

## Introduction

In the cold spray (CS) process, gas-atomized powders are deposited using a super-sonic gas stream at relatively low temperatures. If the powder particles exceed a material-dependent critical velocity, then they bond to the substrate surface forming a dense, adherent coating. CS is an ideal candidate for repair and additive manufacturing because the thermal transients during deposition are low, avoiding the formation of a mechanically compromised heat-affected zone, and the material deposition rates are very high.

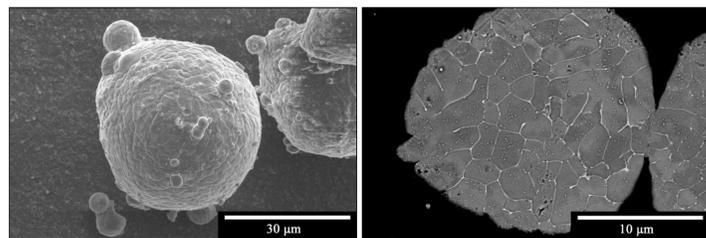
However, the nature of the CS process yields coatings which are in a highly cold-worked state, and have experienced thermal transients on the prior-particle boundaries. These factors result in significant microstructural and property differences between the interiors and boundaries of prior-particle regions.

Few systematic studies have been performed on the heat-treatment of CS coatings, and so the characterization team at UConn has used a combination of SEM, EBSD, and STEM techniques to investigate the microstructural evolution of CS Al 6061 coatings with *ex-situ* heat-treatment.

## Experimental

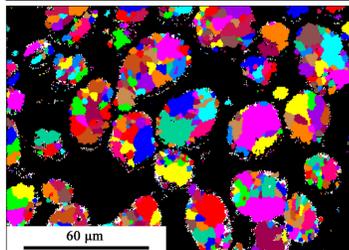
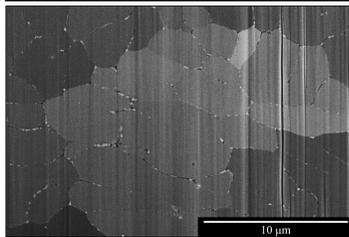
A thick coating of CS Al 6061 was milled into dog-bone-shaped tensile specimens for macro-mechanical testing. The grips of the broken tensile bars were sectioned and utilized for *ex-situ* heat-treatment experiments for 2h at 100, 200, 300, 400, and 500 °C. Heat-treated samples were electropolished into 3mm TEM foils to be used for SE SEM imaging to highlight secondary phases, BF STEM to determine dislocation density (DD), and EBSD to analyze grain size. OIM software was used to process EBSD unique-color grain maps, delineate high- and low-angle grain boundaries, and extract grain size data.

## Feedstock Powder Microstructure



Al 6061 feedstock powders were heat-treated to meet "mill spec" standard. These powders microstructures are characterized by:

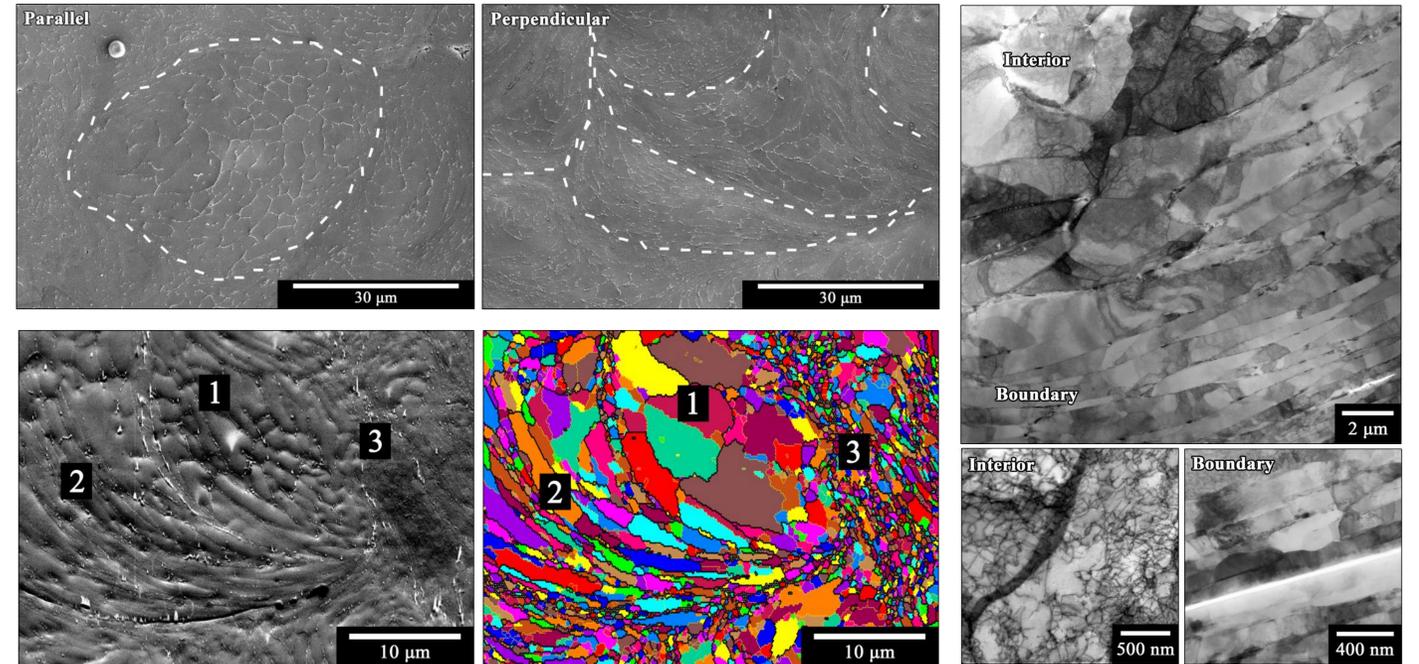
- Spherical morphology with ridged surfaces
- Internal network of thin-films of secondary phases delineating discrete "cells"
- Secondary phases are a combination of Mg<sub>2</sub>Si and an amorphous Fe-rich phase
- Clusters of connected cells constitute grains
- High degree of grain size variation between powders



## Initial Microstructure

CS deposition successfully formed a dense, adherent coating. Macro-tensile testing revealed mechanical properties similar to Al 6061 in the T6 condition. The main microstructural observations for this coating are:

