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Heat Treatment of Cold-Sprayed Titanium and Aluminum Alloys

by Nick Sotiropoulos, Issac Nault, Tristan Bacha, Adolfo Blassino, and Joshua McGuigan

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14. ABSTRACT

Cold-sprayed aluminum and titanium microstructure and mechanical properties were evaluated in terms of how they would perform in both the as-sprayed and heat-treated conditions. While the aluminum alloys formed a dense as-sprayed deposit, the oxygen and hydrogen present on the surface of the feedstock material led to blistering or significant increases in porosity during heat treatments at temperatures greater than 400 °C. Conversely, the as-sprayed, commercially pure titanium had about 10%–14% porosity, but there was no significant increase to the porosity during heat treatment. It was concluded that decreasing and managing the hydrogen and oxygen content in the aluminum feedstock powder is critical if the deposited material will receive heat treatment. Further evaluation of the feedstock, commercially pure titanium will be required to achieve the density, and therefore ductility, that can be found in literature for similar materials.

15. SUBJECT TERMS
Weapons Sciences, cold spray additive manufacturing, aluminum, titanium, mechanical testing

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Contents

Preface

This report outlines the work completed on heat treating cold-sprayed aluminum alloys and commercially pure titanium. It was found that during heat treatment, porosity that was not present in the as-sprayed condition would form. It was hypothesized that the porosity was a result of the varying levels for oxygen and hydrogen on the surface of the aluminum powders. As the samples were heat treated, the oxygen and hydrogen in solution at the surface of these powders would begin to break down, turning into gases. The increase in gas produced, coupled with the increase in temperature, would increase the pressure in these localized pockets. Simultaneously, the aluminum begins to soften at increased temperatures leading to a decrease in yield strength. This leads to a significant increase in porosity as the heat treatment temperature increases, in some cases causing enough pressure buildup to cause delamination between layers.

Commercially pure titanium was sprayed using helium gas and subsequently heat treated for 1 h at 1100 °C in vacuum. Titanium did not see a significant increase in porosity following heat treatment, but the as-sprayed density of the commercially pure titanium was already quite poor. The mechanical properties of the heat-treated, commercially pure titanium were measured using subsized tensile bars in accordance with ASTM E8 standard[1] and compared with previously recorded data.

[1] ASTM E8. Standard test methods for tension testing of metallic materials. ASTM International; 2024 Mar 5.

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BLISTERING OF AI DURING HT

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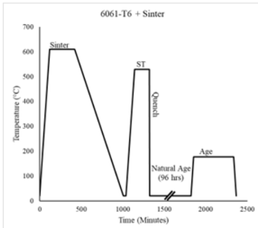
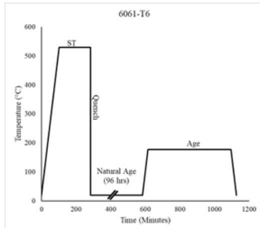
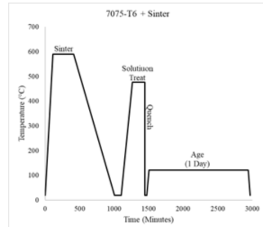
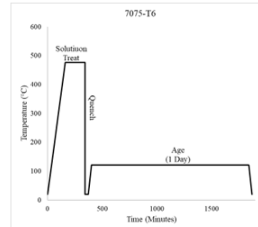
HEAT TREATMENTS

	5056	6061	7050	7075
Solidus (°C)	568.3	582.0	488.0	477.0
Liquidus (°C)	638.0	651.7	629.4	635.0









6061-T6 + Sinter			
Action	Time (min)	Temp (°C)	Cooling
Sinter	300	610	CFC
Solution	180	530	Quench (Warm/ hot water to minimize distortion)
Natural Age (4 Days)	5760	20	NA
Age	480	177	Air cool

7075-T6 + Sinter			
Action	Time (min)	Temp (°C)	Cooling
Sinter	300	590	Furnace Cool
Solution	180	477	Quench (Warm/ hot water to minimize distortion)
Age (1 Day)	1440	121	Air cool

Based on: AMS2772 – AEROSPACE MATERIAL SPECIFICATION: Heat Treatment of Aluminum Alloy Raw Materials

