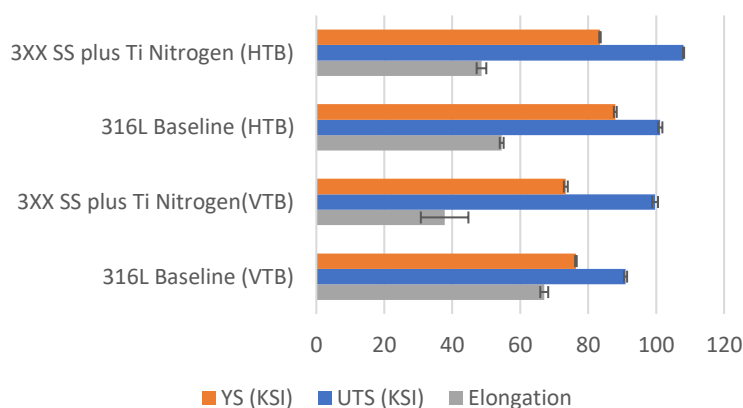


Figure 26: Graph of average mechanical testing results for 3XX SS plus Ti and 316L Baseline samples.

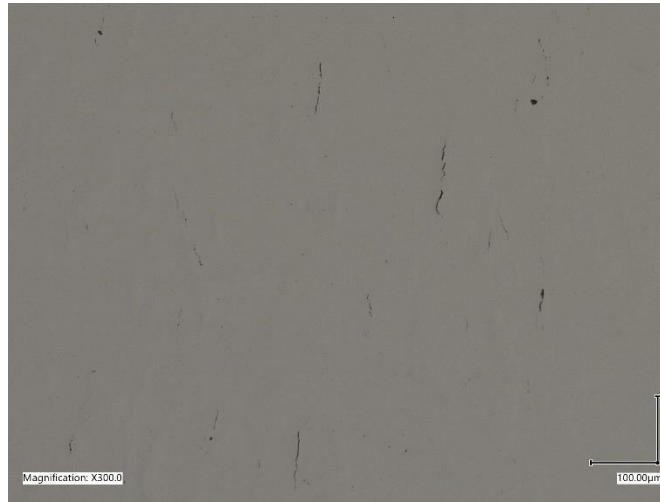


Figure 27: The micrograph shows the microcracks in the microstructure of test alloy 6 resulting from the increase in carbon.

All test alloy powders were analyzed using ICP chemical analysis. The titanium stands out because the powder appears to have less than predicted, but the printed product shows the correct amount. The change in Ti from powder to printed parts suggests that the sampling for testing was inadequately prepared for pure Ti powder within the test alloys. The resulting Ti in the printed part shows that the Ti was fully dissolved after laser welding- confirming the correct amount of Ti was mixed. Silicon within the chemistry was high across all alloys-including the baseline-suggesting that either using ICP to detect silicon is not sufficient, or there was more silicon in the baseline material than previously predicted. Oxygen results show that there is an increase in oxygen after powder preparation and a decrease after printing. The increase in oxygen suggested that open air can contribute to oxidation. The resulting reduction in oxygen after print shows the laser welding is ablating oxygen from the powder during printing. There is more information to pull from the ICP data, and the team is planning to create a statistical matrix comparing all the test alloys result to find a suitable chemistry range for alloy system 1.

The differences from the designed composition shown in Table 33 Appendix A demonstrate potential for compositional changes during the printing process. Because the feedstocks utilized additions comprised of elemental and master alloy powders, the ICP dissolution methods may have been inadequate for hard to dissolve constituents like elemental titanium and therefore may not report accurate results for the feedstock material for all elements. The fractional elemental changes in the printed specimens are shown graphically in Figure 67 Appendix A. It is important to note that in this figure the direction of change is more reliable than the magnitude. The changes measured are generally consistent with expectations based on elemental vapor pressures at temperatures predicted for laser AM processing. More volatile elements like manganese and oxygen show a greater loss while elements with a lower volatility like molybdenum, titanium, and nickel show fractional gains. This data can be utilized to determine the feedstock compositions that will produce the target printed part compositions by accounting for composition changes during the AM processing.

Initial hardness data for the model verification systems is shown in Figure 28. The data reveals that the compositions with increased carbon content had the highest hardness values. The model verification alloys all had increased hardness compared to the baseline composition, revealing that the changes in composition have affected mechanical properties.

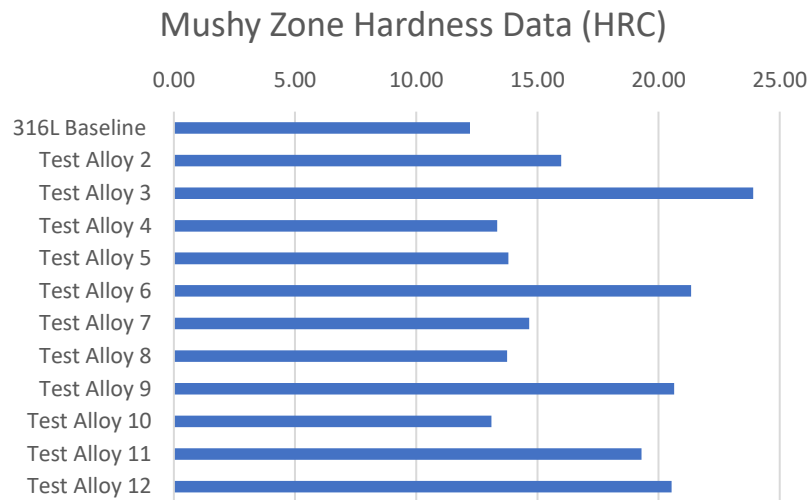


Figure 28: Hardness data in the as-printed condition for model verification test alloys and the 316L baseline. Test alloy 3, 6, 9, and 12 are believed to have increased in hardness due to the increase in carbon.

Alloy 12 was calculated to have a large mushy zone but was not observed to have microcracking. This alloy also had elemental molybdenum additions and the microstructure showed circular inclusions believed to be undissolved molybdenum. Figure 29 displays test alloys 10, 11, and 12 with increasing mushy zone widths.

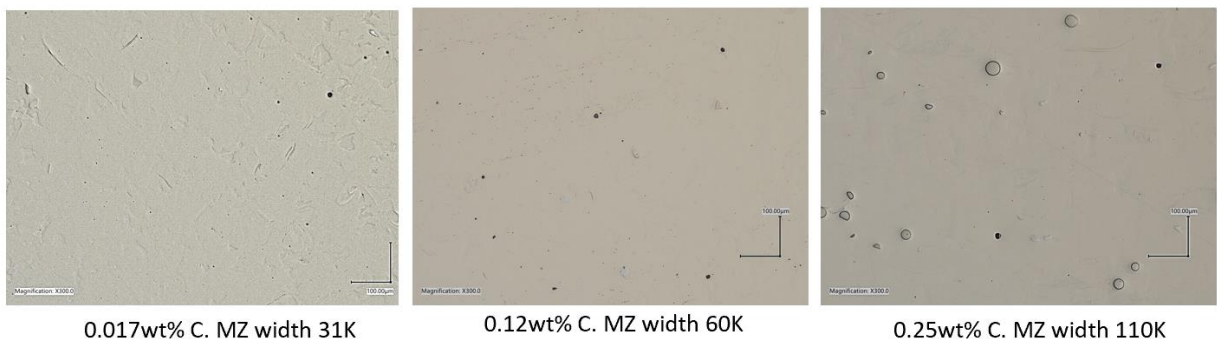


Figure 29: From left to right displays test alloys 10, 11, and 12. The test alloys isolated the change in the mushy zone, bcc, and slag phase present. The change in the mushy zone was created primarily with carbon additions.

Images from an SEM are shown in Figure 30 and Figure 31 from verification alloy 6 and 7, respectively. Figure 30 revealed microcracks across the surface of the sample in an alloy containing increased carbon content with a large mushy zone range. The team will use Electron Backscatter Diffraction (EBSD) and Energy-dispersive X-ray (EDX) to further investigate the origins of the cracking. Figure 31 shows no cracking across the verification alloy 7 specimen, but there appears to be an increase in porosity across the sample. This sample was made to study the effects of the slag phase with a small mushy zone.

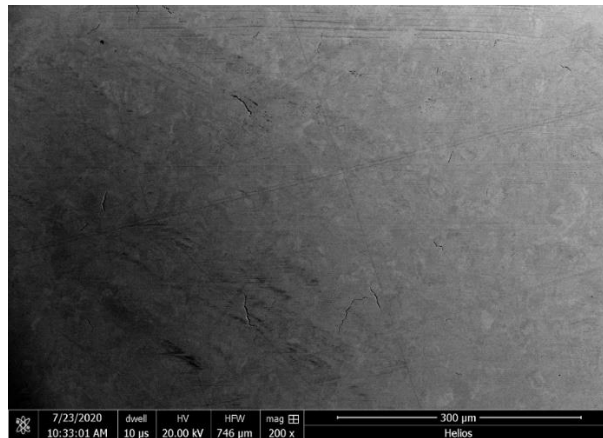


Figure 30: Verification alloy 6 SEM image. The image displays cracking across the polished surface. More investigation is needed to understand the cracking, but the alloy had added carbon to create a large mushy zone range. Carbon is known to increase strength and crack susceptibility in iron-based materials.

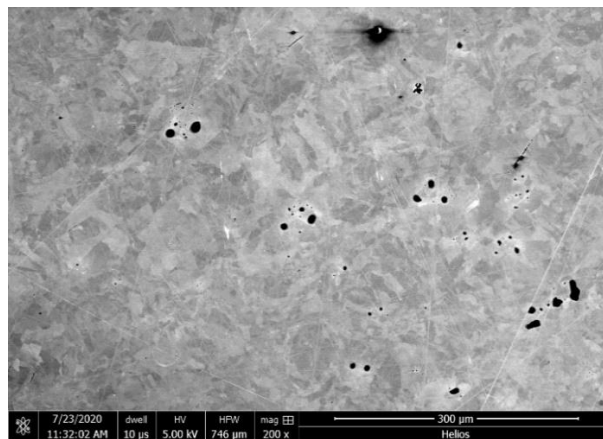


Figure 31: Verification alloy 7 SEM image. The image displays an inclusion and an increase in porosity across the polished surface.

Figure 32 and Figure 33 show SEM micrographs for mushy zone test alloy 3 and 316L baseline, respectively. The mushy zone test 3 appears to have large inclusions and microcracks throughout the microstructure suggesting the changes in chemistry have affected the performance of 316L compared to the Phase 1 316L baseline alloy, Figure 33. This result is in-line with the team's theoretical predictions and serves to verify assumptions behind the theoretical model. The team is conducting a chemical analysis to identify the compositions of these inclusions.

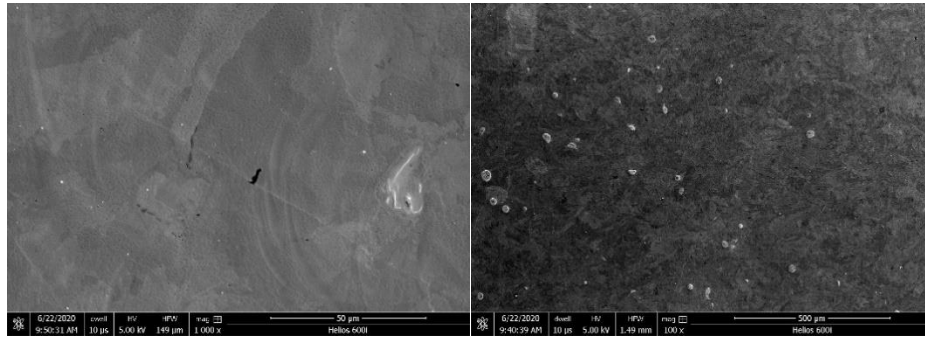


Figure 32: SEM micrograph of test alloy 3 from figure 1. Left shows the microcracking through the microstructure. Right shows the inclusions across the microstructure.

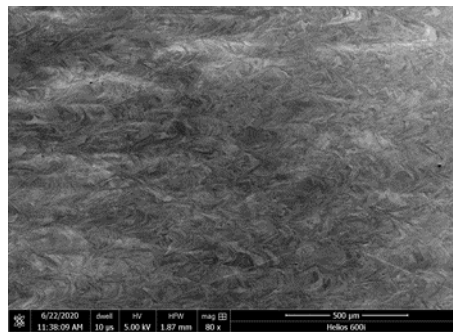


Figure 33: Shows the micrograph of AM 316L baseline from phase 1, showing fewer inclusions and no observable microcracking.

4.1.2.1 Environmental Exposure Testing (Stainless Steel Alloy)

While in service, source powders may be exposed to a range of conditions during storage. These conditions may have an impact on the printability of the powder and, therefore as an operational constrain, acts as a design parameter. The team designed an experiment to understand the effect that exposing source powder to high humidity environments for more than seven days has on the printability of powders. To better establish this design parameter, part microstructures that develop in parts printed from these powders were examined at CSM in scanning electron microscope (SEM) and transmission electron microscope (TEM) images. Results from Phase I will guide powder specifications and provide baseline information for developing procedures and protocols for the use or exclusion of powders based on their storage histories.

The team conducted scanning electron microscopy (SEM) and transmission electron microscopy (TEM) on Phase I humidified samples to find differences in the microstructure.

An incubator was procured (not using funds dedicated to Phase II) to create an artificial environment to test the resilience of 316L powder in an extreme, humid environment. The experiment is being scaled up from Phase I by increasing time exposure and the amount of powder exposed to humidity. The experiment will use the following controls to create the needed powder.

- Humidity set at 80%.
- Temperature set at 40°C.

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- 6 to 8 kg of 316L per humidification period.
- The periods will last 7, 14, and 30 days.

The goal is to evaluate the changes in physical properties from artificial humidification. Leading to a deeper understanding of how changing environment effect print quality for alloy system 1.

Environmental exposure effects on printed properties are important to understand and quantify a reliable performance range as well as develop exposure resistant feedstocks. Three 8kg batches of 316L powder were measured out and placed in an incubator with 87% relative humidity at 40°C. The water level, temperature, and humidity are checked and recorded periodically during the day. Each build consists of eight density cubes, six tensile bars, and four corrosion samples. All but three cubes were printed using standard EOS M290 parameters for 316L. The remainder of the printed cubes use varying laser speeds to determine if parameters can be improved upon for the evaluated powder. The powder samples were subjected to 7, 14, and 30 days of humidification. A non-humidified sample was kept as baseline.

It was determined that the 316L powder absorbed water when exposed to a humid environment. The humidification created a crust of clumped powder on each sample, but 1 to 3 mm below the surface, the powder visually appeared unchanged by the humidity. Table 13 shows the humidity, temperature, and change in weight after the humidification. Figure 34 displays the 14-day humidified powder sample showing the top surface clumping. It was not immediately clear why the 30-day humidified powder had a lower recorded mass gain than the 14-day powder.

Table 13: Change in powder weight after humidification.

Humidifying period	Initial powder weight	Final powder weight	Weight Gained	Humidity	Temperature
Days	(g)	(g)	(g)	(%)	(°C)
7	7994.7	7995.1	0.4	87%	39
14	8003.1	8005.1	2.0	88%	40
30	7997.1	7997.6	0.5	90%	40



Figure 34: Displays the 316L powder after 14 days of humidification. The powder exhibited clumping and fusion in the upper exposed surface.

The density results for the four test criteria for the environmental exposure test can be seen in Figure 35. Figure 36 compares the tensile results for the humidified samples for 7, 14, and 30 days to the 316L baseline. The tensile results show there are little changes in UTS and YS for each test alloy. The elongation appears to decrease as humidity exposure increases.

Mechanical test results obtained using the tensile bars show that after 30 days of humidification, and after breaking up the agglomerates, the powder could be used to build parts that are mechanically similar to those built from the 316L baseline humidified powder. Thus, while humidity influences stainless steel mechanically if exposure is prolonged and the humidity levels are high, the 316L stainless steel baseline material performs better than any other test material both before and after humidification. Figure 36 and Table 12 show the results obtained during from tensile testing. After 30 days in a humidifier at 40°C at 87% ambient humidity the Al₂O₃ ALD-coated stainless steel resulted in parts with increased test variability compared to the other test materials.

