



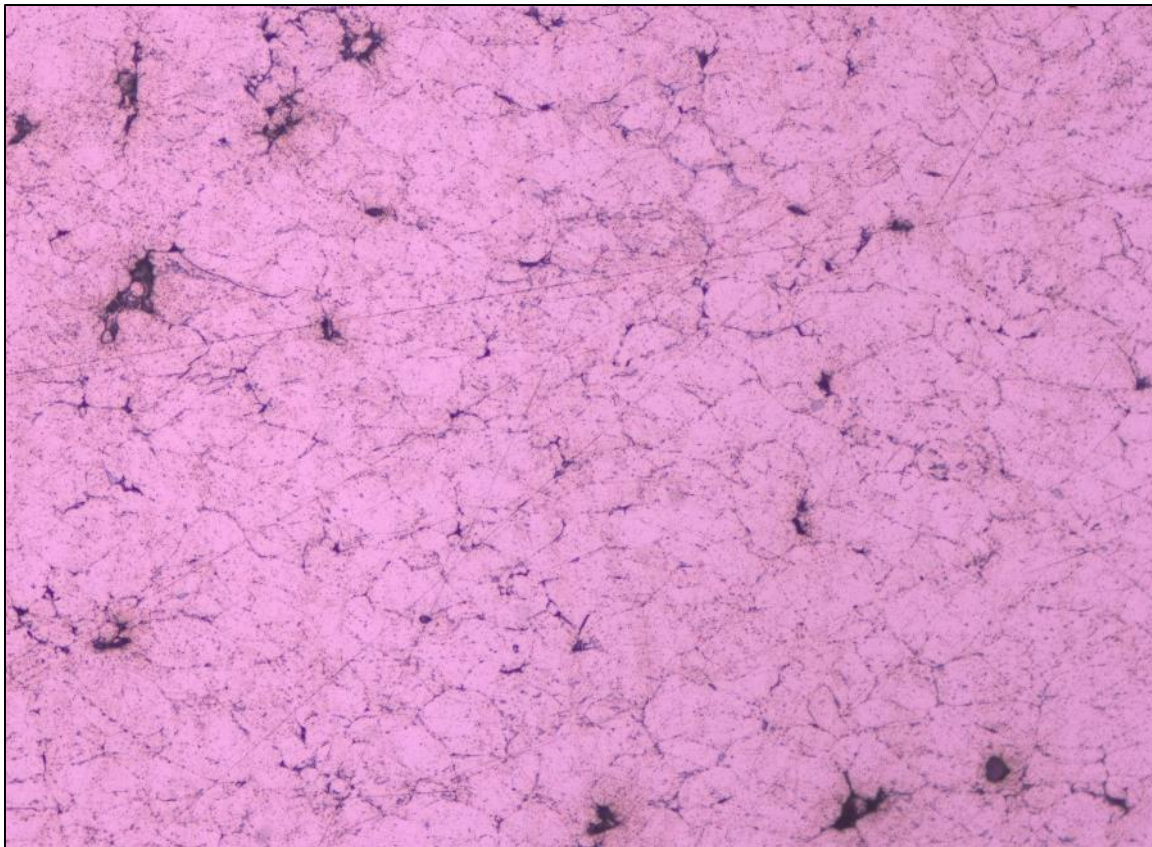
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Analysis of 6061 Aluminum Cold Spray Material

Research and Development Office

Science and Technology Program

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Cover Photograph: Micrograph of cold spray 6061 aluminum (Reclamation/Matthew Jermyn).

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The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

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Technical Memorandum No. 8540-2021-018

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Acronyms and Abbreviations

Al	aluminum
Al 6061	6061 aluminum
AM	additive manufacturing
ASTM	ASTM International, formerly American Society for Testing and Materials
F	face
FPY	fractured prior to yield
HRB	Hardness Rockwell B
Mg ₂ Si	magnesium silicide
NaOH	sodium hydroxide
Reclamation	Bureau of Reclamation
RPM	rotations per minute
UTS	ultimate tensile strength
X	cross-section

Symbols and Measurements

%	percent
°C	degree Celsius
cSt	centistokes
D_s	sintered density
g/cc	grams per cubic centimeter
in ²	square inch
ksi	kilopounds per square inch
lb	pound
lb/in ²	pound per square inch
ρ_w	density of water
μm	micrometers

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Introduction

Background

The Bureau of Reclamation (Reclamation) undertook a study to investigate and demonstrate the capabilities of additive manufacturing (AM) for hydropower applications. The study was broken into two phases. Phase 1 investigates three AM techniques of interest thus to evaluate the processes and resulting material properties—Direct Energy Deposition, Laser Powder Bed Fusion, and Cold Spray. Phase 2 presents three case studies in which a component of interest is reproduced using knowledge gained during phase 1.

This final report focuses specifically on the investigation and analysis of 6061 aluminum (Al 6061) cold spray material and is intended to be incorporated into the larger context of AM for hydropower applications, which will be presented as Final Report No. ST-2021-19085-1, Technical Memorandum No. 8540-2021-015.