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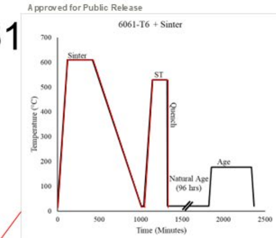
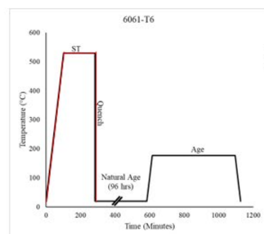
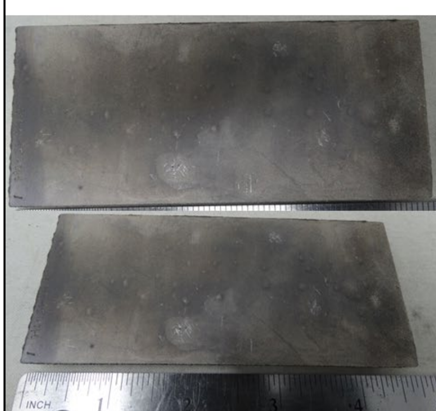
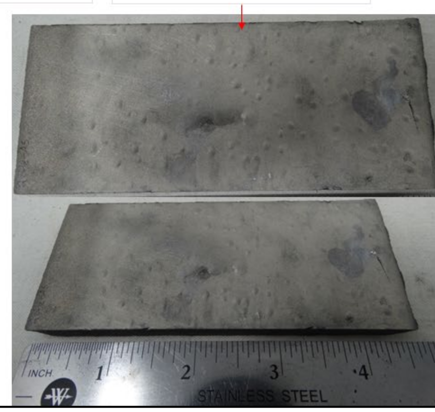
INITIAL OBSERVATION 6061

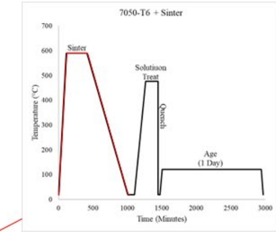


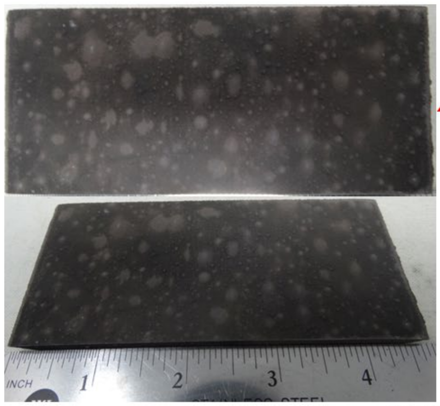






INITIAL OBSERVATION 7075





INITIAL ATTEMPT AT DEGASSING (7050)

- The goal was to determine if we could degas the deposits at a low temperature prior to the heat treatment following deposition.
- Three different degas heat treatments were proposed:
  1. Heat to **205°C** in **vacuum**, hold for 24 hours, cool to room temperature
  2. Heat to **205°C** in **air**, hold for 24 hours, cool to room temperature
  3. Heat to **405°C** in **vacuum**, hold for 24 hours, cool to room temperature
- Following the degas heat treat the samples would be sintered at 590°C

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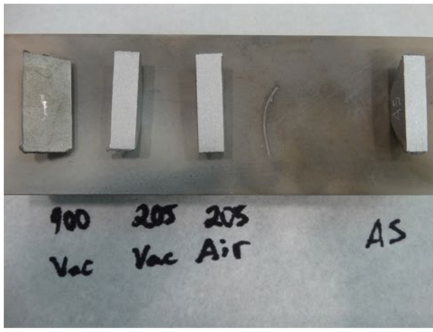
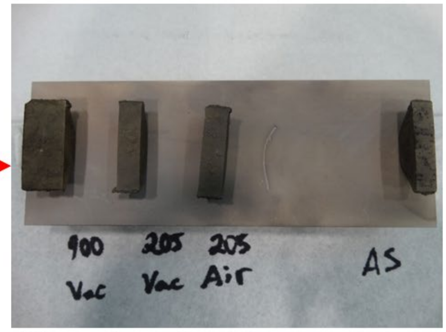
RESULTS OF INITIAL ATTEMPT AT DEGASSING (7050)



