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Micrometric grained Al synthesized via quasi-hydrostatic ultra-high pressure consolidation of micrometric Al powders

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ABSTRACT

This paper reports a study on the synthesis of a fully dense micrometric grained (i.e., grain size around 1 μm) Al via consolidation of spherical micrometric-sized Al powders under a quasi-hydrostatic ultra-high pressure of 5 GPa. Our result shows that during consolidation oxide films on powder surfaces can be effectively disrupted into nano-scale oxide particles, which promoted the formation of metallurgical bonding between powders and the retention of micrometric-sized grains. Consequently, the as-consolidated Al is characterized by micrometric grains (average grain size of 1.2 μm) with a high density of nanoscale oxide particles (average size of ~25 nm) at grain boundaries. The as-consolidated Al exhibits both high strength (ultimate tensile strength of ~130 MPa) and ductility (uniform elongation of ~10%).

1. Introduction

Strength of materials increases with a decrease in grain size as described by the well-established Hall–Petch equation, which provides a theoretical basis for the design and development of a variety of nanostructured (NS, grain size < 100 nm) and ultrafine grained (UFG, grain size 100 to 1000 nm) materials. Despite ultra-high strength imparted by substantial grain refinement, NS and UFG materials generally suffer from poor ductility due to the ability of limited dislocation multiplication [grain boundary (GB) dislocation sources only] and the lack of work hardening [1,2], which confines their widespread applications. In an effort to optimize the combination of strength and ductility, fine-grained (FG, grain size 1 to 10 μm) materials have attracted considerable attention [3,4]. Although strength of FG materials is lower than that of their NS and UFG counterparts, FG materials still exhibit sufficiently high strength in accordance with that required by structural engineering applications, which can be attributed to their FG structure; moreover, FG materials present significantly higher ductility than that of their NS and UFG counterparts as a result of adequate GB and intragranular dislocation sources and high work hardening ability [5]. Among various FG materials, those having grain size around 1 μm (defined hereafter as micrometric grained materials) are of particular interest, given that grain refinement strengthening makes a much larger contribution to the overall strength in micrometric grained materials as compared to that in FG materials with grain size above 1 μm.

Although FG materials can be produced via grain coarsening of NS and UFG materials during annealing [6,7] and via well-controlled thermo-mechanical processing [8], it is difficult to manipulate the processing parameters to attain micrometric-sized grains. In contrast, consolidation of micrometric powders provides a relatively straightforward approach to synthesize micrometric grained materials. Oxide films on micrometric powder surfaces, however, present a challenge for the formation of metallurgical bonding between powders as well as for the mechanical properties of the consolidated materials. Inspection of published studies reveals that oxide films cannot be satisfactorily disrupted even though either conventional [9] or severe [10] plastic deformation operations were applied during consolidation. In view of the aforementioned discussion, it is the objective of the present study to explore the feasibility for the disruption of oxide films on micrometric Al powder surfaces and then for the consolidation of micrometric Al powders via appreciable three-dimensional elastic deformation induced by a quasi-hydrostatic ultrahigh pressure (5 GPa).

2. Experimental

The micrometric Al powders used in the present study were produced by gas atomization method. The specified composition of the as-received powders (except for O) was as follows: Al 99.81, Cu 0.0005, Fe 0.1071 and Si 0.0701, wt%. The Al powders were first

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compacted into a cylindrical billet of 10 mm in diameter and 8 mm in height under vacuum, and then the billet was transferred to the chamber of a six-face presser. The presser can generate a quasi-hydrostatic ultra-high pressure (UHP) on the order of a few GPa, which is imposed on the consolidated powders through the pressure-transferring medium (pyrophyllite). In the present study, the consolidation was performed under the quasi-hydrostatic UHP of 5 GPa at ~560 °C for 3 h. Following consolidation, cylindrical billets with ~9 mm in diameter and ~8 mm in height were produced.