6061 Aluminum and the Cold Spray Process

This work sought to evaluate the mechanical properties and microstructure of Al 6061 additively manufactured through the cold spray process. Cold spray involves the high velocity acceleration of fine powder particles in a compressed gas stream onto a substrate. Upon impact, the particles bond strongly together to form a layer without damaging the underlying substrate. One major benefit of cold spray is the ability to apply and build up material directly onto a part. Additionally, the print material could also be sprayed onto a disposable backing substrate that is easily removed once the build is finished.

Figure 1 shows two cold spray Al 6061 blocks. One block was printed in the “Z” direction and the other was printed in the “XY” direction to allow researchers to obtain test specimens with print directions along different axes.



Figure 1.—Cold spray Al 6061 blocks printed in the Z (top) and XY (bottom) directions.

Methodology

Cold Spray Additive Manufacturing

The cold spray printer had a maximum deposition rate of 100 grams of material per minute with a deposition spot size of 6 millimeters. After printing, the two blocks underwent a T6 heat treatment, which is a two-step process. First, the material is heated to 500 degrees Celsius (°C) for 9 to 10 hours and quenched to achieve a single-phase alloy. Next, a second heat treatment is performed at 180 °C for 9 to 10 hours, followed by air cooling, which results in precipitation hardening of the material.

Test Specimens

Five ASTM E-8/E8M-21 Standard (ASTM International, 2021) tensile specimens (labeled Z1 through Z5 and XY1 through XY5) were machined from each of the two blocks for testing and a strip of material from each block was reserved for hardness traverses (figure 2).

In addition to these “Z” and “XY” tensile specimens, the manufacturer provided test specimens (labeled A1 through A5) that were printed using a slightly different Al 6061 alloy. These “A” specimens were printed in the same orientation as the XY specimens.

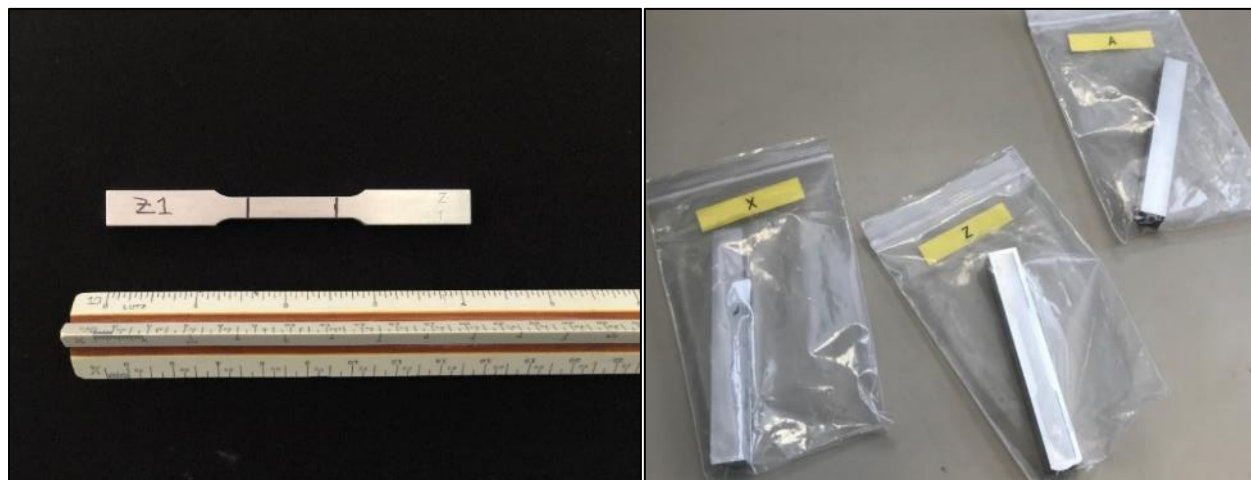


Figure 2.—Representative tensile specimen (left) and test bars for hardness traverses (right) machined from the cold spray Al 6061 blocks.

Metallographic Analysis

Tested tensile specimens were used for metallography. For each specimen group (Z, XY, and A), metallography was performed on the face (F) and cross-section (X). A total of six samples were

analyzed. Figure 3 gives a schematic of the Z specimens (left) and XY/A specimens (right) indicating the face and cross-section regions.