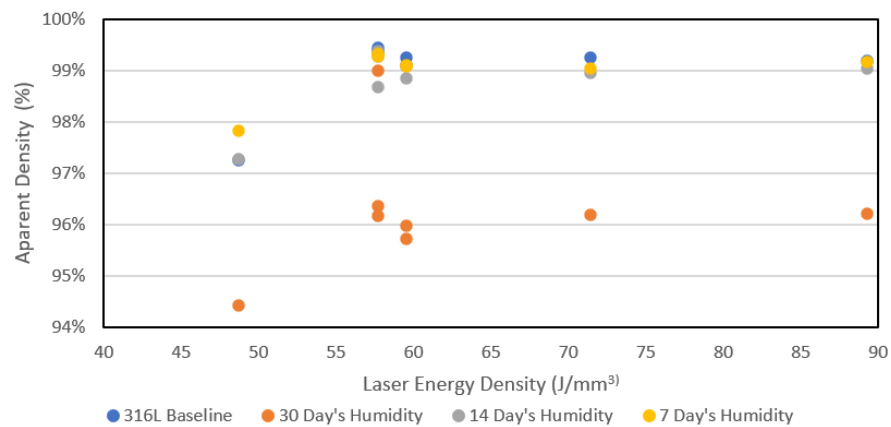


Figure 35: The results show that the 30 days humidified samples have a decrease in density compared to the other samples across all the energy density tested

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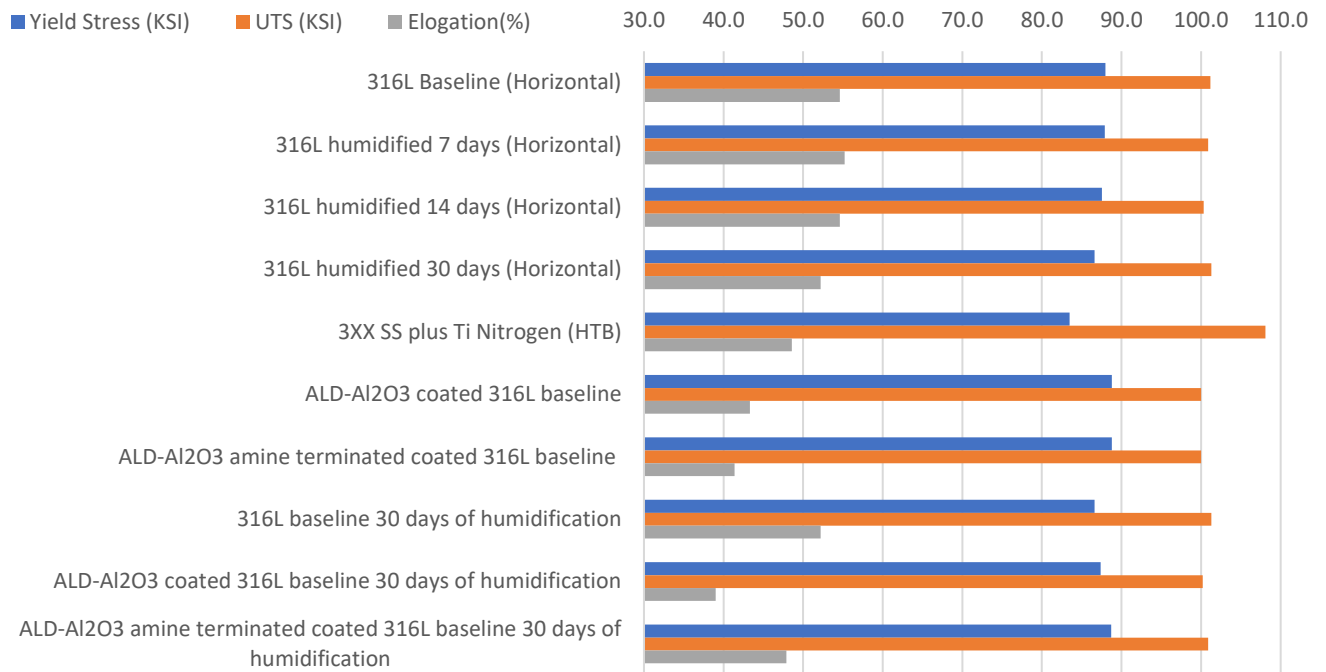


Figure 36: 316L resulting tensile tests comparing baseline 316L to the ALD coated 316L powders before and after humidity.

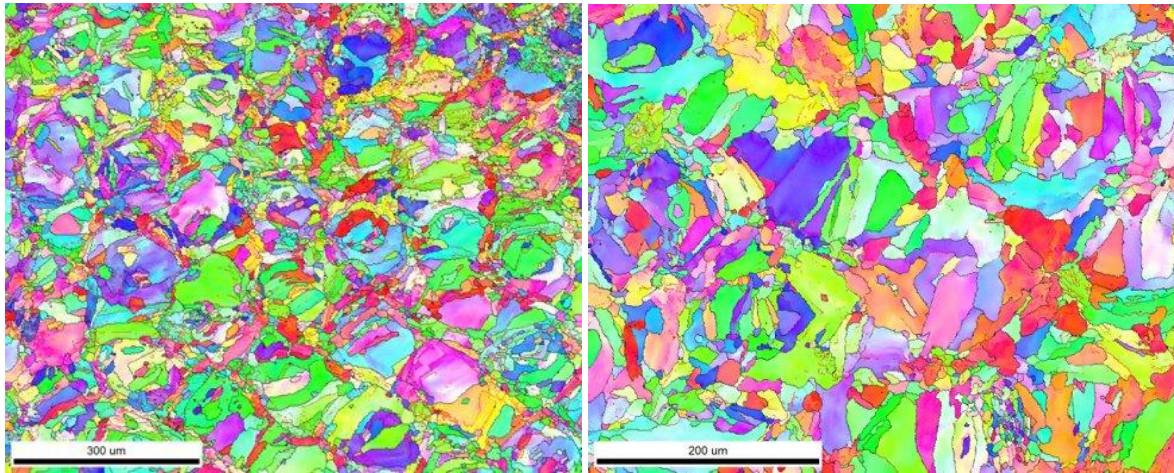


Figure 37: EBSD images taken of 316L baseline left and 316L right after 30 days of humidification. Note the scale difference between the EBSD images.

Two EBSD images are displayed in Figure 37 comparing the 316L baseline to a 30-day humidified sample. The environmental exposure testing has shown that the 316L alloy has exceptional consistency in tensile properties. The 316L alloy system retained its tensile properties when exposed to humid conditions for up to 30 days, despite an average decrease of 2.79% relative density in the printed part using the humidified. These results suggest that 316L can be exposed to an ambient humidity of 90% humidity at 40°C for up to 30 days and still produce parts with repeatable tensile properties.

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It was confirmed that the 316L microstructure increases in porosity after the powder feedstock has been exposed to an environment of 90% humidity at 40°C for 30 days. Figure 38 shows SEM images of the 30-day humidified sample which highlights the microstructure and porosity present in 316L. Figure 39 confirms the increase in porosity by comparing micrographs from each test criterion; as the humidity exposure increases, more porosity is present in the 316L alloy. Each test used the same 316L baseline material and was exposed to varying periods of humidity.

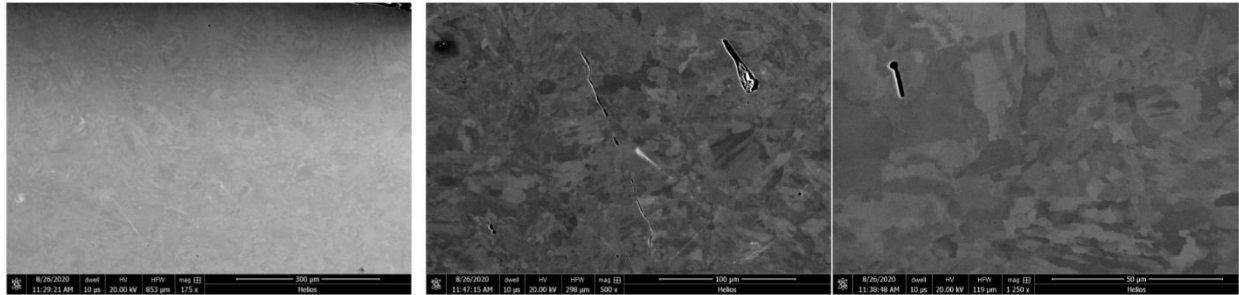


Figure 38: 30 day humidified SEM images displaying a present increase in porosity after 30 days of humidity exposure of 90% humidity at 40°C.

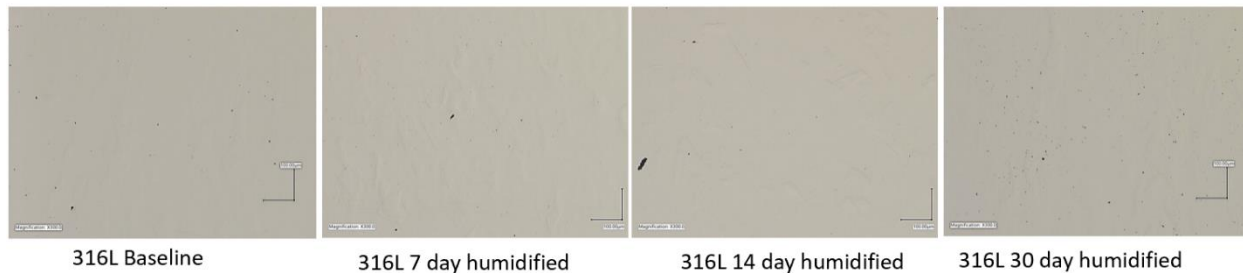


Figure 39: The environmental exposed experiment, the images show the 316L baseline subjected to increasing periods of controlled humidity of 90% at 40°C.

The EEET results have shown that there is an increased porosity after 30 days of humidity exposure, but this has not significantly affected the mechanical properties. To improve powder stability and shelf life when exposed to humidity, atomic layer deposition (ALD) coatings were evaluated on the stainless steel powder. The 316L powder was subjected to three different ALD treatments; Al_2O_3 , Fluorine terminated Al_2O_3 , and Amine terminated Al_2O_3 to evaluate moisture resistance, and printability effects. The 316L powder coated with amine terminated and fluorine terminated ALD was designed to create hydrophobic powder. Table 14 shows aluminum content measured by inductively coupled plasma (ICP) analysis, moisture content, and thermogravimetric analysis (TGA) oxidation onset temperature results from 30°C to 800°C. Humidification was performed at 75% relative humidity at 70°C for 24hr. The coatings all increased the onset oxidation temperature demonstrating improved oxidation resistance. However, the coatings did not appear to help reduce moisture content after humidification. Paradoxically, the fluorine terminated alumina coatings showed marked moisture increases even though they were designed for hydrophobicity.

Table 14: Moisture results for ALD coated and baseline powders.

Description	Aluminum ICP [ppm]	Moisture before humidification [ppm]	Moisture after humidification [ppm]	Onset oxidation after TGA [°C]
Uncoated 316L Baseline material	805	4.9	12.6	547
~0.5nm Al ₂ O ₃ ALD Coated	865	5.4	21	562
~2.5nm Al ₂ O ₃ ALD Coated	1060	2.6	20.9	623
~5nm Al ₂ O ₃ ALD Coated	1360	2.4	17.1	596
~7nm Al ₂ O ₃ ALD Coated	1500	1.4	22.9	675
Fluorine terminated ~2.5nm Al ₂ O ₃ ALD Coated	1040	0.9	13.1	599
Fluorine terminated ~5nm Al ₂ O ₃ ALD Coated	1210	0.6	501.9	694
Fluorine terminated ~7nm Al ₂ O ₃ ALD Coated	1480	2.5	201	741
Amine terminated ~2.5nm Al ₂ O ₃ ALD Coated	1050	0.7	11.3	559
Amine terminated ~5nm Al ₂ O ₃ ALD Coated	1290	1.3	9.3	616
Amine terminated ~7nm Al ₂ O ₃ ALD Coated	1480	5.2	13	696

Each atomic layer depiction (ALD) coated 316L powder was printed into a standard set of tensile bars and cubes for test alloys (Figure 40). Density, hardness, microstructure, and mechanical properties was evaluated and compared to the 316L baseline in the as-printed and environmentally altered over 30-days states. The printing of ALD coated materials can open ideas of adding alloying elements to the AM powder's surface that can provide environmental resistance and intriguing alloy design opportunities.

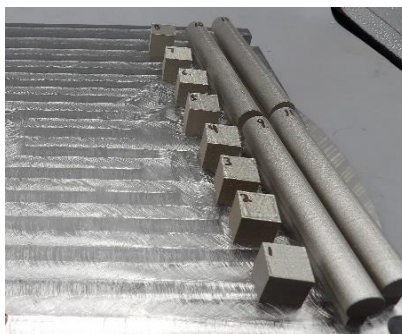


Figure 40: The Al₂O₃ ALD coated 316L print, built to understand if ALD can perform in metal Additive manufacturing.

Table 15: List of the average mechanical properties measured for the 316L baseline and ALD coated 316L samples. Tensile bars printed in the XY-Direction.

Material	Samples tested	YS (KSI)	YS Std. Dev. (KSI)	UTS (KSI)	UTS Std. Dev. (KSI)	Elongation %	Elongation% Std. Dev.
316L baseline	8	88.0	0.4	101	0.6	54.6	0.57
ALD-Al ₂ O ₃ coated 316L baseline	4	88.8	0.6	100	0.4	43.3	5.1
ALD-Al ₂ O ₃ amine terminated coated 316L baseline	4	88.8	1.1	100	0.8	41.4	9.4
316L baseline 30 days of humidification	4	86.6	0.6	101.3	0.6	52.2	0.8
ALD-Al ₂ O ₃ coated 316L baseline 30 days of humidification	4	87.4	0.8	100.2	1.5	39.0	20.3
ALD-Al ₂ O ₃ amine terminated coated 316L baseline 30 days of humidification	4	88.7	0.7	100.9	0.2	47.9	3.1

It was also observed that after humidification, the coated powders did not maintain the flowability typically required of AM powder. After 30 days of humidification, the coated powders became clumpy and hard; this was however not observed with the uncoated 316L baseline powder. Figure 41 and Figure 42 display the effects of the humidification on both coated powders compared to the uncoated baseline. To prepare these powders for printing, they will need to be both tumbled and sieved to break up the clumps.



Figure 41: Stainless steel powder after 30-day humidification. 316L baseline coated in 7nm of Al₂O₃ (left), 316L baseline coated in 7nm of Al₂O₃ amine-terminated (middle), and 316L baseline (right).



Figure 42: ALD-coated powders after humidification. 316L baseline coated in Al₂O₃ amine terminated (left); 316L baseline coated in Al₂O₃ (right).

Further, ALD-coated stainless steel microstructures were evaluated before and after 30 days of humidification. The goal was to understand the effects of humidity on ALD coated stainless steel powder and its usability in L-PBF. Figure 43 displays cubes printed with Al₂O₃ coated powder before and after 30 days of humidity exposure. Each cube was built with the same laser parameters during printing as a

control to isolate the powder as the changing factor. The porosity appears to have increased post-humidification for this coated material. Figure 44 displays the cubes printed with Al_2O_3 amine-terminated coated powder before and after 30 days of humidity. These cubes' porosity, after humidification, visually appears to increase after printing compared to the control. The microstructures for both sets of cubes appear to have been affected by humidity, with the Al_2O_3 amine-terminated steel cube having visually less porosity across its surface.

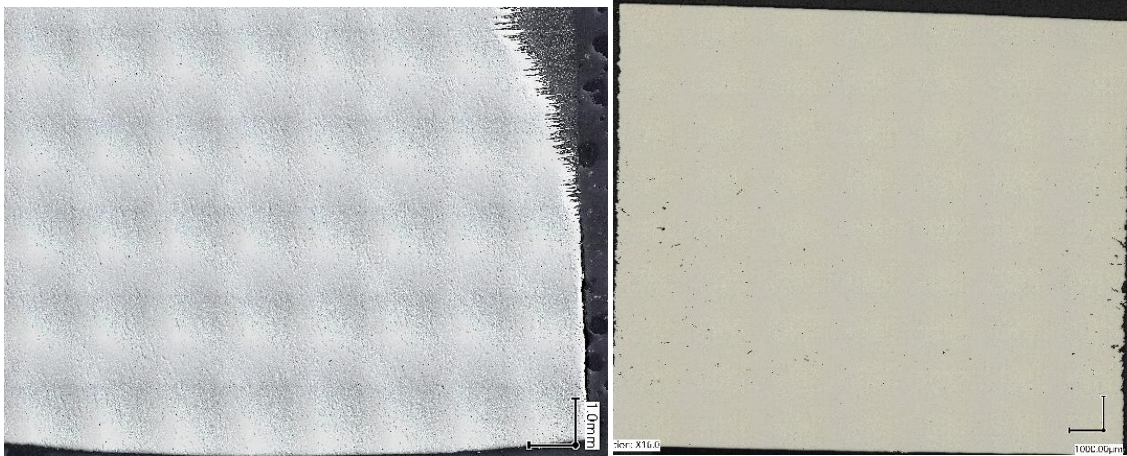


Figure 43: Printed Al_2O_3 coated 316L stainless steel cube before humidification (Left) and after 30 days humidification (Right). The left image pattern is an artifact of the digital image stitching.

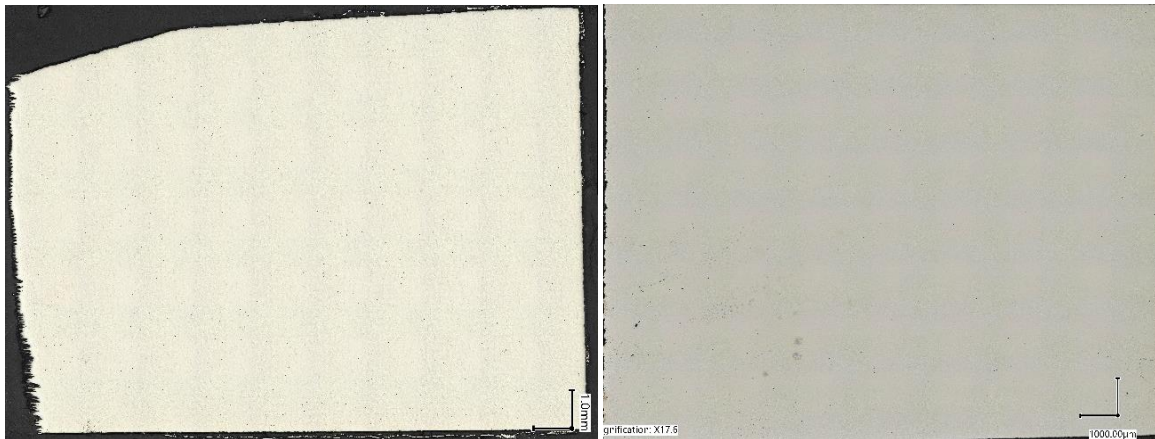


Figure 44: Printed Al_2O_3 amine-terminated coated steel cube before humidification (Left) and 30 days after humidification (Right).

4.1.2.2 Corrosion Resistance (Stainless Steel Alloy)

Corrosion testing was conducted on the 3XX-TiAM-machined, 3XX-TiAM AS-printed, 316L baseline, artificially-humidified 316L baseline (humidified for 30 days at 90% relative humidity at 40 °C), and commercial wrought 316L. Figure 45 shows the materials' performance (from a testing procedure adhering to ASTM G5) in corrosion rate electrochemical measurements. The polarization curves represent the anodic (corroding) half-cell, and the intersection of these curves with a cathodic polarization curve (e.g. H^+ or O_2 reduction), would represent the corrosion current in the x-axis. This

would then be proportional to the degradation rate as per Faraday's law. In other words, an anodic polarization curve pushed towards the left would result in a lower corrosion rate. From the results the following trends can be identified: After the 6 tests were performed, the AM produced 316L exhibited greater corrosion resistance than the commercially purchased wrought samples; Furthermore, the 3XX-TiAM Machines sample, has steeper slope, which would result in a more consistent rate of degradation if subjected to different corrosion conditions (e.g. varying pH or O₂).