Sinter @ 590°C for 5 hours



Observations after degas:
- Surface color only changed for sample degassed at 405°C, and discoloration was minimal
- No Obvious blistering

Observations after sinter:
- Surface color changed for all samples. Blistering was exceptionally dark for the sample that did not receive a degas heat treatment (AS)
- All samples indicated blistering on the surface

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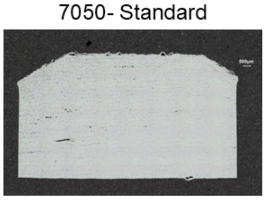
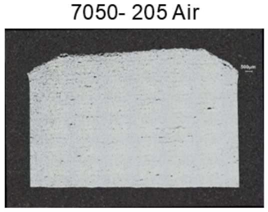
CROSS SECTIONS

Material	Sample	Process	Optical Porosity (%)
AA7050	7050 Standard	Sintered at 590°C in air. No degas	0.723
AA7050	205Air	Sintered at 590°C in air. Degassed at 205 in Air	1.193
AA7050	205Vac	Sintered at 590°C in air. Degassed at 205 under vacuum	1.121
AA7050	400Vac	Sintered at 590°C in air. Degassed at 405 under vacuum	6.077
AA6061	6061 Standard	Sintered at 610°C in air. No degas	7.507
AA6061	Stepwise	Sintered at 610°C in air. Stepwise degas	8.294
AA6061	Smaller Layers	Sintered at 610°C in air. No degas. Smaller layer thickness	15.039

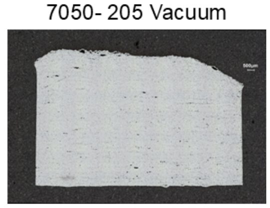
7050- Standard



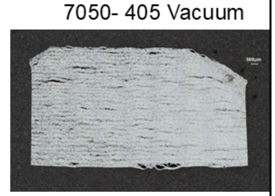
7050- 205 Air



7050- 205 Vacuum

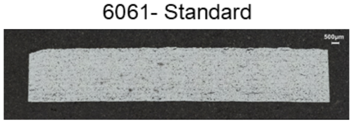


7050- 405 Vacuum

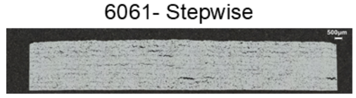


Porosity: 0.723% 1.193% 1.121% 6.077%

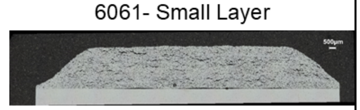
6061- Standard



6061- Stepwise



6061- Small Layer



Porosity: 7.507% 8.294% 15.039%

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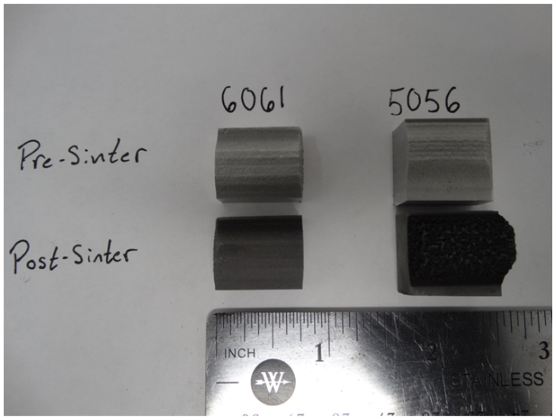
610°C SINTER OF 5056 AND DEGASSED 6061



Observations after HT:
- 5056
  - Significant blistering
  - Significant color change
  - Sample looked like “black coral”
    - Color change is likely due to going to far above the solidus and the material absorbing oxygen and nitrogen from atmosphere.
- 6061
  - Non-obvious blistering of sample
    - Cross-sectional analysis will be required to confirm level of blistering and interlayer delamination.
  - Color change was evident, but not to the extent of the 5056 sample.