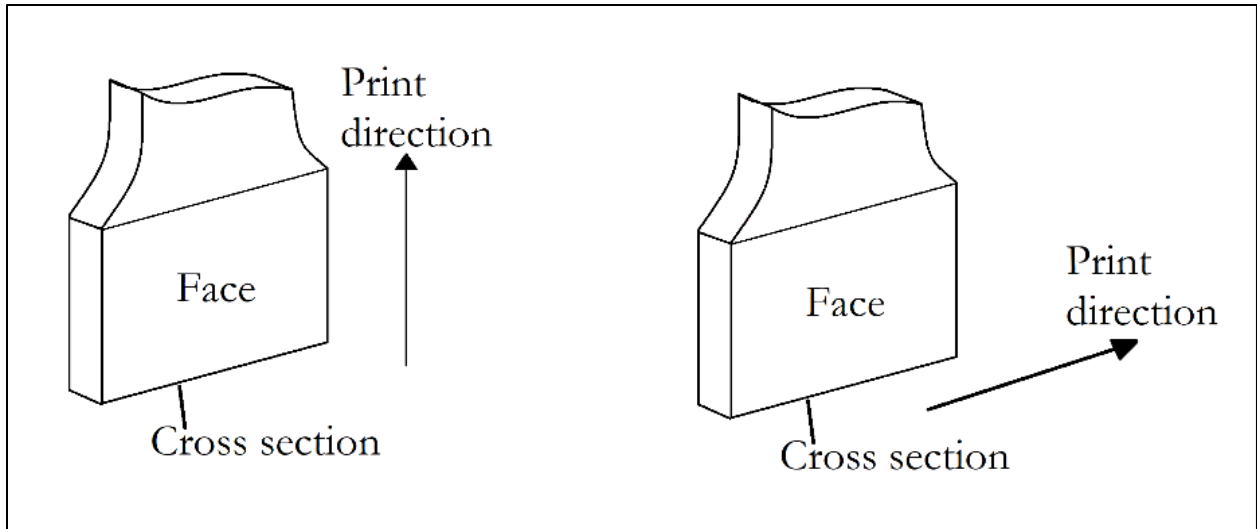


Figure 3.—Schematic showing print direction, face, and cross-section regions of the Z (left) and XY/A specimens (right). Metallography was performed on the face and the cross-section.

Metallography gives a representative image of the metal without any influence from cold working or other processing damage. The following procedure was used for each metallographic specimen:

1. Section metallographic specimens from the tested tensile specimens using an abrasive metallography saw.
2. Cold mount in epoxy and harden for a minimum of 24 hours
3. Grind from coarser to finer grits (force: 40 Newtons; head and wheel rotations: 100 rotations per minute [RPM]):
 - a. 180 for 2 minutes
 - b. 400 for 2 minutes
 - c. 600 grit for 2 minutes
4. Polish to a mirror finish:
 - a. 9-micron diamond polish for 4 minutes
 - b. 6-micron diamond polish for 2 minutes
 - c. 3-micron diamond polish for 2 minutes
 - d. 1-micron diamond polish for 1 minute
5. Etch with a solution of 1 gram sodium hydroxide (NaOH) in 100 milliliter water for 15, 20, or 25 minutes until desired etch is achieved.
6. Image at 20X, 50X, and 100X magnifications

Density Measurement

The density of the six samples was tested using ASTM B962-17, “Standard Test Methods for Density of Compacted or Sinter Powder Metallurgy (PM) Products Using Archimedes’ Principle” (ASTM International, 2017). This standard gives procedures for infiltrating specimens with oil and recommends vacuum infiltration. Due to material and time constraints, researchers lowered the surface pressure by boiling rather than using vacuum infiltration. Since boiling causes the surface pressure on a submerged specimen to drop below one atmosphere, boiling specimens in hydraulic mineral oil with viscosity between 20 and 65 centistokes (cSt) at 38 °C will allow the oil to infiltrate any surface-connected porosity. For these tests, specimens were boiled in mineral oil with a viscosity of 32 cSt for 1 hour.

The adjusted procedure requires three mass measurements with a balance:

1. Mass of sintered part in air, A
2. Mass of oil-impregnated part in air, B
3. Mass of oil-impregnated part in water, C

Each mass reading was taken three times per specimen and averaged to obtain the final reading. Using these masses and the density of the water, ρ_w (dependent on water temperature), the density was calculated using the following equation:

$$\text{Sintered Density} = D_s = \frac{A\rho_w}{B - C}$$

Notes:

1. A balance was not available, so a testing apparatus using a scale was devised to obtain the mass of a hanging specimen. Variation due to this deviation is likely negligible.
2. All density test specimens were sectioned from previously tested tensile; this could provide a potential source of variation from the as-printed material.

Hardness Testing

Hardness testing was performed using a Rockwell Hardness B tester across the test bars for each specimen group at 0.25-inch step increments in accordance with ASTM E18-02, “Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials” (ASTM International, 2002). Test bars were 4.5 inches long.

Tensile Testing

Tensile testing was performed in accordance with ASTM E8/E8M-21, “Standard Test Methods for Tension Testing of Metallic Materials” for subsize specimens at room temperature (ASTM International, 2021). The strain rate was 0.0625 inch per minute.

Fractography

Macro images of the fracture surfaces that resulted from tensile testing were taken using a DSLR camera with lighting adjusted to highlight fracture features.