The jagged nature of the plots is related to the surface finish of the samples; the rougher the surface, the more jagged the plot appears. The 3XX-TiAM was machined to a mirror finish when tested and results in a smooth line. The test gave a preliminary understanding of the corrosion properties of the AM 316L. Six tests were conducted with two being wrought 316L controls and 4 being printed 3XX stainless steel, but the AM produced samples all measure lower than the wrought samples. The results suggest that AM-produced 3XX stainless steel has improved corrosion properties. More research is needed to understand corrosion more closely in AM and how microstructure, and final composition is related to the results. This could involve examining the corrosion products and the post-polarization microstructure. An investigation into improving surface finish may also improve corrosion resistance.

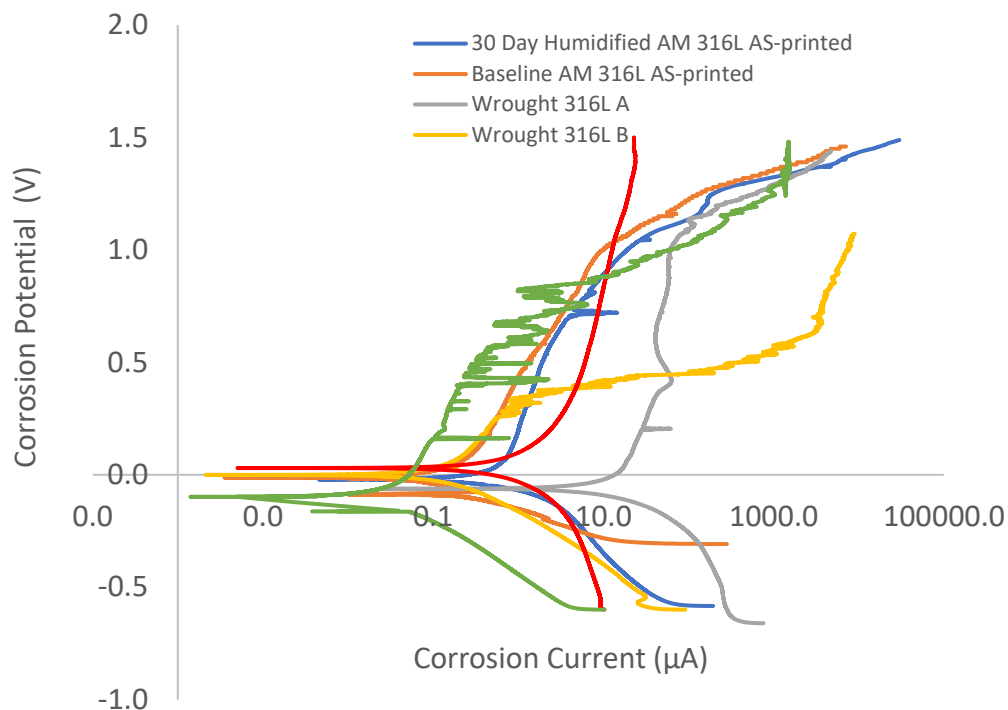


Figure 45: Corrosion Potential (V) vs Corrosion Current (μA). The plot shows that both the AM 316L—the baseline—and 30-day humidified samples performed better than the wrought 316L sample. The overall resistance generated is less in the AM parts than the wrought.

4.1.3 Property Reproducibility Tensile Testing (3XX-TiAM)

Over the past two years, the team put together two material feedstocks: a 3XX stainless steel and an aluminum alloy. After intensive ICME work, a decision was made to improve 316L stainless steel and Al5083 alloy. The Phase II end goal was to have two material feedstocks that had improved isotropic mechanical properties largely insensitive to build orientation and differences between printers. After designing these alloys, titanium was added to the stainless steel to improve the isotropic properties of the material through grain refinement. Table 16 shows the average tensile results of the 3XX-TiAM across the three L-PBF printers used in the Phase II. Figure 1 shows a micrograph of the first deliverable build from printer 1. The 3XX-TiAM shows minimal porosity across the surface of the density cube and shows that the material can be printed using EOS's default parameters for stainless steel. The 3XX-TiAM can therefore be successfully printed with minimal or no changes to industry standard laser parameters for 316L. The sample strength is in line with AM-standard 316L. The greatest variation seen in the 3XX-TiAM was for elongation in the Z-direction across the builds. Figure 47 displays the four builds of 3XX-TiAM material. The parts were produced to evaluate the isotropic behavior of the developed material. The vertical samples shown in Figure 47.B were removed from the mechanical properties evaluation because the parts were adversely affected during the printing due to printer equipment errors. Figure 47.C shows the build printed to replace these failed bars.

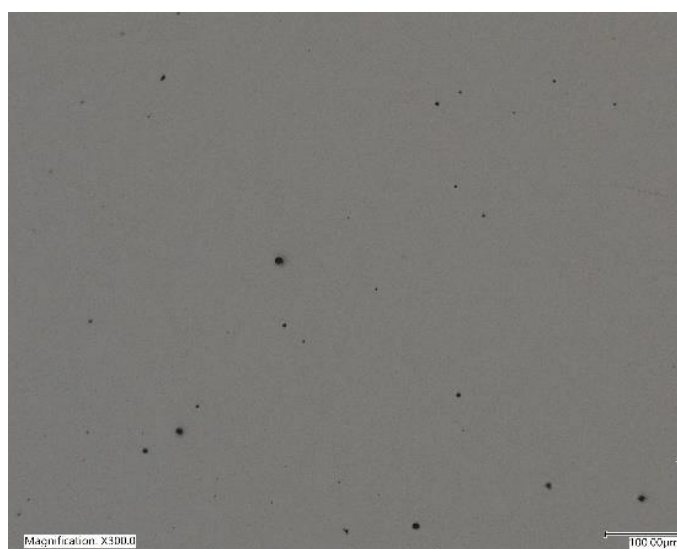


Figure 46: 3XX-TiAM Stainless steel micrograph from printer 1.

Table 16: Average tensile properties for 3XX-TiAM across the three printers, consisting of 27 and 18 samples printed in the XY direction and Z direction, respectively.

	YS (ksi)	UTS (ksi)	El%
3XX-TiAM	69.7 +/- 3.6	91.5 +/- 4.6	47.4 +/- 16

The stainless steel 3XX composition that was chosen for future work with AM is seen in Table 17. The selected composition resulted in a finer microstructure after printing, increased ultimate tensile strength (UTS) and fatigue life, and better utilization of nitrogen-rich printing environments from the addition of elemental titanium. The 3XX-TiAM range in Table 17 would be atomized by Praxair Surface

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Technologies and the composition in Table 18 would be created for the stainless-steel feedstock for statistical evaluation.

Table 17: 3XX-TiAM stainless steel composition specification.

3XX-TiAM	Fe	Cr	Ni	C	N	O	Si	Ti	P	S	Mn	Cu	Mo
WT%	Bal	17-18	11-14	0.03	0.09	0.03	1	0.5-0.8	0.045	0.03	1	0.01	2.0-3.0
Single Numbers are given as maxima													

Table 18: The resulting composition of the atomized 3XX-TiAM stainless steel produced by Praxair.

	Fe	C	Cr	Cu	Mn	Mo	N	Ni	Ti	O	P	S	Si
3XX-TiAM	Bal.	0	17.4	0.01	0	2.4	0.01	12.6	0.5	0.02	0.007	0.01	0

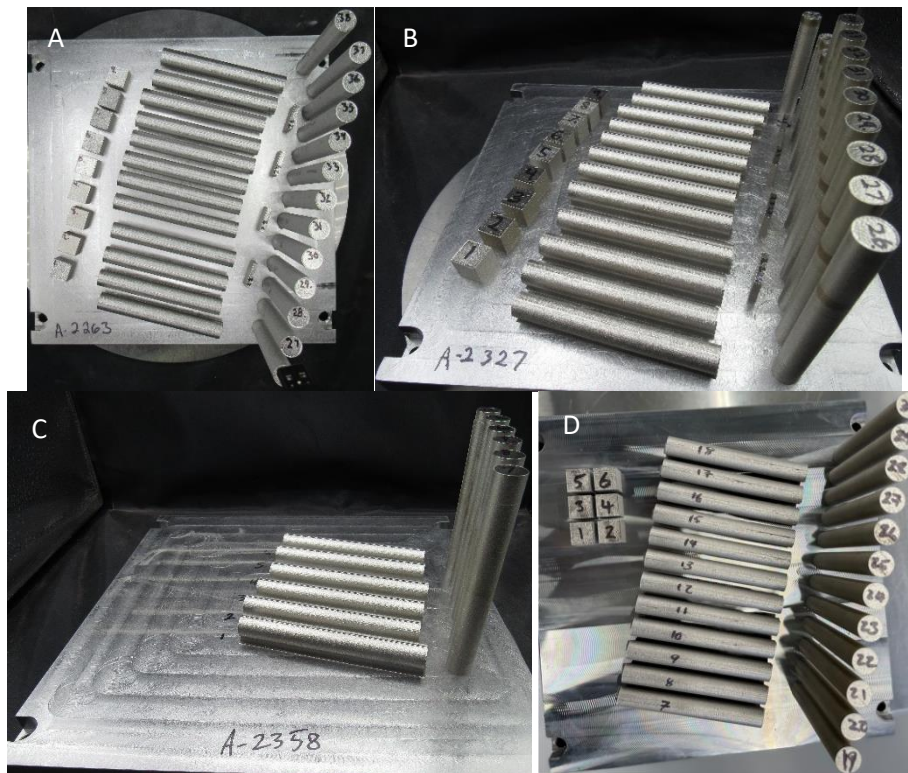


Figure 47: Four builds printed on three different printers to evaluate the isotropic properties of 3XX-TiAM. A) Printer one: EOS M290. B and C) Printer two: EOS M290. D) Printer three: EOS M280 (printed at Georgia Tech).

Figure 48 shows the three 3XX-TiAM prints compared to industry standards and Phase II results. Table 19 outlines the resulting mechanical properties for the all the 3XX-TiAM mechanical testing and resulting coefficients of variation. The 3XX-TiAM gives comparable mechanical properties results to industry standard printed 316L but shows a great improvement in isotropic properties irrespective of

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the printer. Notably, the differences in ultimate tensile strength (UTS) between XY- and Z-printed samples produced using three different printers were within 4 ksi. On average, the coefficients of variation for specimens from each printer/orientation combination are less than 1%, suggesting a high degree of repeatability in mechanical properties. The ability of 3XX-TiAM to produce reliably isotropic mechanical properties will boost confidence and trust in the performance of final-use components made from the material. Every 3XX-TiAM sample was tested in the as-printed state; this put the material front and center in evaluating the performance of L-PBF for producing consistent tensile results. Table 19 shows clearly that 3XX-TiAM outperformed 316L materials supplied by EOS and Carpenter Additive in XY versus Z-direction printing for mechanical testing. The ratio for equivalent XY and Z-direction UTS performance of printed parts increased from 81-91% for commercial 316L offerings for AM up to 96-98% for 3XX-TiAM.

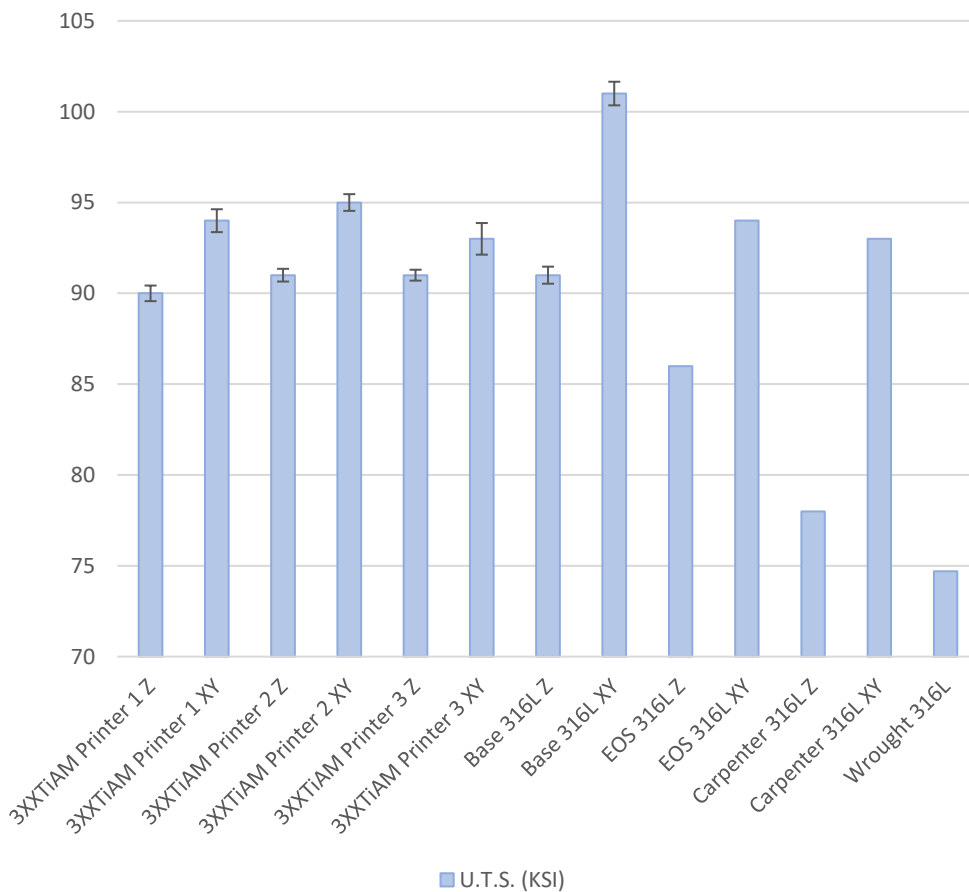


Figure 48: Bar graph representing the Ultimate tensile strengths taken during Phase II.

Table 19: 3XX-TiAM tensile results compared to wrought 316L, EOS 316L, Carpenter Additive 316L (“Base 316L” refers to 316L from Carpenter printed by E3D), and Base 316L + Ti. Data from vendor-supplied data sheets are indicated. Coefficients of variation are presented for the tested materials.

Material	Print Atmosphere	orientation	Y.S. (KSI)	U.T.S. (KSI)	UTS CV	El%
3XX-TiAM Printer 1	Nitrogen	Z	66 ± 0.63	90 ± 0.43	0.48%	43 ± 17
3XX-TiAM Printer 1	Nitrogen	XY	73 ± 0.53	94 ± 0.63	0.67%	54 ± 11
3XX-TiAM Printer 2	Nitrogen	Z	67 ± 0.24	91 ± 0.35	0.38%	53 ± 6.1
3XX-TiAM Printer 2	Nitrogen	XY	74 ± 0.59	95 ± 0.46	0.48%	56 ± 10
3XX-TiAM Printer 3	Nitrogen	Z	67 ± 0.17	91 ± 0.30	0.33%	43 ± 14
3XX-TiAM Printer 3	Nitrogen	XY	72 ± 0.20	93 ± 0.87	0.94%	50 ± 8.3
Base 316L Printer 1	Argon	Z	76 ± 0.23	91 ± 0.47	0.52%	67 ± 1.2
Base 316L Printer 1	Argon	XY	88 ± 0.42	101 ± 0.65	0.64%	55 ± 0.6
Base 316L Printer 1	Nitrogen	XY	80 ± 7.1	101 ± 0.6	0.59%	39 ± 7.6
316L EOS Data Sheet	Argon	Z	71	86	-	40
316L EOS Data Sheet	Argon	XY	80	94	-	45
Carpenter Data Sheet	Argon	Z	68 – 86	86 – 100	-	30-70
Carpenter Data Sheet	Argon	XY	68 – 86	70 - 86	-	25-55
Wrought 316L	-	-	29.7	74.7	-	60

Table 20 shows the statistical analysis of 3XX-TiAM UTS values after three prints of this material and shows that the differences in UTS between sample print orientation and printers are largely statistically significant at the 95% confidence level. Specifically, 3XXTiAM samples printed between printer 1 & 2, printer 2 & 3, and in different orientations have statistically significant differences in UTS; however, a comparison between the UTS of samples printed on printers 1 and 3 did not show a significant difference in UTS. Given the very low variability of the print process, as evidenced by the low standard deviation in UTS values for a given sample set, it is not surprising that the UTS differences are statistically significant. Practically speaking, the UTS values are within a very small range between different printers and print orientations. Perhaps most surprising is that even with such low variability and a relatively large sample size, printers 1 and 3 produced samples whose UTS could not be distinguished statistically. This also points broadly to the consistency of the process in producing reliable mechanical properties superior to wrought.

Table 20: The statistical analysis conducted on the 3XX-TiAM UTS comparing the printers used and the build orientation.

UTS (ksi)								
Printer 1-2			Printer 1-3		Printer 2-3		Orientation	
t-Test: Two-Sample Assuming Unequal Variances			t-Test: Two-Sample Assuming Unequal Variances		t-Test: Two-Sample Assuming Unequal Variances		t-Test: Two-Sample Assuming Unequal Variances	
	Variable 1	Variable 2	Variable 1	Variable 2	Variable 1	Variable 2	Variable 1	Variable 2
Mean	91.96	94.02	92.0	92.1	94.0	92.1	94.3	90.4
Variance	5.50	3.00	5.5	2.1	3.0	2.1	0.8	0.6
Observations	17	16	17	12	16	12	27	18
Hypothesized Mean Difference	0		0		0		0	
df	29		27		26		39	
t Stat	-2.88		-0.25		3.13		15.47	
P(T<=t) one-tail	0.00		0.40		2.16E-03		1.6E-18	
t Critical one-tail	1.70		1.70		1.71		1.68	
P(T<=t) two-tail	0.01		0.80		4.3E-03		3.1E-18	
t Critical two-tail	2.05		2.05		2.06		2.023	
Reject Null? Yes.			Reject Null? No. (-2.05 < -0.25 < 2.05)		Reject Null? Yes.		Reject Null? Yes.	
Printers 1,2 produce statistically distinct UTS.					Printers 2,3 produce statistically distinct UTS.		Orientation produces statistically distinct UTS.	