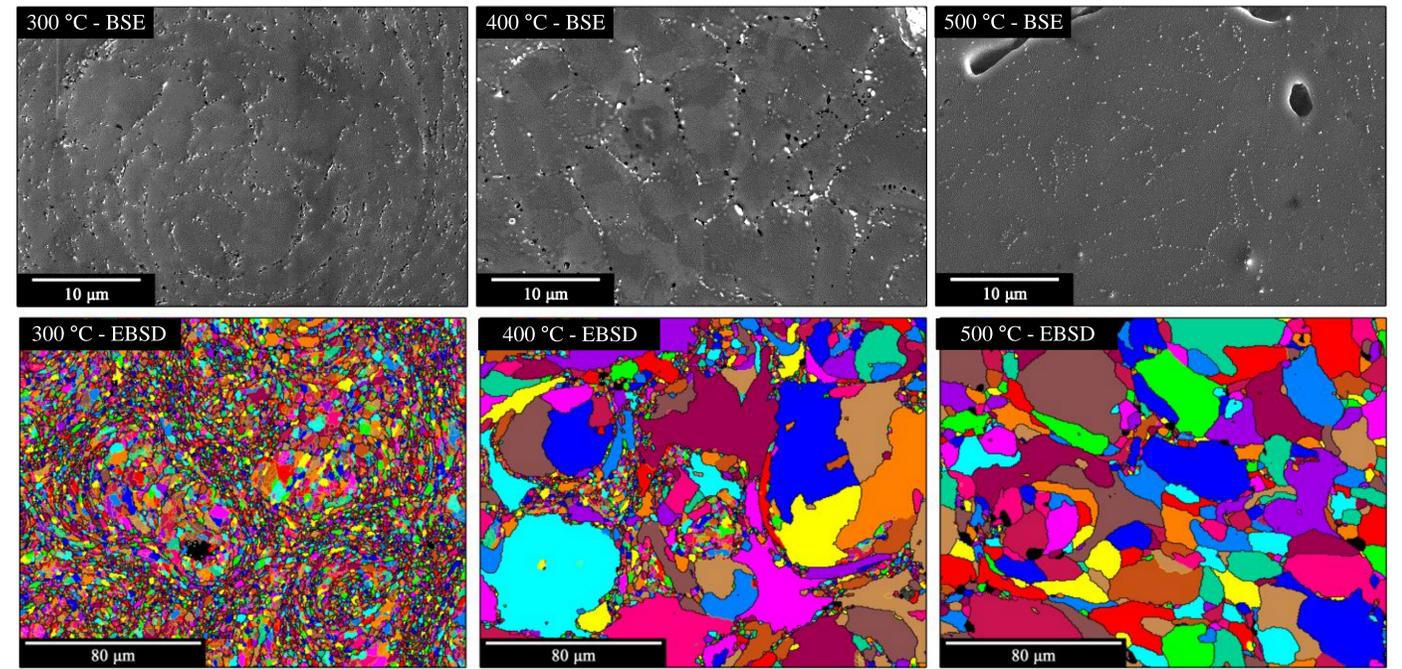
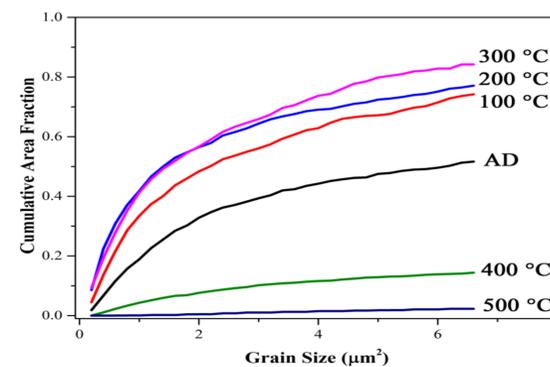
- Repeating "splat"-shaped microstructural motifs with anisotropy in sections cut parallel and perpendicular to the deposit surface
- Preservation of feedstock powder microstructure in the interior of splat regions (1)
- Severe disruption of feedstock powder microstructure at the prior-particle boundaries
- EBSD analysis reveals two types of boundary condition: high-aspect ratio grains in areas where cells appear squashed (2), and an equiaxed, fine-grained structure in boundary regions where the cell structure is not observed (3)
- BF STEM imaging reveals high DD within the coarse grained splat interior, intermediate DD within high-aspect ratio boundary grains, and a low DD in fine, equiaxed boundary grains



## Heat-Treated Microstructure

Heat-treatment and subsequent SEM and EBSD analysis reveals:

- Secondary phase coarsening and spherodization at 300 °C and above
- Recovered and recrystallized grains constitute ~45% of the as-deposited area fraction in EBSD maps
- Increased recovery and recrystallization with heat-treatment, peaking at ~85% area fraction at 300°C for 2h
- Dramatic grain coarsening appearing to initiate within splat interiors in 400 and 500°C heat-treatments.



## Conclusions

Advanced characterization and heat-treatment studies on cold sprayed Al 6061 coatings have revealed certain key microstructural features:

- Anisotropy with respect to CS spray direction
- Hierarchical grain shape and size differences between interior of splats and boundaries between splats
- Two types of grain structure at the boundary: high-aspect ratio grains in areas with squashed cell structure and fine-equiaxed grains in regions with severely disrupted cell structure.
- High DD in the interiors of splats, intermediate in high-aspect ratio grains, and very low in fine, equiaxed grains
- HT at up to 300°C causes coarsening of secondary phases, and the onset of recovery and recrystallization
- HT at 400 °C and 500 °C leads to dramatic grain growth and formation of pores at prior particle boundaries