Results and Analysis

Metallographic Analysis

The metallographic analysis allowed for characterization of the microstructure, which can provide insight into mechanical test results and other material properties. Researchers also collected porosity data through micrograph analysis. The microstructure of wrought Al 6061 is characterized by an aluminum matrix with varying amounts of magnesium silicide (Mg_2Si) inclusions, which appear on the micrograph as small, dark spots [1]. Grains—delineated by fine grain boundaries—range in size depending on stress, heat treatments, or other processes.

Microstructure

The micrograph of the face of specimen XY, XY(F), shown in figure 4 is representative of the general microstructure of cold spray Al 6061, and is very similar to that of the wrought version. This micrograph shows large dark regions (pores), fainter dark lines, and small dark spots (inclusions). The smallest of these dark spots are precipitate phases of Mg_2Si , which is typical of Al 6061 [1]. There are also light grey regions that could be other phases not typically found in wrought Al 6061. Another major difference between the cold spray and wrought microstructures is the prevalence of pores in the cold spray microstructure.

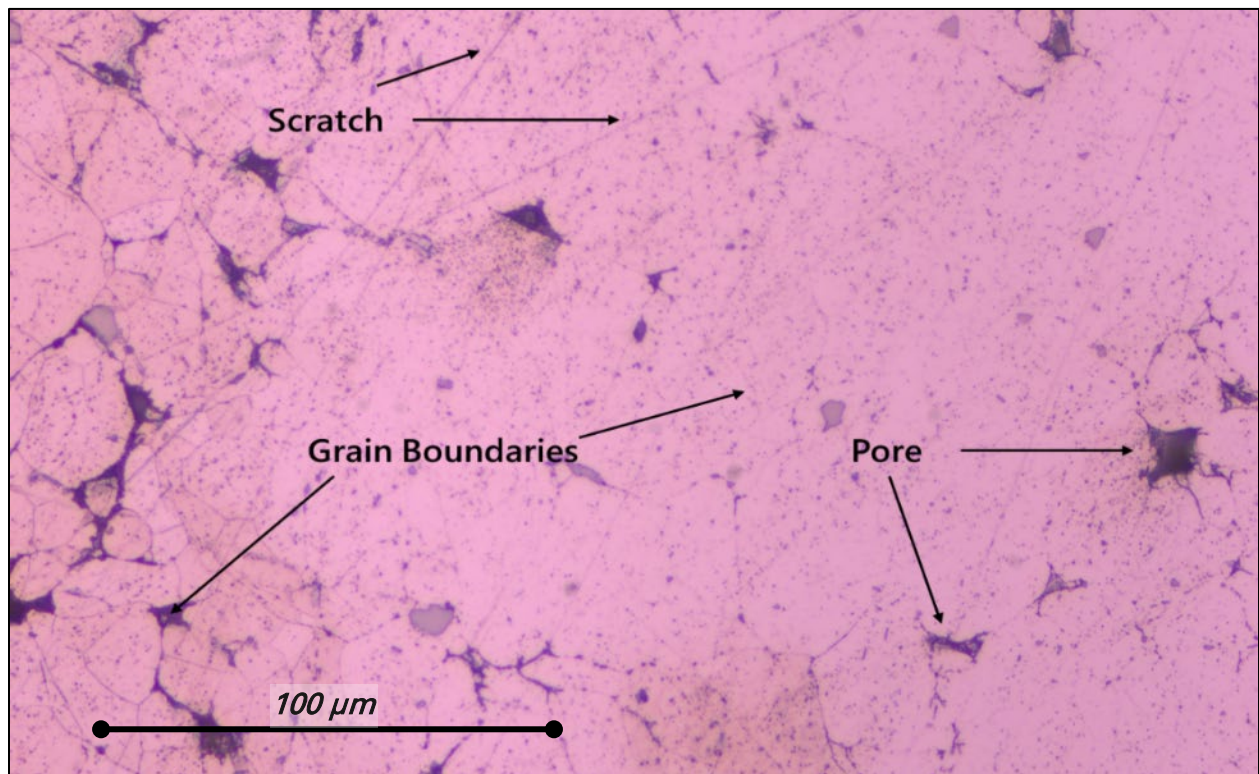


Figure 4.—Key features are labeled on a representative micrograph of the face of specimen XY etched for 15 minutes in NaOH and imaged at a magnification of 20X.

Porosity Measurement

To quantify porosity, researchers performed an analysis of the micrographs obtained through metallography, calculating the area fraction of pores in relation to the whole image. The results of porosity area analysis are shown in table 1.

Table 1.—Calculated Porosity

Specimen Sample	Average Percent Porosity (%)	Standard Deviation
A(F)	2.38	1.09
XY(F)	1.46	0.22
XY(X)	1.83	0.80
Z(F)	2.22	0.59
Z(X)	3.56	2.24
Cold Spray Al (reported) [2]	1.53–2.66	0.61–1.71
Wrought Al 6061	Variable	–

The porosity values and variability are within the range of porosity values for cold spray aluminum found in literature. Specimens A(F) and Z(X) are the most porous while XY(F) and XY(X) are the least porous. The cold spray Al 6061 is less porous in the XY direction than in the Z direction. The alloy of the A specimens resulted in more porous samples than the XY specimens despite being printed in the same direction.