Table 21 displays the XY:Z ratios of the UTS of printed tensile samples. The results indicate that the 3XXTi-AM could achieve a closer XY:Z ratio of mechanical properties than the base commercial 316L used in Phase II (from Carpenter Additive) and the AM industry standard 316L offered by EOS. The 3XXTi-AM material was able to improve the isotropy of as-printed mechanical properties across three different L-PBF systems. Overall, the simple change in composition made by adding titanium to 316L has greatly improved the achievable isotropic properties of stainless-steel components printed using L-PBF. The new 3XX-TiAM material is still in its infancy, and a plan for commercialization of this material would involve printing from multiple lots of atomized powder and microstructure characterization to develop upper and lower limit alloy specifications as well as creating a large statistical data set to meet S-basis standards outlined in the Metallic Materials Properties Development and Standardization (MMPDS) handbook.

Table 21: The average UTS ratio for XY and Z-direction tensile results.

	Average UTS XY- Direction (ksi)	Average UTS Z- Direction (ksi)	UTS Z:XY ratio
3XX-TiAM Printer 1	94 ± 0.63	90 ± 0.43	95.7%
3XX-TiAM Printer 2	95 ± 0.46	91 ± 0.35	95.8%
3XX-TiAM Printer 3	93 ± 0.87	91 ± 0.30	97.3%
3XX-TiAM Average	94 ± 0.88	90 ± 0.80	95.8%
Base 316L Pinter 1	101 ± 0.65	91 ± 0.47	90.1%
EOS 316L Data sheet (40µm)	94	86	91.5%
Carpenter Additive 316L	86 – 100	70 – 86	81% - 86%

Table 22 shows how 3XX-TiAM compares to commercial wrought 316L and powder 316L for AM. The 3XX-TiAM gives UTS values that are 123% of current ASM handbook volume 6-reported wrought 316L values and 102% of standard AM 316L offerings by EOS and Carpenter Additive. [4] [5] The goal of developing the 3XX-TiAM was to make a material that performed to 90% of industry standards; however, it was able to perform better than current wrought material by a significant margin. The 3XX-TiAM performs in a manner consistent with commercial AM offerings in terms of UTS, but well exceeds commercial AM offerings in terms of mechanical isotropy.

Table 22: 3XX-TiAM UTS compared to wrought commercial, AM Commercial, and the Phase II base 316L (Carpenter Additive 316L printed in house at E3D).

	UTS Wrought 316L	UTS EOS Commercial 316L	UTS AM Base 316L
3XX-TiAM Printer 1	123%	102%	92%
3XX-TiAM Printer 2	121%	101%	91%
3XX-TiAM Printer 3	123%	102%	92%
316L plus Ti	129%	107%	96%
Base 316L	133%	111%	100%
EOS 316L 40um	129%	100%	90%

4.2 New High Strength Aluminum Development (A5083-RAM7)

The next alloy target was development of an aluminum alloy with highly-reliable high-strength properties without the need for post process heat treatment such as aluminum alloy 5083 which is used extensively in the military in applications ranging from armor to fuel storage. High strength wrought alloys fitting these criteria, such as aluminum alloy 5083, suffer from severe solidification cracking (hot tearing) during fusion-based AM processes. ICME calculations evaluated the effects of alloy composition centered around aluminum alloy 5083 to reduce susceptibility to solidification cracking and produce consistent and high tensile strength.

4.2.1 Integrated Computational Materials Engineering (Aluminum Alloy)

E3D and CSM have considered a range of different material types, including high strength low alloy steel, ferritic or martensitic stainless steel, copper based, and aluminum-based alloys. Through discussion, the team concluded that an aluminum alloy might be best suited as a second material for this project. Aluminum alloys can offer high strength to weight ratios as well as economic part production due to high print speeds and low volumetric feedstock costs. High strength aluminum alloys designed for wrought processing typically suffer from hot tearing in fusion-based AM processes and therefore are often considered unprintable. Elementum 3D has developed reactive additive manufacturing (RAM) methods that enable the creation of printable and reliable high strength aluminum alloys. Elementum 3D has already commercially released RAM versions of aluminum alloy 6061 and 2024 as well as other aluminum based alloys that offer excellent printability and performance in standard commercial L-PBF AM systems.

The team has conducted CALPHAD analysis on a second alloy system to investigate predicted phases and examine the effects of compositional changes. The team studied compositions based on Elementum 3D's RAM aluminum alloys that have been demonstrated to offer good printability, high strengths, and good wear resistance. These aluminum alloys contain ceramic reinforcements within the alloy matrix which favorably affect the solidification behavior during the LPBF process. The CALPHAD simulation will investigate the compositional space around an A1000-RAM10 aluminum alloy to develop a reliable, light weight, general purpose alloy with good strength without need for post process heat treatment.

The CALPHAD calculated phase fractions are displayed in Figure 49 over a wide temperature range for this aluminum matrix composite.

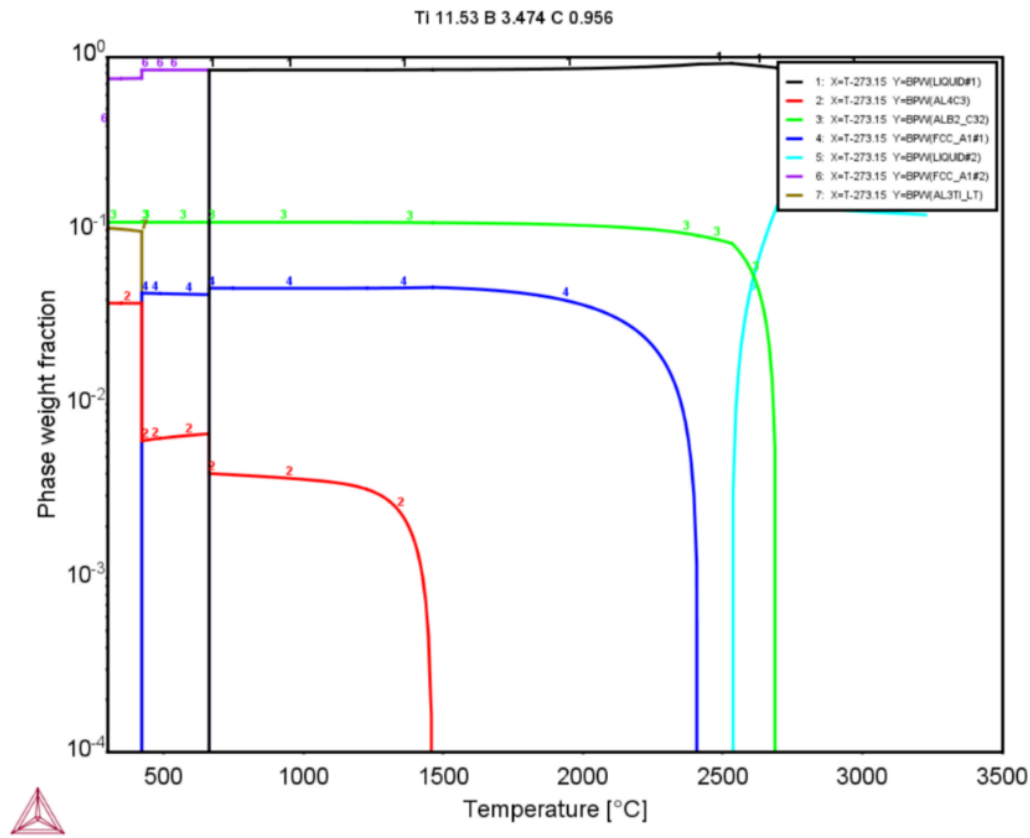


Figure 49: CALPHAD simulation created for an aluminum metal matrix composite revealing the predicted phase fraction changes with temperature.

The same methodology developed for alloy system 1 was utilized starting with the CALPHAD and ML framework and then followed by experimental testing and verification.

High strength aluminum alloys are traditionally produced by wrought processes, but these alloys often suffer from hot tearing solidification cracking making them unsuitable for fusion-based AM processes. The team is investigating aluminum alloy systems computationally with potential to offer good printability as well as good strength. Another goal is to achieve these properties without the need for a post process heat treatment, good weldability, and more isotropic microstructure and properties. Wrought alloys based on 3XXX and 5XXX series aluminum use Mn and Mg additions respectively to strengthen the alloy [3]. Both alloys use Mg and Mn in different concentrations with other alloying elements to create standard alloys; i.e., 3003 and 5083.

Aluminum alloys based on the 5XXX series are promising candidates because they can provide good strength without need for precipitation strengthening heat treatments. The team has selected alloys 5083, 5086, and 5182 as baselines for computational investigation and literature property validation. These selected alloys have similar compositions with varying manganese, magnesium, and chromium content and are expected to have variation in susceptibility to hot tearing as well as different mechanical properties. The higher strength alloys like 5083 are known to suffer from extensive hot tearing during fusion AM processes leading to poor printability and unsuitable mechanical performance. Elementum 3D's reactive additive manufacturing (RAM) methods will also be investigated for aluminum

alloy design. This RAM process has been utilized for other aluminum alloys to greatly refine the grain structure and eliminate hot tearing. Figure 50 shows the grain refinement and hot crack prevention in E3D's 6061-RAM2 compared to the standard wrought AA6061 composition.

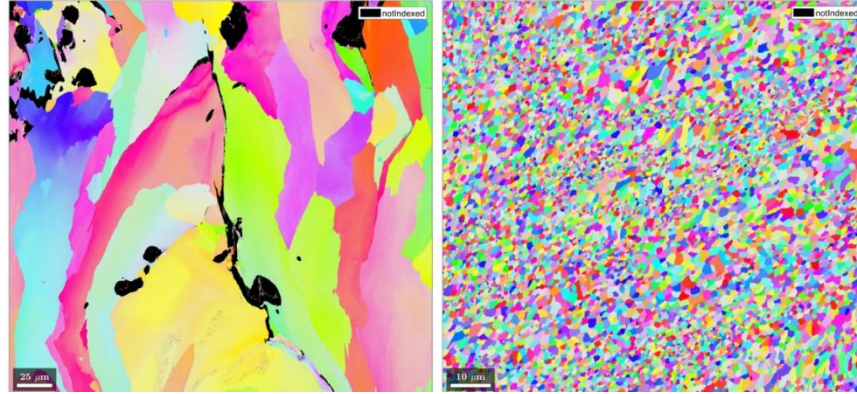


Figure 50: (Left) Traditional 6061 aluminum alloy composition printed by laser powder bed fusion (L-PBF) shows large elongated grains and solidification cracking (hot tearing). (Right) Elementum 3D's 6061-RAM2 aluminum alloy printed by L-PBF shows a fine equiaxed grain structure that offers excellent printability and properties.

A CALPHAD modeling framework (AMALLOY) has been built on the success of the CALPHAD modeling used to identify phenomena important to printability of steel alloys. Earlier modeling revealed a low temperature oxide (slag) phase, high temperature BCC (δ -ferrite) phase, and a variability in the solidification range (mushy zone) as a function of composition. These three phenomena became the foundation for an experimental design centered around optimizing printability and mechanical property heuristics based on the presence or absences of slag, the presence or absence of δ -ferrite, and a small, medium, and large mushy zone. In the latest modeling-based development work, this idea has been extended to improve the predictive performance of CALPHAD simulations for aluminum.

Where earlier optimization of the steel alloy depended on an experimentally evaluated figure of merit, the goal of AMALLOY is to extend this concept to a calculable figure of merit. This simplifies the optimization process by relying on relative measurements between simulated quantities, which are often more reliable than the absolute values of those quantities. The ThermoCalc CALPHAD simulation software exposes a python application programming interface (API) that allows CALPHAD simulations to be executed and examined programmatically. Approximately 50 phase equilibria, physical, kinetic coefficient, solidification, mechanical diffusion and precipitation properties were evaluated to determine the subset of calculable properties—properties such as hot cracking susceptibility (sometimes referred to in literature as hot tearing tendency), yield strength, and solidification range—that most suitably contribute to a figure of merit for printability and performance. Each step in the multi-step property calculation must be developed and tested independently. Over the course of this Phase II, the liquidus temperature, yield strength, and hot cracking susceptibility steps have been developed and tested. Additional work was needed to more fully automate thermodynamic driving force and precipitation kinetic calculations before these properties can be used in the final figure of merit. In parallel, the software framework that generalizes the execution and analysis of these CALPHAD calculations has been developed and tested. Legacy data was used to perform checks and validations for these calculations to ensure similar trends between composition and printability/performance. Figure 51 highlights the workflow for the AMALLOY.

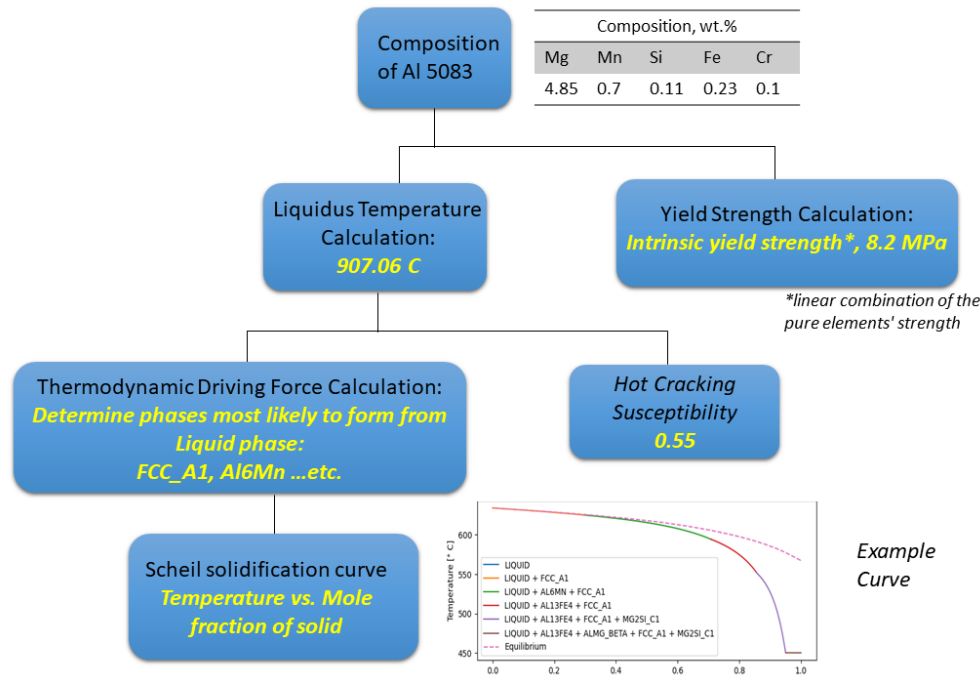


Figure 51: Workflow of AMALLOY. A merit function using yield strength and hot cracking susceptibility estimated from these simulations was used to determine an optimal composition for printability and performance.

The next step was to develop a Figure of Merit (FOM) that captures both quantitative physical properties and more qualitative processability metrics. To address weldability heuristically, the range of 5XXX series aluminum alloy compositions includes eleven known compositions and their corresponding filler metals. This information has been used to produce a weldability heuristic, “Average weldability” in Figure 51. The following logic has been used to develop a figure of merit:

- Some properties, such as elongation to failure, should be maximized⁴. For such properties, a heuristic $FOM \propto x$
- Some properties, such as yield strength, should be maximized but bounded. That is, they may prove detrimental if they become too large, so the increase in the merit function should approach an asymptote as the property increases past some target. Conversely, a reduction in these properties should be penalized heavily. Several functions display this type of asymptotic behavior, but we have chosen $FOM \propto 1 - e^{-\beta(x - x_{\text{target}})}$. Asymptotically, values over x_{target} benefit the figure of merit, values at the target have no impact on the FOM, and values below the target reduce the FOM.

The final FOM combines weldability (x_1), yield strength (x_2), ultimate strength–yield strength ratio (x_3), and elongation (x_4),

$$FOM = w_1 x_1 + w_2 (1 - e^{-0.3(x_2 - 17)}) + w_3 x_3 + w_4 x_4.$$

⁴ Maximization and minimization are mathematically equivalent: $\max x = \min -x$. Therefore, and without loss of generality, this description assumes all properties are to be maximized.

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The weights of each term are adjusted to ensure the “Alloy Ranking” order established in Table 23 for known alloys is respected. The resulting FOM will then be used as part of a metaheuristic algorithm (particle swarm optimization, PSO) to find an optimal alloy in the space spanned by these 11 AL 5XXX compositions. Table 23 outlines the ASM values for UTS, Yield Strength (YS), weldability average to rank the alloys, and the CALPHAD hot cracking susceptibility (HCS) analysis. The UTS and YS were used to create FOM and average weldability based on the alloys’ weld performance with 5183 filler metal. The alloys that ranked highest are 5086, 5083, 5182, and 5056. The results are show that many of alloys are highly weldable and the team will continue with our top selected to begin practical experimentation. Al 5083 was choose for its of use in DoD operation and is known to suffer from hot cracking when welding. Allowing the team to solve a microstructure problem and ICME can directly improve the weldability of a alloy with know welding issues.

Table 23: Weldability and UTS/YS calculations to create Figure of Merit for CALPHAD and machine learning. The UTS and YS were used to create a discriminating figure of merit. Weldability is based on the alloys’ literature reported weld performance with 5183 filler metal. The alloys that ranked highest are 5086, 5083, 5182, and 5056.

Alloy	Exp. FOM	CALPHAD HCS	Weldability & UTS/YS	Ranking
5456	0.74	0.54	2.85	3
5056	1.63	0.58	2.7	1
5182	0.94	0.63	2.85	3
5083	1.25	0.75	2.88	4
5086	0.97	0.8	2.99	5
5254	0.81	0.86	2.73	1
5154	0.81	0.88	2.73	2
5052	-1.42	1.03	2.88	4
5454	0.67	1.06	2.76	2
5652	-2.63	1.07	2.97	5
5252	-2.51	1.11	2.76	2

Figure 52 displays the liquidus temperatures for the aluminum alloys being developed through Thermocalc. The different colors outline the temperatures where certain phases begin to solidify. The red and green phases in Figure 52 represent titanium aluminide (Al_3Ti) intermetallics with higher melting temperatures than the aluminum alloy. Al_3Ti is a brittle intermetallic that is unwanted in the reactive additive manufacturing (RAM) process. These phases are created during the RAM process when the two RAM constituents react with the aluminum alloy instead of making the in-situ strengtheners and grain refiners. As a result, the Al_3Ti can hamper the performance of the developed alloy by lowering its mechanical properties. The ICME design will continue until powder is received from aluminum powder suppliers to develop the final 5XXX series up-alloyed L-PBF test alloy.

