SAND2005-7691C

Mechanisms Resulting in Improved Ductility of Cold Spray Coatings after Annealing

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Abstract

An earlier study reported an investigation of the mechanical properties of cold sprayed aluminum and the effect of annealing on those properties [1]. In that study, cold spray coatings approximately one centimeter thick were prepared using three different feedstock powders: Valimet H-10, Valimet H-20, and Brodman Flomaster. ASTM E8 tensile specimens were machined from these coatings. Each material was tested in two conditions: as-sprayed and after a 300°C, 22 hour air anneal. The as-sprayed material showed high ultimate strength and low ductility, < 1% elongation. The annealed samples showed a reduction in ultimate strength but a dramatic increase in ductility, up to 10% elongation. Microstructural examination and fractography clearly showed a change in fracture mechanism between the as-sprayed and annealed material, but insufficient data was available to conclusively explain the ductility increase at that time. Since then, Kikuchi mapping of the Valimet H-10 material in the as-sprayed and annealed conditions has been conducted. The data shows that significant recrystallization within the splats upon annealing has occured. No significant crystal growth across splat boundaries is observed. The data demonstrate that the mechanism of ductility increase in annealed cold spray deposits is recrystallization of the base aluminum material.

Introduction

In the cold spray process, finely divided metal particles are accelerated in an inert gas jet to velocities in excess of 500 m/s. When accelerated to velocities above a material-dependent critical velocity, V_C, metal particles will bond to the substrate and form a dense, well-adhered deposit. This is the foundation of the cold spray process [2 - 4]. The cold spray process is capable of preparing deposits of low-oxide content in air at near-room-temperature conditions. This makes cold spray very attractive for many applications. Some of those applications, such as spray forming, require the cold-sprayed material to exhibit a modest amount of ductility. In their as-sprayed state, cold-sprayed metals tend to be brittle because the particles are heavily cold worked during the deposition process and interparticle bonding tends to be incomplete. The goal of this work is to better understand the factors affecting ductility of cold-sprayed aluminum deposits. This will allow new applications for cold spray processing such as spray forming.

Experimental Procedure

Cold spray tensile bars prepared from gas atomized aluminum powders Valimet (Stockton, CA) H-10 were investigated in this study. The H-10 powder had a volumetric mean particle size of 11.8 µm. It was cold sprayed using a helium accelerating gas pressure of 1723 kPa at 350˚C. The calculated average center-line velocity for the H-10 powder at these conditions was 1117 m/s. Powder feed gas pressure was maintained 5% higher than the accelerating gas pressure.

Spray deposits measuring 100 cm X 100 cm X 13 cm were laid down using a simple X-Y raster pattern. Tensile bars were machined from the as-sprayed deposits according to a modified ASTM E8 geometry. The gage diameter of these samples was reduced from its normal value of 6.35mm to 4.57mm because the 6.35mm gage diameter is so close to the root diameter of the 5/16”-24 UNF threads that the as-sprayed tensile samples were fracturing in the threads. Tensile bars were wrapped in aluminum foil then annealed at 300°C for 22 hours in air. Eight H-10 tensile bars were tested, 6 in the as-sprayed condition and two in the annealed condition.

The microstructures, fracture surfaces, and cross sections near the fracture of both the as-sprayed and annealed bars were examined using optical and scanning electron microscopy (SEM).

Results and Discussion

Micrographs of the as-sprayed deposits showed that the deposits produced from H-10 powder produced deposits have densities > 99.9% (Figure 1). No grain growth was observed in the annealed microstructure using optical microscopy (Figure 2).

