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Microstructural stability of ultrafine grained cold sprayed 6061 aluminum alloy

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ABSTRACT

The microstructural stability of ultrafine grained (UFG) cold spray 6061 aluminum deposits produced by high pressure cold spray were investigated by in situ heating to a fully annealed state via a hot-stage transmission electron microscope (TEM). It was possible to observe the precise locations and temperatures of different microstructural changes, like dislocation movement and other restoration processes. Even after heating up to the annealing temperature for this alloy, the deposited layer in the perpendicular direction was found to preserve the UFG structures, which were the result of different recrystallization mechanisms caused by the high strains present during cold spraying. Extensive solute segregation at the grain boundaries acted as an obstruction for grain boundary migration in this direction, thereby preventing grain growth. However, in the direction parallel to the deposited surface, the UFGs were not resistant to grain coursing like the other direction, since the grain boundaries had much less solute segregation.

1. Introduction

Recently, considerable efforts have been devoted to fabricate bulk ultrafine grained (UFG) materials without porosity and a better combination of ductility and strength by imposing severe plastic strain [1–6]. A high degree of deformation coupled with the ability to tailor admixtures of powders during cold spraying (CS) can produce dense homogeneous or functionally graded microstructures unattainable by traditional P/M methods as well as UFG or even nano structure [5–7]. Often UFG and as-deformed structure, which is believed to be thermo-dynamically metastable due to excess amounts of various lattice defects and elastic distortions [8], can work together to provide excellent strength at room temperature, however, for many applications a more thermodynamically stable microstructure with better ductility would be preferred. Hence, thermally activated restoration processes, e.g. recovery, recrystallization and grain growth, that arise during annealing to high temperatures, are of interest here. In many cases, precisely controlled recovery and recrystallization of cold sprayed or severely deformed structures have been used to augment ultrafine grain formation [9–12].

There is also some indirect evidence of grain boundary (GB) segregation in UFG materials processed by cold work [13–17]. GB segregation is an extremely sensitive phenomenon because it may affect significantly material properties like corrosion resistance, mechanical behavior, or thermal stability [17–19]. It is thus of great importance to examine the mechanisms of the deformation-induced solute redistribution and concurrent grain boundary migration that was observed in the as-sprayed deposition of this investigation.

Non-isothermal heat treatments, especially the use of low heating rates, are frequently encountered during thermo-mechanical processing cycles, and hence are of importance for industrial purposes. During non-isothermal heat treatment, recovery and precipitation are likely to occur, generating complex interactions with recrystallization. Precipitation can also strongly influence the recrystallization behavior. The precipitation of dispersoids in the course of an annealing treatment can hinder or even suppress recrystallization [20–22]. Liu et al. [23] attributed the occurrence of large, inhomogeneous grain sizes in AA3105 to the occurrence of concurrent precipitation. Bampton et al. [20] also reported low heating rates should be employed in order to obtain maximum benefit from the fine grain processing technique. It has been demonstrated that the extent of recovery and precipitation also influence the recrystallization temperature [24].

The present study was undertaken in an effort to understand the recovery and recrystallization processes and associated changes in the microstructure during heating and annealing of a highly deformed 6061 alloy layer deposited by high pressure CS. Finding the exact temperatures for the processes involved during heating are important for microstructural control. In this case,

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an in situ hot-stage TEM investigation was found to be a suitable and powerful tool to study and probe the complex interactions and thermal stability of the various inhomogeneous as-deposited microstructures, during non-isothermal heating and annealing.

2. Experimental procedure

In the present study, a 6061 coating was produced with a commercial gas-atomized 6061 Al powder as the feed stock with a size range of 5–50 μm and an average size of 38.7 μm (measured with Microtrac S3000 instrument). Helium was used as the process gas to achieve high impact between incident particles. The deposits were made using a CGT 4000 cold spray system and the pressure and temperature of helium were maintained at 28 bar and 400 °C, respectively. The deposit was made up to reach a total thickness of 10 mm.

The powder morphology and the coating microstructure were examined using SEM samples, sectioned for both the *Z* direction (looking into the deposit parallel to the direction of impact) and the *Y* direction (perpendicular to the impact and in the plane of the deposit). The microstructure of the coatings was also analyzed by TEM using a JEM-2100 LaB_6 equipped with a hot-stage and energy dispersive X-ray spectroscopy (EDX) analysis, using discs of 3 mm diameter punched from different directions of the coating, and then polished, dimpled, and ion milled for 4 h.

