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Structure and strength of aluminum with sub-micrometer/micrometer grain size prepared by spark plasma sintering

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ABSTRACT

A spark plasma sintering (SPS) technique has been applied to prepare fully dense Al samples from Al powder. By applying a sintering temperature of 600 °C and a loading pressure of 50 MPa, fully recrystallized samples of nearly 100% density with average grain sizes of 5.2 μm, 1.3 μm and 0.8 μm have been successfully prepared using a sintering time of less than 30 min and without the need for a nitrogen atmosphere. A similarity between the grain size and powder particle size is found, which suggests a potential application of the SPS technique to prepare samples with a variety of grain sizes by tailoring the initial powder particle size. The SPS samples show higher strength than Al samples with an identical grain size prepared using thermo-mechanical processing, and a better strength–ductility combination, with the 1.3 μm grain size sample showing a yield strength ($\sigma_{0.2\%}$) of 140 MPa and a uniform elongation of more than 10%. This higher strength is related to the presence of oxide particles in the grain boundaries of the samples. It is concluded that SPS is an excellent technique for the production of very fine grained Al materials with high strength, by combining both grain boundary and oxide dispersion strengthening.

1. Introduction

Aluminum alloys are widely used as engineering materials and are used, for example, extensively for aircraft components due to their high strength-to-weight ratio. Nevertheless, achieving an increased strength of Al alloys without the use of expensive alloying additions remains a desired goal. One way to realize such an increase in strength is through reduction of the grain size, and Al alloys with a grain size in the micrometer range are favoured by industry because of a good balance between strength and ductility. Recently, scientific interest has also been focused on metals with grain sizes below 5 μm, where metals may show unusual mechanical behavior, such as an unexpected yield drop phenomenon [1,2], and hardening by annealing and softening by deformation [3]. To better understand the underlying mechanisms for this transitional behavior, simple model samples with grain sizes covering the sub-micrometer/micrometer range are needed in order to establish relationships between material properties and grain size in a range not hitherto explored. For such experiments the microstructure of the model samples should be as simple as possible, i.e., with a random texture, a fully recrystallized microstructure and a low fraction of low angle boundaries (LABs).

Casting and deformation are the two traditional ways to produce Al alloys. In order to produce fine-grained Al, severe plastic deformation techniques (such as equal channel angular pressing or accumulated roll bonding) can be applied. The resulting material is, however, in a deformed state, with a high dislocation density and a high fraction of LABs. Annealing treatments can be given to reduce the dislocation density, however it is difficult to achieve near-micrometer grain sizes without retaining a significant LAB fraction in the material, and more generally it is difficult to control grain growth during such annealing treatments.

A third way to produce materials is by powder metallurgy (PM), which is a versatile technique that can be applied to many materials. However, for Al the initial powders are covered with a surface oxide, such that a major problem is to obtain good bonding between powder particles during processing. A standard process to prepare Al and Al alloys by PM is through cold/hot compaction of powders, followed by extrusion/forging at temperatures in the range of 500–550 °C. This processing sequence results, however, in a sub-grained microstructure containing a high fraction of LABs. Hot isostatic pressing (HIP) has also been applied to prepare Al samples [4,5], but this process requires a large pressure and a long processing time and is not well suited to the preparation of fine grain size material in a fully recrystallized condition.

Recently, the spark plasma sintering (SPS) technique has attracted interest for the preparation of powder samples [6,7]. This technique offers several advantages over conventional sintering

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treatments on account of the high heating rate and low loading pressures typically used, resulting in the use of low sintering temperatures, and short sintering times. Although developed for preparation of ceramic materials, the SPS technique has also been applied to prepare nano-grained metallic bulk samples, such as Al, Ni, Fe, and Cu [8–14]. Recently, the technique has also been applied to prepare coarse grained Al samples [15].

In this work, the SPS technique has been used for the preparation of polycrystalline Al samples with a range of sub-micrometer/micrometer grain sizes. These samples are prepared as starting materials for studies of deformation mechanisms in a grain size range that bridges the gap between the nanometer scale and the micrometer scale, i.e. a range of both scientific and industrial interest.

2. Experimental procedures

2.1. Materials

Atomized Al powders with a purity of 99.9% and of different sizes were purchased from Mengtai Technology Inc. (Beijing, China). Samples were prepared from three different powders, with average powder sizes of 0.9, 1.4, and 5.7 μm, as measured from investigations in scanning electron microscope (SEM) following the guidelines for powder size measurement in ASTM: E2651-10. In the following samples prepared from these powders are referred to as P0.9 μm, P1.4 μm and P5.7 μm. Example images of all three powders are shown in Fig. 1a–c. All the powders were of spherical shape with a unimodal distribution of powder sizes. Fig. 1d shows as an example of the powder size histogram for the P1.4 μm powder. The powders used in this study were stored in a desiccating unit prior to SPS processing. The powder weighing and die filling operations resulted in a handling time in air of approximately 30 min.