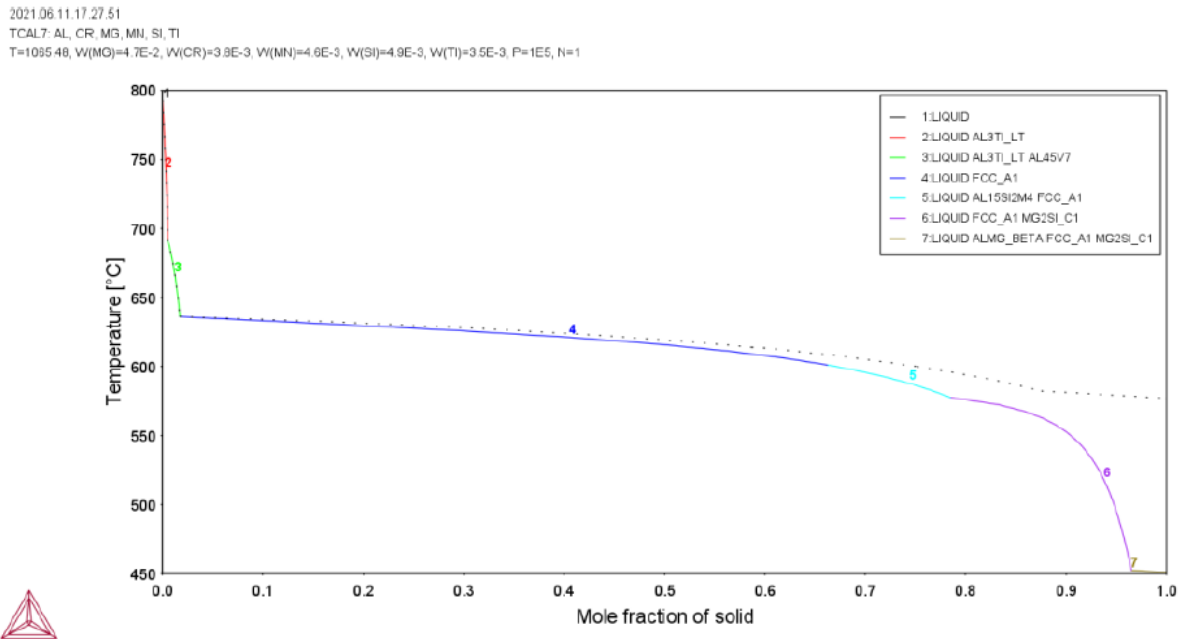


Figure 52: Phase diagram of aluminum alloys with RAM constituents, resulting from a ThermoCalc simulation conducted to understand the predicted phase's liquidus temperature.

To create the CALPHAD framework, Scheil, liquidus, DICTRA, and crack susceptibility simulations were conducted in ThermoCalc to be integrated into the machine learning program

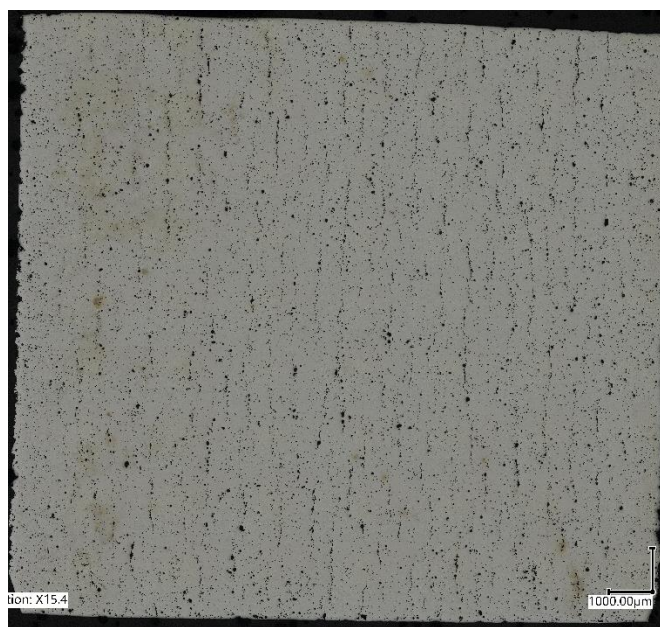
This sequence of calculations begins with melting temperature calculations to identify a composition-dependent starting point for the second step: Scheil calculations. Scheil calculations indicate phase stability as a function of temperature, providing a list of precipitate phases, their compositions, and their thermodynamically stable ranges. In the third step, the thermodynamic driving force is calculated for each precipitate phase 100 °C below when it first appears. While this does not explicitly capture the kinetics of the precipitate, it does provide a proxy for kinetics, as precipitates with a high driving force are more likely to have faster kinetics (i.e., the forward reaction is more energetically favored than the backward reaction). The final step is to calculate the hot cracking coefficient (or, the hot cracking susceptibility) of each composition. Although the initial FOM included hot cracking susceptibility as a criterion, this calculation is prohibitively expensive (computationally) with no hope of finishing the many hundreds of calculations required for a subsequent optimization within the project timeline. Therefore, the alloy composition was optimized on driving force (as a kinetics proxy for nucleation rate) and estimated yield strength. Rather than including hot cracking coefficient in the optimization, the hot cracking coefficient was instead calculated for the top candidates that resulted from multiple independent optimization calculations.

Elementum 3D had chromium, manganese, aluminum-magnesium, titanium, and silicon in stock to alter the composition if needed. Table 24 displays the composition being evaluated by ICME. The ICME simulations are identifying possible phases and the solidification behavior with RAM inoculants added to the Al 5083 composition.

Table 24: The Al 5083 minimum ASTM chemical compositions with RAM additions from 1 to 10 volume percent were used in the CALPHAD simulations to improve printability.

Alloy	Elemental Chemical Composition						
	Al	Mn	Mg	Cr	Ti	B	C
Al5083 Min	Bal	0.4	4	0.05	—	—	—
Al5083 RAM1% Min	Bal	0.39%	3.93%	0.05%	1.25%	0.09%	0.39%
Al5083 RAM2% Min	Bal	0.39%	3.86%	0.05%	2.48%	0.18%	0.78%
Al5083 RAM5% Min	Bal	0.37%	3.66%	0.05%	6.07%	0.43%	1.91%
Al5083 RAM7% Min	Bal	0.35%	3.54%	0.04%	8.38%	0.59%	2.63%
Al5083 RAM10% Min	Bal	0.34%	3.35%	0.04%	11.72%	0.83%	3.68%

Solidification cracking is a known problem when welding Al 5083. When joining Al 5083, Al 5183 filler metal -- which contains slightly elevated Mg and up to 300 ppm Be compared to 5083 to improve yield strength -- is used to overcome the solidification cracking. Unaltered Al 5083 powder was printed to understand how Al 5083 performs during printing. Figure 59 shows a L-PBF printed cube creates solidification cracking and large columnar grain growth in the Z-direction. The team's goal is to generate information from CALPHAD simulations and the RAM technology to create a solution to improve Al 5083 printability.

**Figure 53: Pure Al 5083 printed with no RAM inoculants.**

To address the issues with solidification cracking and columnar solidification mode in L-PBF processing, focus was given to achieve equiaxed solidification mode and low hot cracking susceptibility. Previous literature has shown inoculants to be successful in promoting grain nucleation and equiaxed grain formation during solidification. Ideally, these inoculants would not grow in the alloy melt, and would remain stable throughout solidification. Hence, the thermodynamic driving force to form these particles at high temperatures in the liquid (100 °C below melting points of inoculants), as well as the

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driving force near melting temperature of typical Al 5xxx alloys (700 °C) were chosen as indicators for good inoculation. Potential inoculants were identified for our calculations. Specifically, TiB_2 , Al_3Zr (D023) and Al_3Ti (LT) phases were selected as candidates for inoculation. It is hoped that these precipitates will also provide strengthening through grain refinement (Hall-Petch strengthening) as well as precipitate strengthening in the aluminum matrix.

To optimize printability and performance, hot cracking susceptibility and yield strength calculations in ThermoCalc® were initially planned to be included. However, when the efficacy of both of these calculations were compared with values found in literature, or experimental values, respectively, the yield strength module was determined to be ineffective in ranking the experimental yield strengths. To incorporate a solid solution strengthening contribution, a linear regression was done on the available yield strength values for ASTM Al 5xxx compositions. Following iteration[1], linear regression was used to estimate the yield strength on Mg composition. The following FOM equation was selected to optimize composition,

$$FOM = hcc - \Delta G(Al_3Ti; 1523.15 K) - \Delta G(Al_3Ti; 973.15 K) - \Delta G(Al_3Zr; 1753.15 K) - \Delta G(Al_3Zr; 973.15 K) - \Delta G(TiB_2; T_m) - \Delta G(TiB_2; 973.15 K) - YS_{SS}.$$

hcc is hot cracking susceptibility coefficient, $\Delta G(X; T)$ is the driving force calculation for composition X at temperature T , and YS_{SS} is the solid solution yield strength.

The particle swarm optimizer algorithm was used to run thermodynamic simulations. The goal of the optimizer was to minimize the cost function (FOM) over some input parameter space, in our case, composition. Since the hot cracking susceptibility calculations were highly computationally intensive, the hcc parameter was taken out of the FOM equation, and was performed separately on the optimized compositions by running the optimizer calculation multiple times. Table 26 shows the alloys identified for printing. Table 26 shows the alloys identified for printing.

The resulting calculations sit within a twelve-dimensional hypervolume defined by the composition. The complexity of this parameter space makes it difficult to interpret the effect of composition on the driving force, hot cracking coefficient, yield strength, and ultimately, FOM. Local linear embedding (LLE) was used to visualize all the optimized compositions, as well as RAM compositions developed by Elementum 3D and ASTM Al 5xxx compositions used for experimental rank ordering for yield strengths. Figure 54 shows the compositions used for LLE training. While information is lost when a 12-D space is projected on a 2D surface, as is the case with LLE, Figure 54 and Figure 55 do predict an improvement in the FOM for the RAM compositions over the ASTM compositions, and the PSO optimization seems to have identified additional compositions (those shown in Table 24) that may show improvement even over the RAM compositions.

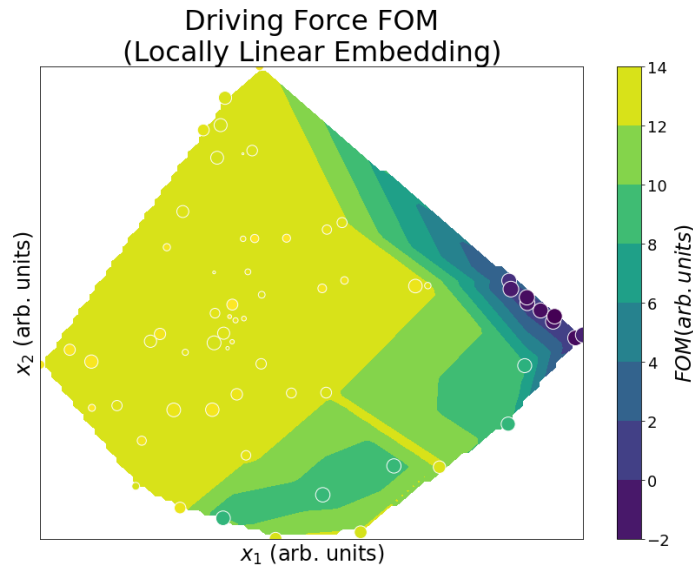


Figure 54: LLE training of the optimized, RAM and ASTM compositions.

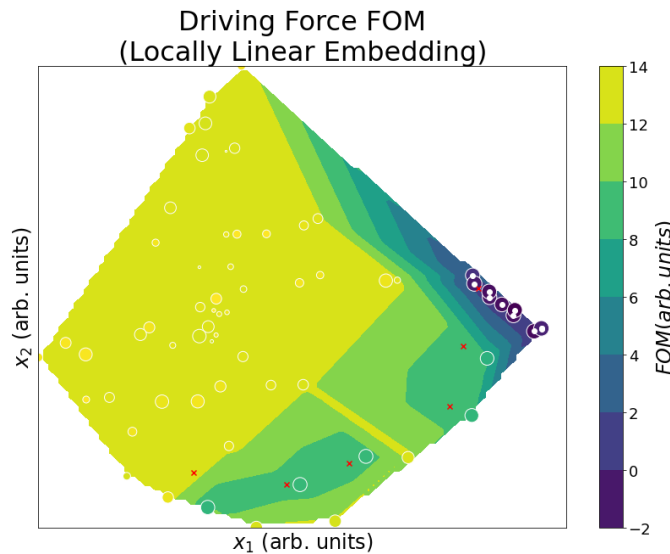


Figure 55: ASTM compositions (white colored circles) and RAM compositions (red x markers) are shown on the LLE plot.

4.2.2 Experimental Development (Aluminum Alloy)

The Al 5083 feedstock was atomized by Valimet, and the powder was found to contain a carbon contaminant after atomization. Figure 56 shows what is believed to be the contaminant within the material. The team accepted the powder with the contaminant present as the lead time needed to re-atomize would not allow the team to meet the final Al 5083 Phase II deliverables. The Valimet 5083 composition can be seen in Table 25. The material was delivered with a higher concentration of magnesium to offset the vaporization of Mg during printing. The printing of Al 5083 began with two and five percent reactive additive manufacturing (RAM) additions. The Al 5083 CALPHAD simulations have been adjusted because the current time frame will not allow the procurement of strontium and

zirconium metals needed to produce the predicted alloys. The Al 5083 material was printed with the RAM constituents instead of strontium and zirconium. Table 26 outlines the compositions predicted by CALPHAD to have a high figure of merit (FOM) for success. The team has agreed to create the two compositions – PSOs 3 and 7 – whose plots indicate the most robust optimization.

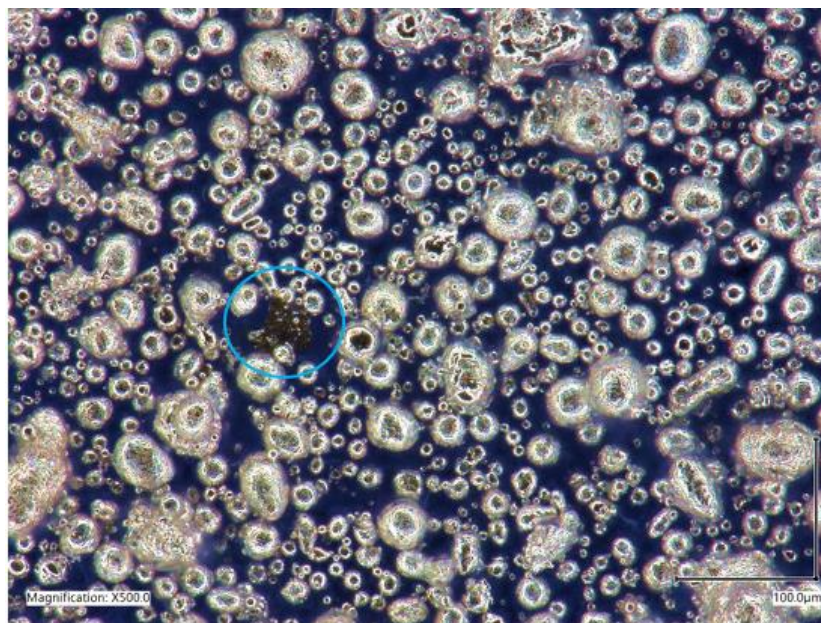


Figure 56: Micrograph of the Aluminum 5083 powder. The blue circle highlights the contaminant found in the powder.

Table 25: The Valimet ordered aluminum 5083 chemical composition.

Al	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn
94.0	0.17	0.01	0.12	4.9	0.73	0.06	0.01	0.01

Table 26: The modified Al5083 test alloys, selected for printing and improving the printability of Al5083. The compositions are presented in weight percent.

Material	Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	B	C	FOM
Al5083 RAM2 Max	Bal.	0.39	0.39	0.1	0.97	4.73	0.24	0.24	2.61	0.17	0.78	X
PSO 3	Bal.	0.15	0.45	0.19	0.84	9.81	0.1	0.2	7.05	2.15	0.85	12.58
PSO 7	Bal.	0.12	0.6	0.27	0.19	9.77	0.24	0.18	6.3	1.95	0.84	12.60
AL5083 RAM2	Bal.	0.06	0.12	0.01	0.73	4.9	0.17	0.01	2.61	0.73	0.2	X
A5083- RAM7	Bal.	0.06	0.12	0.01	0.73	4.9	0.17	0.01	8.47	2.50	0.67	X

The team has successfully printed four modified Al5083 alloys: two standard E3D approaches with the RAM constituents, and two ICME optimized strategies (see Table 27) The modified Al5083 alloys' performance was evaluated through density, microstructure, and mechanical properties

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measurements. The contaminant present in the Al5083 alloy has not been shown to significantly affect the printability of Al5083 after the composition modifications. After the second printing of each Al5083 composition, fully dense parts were achieved after each print. Table 27 outlines the density result obtained for the selected laser parameters.

E3D tested RAM inoculant volume fractions of 2% and 7%. A5083-RAM2 and A5083-RAM7 micrographs were collected for the selected laser parameters equaling an energy density of 51.4 J/mm³. Figure 58 and Figure 59 demonstrate the ability to print aluminum alloys in L-PBF with RAM constituents at 2% and 7% by volume, respectively. These micrographs show little porosity, with the ceramic and metal constituent distributed homogenously throughout the printed cube.

The ICME Al5083 alloy composition we chose to print were PSO3 and PSO7. Al5083 PSO3 and PSO7 micrographs are shown in Figure 60 and Figure 61. The energy density used for Al5083 PSO3 and PSO7 are 61.7 and 56.1 J/mm³, respectively. These micrographs show that a highly dense cube was produced with the added titanium, boron, magnesium, and other alloying elements. The ceramic constituents in this RAM composition appear as dark spots and present similarly to porosity in the images. Porosity in AM is spherical in nature, and the ceramic inoculant appears more angular. During the laser printing process, Al 5083 PSO3 and PSO7 showed a large amount of vaporization. It is expected that magnesium is vaporizing off due to its low boiling point of 1090°C. The team will evaluate the powder feedstock and printed products using SEM and inductively coupled plasma mass spectrometry (ICP-MS) testing on the modified Al5083 alloys to characterize the change in composition after printing.

