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Microstructure and mechanical properties of cold sprayed 7075 deposition during non-isothermal annealing

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ABSTRACT

This study presents microstructure and mechanical property relationships of cold-sprayed 7075 aluminum during non-isothermal annealing. Microstructure evolution during non-isothermal annealing from room temperature to 450 °C was performed using in-situ heating via a hot-stage transmission electron microscope. Additional characterization was performed using differential scanning calorimetry and X-ray diffraction. Grain size, dislocation density, microstrain, lattice parameter, and precipitation phenomena were evaluated as a function of annealing temperature. The results showed that cold spray processing accelerated the precipitation kinetics of strengthening phases in the microstructure, compared to the as-received cold spray powder, but did not affect the overall precipitation sequence. Also, pancaked grain structures, found at particle–particle interfaces within the deposition, were converted, due to recrystallization, to ultrafine-grained structures during annealing. The ultrafine-grained structures experienced limited grain growth during the annealing process. This was attributed to the nucleation of grain boundary precipitates in the as-sprayed material, primarily originating from grain boundary solute segregation present in the cold spray powder. Mechanical properties were evaluated using microhardness testing, after annealing, and correlated with microstructural analysis. When subjected to low temperature annealing (below 370 °C), the cold spray processed material had a lower microhardness than that found in conventional 7075 aluminum subjected to the same thermal treatment, due to the presence of inter-particle porosity in the cold spray microstructure. Annealing at temperatures above 370 °C, however, resulted in an increase in hardness, likely due to a reduction in inter-particle porosity and grain boundary strengthening associated with the retention of an ultrafine grain structure at high temperatures.

1. Introduction

Cold spray processing is a material deposition process which achieves a dense coating or bulk deposition through extensive plastic deformation of fine powder particles upon impact with a target substrate [1–4]. Here small particles (~5 to 50 μm), in the solid state, are propelled towards a substrate at high velocity (typically 300 to 1200 m/s), subsequently developing a solid deposit as a consequence of the particles' kinetic energy on impact [3–6]. Although the deposition process relies on plastic deformation of the powder particles, the plastic strain experienced by the particles is non-uniform and it is well established that the microstructure of the deposited coating is also non-uniform [2,3,5–8]. For instance, several reports have shown the presence of both micron and sub-micron sized grains within the cold-spray deposition. The majority of these reports, however, have been focused on pure metals (e.g. Al [3,4], Ti [5], Cu [1,9], and Ni [10,11]), with few studies [6,7,12–14] examining the microstructure of CSP industrial alloys. With this in mind, among its many applications, cold spray processing has gained significant attention for its potential to perform dimensional restoration and repair of damaged metallic components (e.g. aircraft parts). Thus, there is a need for improved understanding regarding the use of cold spray processing for deposition of industrial alloys.

Recently, Rokni et al. [12] reported on the microstructure and local mechanical behavior of cold spray deposited 7075 aluminum (Al) alloys. Results showed the presence of both micro-scale grains in particle interiors and ultra-fine grained (UFG) structures at particle–particle boundaries. The presence of UFG structures have also been reported in CS processed (CSP) depositions in other materials (e.g. Al [3,15], Ti [5], Cu [11], and Ni [16]). Furthermore, it has been widely reported that the majority of grain boundaries (GBs) within UFG structures formed during severe plastic deformation (SPD) processes are in a non-equilibrium state due to the accumulation of large amounts of dislocations [7,17–21]. As a result, the GBs are thermally unstable, and grain growth may occur easily at moderate temperatures, leading to a variation in mechanical properties. Considering that CSP depositions may be used in applications where the parts experience moderate to high temperatures for extended periods of time, it is important to understand the thermal stability of the CSP microstructure. Furthermore, many of the conventional aluminum alloys, for which CSP deposition may be used, are precipitation hardenable alloys (e.g. 6061 Al and

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7075 Al). Thus, it is important to understand the influence of cold spray on the precipitation of strengthening phases within the microstructure during heating.

The microstructural evolution of Al–Mg–Zn (similar to 7075 Al) alloys subjected to severe plastic deformation (equal channel angular extrusion (ECAP) [22], high pressure torsion (HPT) [23], cryomilling and hot isostatic pressing/extrusion [24]) and subsequent heat treatment has been reported. Results show that SPD processes, resulting in UFG structures, alter the precipitation kinetics of strengthening phases ($MgZn_2$, η'/η) in the microstructure [22–24]. Furthermore, the studies highlight that these materials are strengthened by not only precipitate strengthening mechanism, but also, and more importantly, by grain boundary strengthening due to the presence of the UFG structure after thermal treatment [24]. A subtle difference between these studies and the CSP microstructure is the significant non-uniformity observed in as-deposited CSP microstructures, i.e. microscale grains in particle interiors and UFG structures located at particle–particle boundaries. Furthermore, several reports have demonstrated the presence of extensive grain boundary solute segregation in both gas atomized cold spray powder and CSP depositions [12,17,25–27]. The thermal stability of UFG structures can be maintained, to some extent, by the nucleation of fine, incoherent precipitates at the GBs so that GB migration causing grain growth is hindered by the pinning effect of these precipitates [28–30]. Thus, it is important to develop an understanding of the microstructural and mechanical property evolution of CSP 7075 Al during thermal treatment.