Density Measurement

To characterize the prevalence of voids in the larger fabrication, specimen density was measured and compared to literature values. Table 2 shows specimen and average densities for the three specimen groups.

Table 2.—Density Results

Specimen Identification (ID)	Density (g/cc)	Percent Dense (%)
A2	2.50	92.60
A3	2.52	93.10
A5	2.50	92.50
Average A	2.51	92.73
XY2	2.52	93.40
XY3	2.54	94.20
XY5	2.51	93.10
Average XY	2.52	93.57
Z1	2.51	93.10
Z4	2.52	93.20
Z5	2.52	93.50
Average Z	2.52	93.27
Wrought Al 6061 (reported) [3]	2.70	–

The average density of all cold spray specimens is 2.52 grams per cubic centimeter (g/cc) with a standard deviation of 0.01 g/cc. Standard deviations for density measurements are not reported because they were so close to zero. The average densities of the XY and Z specimen groups (printed with the same alloy) are the same, while the A specimens (printed with a different alloy) were slightly less dense on average. All cold spray specimens were less dense than wrought Al 6061, with typical reported values for the density of wrought Al 6061 around 2.70 g/cc. On average, all cold spray specimens are 93.2 ± 0.5 percent (%) dense, demonstrating a notable amount of internal porosity compared to the conventional Al 6061 material.

Hardness Testing

The reported hardness for T6 tempered Al 6061 is 56.2 Rockwell Hardness B (HRB) and 55 HRB for A356-T6 [4, 5]. For each of the three specimen groups, data from Rockwell Hardness B traverses is compiled in figure 5.

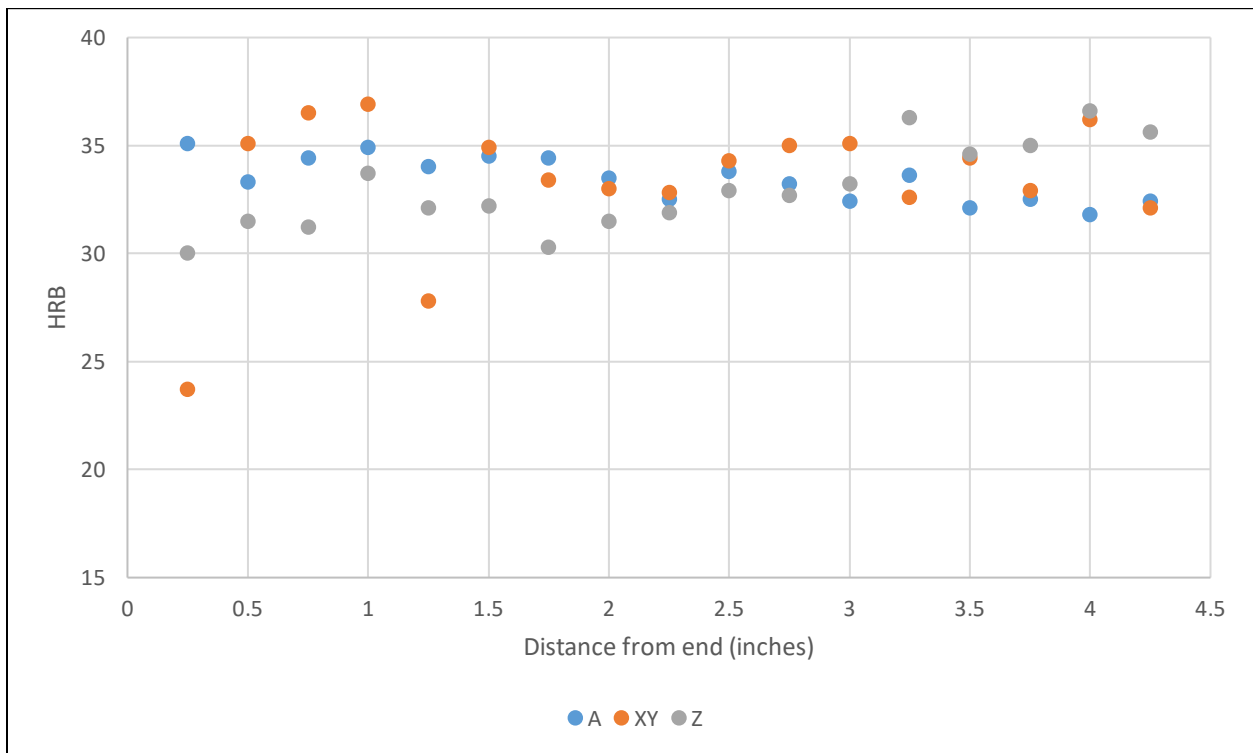


Figure 5.—Rockwell Hardness B traverses for each specimen group’s test bar.