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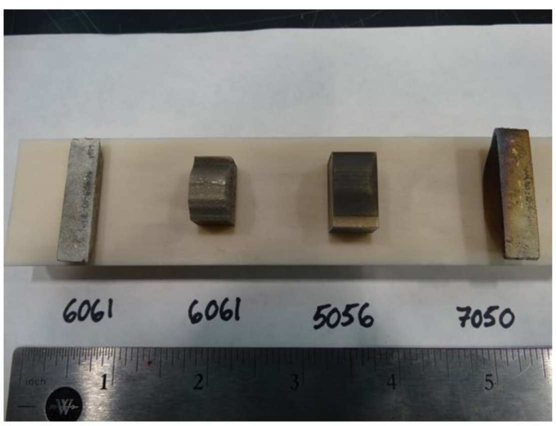
530°C HEAT TREATMENT IN AIR



Observations after HT:
- 5056
  - Slight surface discoloration
  - Slight surface blistering*
- 6061
  - Minimal discoloration on both samples
  - Minimal (if any) blistering on the surface*
- 7050
  - Significant discoloration on sample
  - Significant blistering on the surface visible by eye*

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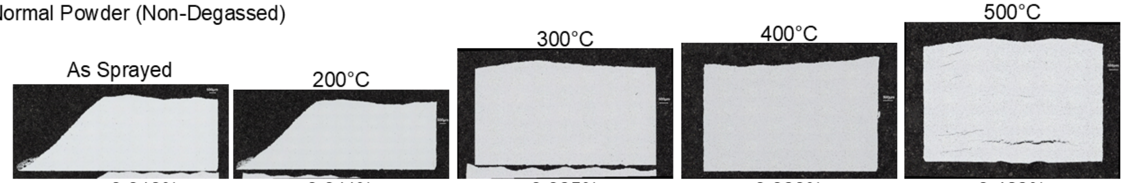
CROSS SECTIONS

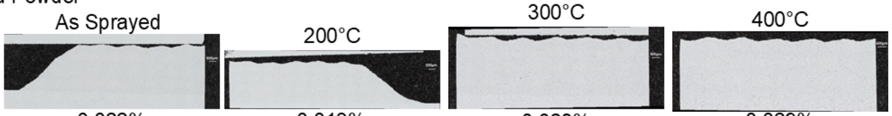
Sample	Process	Optical Porosity (%)
AS	Powder directly from bag. No HT	0.010
AS_Degas	Powder degassed prior to spray. No HT	0.022
200C	Powder directly from bag. CS annealed at 200°C for 5hrs	0.011
200C_Degas	Powder degassed prior to spray. CS annealed at 200°C for 5hrs	0.013
300C	Powder directly from bag. CS annealed at 300°C for 5hrs	0.035
300C_Degas	Powder degassed prior to spray. CS annealed at 300°C for 5hrs	0.028
400C	Powder directly from bag. CS annealed at 400°C for 5hrs	0.030
400C_Degas	Powder degassed prior to spray. CS annealed at 400°C for 5hrs	0.029
500C	Powder directly from bag. CS annealed at 500°C for 5hrs	0.483

Normal Powder (Non-Degassed)