2.2. Sintering process

Sintering temperatures of 400 °C, 450 °C, 550 °C and 600 °C, were evaluated in this study. The initial sintering process was as follows. Firstly, 4 g of an Al powder were placed in a graphite die with a diameter of 20 mm. An initial pressure of 25 MPa was then applied after the die was placed into the chamber of a SPS-1050 (Sumitomo Heavy Industries Ltd., Japan) apparatus. This instrument is capable of supplying a maximum force of 100 kN and is equipped with a power supply producing a pulsed DC signal with a maximum value of 5 kA at 10 V. Joule heating was first applied only after a suitable vacuum (better than 5×10^{-2} torr) was reached in the chamber. Note that this vacuum was maintained through the full processing schedule, thereby contributing to the densification of the samples. A pulse pattern of 12:2 was used (a 12×3.2 ms long current pulse on, followed by a 2×3.2 ms long current pulse off).

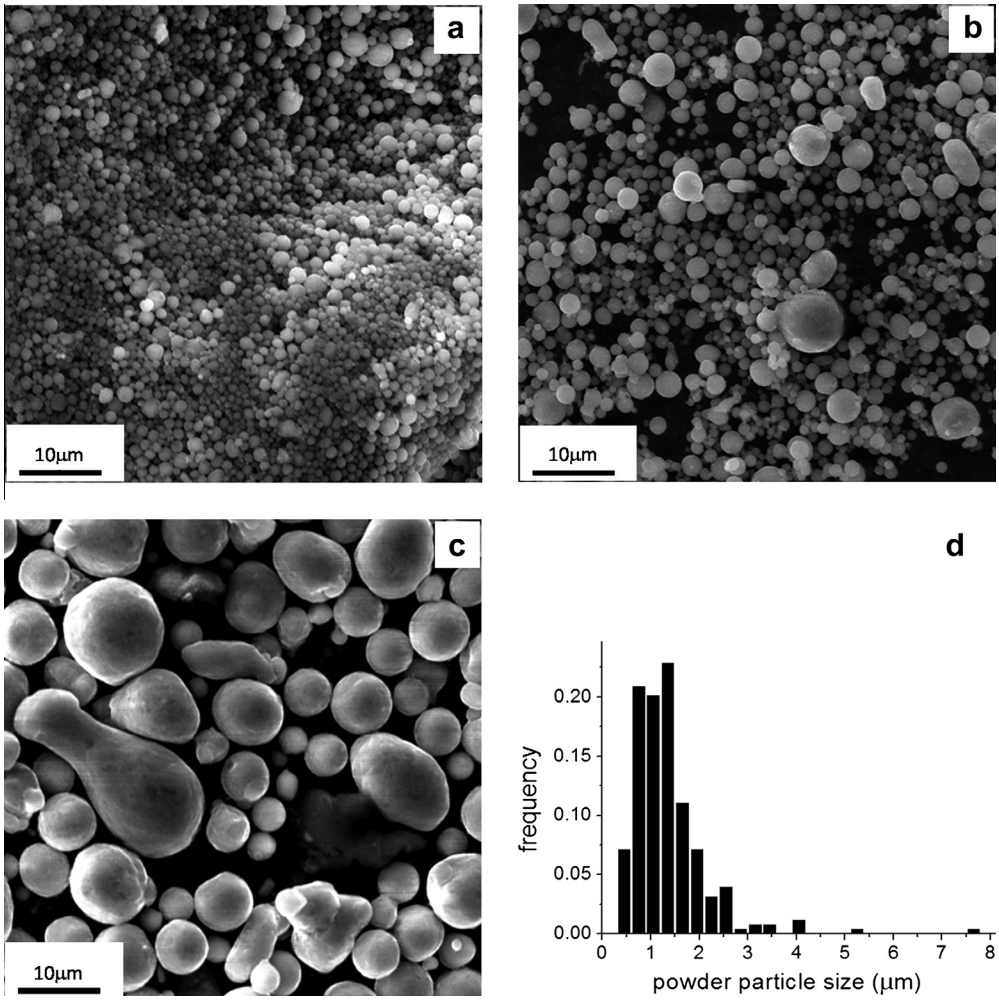

Fig. 1. SEM images of the three powders used in the study with average sizes of: (a) 0.9 μm, (b) 1.4 μm, and (c) 5.7 μm. The histogram (d) shows the powder size distribution for the powder with average size of 1.4 μm.