Near the end of the test bars, there is a wide variation in hardness between the specimens. Excluding the first datapoint (at 0.25 inch), the XY specimen had the highest hardness, exceeding 35 HRB, followed by the A specimen, then by the Z specimen. The hardness values follow that order while gradually converging to approximately 32 HRB near the center of the test bars (between 2 and 2.5 inches). The XY and Z specimens generally had more varied hardness, with values for XY ranging from sub-25 HRB to 37 HRB, and Z hardness ranging from 30 to 36.5 HRB. The A alloy had much more consistent hardness values across the test bar, with a range of 32 to 35 HRB. The

discrepancy between the cold spray and conventional Al 6061 hardness could be due to the high amount of porosity in the cold spray samples, or just as an implication of the printing process.

Tensile Testing

Figures 6 through 8 show the stress-strain curves for each specimen of the three groups, A, XY, and Z, respectively. Table 3 gives the average ultimate tensile strength (UTS), yield stress, percent elongation for the three specimen groups. Complete tensile test results are found in appendix A. UTS and yield stress values were obtained directly from each specimen's stress-strain curve and averaged. The percent elongation was obtained by physically measuring the specimen after breaking and calculating the percent change from the original length. The average elastic modulus was determined by calculating the slope of the elastic region (initial linear portion) of each curve.

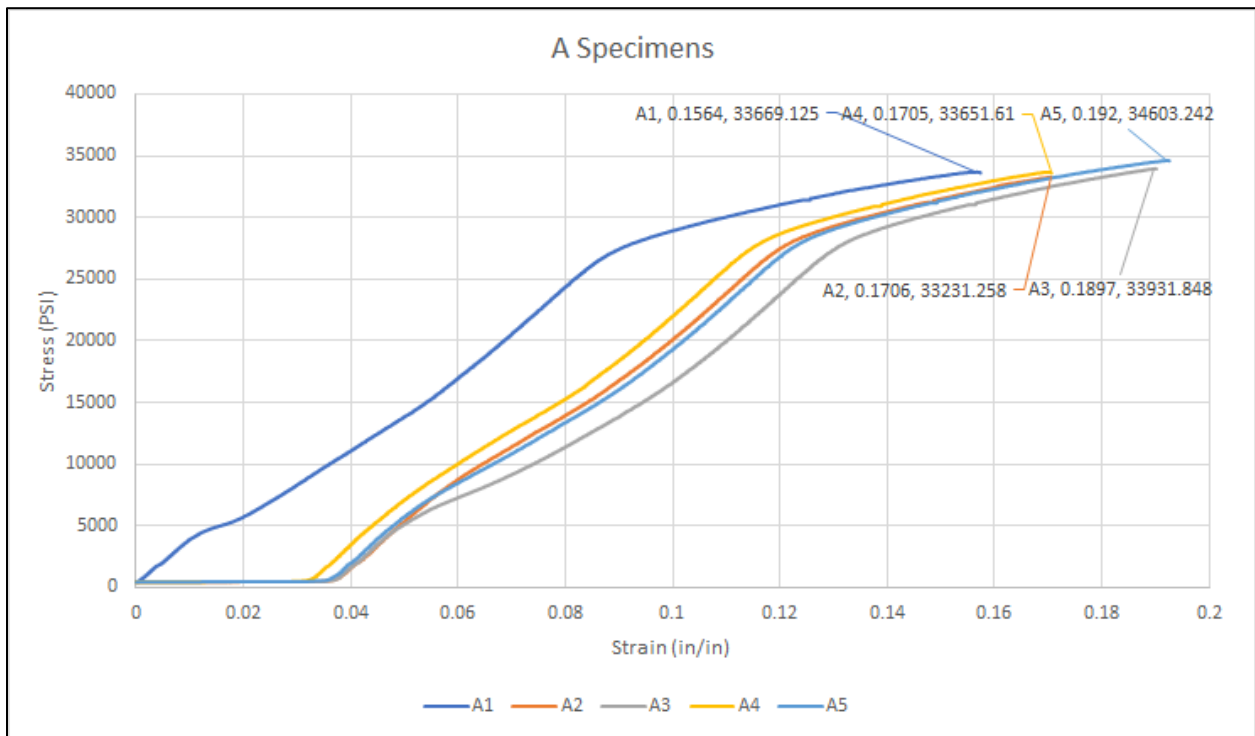


Figure 6.—Compilation graph of the stress-strain curve of five samples fabricated from the A alloy.