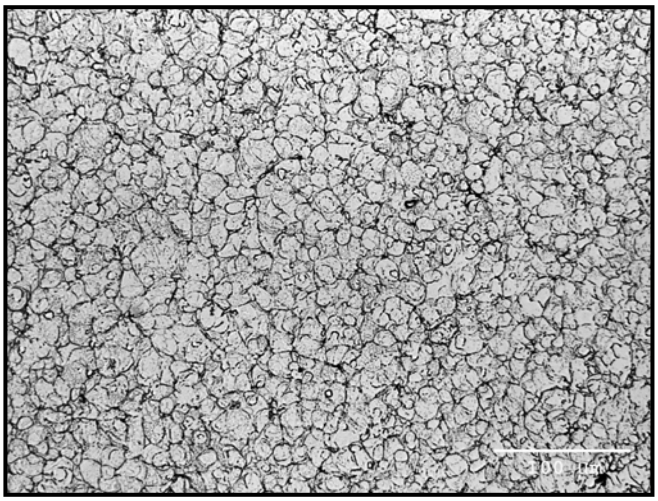

Figure 1. As-sprayed H-10 cold sprayed aluminum.

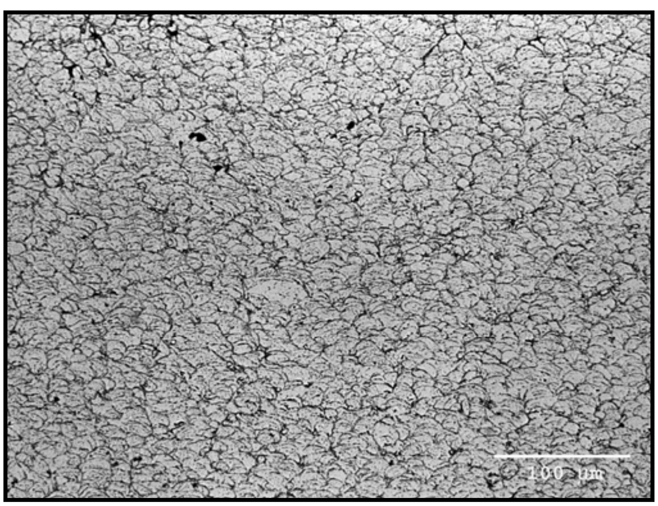

Figure 2. Annealed H-10 cold sprayed aluminum.

In the as-sprayed condition, the H-10 material exhibited brittle behavior; however, after annealing, it exhibited remarkable ductility. The H-10 deposits exhibited a maximum of 1% ductility in the as-sprayed condition. After annealing, its ductility increased by a factor of 10. The mechanical properties of the annealed H-10 material compares favorably with 1100 H14, a typical wrought aluminum [5]. The as-sprayed ultimate tensile strength was higher than that of the annealed samples. There was no significant difference in elastic modulus between the as-sprayed and the annealed samples. Tensile curves from the as-sprayed and annealed materials are shown in Figures 3 and 4.

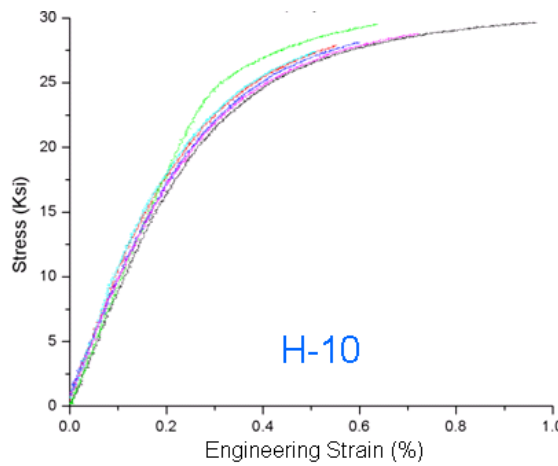

Figure 3: Tensile curve for as-sprayed H-10 aluminum samples showing less than 1% elongation to failure.