Porosity: 0.010% 0.011% 0.035% 0.030% 0.483%

Degassed Powder



Porosity: 0.022% 0.013% 0.028% 0.029%

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POROSITY ACROSS ALL SAMPLES

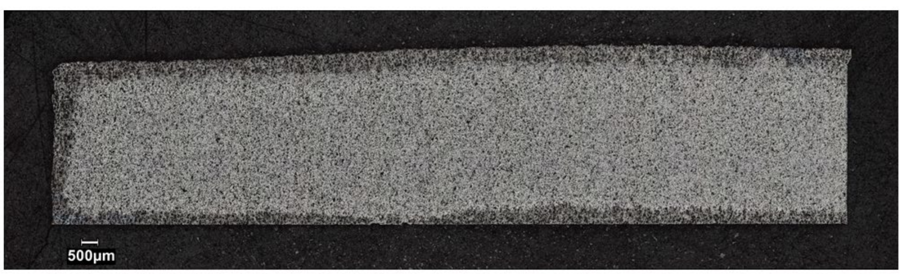
Material	Sample	Process	Optical Porosity (%)	Difference from no HT (%)
7050	VM24-10-2-1-1_7050_AS	No HT	0.131	
7050	VM24-10-2-1-1_7050_HT241115	Heated to 530°C in air	2.531	2.400
7050	VM24-10-2-1-1 Sintered	Sintered at 590°C in air. No degas	0.723	0.593
7050	VM24-10-2-1-1 205Air	Sintered at 590°C in air. Degas HT at 205 in Air	1.193	1.063
7050	VM24-10-2-1-1 205Vac	Sintered at 590°C in air. Degas HT at 205 under vacuum	1.121	0.990
7050	VM24-10-2-1-1 400Vac	Sintered at 590°C in air. Degas HT at 405 under vacuum	6.077	5.947
6061	VM24-11-7-1-1_6061_Degas_AS	6061 powder degassed. No HT	0.027	
6061	VM24-11-7-1-1_6061_Degas_HT241115	6061 powder degassed. Heated to 530°C in air for 5 hrs	0.513	0.486
6061	VM24-11-7-1-1 6061 Degas HT241114	6061 powder degassed. Sintered at 610°C in air	1.869	1.843
6061	VM24-10-31-1-13_6061_HT241115	Heated to 530°C in air	0.982	0.956
6061	VM24-10-31-1-13 Standard Sintered	Sintered at 610°C in air. No degas HT	7.507	7.480
6061	VM24-10-31-1-13 Step Sintered	Sintered at 610°C in air. Stepwise degas HT	8.294	8.267
6061	VM24-11-4-1-1 Sintered (Smaller Layers)	Sintered at 610°C in air. No degas. Smaller layer thickness	15.039	15.012
6061	VM24-10-9-1-1 Tensile Plate (Repurpose)	As Sprayed	0.010	
6061	VM24-12-3-1-1 Degas As-Received	As Sprayed	0.022	
6061	VM24-10-9-1-1_Tensile_Plate (Repurpose)	Annealed at 200°C	0.011	0.001
6061	VM24-12-3-1-1 Degas As-Received	Annealed at 200°C	0.013	-0.009
6061	VM24-10-9-1-1 Tensile Plate (Repurpose)	Annealed at 300°C	0.035	0.025
6061	VM24-12-3-1-1 Degas As-Received	Annealed at 300°C	0.028	0.007
6061	VM24-10-9-1-1 Tensile Plate (Repurpose)	Annealed at 400°C	0.030	0.019
6061	VM24-12-3-1-1 Degas As-Received	Annealed at 400°C	0.029	0.008
6061	VM24-10-9-1-1 Tensile Plate (Repurpose)	Annealed at 500°C	0.483	0.473
5056	VM24-11-12-1-1_5056_AS	No HT	0.091	
5056	VM24-11-12-1-1_5056_HT241115	Heated to 530°C in air	3.872	3.781
5056	VM24-11-12-1-1 5056 HT241114	Sintered at 610°C in air	40.814	40.723

- 5056 had the most significant increase in porosity across all materials. This is followed by the 6061 samples heated to 610C.
- We see in increase in porosity of 6061 when heated to 530C, which is significantly below the solidus temperature for AA6061.
- The degassed powder shows the lowest porosity across all heat-treated samples.

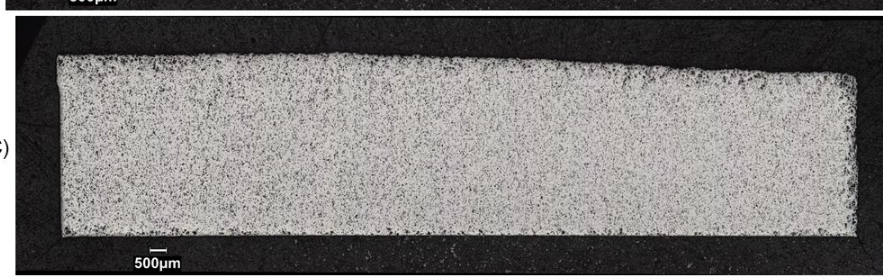
CS-CP-Ti POROSITY

CP-Ti TENSILE PLATE CROSS SECTION

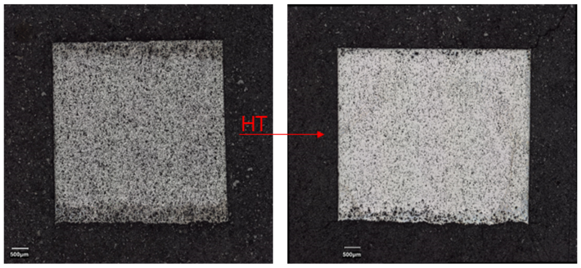
As-Sprayed



Heat Treated
(1hr @ 1100°C)