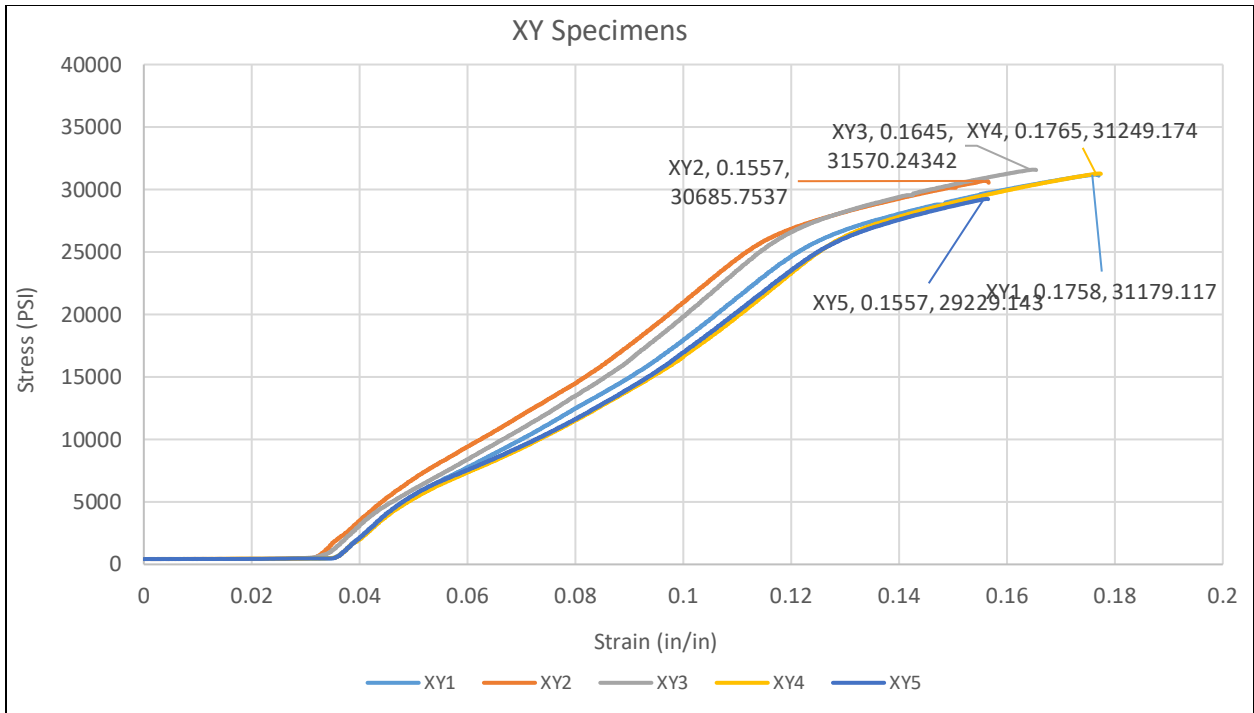


Figure 7.—Compilation graph of the stress-strain curve of five samples printed in the XY orientation.



Figure 8.—Compilation graph of the stress-strain curve of five samples printed in the Z orientation.

Table 3.—Stress and Percent Elongation Data for Cold Spray Al 6061 Tensile Specimens

Specimen Group	Average Ultimate Tensile Strength (ksi)	Average Yield Stress (ksi)	Average Elastic Modulus (ksi)	Average Percent Elongation (%)
A	33.86	27.86	290.3	4.8
XY	30.80	25.82	278.1	3.2
Z	28.24	27.17	274.5	0.4*
T6 Al 6061 (reported) [6, 7]	45	40	1007.6	12

*Note: Two of the five Z specimens fractured prior to yield, resulting in a 0% elongation value recorded for those specimens and lowering the average percent elongation for group Z.

Each specimen group had similar average UTS, yield stresses, and elastic moduli, with the A alloy exhibiting slightly better tensile properties than the XY and Z alloys. The A alloy also resulted in parts with larger percent elongation. The specimens printed in the Z direction had significantly lower elongation than the XY and A specimens, likely due to the force being applied perpendicular to the sprayed layers, which readily broke apart without much elongation. However, it is important to note that only three specimens were used to calculate elongation percent for Z, while five specimens were used to calculate elongation for each of XY and A. Two of the Z specimens failed prior to yielding, resulting in a zero percent elongation.

All cold spray specimens had significantly lower UTS, yield stress, elastic modulus, and percent elongation compared to the T6 Al 6061 specimens reported in literature. These differences are likely all due to the higher porosity within the cold spray specimens, which greatly reduced material strength, elasticity, and ductility.

Fractography

In literature, conventional T6 Al 6061 is described as a “highly ductile” material that exhibits classic cup-and-cone fracture [8]. Representative images of cold spray specimen fracture surfaces are shown in figure 9 for A (left), XY (center), and Z (right) specimens. The relatively flat fracture surfaces indicate brittle fracture modes, with the observation of little to no specimen necking supporting this finding. The very short plastic region of the stress-strain curves is also typical for brittle materials.