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 in the Z and *Y* directions. A custom software code was written to take an image from the microstructure every 5 s in order to make a movie of the whole heat treatment. As a result, all of the notable microstructural changes, like the temperatures at which dislocations started to move, precipitation, recovery and recrystallization, were observed and recorded.

Thermal analysis was also performed in a SDT Q600 differential scanning calorimeter (DSC) in order to investigate the phase evolution of 6061 deposited sample during the continuous heating. Polished alloy disks with a diameter of 3 mm were sealed in Al pans and heated in a flowing Argon atmosphere at the same heating rate used in the TEM. Two DSC runs were successively performed on each sample with the curve from the second run used as the baseline.

3. Results and discussion

3.1. As-received 6061 Al powder

3.1.1. Morphology and microstructure

Fig. 1 shows the microstructure of the feedstock powder in which the spherical shape and size range of particles can be seen. Fig. 1(a), clearly depicts a typical particle size in the range of ~35 μm for the as-received 6061 Al powder particle. In Fig. 1(b) the grain structure of the as-received powder can be observed. According to this image scale, the powder structure consists of grains and subgrains in the range of 1–4 μm. TEM image of the powder in Fig. 1(c) demonstrates the presence of subgrain structures and dislocations in the powder particles. As seen in this figure, the subgrains are in the size range of ~200 nm, which some authors suggest can later transform into ones with well-defined low angle grain boundaries (LAGBs) after cold spray processing [6,25,26].

3.1.2. Phase identification and solute segregation

The back scattered SEM image in Fig. 2(a) confirms the same grain structure observed on the surface is also present inside of the powder particles. As can be seen, the GBs have a different chemical composition compared with matrix, which is evidence of solute segregation in the particles. The EDS analysis of the as-atomized 6061 powder (Fig. 2(b)) shows that the GBs in the powder particles are rich in Si, Mg, Cu and Fe elements. Different compositions of GB

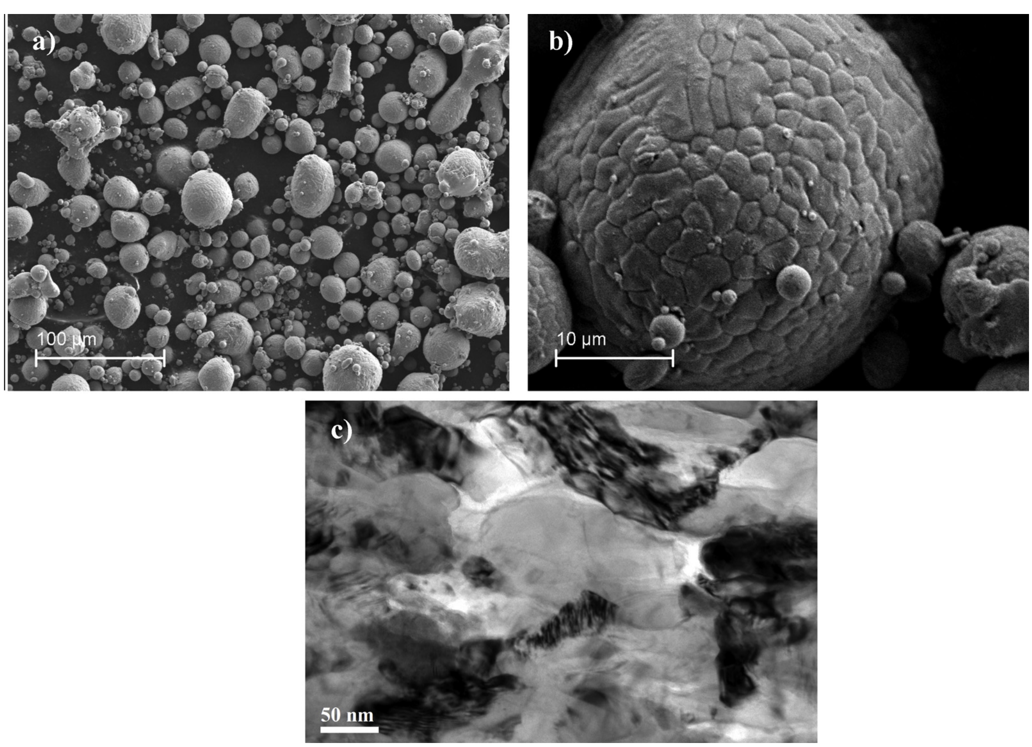

Fig. 1. Images from as-received 6061 Al powder. (a) SEM micrograph about powder morphology, (b) SEM micrograph showing the grain structure of the powder and (c) TEM image showing the presence of subgrain structures and dislocations in the powder particles.