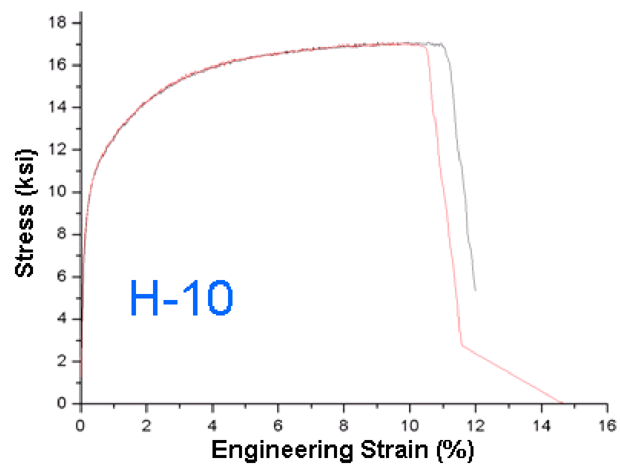

Figure 4: *Tensile curve for annealed H-10 aluminum samples showing over 10% elongation to failure.*

The fracture surfaces of the annealed deposits revealed features that are consistent with the observed increase in ductility. The as-sprayed material exhibited brittle failure with a relatively smooth and blocky fracture surface. The fracture surfaces of the as-sprayed and annealed material are shown in Figures 5 and 6. The fracture surface of the annealed material shown in Figure 6 is typical of ductile failure showing classic micro-void coalescence.

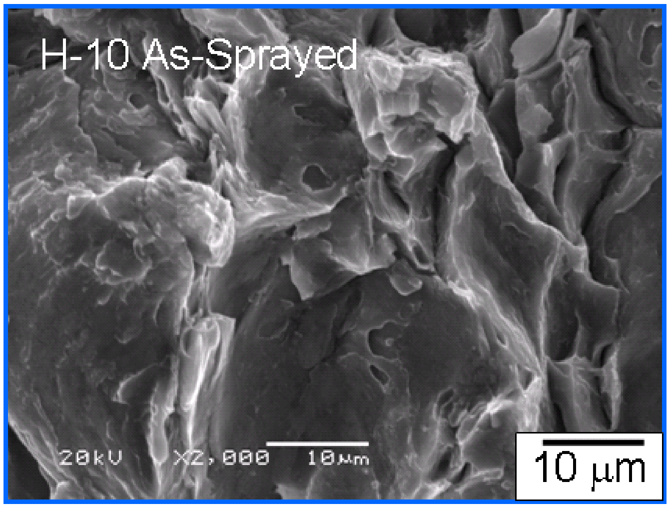

Figure 5: SEM micrograph showing the fracture surface of the as-sprayed H-10 material

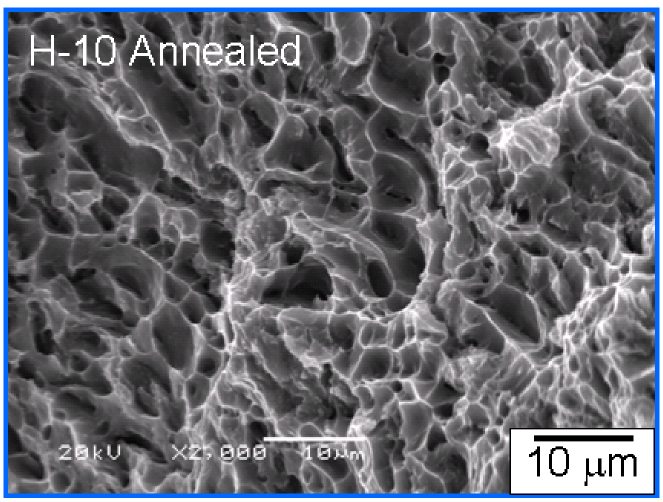

Figure 6: SEM micrograph showing the fracture surface of the annealed H-10 material.

Metallographic sections taken through the fracture region reveal that the as-sprayed specimens exhibit primarily brittle intragranular failure, showing that the splat boundaries are the favored site for crack propagation in the as-sprayed material. Some trans-granular fracture was observed in the as-sprayed material. Micrographs of the annealed material show considerable elongation and flow of the individual grains. The fracture appears to be mixed mode with intergranular failure and a large amount of void-coalescence typical of ductile fracture (Figure 7).