CP-Ti TENSILE PLATE DENSITY MEASURMENTS



Method	Sample	Porosity (%)
Optical	AS	30.939
	HT	14.042
	LS_2	38.915
	LS_3	30.423
Archimedes	AS Pre bubbling	14.060
	AS Post bubbling	11.260
Pyc	AS 1	4.927
	AS 2	9.604
	AS 3	-0.394
	AS 4	-0.698

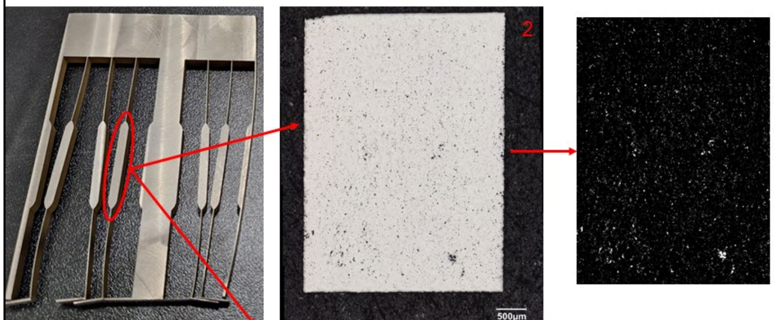
The change in porosity following heat treatment indicates that a significant portion of the porosity in the as-sprayed sample is likely pull out.

Pycnometry doesn't seem to be the most reliable way to measure density. This is because if there is a significant level of open porosity it will give inaccurate measurements (Samples 3 and 4).

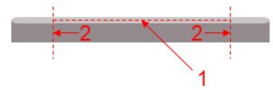
Would estimate porosity is about 14%

CP-Ti CS tensile plate release

TENSILE SPECIMEN MICROSTRUCTURE



Sample	Orientation	Optical Porosity (%)
1	1	3.45
2	2	2.41
3	2	4.28

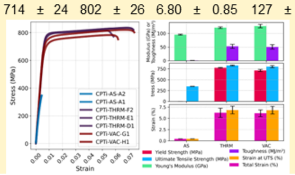


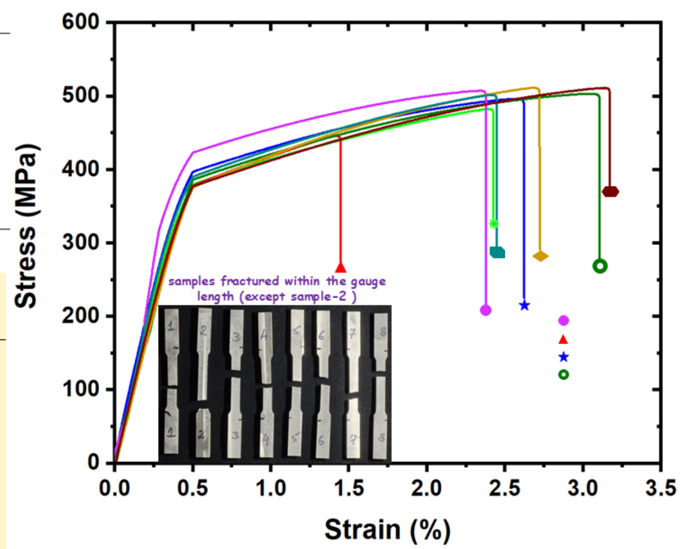
CP-Ti Tensile Results

Sample	0.2% YS Mpa (ksi)	UTS Mpa (ksi)	%El
1	386 (56)	482 (70)	2.43
2	390 (56)	446 (65)	1.45
3	405 (58)	496 (72)	2.62
4	399 (57)	503 (73)	3.11
5	429 (62)	506 (73)	2.38
6	391 (57)	511 (74)	2.73
7	399 (57)	501 (73)	2.45
8	387 (56)	511 (74)	3.18
Avg.	398 ± 13 (57 ± 1.8)	495 ± 21 (72 ± 3)	2.54 ± 0.54

From ARL-TR-10063:

Sample	YS(MPa)	UTS (MPa)	El (%)	Young's Modulus (GPa)	Toughness (MJ/m³)
AS	Fractured before yield	345 ± 2	0.44 ± 0.01	95 ± 2	0.9 ± 0.0
VAC	714 ± 24	802 ± 26	6.80 ± 0.85	127 ± 6	50.7 ± 8.3



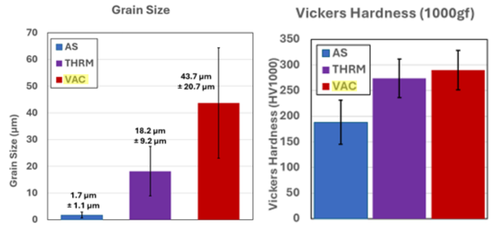


CP-Ti Tensile Results (Continued)

Average hardness (0.1 HV) = 189 ± 64

Average Grain Size = 45.0072 ± 20.34152

From ARL-TR-10063:



- The difference in the Hardness values from the Titan sample and ours is likely due to the increase in porosity in our sample. Since both samples have similar grain sizes following the same heat treatment the grains within the powder likely also started at the same size, meaning that there was likely not significantly more thermal treatment done to one powder and not the other.

CP-Ti POWDER ONH TESTING

Sample		ONH Data			ONH Raw Data		
System	Temp	O (%)	N (%)	H (ppm)	O (%)	N (%)	H (ppm)
					0.444	0.0265	558
					0.418	0.0256	557
GenIII	650	0.427±0.014	0.0254±0.0012	555±4.4	0.420	0.0241	550
					0.448	0.0267	606
					0.454	0.0257	634
GenIV	750	0.454±0.006	0.0271±0.0017	621±14.1	0.459	0.0290	623
					0.437	0.0289	592
					0.438	0.0278	581
GenIV	800	0.439±0.003	0.0283±0.0006	588±6.1	0.443	0.0282	591
					0.433	0.0160	551
					0.411	0.0136	544
TiAM-LITE		0.423±0.011	0.0147±0.0012	547.7±3.5	0.424	0.0144	548

CONCLUSIONS

CS-AA6061

- Significant blistering (or "bubbling) in Al deposits appearing when heated to temperatures greater than 500°C.
  - Since the in-house degassed powder seems to preform the best (most minimal increase in porosity), the blistering seems to be the biproduct of the hydroxide layer that is formed on the surface of Al powder. At elevated temperatures, the embedded hydroxides break down into gas, which builds up pressure and forms porosity when the yield stress of aluminum reaches a critical threshold during heating.
  - When samples were annealed at temperatures between 200°C and 500°C we noticed that there wasn't a significant increase in porosity until about 500°C, indicating we may be able to utilize lower temperature heat treatments to restore some of the mechanical properties.

CS-CP-Ti

- Significant level of porosity in the as sprayed condition. Difficult to accurately measure but it seem to be somewhere between 2-15%.
  - Pull-out during polishing makes accurate optical density measurements unreliable.
  - The substrate material plays a large part in the porosity, with Al substrates having the lowest porosity.
- Overall lower mechanical performance than past sprays indicates that the feedstock powder is leading to the decreased performance.
- No noticeable increase in porosity when CS-CP-Ti is heat treated.

MOVING FORWARD

CS-AA6061

- Look into alternative powders that have a low hydroxide content on the surface. This should allow for minimal blistering during the sintering and solution treating steps of the heat treatments.
- Determine mechanical properties CS-AA6061 heat treated at low temperatures (<500°C).
  - Determine if the required properties can be achieved via a heat treatment besides the T6.

CS-CP-Ti

- Improve as spray density of CS-CP-Ti
  - Look into different powder alternatives that preform better during the cold spray process, resulting in a higher density deposit.
  - Look into using Ti64 blending in with the CP-Ti, this may improve the density of the deposit through in-situ shot peening. Embedded Ti64 will likely have minimal effect on the overall mechanical properties, especially if it improves the overall density.

THANK YOU.

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U.S. ARMY

List of Symbols, Abbreviations, and Acronyms

AL	aluminum
ARL	Army Research Laboratory
CP	commercially pure
CS	cold sprayed
DEVCOM	U.S. Army Combat Capabilities Development Command
HT	heat treatment
Ti	titanium

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