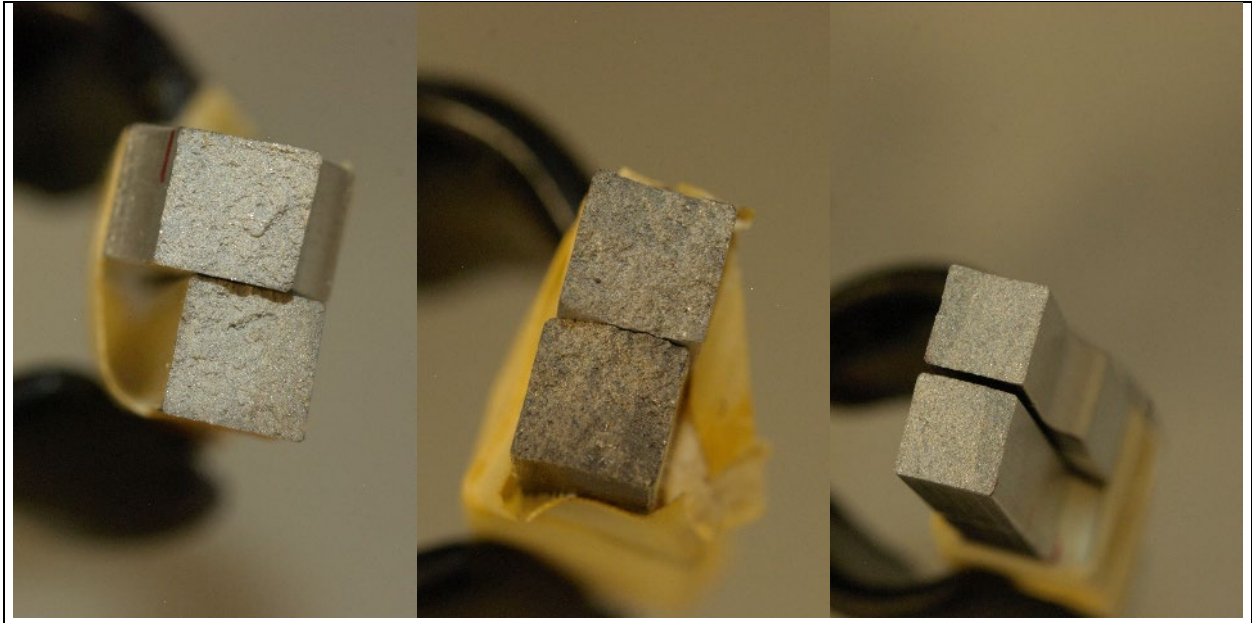


Figure 9.—Fracture surfaces of cold spray Al 6061 tensile test specimens. Left: A, center: XY, right: Z. Fracture surfaces are relatively flat, indicating brittle fracture modes.

Conclusions

Cold spray Al 6061 has significantly poorer mechanical properties compared to conventional Al 6061. While aluminum is typically a very ductile material, the cold spray process produced brittle samples with comparatively low elongation and elastic modulus. Aside from the high percentage of porosity noted in the cold spray microstructure, metallographic evaluation found similar microstructures between cold spray and conventional Al 6061, thus indicating porosity as the likely cause of poor mechanical properties.

Despite all cold spray specimens having relatively high porosity, the A alloy had better mechanical properties than the XY and Z alloys. Ultimately, however, none of the cold spray specimens came close to having mechanical properties that matched conventional Al 6061. Cold spray parts produced with the current printing process (while the porosity issue remains unsolved) should not be used as substitutes for conventional Al 6061 materials in critical applications.

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Appendix A—Tensile Test Results

Table A-1.—Tabulated Cold Spray Tensile Test Data

Specimen Sample	Width (inch)	Thickness (inch)	Area (in ²)	Yield Load (lb)	Yield Stress (lb/in ²)	Ultimate Tensile Load (lb)	Ultimate Tensile Strength (lb/in ²)	Elongation (inch)	Elongation (%)
A1	0.236	0.235	0.0555	1,491	26,900	1,868	33,700	1.05	5.00
A2	0.236	0.235	0.0555	1,551	28,000	1,844	33,300	1.05	5.00
A3	0.236	0.235	0.0555	1,552	28,000	1,883	34,000	1.05	5.00
A4	0.236	0.235	0.0555	1,566	28,200	1,868	33,700	1.04	4.00
A5	0.236	0.235	0.0555	1,562	28,200	1,920	34,600	1.05	5.00
X1	0.236	0.235	0.0555	1,400	25,200	1,730	31,200	1.04	4.00
X2	0.236	0.235	0.0555	1,421	25,600	1,702	30,700	1.03	3.00
X3	0.236	0.235	0.0555	1,467	26,500	1,752	31,600	1.04	4.00
X4	0.236	0.235	0.0555	1,454	26,200	1,735	31,300	1.03	3.00
X5	0.236	0.235	0.0555	1,421	25,600	1,622	29,200	1.02	2.00
Z1	0.236	0.235	0.0555	FPY*	FPY*	1,415	25,500	–	–
Z2	0.236	0.235	0.0555	FPY*	FPY*	1,461	26,300	–	–
Z3	0.236	0.235	0.0555	1,507	27,200	1,617	29,200	1.00	0.00
Z4	0.236	0.235	0.0555	1,514	27,300	1,658	29,900	1.01	1.00
Z5	0.236	0.235	0.0555	1,497	27,000	1,679	30,300	1.01	1.00

*Note: FPY = fractured prior to yield.