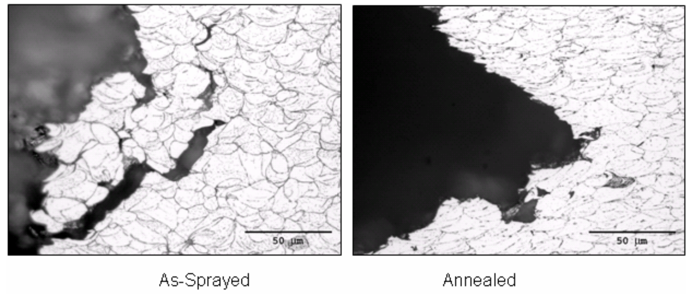

Figure 7. Metallographic section through the fracture surface of as-sprayed and annealed H-10 tensile bars.

Kikuchi maps were prepared in the grip section of both the as-sprayed and annealed tensile bars (Figure 8 and Figure 9). The grip sections of the tensile bars were analyzed to insure that deformation from the tensile test did not confound the results.

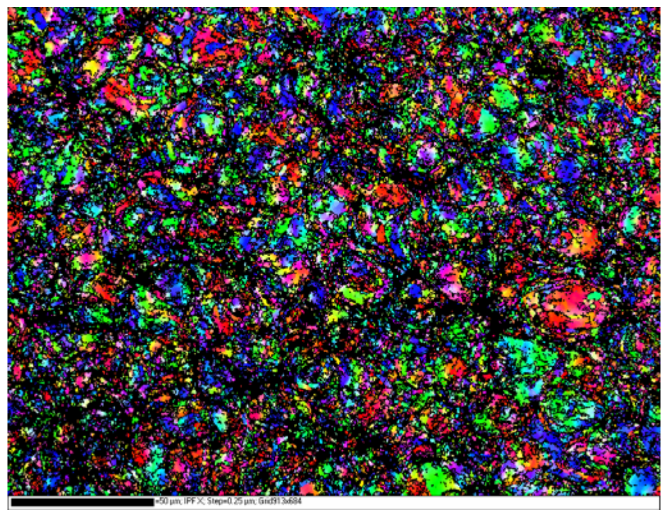

Figure 8. Kikuchi map of as-sprayed H-10 material.

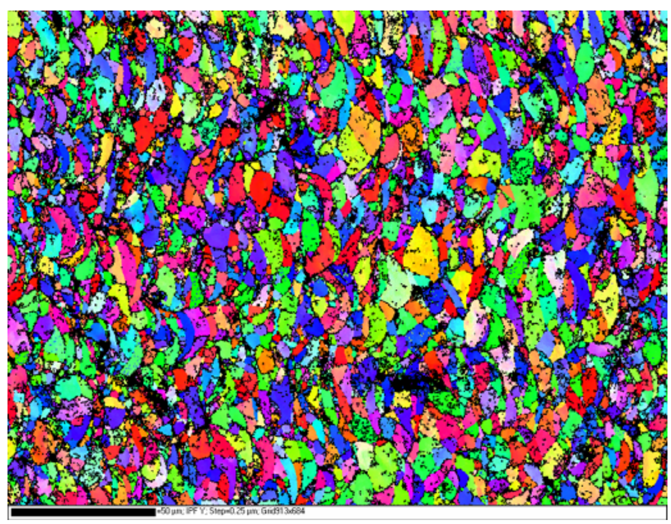

Figure 9. Kikuchi map of annealed H-10 cold sprayed material.

Figures 8 and 9 show grain orientation as color maps. Black regions indicate areas where the SEM system was not able to index the kikuchi pattern due to low contrast. Typically this results from the presence of voids (porosity) or very small grains. Figure 10 is an index to the color mapping shown in Figure 8 and 9. Grain orientation is referenced with respect to the beam direction.

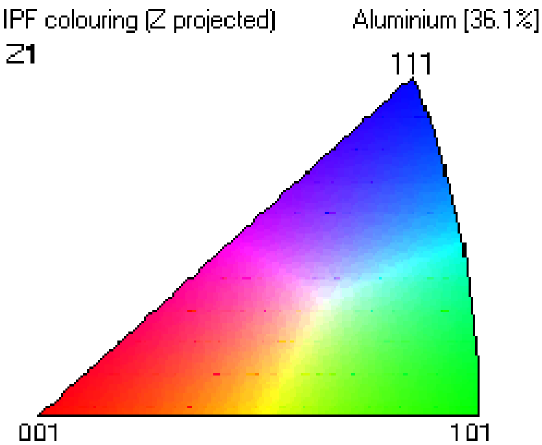

Figure 10: Index to Figures 8 and 9. Orientation is referenced with respect to the beam direction.