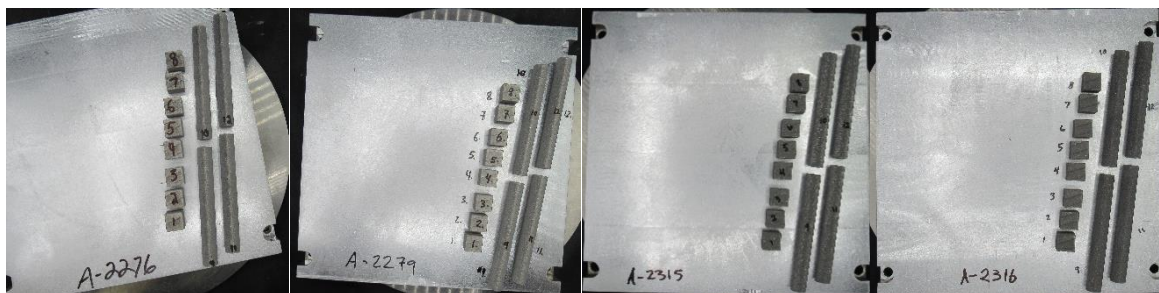


Figure 57: The four aluminum Al5083 test sample builds from left to right A5083-RAM2, A5083-RAM7, A5083-PSO3, and A5083-PSO7.

Table 27: The modified Al5083 test compositions' resulting laser parameter energy density, theoretical density, printed density, and standard deviation for the second development builds.

Test Alloy	Cubes Printed	Energy Density (J/mm ³)	Theoretical Density (g/cc)	Average Relative Density	Std. dev.
A5083-RAM2	8	47.4-56.1	2.70	99.83%	0.12%
A5083-RAM7	8	47.4-56.1	2.76	99.64%	0.24%
A5083-PSO3	8	47.4-58.7	2.64	99.85%	0.18%
A5083-PSO3	8	47.4-58.7	2.60	99.99%	0.17%

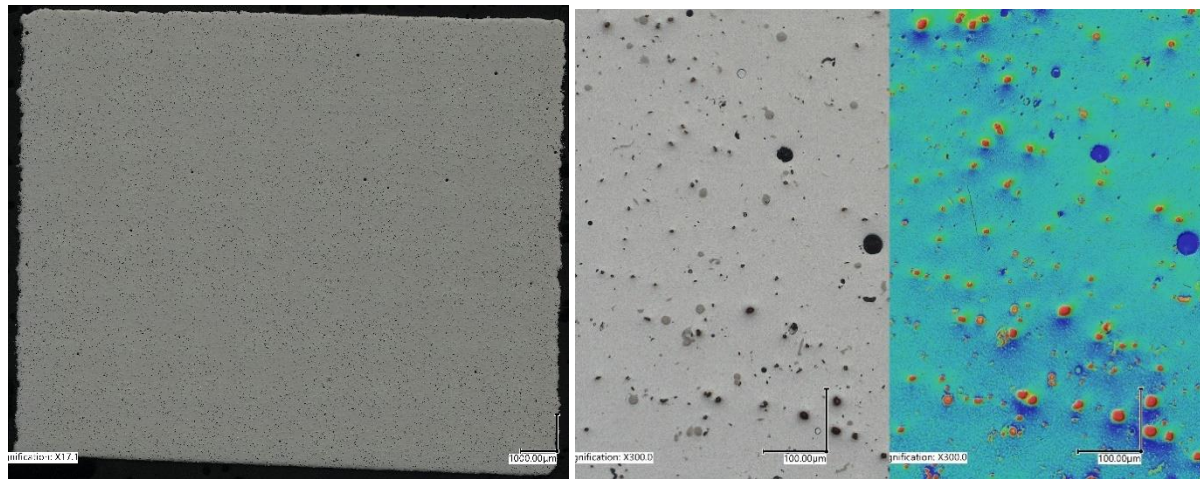


Figure 58: Al5083 RAM2 micrographs show a small degree of porosity from build A-2254 cube eight with an energy density of $51.4\text{J}/\text{mm}^3$. The right image highlights the ceramic constituents and the porosity, with the red spots being ceramic and the dark blue being pores. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube.

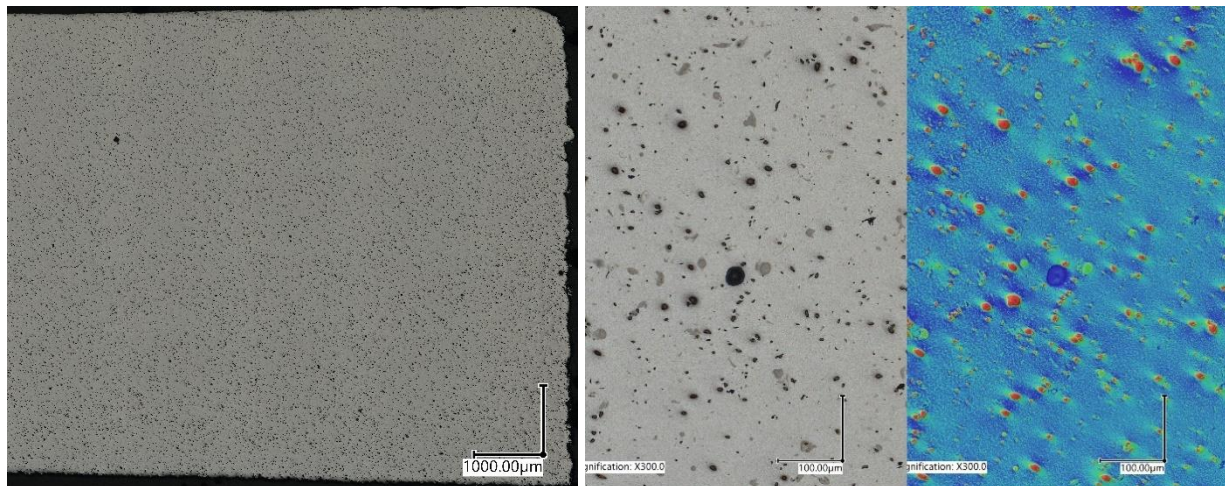


Figure 59: Al5083 RAM7 micrographs show a small degree of porosity from build A-2255 cube eight with an energy density of $51.4\text{J}/\text{mm}^3$. The right image highlights the ceramic constituents and the porosity, with the red spots being ceramic and the dark blue being pores. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube.

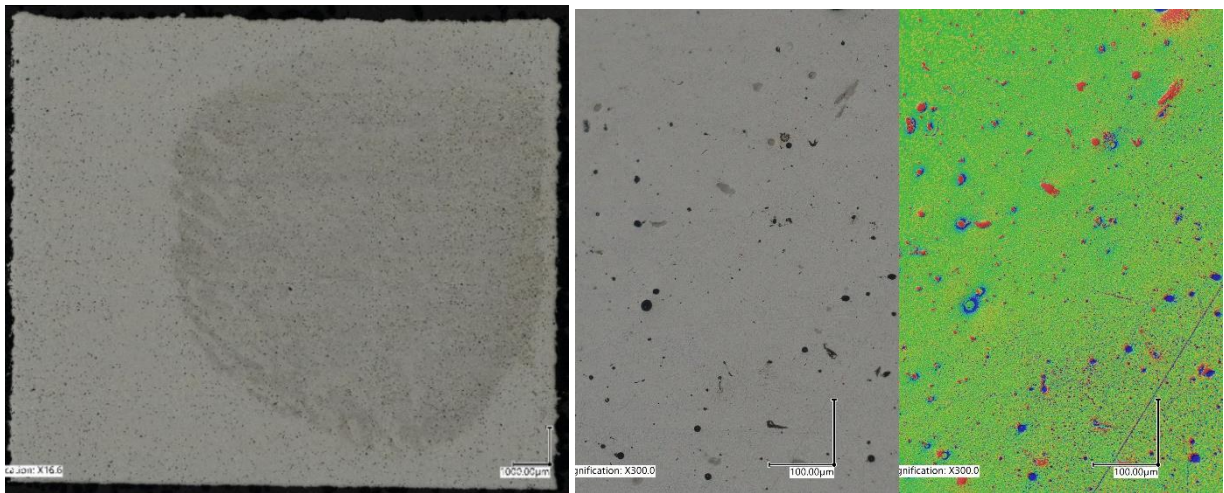


Figure 60: Al5083 – PSO3 micrographs show a small degree of porosity from build A-2296 cube seventeen with an energy density of 61.7 J/mm³. The right image highlights the ceramic constituents and the porosity, with the red spots being ceramic and the dark blue being pores. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube.

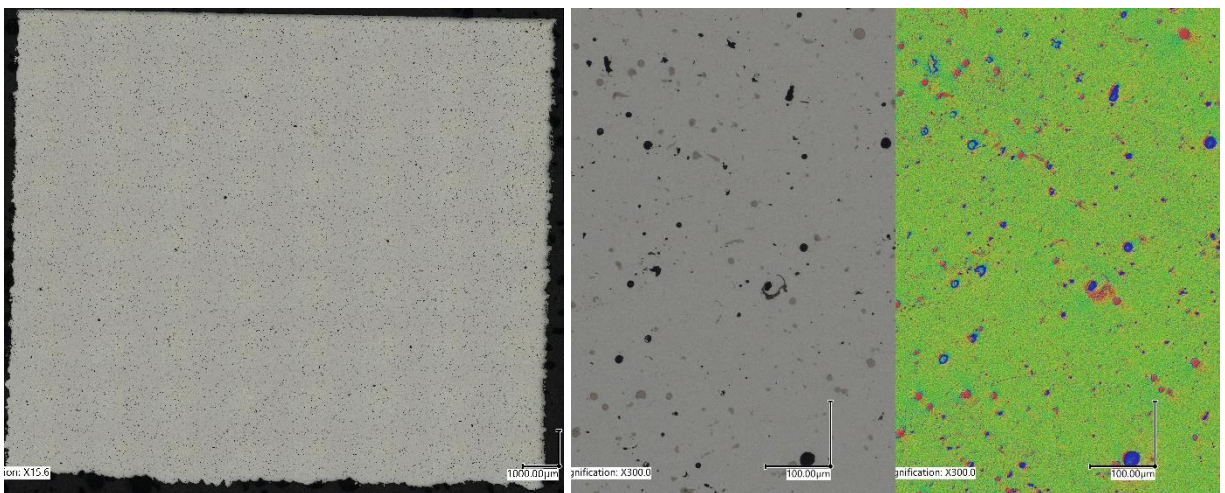


Figure 61: Al5083 – PSO7 micrographs show a small degree of porosity from build A-2302 cube eighteen with an energy density of 56.1 J/mm³. The right image highlights the ceramic constituents and the porosity, with the red spots being ceramic and the dark blue being pores. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube.



Figure 62: The printing layout for the A5083-RAM7 deliverable builds.

The A5083-RAM7 as-printed material performed the most isotropic out of the 4-test compositions with the lowest standard deviation for UTS, YS, and elongation. The overview of the mechanical testing can be seen in Table 28. The four alloys overall outperformed commercial alloys Al5083-O and Al5083-H321 for UTS and YS. [3] The clear best performing candidate was A5083-RAM7. A5083-RAM7 showed the highest UTS and YS, but the elongation was 9.6% lower than Al5083-O and only 3.6 % lower Al5083-H321. The lower elongation is expected to be related to the increased strengths observed. The elongation for A5083-RAM7 did have the smallest standard deviation between the samples tested. Figure 63 shows the resulting stress-strain curves for A5083-RAM7.

Table 28: The aluminum composition average tensile results compared to ASM volume 2 standards for Al 5083. The mechanical testing was conducted by GT following the ASTM E8 standards. The tensile results are based on four test specimens for each composition.

Alloy	YS (KSI)	UTS (KSI)	Elongation%
A5083-PSO7	56.7 ± 4.3	59.2 ± 4.8	5.3 ± 2.8
A5083-PSO3	44.9 ± 1.0	56.1 ± 6.5	4.9 ± 4.3
A5083-RAM2	44.6 ± 0.7	53.9 ± 4.5	13.7 ± 8.2
A5083-RAM7	56.8 ± 0.7	64.8 ± 1.9	12.4 ± 2.2
Al 5083-O	21.0	42.1	22
Al 5083-H321	33.4	45.7	16

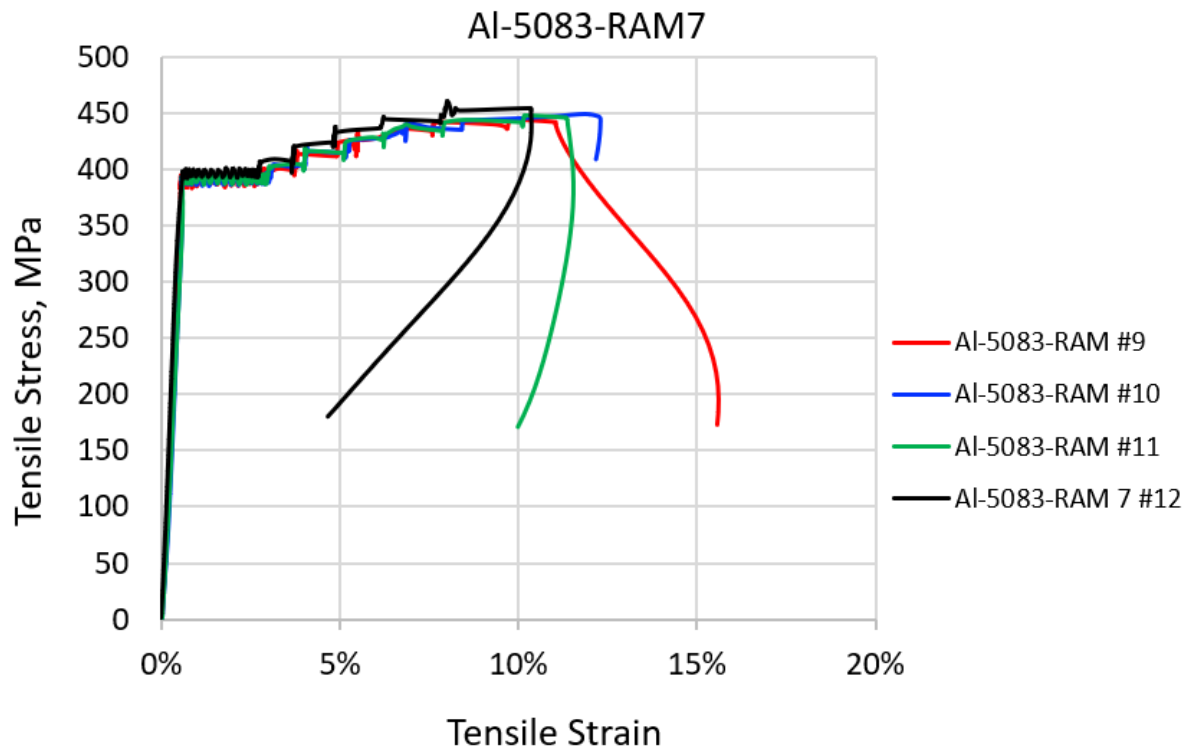


Figure 63: AI-5083-RAM7 stress strain curves for the four horizontally printed ASTM E8 tensile specimens.

4.2.3 Property Reproducibility Tensile Testing (A5083-RAM7)

For the second alloy for investigation in Phase II, we chose an aluminum alloy. The team felt that including a fast printing, light weight, single step printable aluminum alloy requiring minimal postprocessing would greatly improve the Army's ability to deliver AM-fabricated, field-ready parts to the warfighter. The 5XXX series aluminum alloys were chosen as solid solution, Mg-based aluminum alloys that forgo the need for post-print heat treatment after fabrication. Al5083 is known to suffer solidification cracking in standard welding practices and a filler metal is needed to perform a weld. Al5083 was therefore selected for the second alloy. We investigated into how the material printability could be improved through reactive additive manufacturing (RAM) (in situ inoculation) with the addition of Ti and B₄C into the aluminum powder feedstock. The results for Al5083 with RAM in situ inoculants show increased mechanical performance and major improvements in the printed isotropic properties. A5083-RAM7 was selected with seven volume percent of the RAM inoculants. The resulting tensile properties are presented in Table 29. Figure 64 shows a micrograph of A5083-RAM7 showing very dense sample.

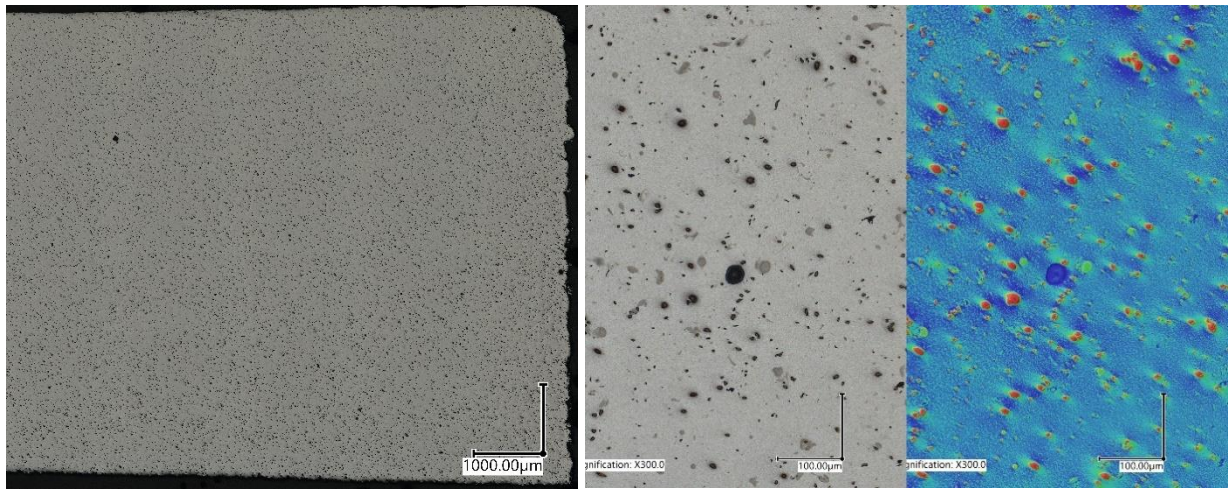


Figure 64: Al5083-RAM7 micrographs show a small degree of porosity from build A-2255 cube eight with an energy density of 51.4 J/mm^3 . The right image highlights the ceramic constituents and the porosity, with the red spots being ceramic and the dark blue being pores. Right image: 100x stitch of the whole cube; Left image: 300x image of the same cube.