In this work, the microstructural and mechanical property evolution of CSP 7075 Al are reported during non-isothermal annealing from room temperature to 450 °C. Specific attention has been devoted to studying both the precipitation and restoration processes in the microstructure. In-situ hot-stage transmission electron microscopy (TEM), differential scanning calorimetry (DSC), and X-ray diffraction (XRD) were used to monitor the changes in the CSP microstructure (i.e. grain size, dislocation density, microstrain, lattice parameter, and precipitation sequence) during heating. Furthermore, Vickers microhardness was performed to correlate observed microstructure changes with resulting mechanical behavior.

2. Experimental procedure

2.1. Coating preparation

7075 aluminum (Al) (Al-6.09 Zn-2.68 Mg-1.28Cu-0.18Fe-0.13 wt.% Si) coatings were produced using commercially available gas-atomized 7075 Al powder (Valimet, Stockton, CA, USA), 18.6 ± 8.2 μm in size. Feedstock powder size was measured using a laser diffraction particle size analyzer (Microtrac s3000, Microtrac Software Co., USA). Cold spray deposition was performed using a VRC Gen III high-pressure cold spray system (VRC Metal Systems, Rapid City, SD, USA). Deposition was carried out onto wrought 7075 Al substrates using helium process gas while maintaining a gas pressure of 2.8 MPa and temperature of 400 °C at the heater exit. Coatings were deposited using a nozzle stand-off distance of 25 mm, deposition angle of 90°, powder feed rate of 12 g min^{-1}, and a nozzle traveling speed of 600 mm s^{-1}. Finally, a total deposition thickness of ~8.5 mm was achieved in this study.

2.2. Non-isothermal annealing

The annealing behavior of the cold spray deposited material was characterized by continuous in-situ heating of the sample to 450 °C in 45 min (10 °C/min) from ambient temperature using the TEM heating stage followed by microstructural characterization. A custom software code was written to capture a TEM image of the microstructure every 7 s. A total of 420 TEM images were combined to generate a video, which documented the microstructural evolution throughout the non-isothermal annealing process (RT to 450 °C). The video has been provided as a supplemental file (Video) with this publication. For this purpose, thin disks, 3 mm in diameter, were excised from perpendicular direction to the particle impact vector of the deposition, and then polished, dimpled, and ion milled for 4 h.

2.3. Thermal analysis

Thermal analysis was performed in a DSC machine (SDT Q600, TA Instruments, DE, USA) to investigate the precipitation sequence of the CSP 7075 deposition and the as-received powder during annealing process. The DSC was calibrated by using standard samples, giving an accuracy of ±0.3 °C for the temperature and ±0.02 mW for the heat flow measurements. Polished alloy disks with a diameter of 3 mm were sealed in aluminum pans and heated in a flowing Argon atmosphere at the same heating rate used in the TEM (10 °C/min). An empty cup was used as a reference to serve as a base line in this study.

2.4. Microstructure characterization

The microstructure of the as-received 7075 powder (ARP sample) and CSP 7075 Al deposition (CSP sample) annealed at various temperatures was evaluated by TEM, scanning electron microscopy (SEM), and XRD. TEM micrographs were obtained by utilizing a JEM-2100 LaB_6 operating at 200 kV. Thin disks of 3 mm diameter were excised from the deposition, and then polished, dimpled, and ion milled for 4 h. The grain size measurements were carried out particle–particle interfaces with pancaked structure using TEM analysis. 100 TEM grain size measurements were made at each temperature and the average and standard deviation are reported.

XRD experiments were performed using a D8 Bruker diffractometer (Bruker AXS, Karlsruhe, Germany) with negligible instrumental broadening using Cu $K\alpha_1$ radiation in the range $2\theta = 35–105°$ with a step size of 0.02° and counting time of 1 s per step. The XRD results were also used to calculate the mean grain diameter, d, and microstrain, ε, of the CSP samples at RT and following annealing at various critical temperatures. These values were calculated using Eq. (1)[24,31]:

$$B\cos\theta_B = \frac{K\lambda}{d} + \varepsilon \sin\theta_B, \tag{1}$$

where λ is the wavelength of Cu $K\alpha_1$ radiation, i.e. 1.54 ÅK is ~0.9, ε is the microstrain, θ_B is the Bragg angle, and B is the peak broadening term. The values of d and ε were obtained from the slope and intercept of the $B\cos\theta_B$ vs. $\sin\theta_B$ curve by performing a linear regression analysis. Additionally, following the Williamson–Hall method [32], the full width at half-maximum (FWHM) values were used to calculate peak broadening, B, which was derived from Eq. (2):

$$\sqrt{B_{obs}^2 - B_{inst}^2}, \tag{2}$$

where B_{obs} is the observed peak broadening and B_{inst} is the instrumental broadening.

2.5. Mechanical property evaluation

Microhardness measurements were performed using a Vickers microhardness tester (HMV-2, Shimadzu, Tokyo, Japan) with a load of 300 gf and a loading time of 10 s. The indenter was the Vickers diamond pyramid. For all the microhardness values reported in this paper 10 measurements have been carried out and the standard deviations have been calculated based on the obtained data.