Analysis of the data presented in Figures 11 and 12 shows the location of high and low angle grain boundaries.

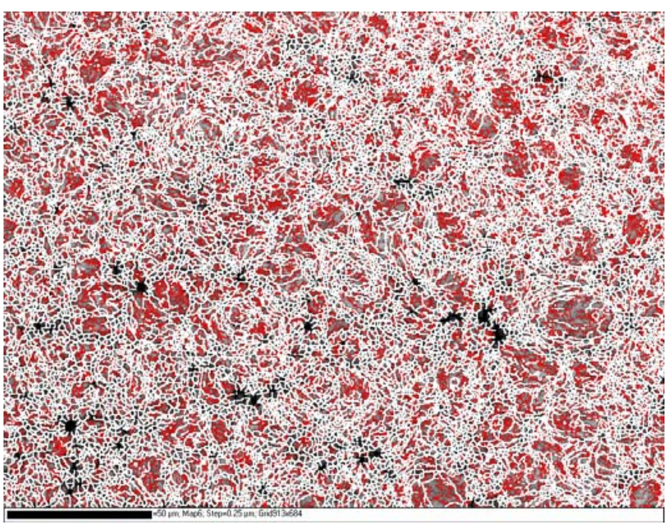

Figure 11: High angle grain boundaries (>10˚), shown in white, and low angel grain boundaries (<3˚), shown in red, for as-sprayed H-10 aluminum.

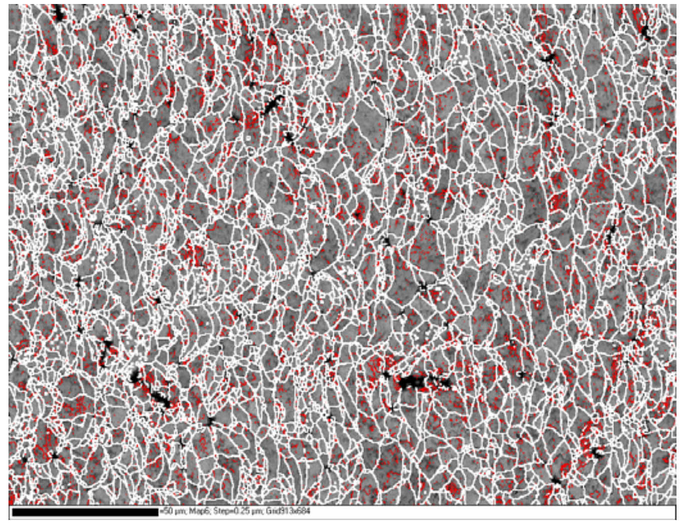

Figure 12: High angle grain boundaries (>10˚), shown in white, and low angle grain boundaries (<3˚), shown in red, for annealed H-10 aluminum.

These data clearly shows that the as-sprayed H-10 material contains significantly more high angle grain boundaries than the annealed material. The grains in the as-sprayed are 1 to 5 microns in size. This is significantly smaller than the mean incoming particle size of 11.8 microns. The small grain size in this material is due to the high deformation associated with the cold spray process. The size and shape of the grains in the annealed material suggest that their boundaries are associated with the splat boundaries. It is also clear that the as-sprayed material contains significantly more low angle grain boundaries than the annealed material. This suggests that the aluminum crystals in as-sprayed material have higher dislocation density than the crystals in the annealed material. Clearly, significant recrystallization and associated dislocation recovery has occurred in the annealed material. Interestingly, significant grain growth beyond the splat boundaries does not appear to have occurred in the annealed material. SEM images of the polished sample surface show this (Figures 13 and 14). Figure 13 shows the polished surface of the as-sprayed sample. Porosity at the splat boundaries can be clearly seen in both the as-sprayed sample and the annealed sample. This porosity is consistent with the idea that consolidation of cold sprayed material occurs through partial metallurgical bonding between the splats. Interestingly, the porosity along the splat boundaries does not appear to be significantly different when the as-sprayed and annealed samples are compared. This observation, combined with a lack of grain growth across the splat boundaries indicates that the splat boundaries have not changes significantly as a result of annealing.