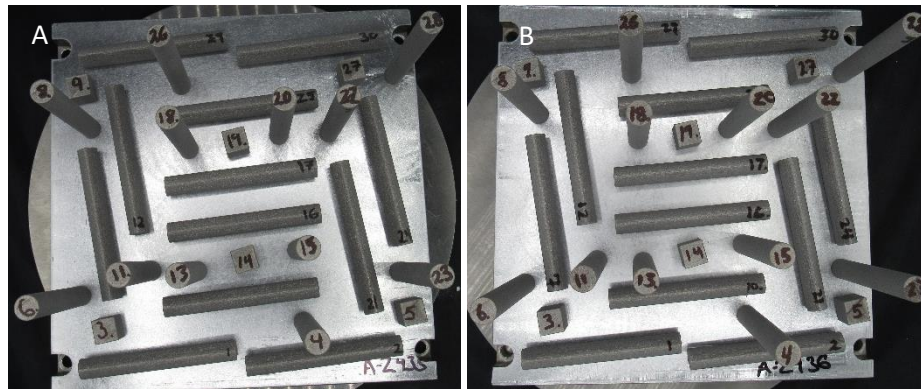


Figure 65: The Al5083 RAM7 builds from A) printer 1 and B) printer 2.

Figure 66 displays the UTS of the A5083-RAM7 obtained during Phase II compared to commercial equivalents. The A5083-RAM7 increased Al5083 yield strength by 26 ksi in the H321 state and the UTS was increased by 26.3 ksi with a 56% reduction in elongation for the as-printed state. A stress relief procedure following the Al5083-O requirement (650 °F held for 2 hr) was observed not to have a significant effect on the mechanical properties of A5083-RAM7. The resulting tensile properties after stress relief were statistically similar to the as-printed samples above the 95% confidence level in all cases.

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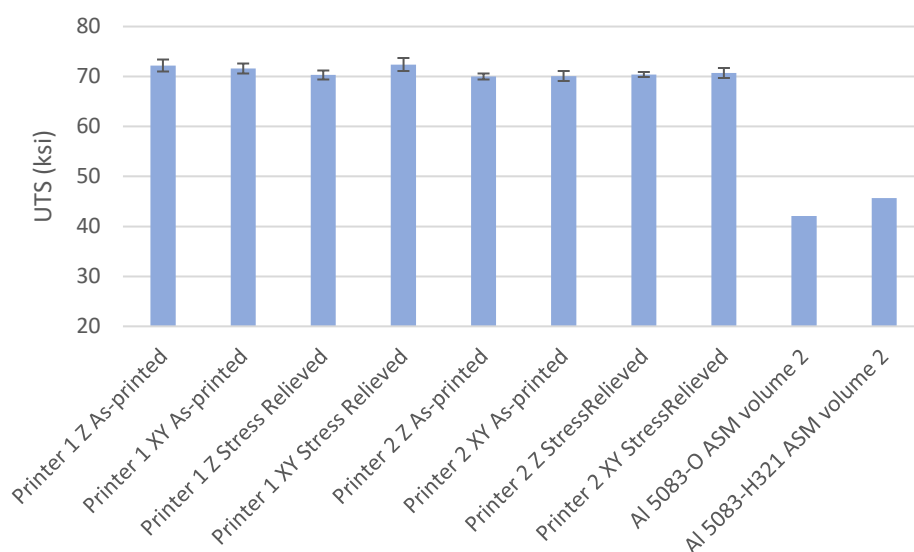


Figure 66: A5083-RAM7 UTS compared to current ASM values to wrought AL 5083 alloys.

Table 29: The A5083-RAM7 feedstock mechanical properties compared to the ASM standard and the UTS variation in the printed specimens. Every criterial was consisted of six specimens.

A5083-RAM7		Orient ation	YS (ksi)	UTS (ksi)	UTS coefficient of variation < 3.5%	El%
Printer 1	AS-printed	Z	59.8 ± 0.2	72.2 ± 1.2	1.7%	6.7 ± 1.2
Printer 1	AS-printed	XY	61.5 ± 0.6	71.6 ± 1.0	1.4%	7.5 ± 0.4
Printer 1	Stress Relieved	Z	59.0 ± 0.4	70.3 ± 0.9	1.2%	5.9 ± 1.0
Printer 1	Stress Relieved	XY	61.6 ± 0.3	72.4 ± 1.3	1.8%	5.1 ± 0.8
Printer 2	AS-printed	Z	58.9 ± 0.4	70.0 ± 0.6	0.9%	7.3 ± 1
Printer 2	AS-printed	XY	58.1 ± 0.6	70.1 ± 1.0	1.4%	7.2 ± 0.9
Printer 2	Stress Relieved	Z	58.8 ± 0.5	70.4 ± 0.5	0.7%	6.5 ± 0.6
Printer 2	Stress Relieved	XY	60.1 ± 0.5	70.7 ± 1.0	1.4%	6.4 ± 1.0
ASM Volume 2	Al 5083-O (Wrought)	-	21.0	42.1	-	22
ASM Volume 2	Al 5083-H321 (Wrought)	-	33.4	45.7	-	16

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Table 30 show the statistical means comparison for UTS of A5083-RAM7 samples printed either on printers 1 and 2, either as-printed or with stress relief treatment, or either in the XY- or Z-direction. While UTS differences were significant depending on the specific printer and the print orientation, remarkably, no significant difference was observed between as-printed and stress relieved samples, indicating that for A5083-RAM7 uniquely, stress relief provides no additional strength. This fact serves as a strong argument in favor of using this material in Army applications, as time-intensive post-processing procedures may not be necessary prior to deployment of parts printed from this material.

Table 30: The statistical analysis conducted on the A5083-RAM7 UTS comparing the printers used and the statistical relevance of printing orientation.

Printer 1-2			AS-Printed Vs Stress Relieved		Print Orientation	
t-Test: Two-Sample Assuming Unequal Variances			t-Test: Two-Sample Assuming Unequal Variances		t-Test: Two-Sample Assuming Unequal Variances	
	<i>Variable 1</i>	<i>Variable 2</i>	<i>Variable 1</i>	<i>Variable 2</i>	<i>Variable 1</i>	<i>Variable 2</i>
Mean	71.6	70.3	70.95	70.9	71.33	70.55
Variance	1.7	0.7	1.72	1.5	2.03	0.90
Observations	24	24	24	24	24	24
Hypothesized Mean Difference	0		0		0	
df	38		46		40	
t Stat	4.03		0.05		2.241	
P(T<=t) one-tail	1.30E-04		0.48		0.015	
t Critical one-tail	1.7		1.68		1.684	
P(T<=t) two-tail	2.60E-04		0.96		0.031	
t Critical two-tail	2.02		2.01		2.021	
Reject Null? Yes.			Reject Null? No. (-2.01 < 0.05 < 2.01)		Reject Null? Yes.	
Printer 1,2 produce statistically distinct UTS.					Orientation statistically impacts UTS.	

Table 31 results show the isotropy of the A5083-RAM7 samples printed on different printers and with or without stress relief heat treatment. Regardless of specific printer or stress relief treatment, all samples showed an isotropy greater than 97%. On average, the as-printed isotropy was a remarkable 99.6%. These represent industry-leading results. Further development is needed to commercialize the A5083-RAM7 would be minimal, and the application potential as a highly printable, lightweight, corrosion-resistant alloy that does not require post-build heat treatment. The development of A5083-RAM7 represents yet another non-weldable alloy that Elementum 3D has been able to make printable, and importantly, these results indicate that when commercialized, it may be the most isotropic aluminum-based material for L-PBF on the market.

Table 31: A5083-RAM7 metal matrix composite XY and Z-direction UTS and UTS ratio in as-printed and stress-relieved samples printed on two different printers.

	UTS XY Direction (ksi)	UTS Z Direction (ksi)	UTS Z:YX ratio
Al5083 – RAM7 AS-printed Printer 1	71.6 ± 1.0	72.2 ± 1.2	99.2%
Al5083 – RAM7 Stress Relieved Printer 1	72.4 ± 1.3	70.3 ± 0.9	97.1%
Al5083 – RAM7 AS-printed Printer 2	70.1 ± 1.0	70.0 ± 0.6	99.9%
Al5083 – RAM7 Stress Relieved Printer 2	70.7 ± 1.0	70.4 ± 0.5	99.5%
Al5083 – RAM7 AS-printed Average	71.1 ± 1.5	70.8 ± 1.1	99.6%
Al5083 – RAM7 Stress Relieved Average	71.5 ± 1.4	70.3 ± 0.7	98.3%

Table shows ratios of the UTS of A5083-RAM7 to current wrought Al 5083 reported in the ASM handbook, volume 2 for annealed and hot worked Al5083. The printed A5083-RAM7 material produced samples with UTS values far greater than reported industry standard, with UTS values on average 169% and 155% those of Al5083-O and Al5083-H321 conditions, respectively. The Al 5083-RAM7 has also outperformed materials reported in a study describing the development of an L-PBF-printable variation of Al5083. [2]

Table 32: Al5083 – RAM7 comparison to commercial Al 5083 alloy in the two ASM volume 2

	UTS ASM Al 5083 – O	UTS ASM Al 5083 – H321
Al5083 – RAM7 AS-printed Printer 1	171%	153%
Al5083 – RAM7 Stress Relieved Printer 1	170%	156%
Al5083 – RAM7 AS-printed Printer 2	167%	153%
Al5083 – RAM7 Stress Relieved Printer 2	168%	154%
Al5083 – RAM7 AS-printed Average Tensile	169%	155%
Al5083 – RAM7 Stress Relieved Average Tensile	169%	155%

5 Conclusions

The successful execution of the ARMY Phase II project, “Design of Easy-to-Use Structural Alloy Feedstocks for Additive Manufacturing” has shown that alloy design through ICME and experimental validation can produce a more robust, isotropic material for metal additive manufacturing. Phase II produced two alloy feedstocks 3XX-TiAM, a more isotropic alternative to 316L stainless steel, and A5083-RAM7, an alloy with excellent printability, superior strength, nearly perfectly isotropic mechanical properties. The following are takeaways of note:

- The 3XX-TiAM stainless steel reduced the variation in UTS between the XY and Z-directions greatly. The Z:XY UTS ratio of commercial 316L AM powders was reported to be as low as 86% for Carpenter Additive powder and EOS 316L material, whereas an average of 95% was observed for the developed 3XX-TiAM. For A5083-RAM7, the Z:XY UTS ratio was nearly 100% isotropic at 99.6%.
- Statistical analysis of 3XX-TiAM has shown that the coefficient of variation of UTS was generally < 1%. The A5083-RAM7 UTS maintains a coefficient of variation < 1.8%. This clearly fulfills the requirement that samples should have ultimate tensile strength (UTS) measurements with a coefficient of variation of < 3.5 % (parts printed in the same orientation).
- UTS measurements for printed A5083-RAM7 showed no statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens when the sample size was n=6. UTS measurements for printed 3XX-TiAM did show a statistical difference (at the 95% confidence level) between XY- and Z-oriented tensile specimens when the sample size was n=6. However, this is likely due to the high repeatability and thus low standard deviation of the printing process and tensile measurements. Indeed, the ratio between the UTS values measured in the Z-direction to the XY-direction is up to 97.3%. In contrast, datasheets for common 316L AM feedstocks give ratios of 91.5% (EOS) and 86% (Carpenter).
- Al5083-RAM7 feedstock proved to be the highest-performing material designed during Phase II. The creation of a metal matrix composite through inoculation of an aluminum 5083 alloy improved the as-printed properties and resulted in a nearly fully isotropic material by eliminating hot cracking during welding.
- Stress-relieved A5083-RAM7 was not significantly stronger than as-printed A5053-RAM7, and both resulted in samples that outperformed industry-standard Al5083-O by 36 ksi for UTS and 39 ksi for YS.
- AM 316L powder effectively maintains mechanical strength properties exposed to 30-days of intense humidity. Atomic layer deposition coating was explored to improve this shelf life but proved ineffective in maintaining consistent flowability after humidification. The findings show that Base 316L stainless steel metal powder exposed to 87% ambient humidity at 40 °C is usable up to 30 days before prints suffer from increased porosity.
- ICME can be implemented to improve alloy design and selection by eliminating options that are likely to result in poor printability. Here, ICME tools predicted the most fruitful ways to improve printability of L-PBF metal powder feedstocks by establishing a theoretical basis for the design of 3XX-TiAM and A5083-RAM7.

6 Future Work & Commercialization

The research carried out in this Phase II program focused on increasing the printability and reliability of austenitic stainless steel and aluminum alloy feedstocks for L-PBF. The work has solidly established 3XXTiAM and A5083-RAM7 as superior to other commercial offerings. In the case of A5083-RAM7, the material could represent a true breakthrough in isotropic properties for an AM aluminum material. These materials are therefore very likely to be commercialized and add to Elementum 3D's growing portfolio of superior AM materials.

For Phase III commercialization efforts, the team plans to expand the experimental part of the work in the following areas:

- Demonstration of the isotropic nature of the selected feedstocks through statistical testing and analysis.
- Demonstration of the isotropic nature of the austenitic stainless steel and aluminum powder feedstocks across multiple L-PBF printers.
- Demonstration of the prototype powder feedstock to our endusers through printed prototypes and the statistical data generated
- Demonstration of the ease-of-use of austenitic stainless steel and aluminum metal matrix composite in a real-world application with relevance to Army needs

The feedstock materials will then be transitioned to Army advanced manufacturing centers and mobile labs/production operations for use in rapid prototyping and point of need manufacturing. Components with the new alloys designed in will also be part of the Phase II sequential and Phase III efforts. Component level qualification with the Army and prime contractors will be a key next step for serial production use of the materials.

Elementum 3D's core business is to commercialize and supply new metal feedstocks, and the E3D team has gained extensive experience doing this. Over the past three years, the team has successfully commercialized more than 6 new AM materials with at least four of these materials now in wide usage with multiple customers including volume part production for critical applications. Elementum 3D's transition and commercialization strategy for this project's new highly reliable AM alloys will utilize this experience and follow a similar pathway to the market. The commercialization plan includes directly supplying the new highly reliable AM feedstocks, processes, and statistical performance data to original equipment manufacturers and contract manufacturers. Elementum 3D will also support component-level application development and low to moderate volume parts production on our in-house AM systems to help facilitate early manufacturing adoption.

Component qualification is a critical barrier to wide AM adoption with the qualification of critical parts potentially costing tens or even hundreds of millions of dollars such as in the case of the GE LEAP engine fuel nozzle. Highly reliable materials with MMPDS equivalent design allowable data are needed to de-risk AM production and component qualification. Elementum 3D is in direct regular contact with advanced AM users in multiple industries and recognizes the demand for highly reliable feedstocks, AM processes, and statistical design allowable quality data to facilitate qualification of critical AM produced components. Several of these advanced AM users, including Northrop Grumman Corporation, are looking forward to the substantial cost savings and accelerated production timelines that these new feedstock materials and statistically based design allowable property data will provide.

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These easy-to-use and highly reliable feedstocks support the industry's need for economic and ecological components that are reliable, isotropic, and more amenable for printing than products on the market today. There is a strong market need for alloys that enable successful first-try metal part fabrication without extensive knowledge of metallurgy, support design, or application specific parameter development. The team's approach is to introduce alloys that serve as a default material choice for metals AM and allow anyone with basic knowledge found in a simple five-page handbook to make viable replacement parts with highly consistent properties. This product will reach a wide range of markets, including DoD (FOBs and established bases), aerospace, automotive aftermarket, machine shops (jigs), universities, mechanics, and ultimately hardware stores.

The field of additive manufacturing had a market size of \$5.2 billion in 2015 and is predicted to grow to \$26.7 billion in 2027. [6] [7] The explosive growth of metal additive manufacturing in the last five years is in direct correlation with the incredible demand for metal powders. The additive metal powders market is expected to expand at a robust 27% CAGR for the next several years. At this pace, the market's valuation of \$310.6 million in 2016 [6] will rise substantially to become \$1.1 billion by the end of 2024 [8]. Elementum 3D expects a multipurpose steel product that is highly reliable and easily printable to earn roughly 2% of the metal powders market share by 2026, equaling approximately \$35 million in revenue with the multipurpose aluminum alloy offering similar or better market share potential.

Elementum 3D is a rapidly growing materials company that is already generating growing revenue through materials sales of several 'designed for AM' feedstocks. Elementum 3D is ISO 9001 certified and has recently expanded production capabilities for their RAM feedstocks to enable production of more than 1 ton of feedstock powder per day. Elementum 3D is ITAR registered, NIST 800-171 compliant and understands the importance of customer confidentiality, especially when it comes to national defense or dual use technologies.

Elementum 3D has marketing and sales expertise in house and has established extensive connections and relationships throughout multiple industries that use additive manufacturing. Elementum 3D has also established agreements with two of the largest AM equipment manufacturers for them to directly resell our materials to customers using their systems. Our business model is agnostic with respect to who makes the final part, and our intent is to grow the entire market. Current Elementum 3D customers include companies in defense, space, aerospace, motorsport, automotive, thermal management, and contract manufacturing. Many of these same customers are interested in a general-purpose metal that can be used to cost-effectively print reliable parts on the first attempt.

A major barrier to adoption of AM is a lack of adequate statistical testing and qualification data for AM materials. Metal AM has a stigma of not having repeatable, reliable, and isotropic materials. Based on customer input, Elementum 3D has determined the best plan of righting this stigma is through the introduction of made-for-AM materials and the adoption of statistical material property data in-line with MMPDS S-basis data. Elementum 3D plans to collect statistical data comparable to S-basis MMPDS data for these new materials with statistical testing including multiple heats, lots, builds, printers, and post processing batches. Testing will include tensile, fatigue, fracture toughness, compression, and thermal performance. This data along with the reliable and easy-to-use material will reduce E3D's

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customers' cost and time burden of designing and qualifying components to promote adoption of these materials as well as increase the adoption of AM.

Elementum 3D will work with potential customers including our endorsers at Northrup Grumman and the US Army DEVCOM Aviation & Missile Center to identify promising components for adoption of these new AM feedstocks. To help accelerate and expand the market reach of our products, the Elementum 3D team is planning a \$20 million Series B funding round in 2021. The funds will be used in part to expand our material development, testing, and production capabilities. A portion of this planned funding is dedicated to promoting adoption of new products including the new feedstock products from this ARO STTR project.

Other AM materials producers could be viewed as potential competitors, but they typically lack the extensive in-house AM equipment, AM process development ability, and material and component qualification expertise that Elementum 3D has developed. For this reason, other material producers are commonly partnering with E3D for development of the AM processing strategy, printed property data, and detailed feedstock specifications. Elementum 3D will leverage these existing relationships with metal atomizers to ensure adequate production including a qualified second supplier of these feedstock materials. DoD funding for this project will allow Elementum 3D to accelerate commercialization and adoption of these highly reliable stainless steel and aluminum feedstocks.

While the new materials feedstocks are the primary products being commercialized, the innovative and systematic computational materials discovery framework developed in this project also has ongoing commercial value. The engineered platform incorporates multi-scale modeling coupled with machine learning (ML) which and this methodology could transform and accelerate the way alloys are discovered for other applications. This platform can significantly contribute to manufacturing cost reduction and create new revenue opportunities. Such a tool would be indispensable to steel producers who must vie in a market characterized by intense competition and enormous potential across the world. Elementum 3D will work with the Georgia Institute of Technology and the KMMD technical team to develop the market-facing aspects of this technology including the development of a sustainable business model and go-to-market strategy.

7 References

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8 Appendix A

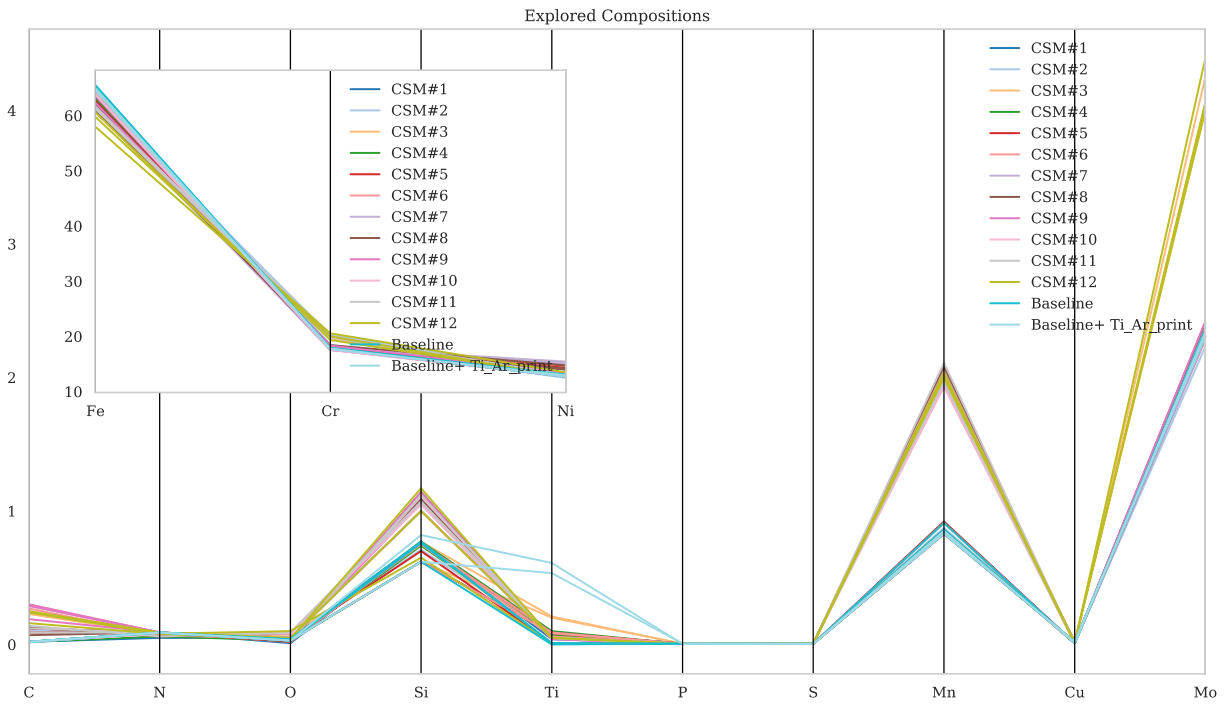


Figure 67: Compositions selected from CALPHAD and ML to achieve specific combinations of slag/no-slag and δ -ferrite/no- δ -ferrite for experimental verification.

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Table 33: ICP data comparing the designed composition with the analyzed feedstock and printed part compositions.

Material	Fe	Cr	Ni	C	N	O	Si	Ti	P	S	Mn	Cu	Mo
Test Alloy #1	65.49	17.80	12.70	0.017	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31
Powder	65.26	17.75	12.82	0.020	0.05	0.05	0.77	0.040	0.005	0.01	0.92	0.01	2.30
Printed	64.64	17.94	13.18	0.020	0.09	0.01	0.77	0.090	0.005	0.01	0.87	0.01	2.37
Powder difference	-0.23	-0.05	0.12	0.003	-0.04	0.02	0.15	-0.045	-0.002	0.00	0.09	-0.01	-0.01
Printed difference	-0.85	0.14	0.48	0.003	0.00	-0.02	0.15	0.005	-0.002	0.00	0.04	-0.01	0.06
Test Alloy #2	63.18	20.00	12.70	0.120	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31
Powder	64.90	18.48	12.48	0.110	0.07	0.06	0.71	0.070	0.005	0.01	0.87	0.01	2.23
Printed	62.07	20.30	13.29	0.090	0.09	0.02	0.77	0.090	0.005	0.01	0.85	0.01	2.41
Powder difference	1.72	-1.52	-0.22	-0.010	-0.02	0.03	0.09	-0.015	-0.002	0.00	0.04	-0.01	-0.08
Printed difference	-1.11	0.30	0.59	-0.030	0.00	-0.01	0.15	0.005	-0.002	0.00	0.02	-0.01	0.10
Test Alloy #3	61.25	20.00	12.70	0.250	0.09	0.03	0.62	0.200	0.007	0.01	0.83	0.02	4.00
Powder	61.38	19.61	12.90	0.230	0.08	0.06	0.75	0.010	0.005	0.01	0.91	0.01	4.05
Printed	60.72	19.77	13.07	0.230	0.08	0.03	0.77	0.210	0.005	0.01	0.85	0.01	4.25
Powder difference	0.13	-0.39	0.20	-0.020	-0.01	0.03	0.13	-0.190	-0.002	0.00	0.08	-0.01	0.05
Printed difference	-0.53	-0.23	0.37	-0.020	-0.01	0.00	0.15	0.010	-0.002	0.00	0.02	-0.01	0.25
Test Alloy #4	63.19	17.80	15.00	0.017	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31
Powder	63.26	17.53	15.14	0.020	0.06	0.03	0.74	0.070	0.005	0.01	0.84	0.01	2.29
Printed	62.65	17.80	15.30	0.020	0.09	0.03	0.77	0.100	0.005	0.01	0.86	0.01	2.36
Powder difference	0.07	-0.27	0.14	0.003	-0.03	0.00	0.12	-0.015	-0.002	0.00	0.01	-0.01	-0.02
Printed difference	-0.54	0.00	0.30	0.003	0.00	0.00	0.15	0.015	-0.002	0.00	0.03	-0.01	0.05
Test Alloy #5	64.08	17.80	14.00	0.120	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31
Powder	63.67	17.81	14.32	0.120	0.09	0.05	0.70	0.010	0.005	0.01	0.92	0.01	2.29
Printed	62.76	18.21	14.70	0.090	0.09	0.02	0.76	0.090	0.005	0.01	0.85	0.01	2.41
Powder difference	-0.41	0.01	0.32	0.000	0.00	0.02	0.08	-0.075	-0.002	0.00	0.09	-0.01	-0.02
Printed difference	-1.32	0.41	0.70	-0.030	0.00	-0.01	0.14	0.005	-0.002	0.00	0.02	-0.01	0.10
Test Alloy #6	65.20	17.80	12.70	0.300	0.09	0.03	0.62	0.085	0.007	0.01	0.83	0.02	2.31
Powder	65.10	17.69	12.79	0.270	0.07	0.04	0.75	0.060	0.005	0.01	0.91	0.01	2.30
Printed	64.63	17.82	13.08	0.290	0.09	0.02	0.76	0.090	0.005	0.01	0.85	0.01	2.35
Powder difference	-0.10	-0.11	0.09	-0.030	-0.02	0.01	0.13	-0.025	-0.002	0.00	0.08	-0.01	-0.01
Printed difference	-0.57	0.02	0.38	-0.010	0.00	-0.01	0.14	0.005	-0.002	0.00	0.02	-0.01	0.04
Test Alloy #7	61.65	17.80	15.00	0.017	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	2.31
Powder	61.65	17.53	15.16	0.020	0.08	0.09	1.05	0.040	0.005	0.01	2.08	0.01	2.28
Printed	60.87	18.01	15.43	0.020	0.08	0.04	1.14	0.050	0.005	0.01	1.96	0.01	2.38
Powder difference	0.00	-0.27	0.16	0.003	-0.01	0.04	0.05	-0.010	-0.002	0.00	0.08	-0.01	-0.03
Printed difference	-0.78	0.21	0.43	0.003	-0.01	-0.01	0.14	0.000	-0.002	0.001	-0.04	-0.01	0.07
Test Alloy #8	62.55	17.80	14.00	0.120	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	2.31
Powder	62.25	17.73	14.20	0.130	0.06	0.08	1.09	0.050	0.005	0.01	2.08	0.01	2.31
Printed	61.01	18.40	14.75	0.070	0.09	0.02	1.14	0.050	0.005	0.01	2.04	0.01	2.41
Powder difference	-0.30	-0.07	0.20	0.010	-0.03	0.03	0.09	0.000	-0.002	0.001	0.08	-0.01	0.00
Printed difference	-1.54	0.60	0.75	-0.050	0.00	-0.03	0.14	0.000	-0.002	0.001	0.04	-0.01	0.10
Test Alloy #9	63.67	17.80	12.70	0.300	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	2.31
Powder	63.79	17.54	12.79	0.290	0.09	0.04	1.06	0.040	0.005	0.01	2.05	0.01	2.29
Printed	62.39	18.31	13.35	0.190	0.09	0.03	1.14	0.040	0.005	0.01	2.02	0.01	2.42
Powder difference	0.12	-0.26	0.09	-0.010	0.00	-0.01	0.06	-0.010	-0.002	0.001	0.05	-0.01	-0.02
Printed difference	-1.28	0.51	0.65	-0.110	0.00	-0.02	0.14	-0.010	-0.002	0.001	0.02	-0.01	0.11
Test Alloy #10	63.95	17.80	12.70	0.017	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	2.31
Powder	63.89	17.66	12.81	0.020	0.08	0.08	1.05	0.050	0.005	0.01	2.04	0.01	2.30
Printed	63.94	17.57	12.92	0.020	0.09	0.03	1.11	0.050	0.005	0.01	1.93	0.01	2.32
Powder difference	-0.06	-0.14	0.11	0.003	-0.01	0.03	0.05	0.000	-0.002	0.001	0.04	-0.01	-0.01
Printed difference	-0.01	-0.23	0.22	0.003	0.00	-0.02	0.11	0.000	-0.002	0.001	-0.07	-0.01	0.01
Test Alloy #11	61.65	20.00	12.70	0.120	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	2.31
Powder	61.52	19.81	12.81	0.140	0.06	0.09	1.07	0.050	0.005	0.01	2.11	0.01	2.32
Printed	61.28	19.98	13.02	0.090	0.09	0.03	1.12	0.050	0.005	0.01	1.97	0.01	2.35
Powder difference	-0.13	-0.19	0.11	0.020	-0.03	0.04	0.07	0.000	-0.002	0.001	0.11	-0.01	0.01
Printed difference	-0.37	-0.02	0.32	-0.030	0.00	-0.02	0.12	0.000	-0.002	0.001	-0.03	-0.01	0.04
Test Alloy #12	59.83	20.00	12.70	0.250	0.09	0.05	1.00	0.050	0.007	0.01	2.00	0.02	4.00
Powder	60.62	19.33	12.85	0.240	0.08	0.10	0.65	0.010	0.005	0.01	2.04	0.01	4.06
Printed	58.03	20.56	13.49	0.160	0.08	0.04	1.17	0.050	0.005	0.01	2.01	0.01	4.39
Powder difference	0.79	-0.67	0.15	-0.010	-0.01	0.05	-0.35	-0.040	-0.002	0.001	0.04	-0.01	0.06
Printed difference	-1.80	0.56	0.79	-0.090	-0.01	-0.01	0.17	0.000	-0.002	0.001	0.01	-0.01	0.39
316L Baseline	65.57	17.80	12.70	0.017	0.09	0.03	0.62	0.000	0.007	0.01	0.83	0.02	2.31
Powder	65.21	17.78	12.84	0.020	0.09	0.04	0.77	0.010	0.005	0.01	0.91	0.01	2.31
Printed	64.84	17.93	13.08	0.020	0.09	0.03	0.76	0.010	0.005	0.01	0.86	0.01	2.36
Powder difference	-0.36	-0.02	0.14	0.003	0.00	0.01	0.15	0.010	-0.002	0.00	0.08	-0.01	0.00
Printed difference	-0.73	0.13	0.38	0.003	0.00	0.00	0.14	0.010	-0.002	0.00	0.03	-0.01	0.05
316L Plus 0.535wt%Ti	65.04	17.80	12.70	0.017	0.09	0.03	0.62	0.535	0.007	0.01	0.83	0.02	2.31
Powder													
Printed	64.58	17.67	12.97	0.020	0.09	0.03	0.82	0.610	0.005	0.01	0.85	0.01	2.34
Powder difference													
Printed difference	-0.46	-0.13	0.27	0.003	0.00	0.00	0.20	0.075	-0.002	0.00	0.02	-0.01	0.03

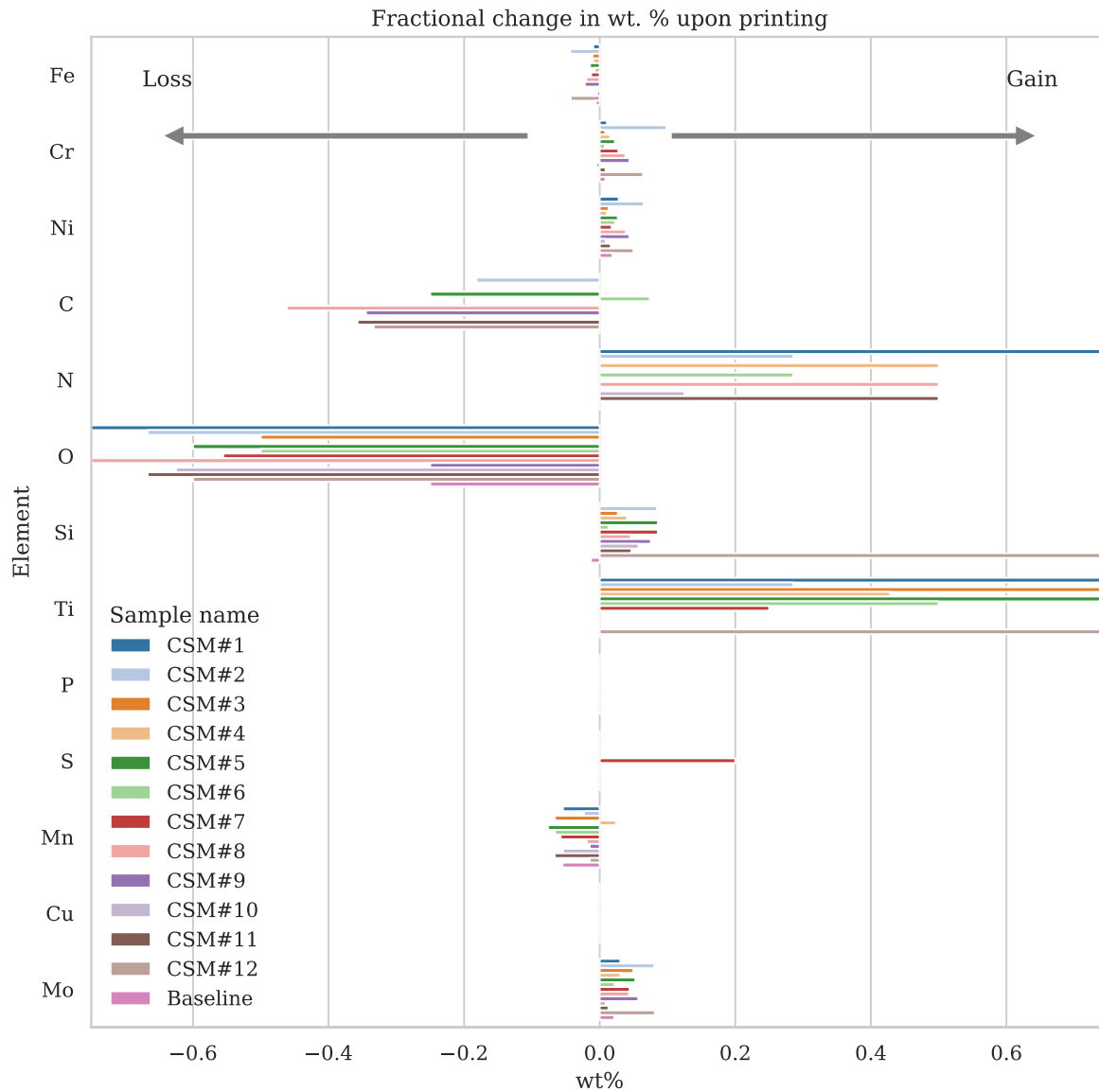


Figure 68: The fractional change in the weight percent of each element, $wt\% = \frac{c_{\text{printed}} - c_{\text{powder}}}{c_{\text{powder}}}$, from powder-to-print. The large disparity in the magnitude of each element results from the fact that the fractional change is normalized by the starting concentration in the powder and the direction of change is considered more reliable than the magnitude.