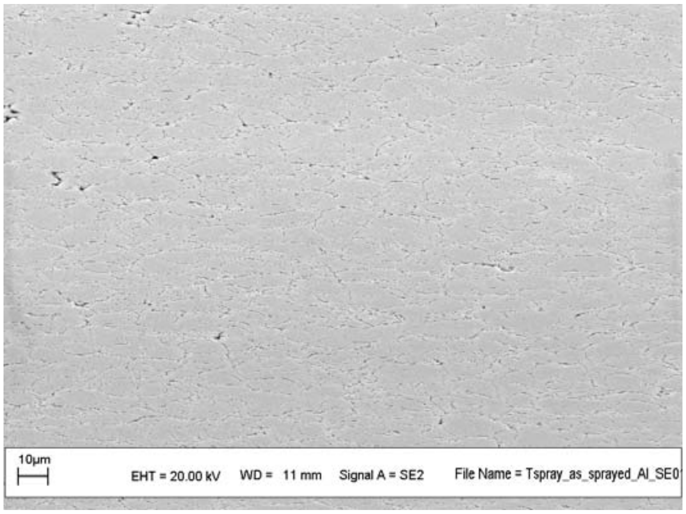

Figure 13: SEM micrograph showing the polished surface of the as-sprayed material. Porosity can be seen along the splat boundaries.

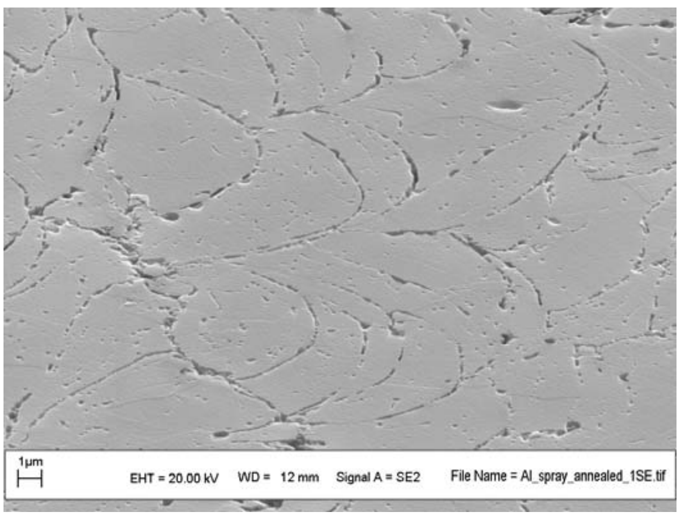

Figure 14: SEM micrograph showing the polished surface of the annealed material. Porosity can be seen along the splat boundaries. Note: this image is higher magnification than Figure 13.

The data shown above explain the change in fracture mechanism and increase in ductility observed upon annealing as recrystallization and reduction of dislocation density of the aluminum material within the splats. No macroscopic changes in splat boundaries were observed upon annealing. Clearly the orientation relationships along the splat boundaries changed as recrystallization occurred within the splats, but no coarsening of the material structure beyond the splat boundaries was observed. Thus, only recrystallization within the splats and reduction in dislocation density in the aluminum crystals can be responsible for the observed changes in mechanical properties and fracture mechanism.

Conclusions

1. Annealed cold spray deposits approximately 1 cm thick exhibit elastic moduli, ultimate tensile strength, and elongations similar to wrought aluminum. This indicates that cold spray technology may be used for spray forming aluminum components.

2. The primary mechanism for mechanical property changes in commercially pure cold spray deposits exposed to a moderate anneal is recrystallization and reduction of dislocation density within the aluminum splats.

Acknowledgments

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94-AL85000.

References

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