The as-received Al powders and the as-consolidated bulk Al were characterized using field emission gun (FEG) scanning electron microscopy (SEM) Hitachi S-4800. The tensile tests were conducted at room temperature using an Inspekt Table tester (3 kN) (Hegewald & Peschke, Germany) with an initial strain rate of $5 \times 10^{-4}\ s^{-1}$. In order to acquire consistent tensile properties, at least three specimens were tested. The miniature tensile specimens with ~2 mm in gauge length and 1 mm × 1 mm in cross-section were sectioned from the consolidated cylindrical billets parallel to the axes via electrodischarge machining and then mechanically polished. Given the gauge length as small as 2 mm, it is difficult to accurately measure the displacement of the gauge section even using a video extensometer. In order to render this problem tractable, in the present study only maximum uniform elongation [corresponding to the ultimate tensile strength (UTS)] and elongation to failure are determined via postmortem measurements, which are implemented to represent the plasticity of materials. The suggested strategy is described as follows: first, tensile testing of a specimen is performed until fracture; based on the engineering stress–strain curve, the UTS (engineering) is determined (corresponding to the maximum tensile load). Second, a new tensile specimen is loaded to attain the UTS and then unloaded immediately; a digital picture of the specimen is taken, and the elongation of the specimen (i.e., the maximum uniform elongation) is assessed by measuring the change in length of the pre-marked gauge section (i.e., the distance between the inner sides of the marks) based on a properly enlarged digital picture (in order to minimize the measurement errors). Third, using the same method as that for the maximum uniform elongation, the elongation to failure is determined based on the aforementioned fractured specimens. The fractured surfaces were also studied using FEG SEM Hitachi S-4800.

3. Results

SEM image in Fig. 1a shows the morphology of as-received Al powders. The diameter distribution of spherical powders is reported in Fig. 1b (1338 powders), with average diameter of ~1.0 μm (fitting by lognormal probability function). Fig. 2a demonstrates the typical microstructure of the as-consolidated bulk Al. Our result shows that oxide films on Al powder surfaces were disintegrated into nanoscale oxide particles (~25 nm of average size) and these oxide particles are predominantly distributed at GBs, which are more clearly displayed in Fig. 2b. The rod-like particles that can be occasionally observed at both grain interiors and GBs may be Al–Fe intermetallics. Based on the statistical distribution of grain size as shown in Fig. 2c (500 grains), the average grain size is evaluated as 1.2 μm. Comparison between the average powder diameter and the average grain size of the as-consolidated Al reveals that grain growth was essentially inhibited during UHP consolidation.

A typical engineering stress–strain curve obtained without an extensometer used is shown in Fig. 3a. The average UTS of several tests is 130 ± 15 MPa, approximately twice that (69 MPa [11]) of a coarse-grained (CG) commercial pure Al 1060 (Al 99.6 wt%). The insets in Fig. 3a display the initial tensile specimens with the pre-marked gauge section, as well as the specimens strained to the UTS and to fracture. The maximum uniform elongation and elongation to failure were measured to be ~10 ± 1% and 40 ± 2%, respectively. Necking is evident in the fractured specimen (the right inset in Fig. 3a) and consistently, the fractured surface as shown in Fig. 3b features a morphology of dimples, suggesting a nature of ductile fracture.

4. Discussion

During quasi-hydrostatic UHP consolidation, the percentage of volume change ($\Delta V/V$) of the consolidated materials can be evaluated by: $\Delta V/V = P/B$, where P is the quasi-hydrostatic pressure applied, and B is the bulk modulus that is both P and temperature (T) dependent. Taking into account the effect of P and T, B can be taken as 73.9 and 251.5 GPa for Al [12,13] and Al_2O_3 [14,15] in the present study (P=5 GPa and T=560 °C), respectively, and then the difference in $\Delta V/V$ between Al and Al_2O_3, which represents the extent of inconsistence in elastic deformation between Al powders and Al_2O_3 films on their surfaces, is calculated to be 4.78%. Under regular hot isostatic pressing (HIP) conditions, the hydrostatic pressure ranges from 100 to 200 MPa for Al alloys [9,16,17], the difference in $\Delta V/V$ between Al powders and Al_2O_3 films on powder surfaces is estimated to be 0.22% (B=65.9 and 237.5 for Al [13] and Al_2O_3 [14] at 560 °C under 200 MPa, respectively). Inspection of the published literature reveals that during consolidation of Al and Al alloys via HIP Al_2O_3 films on powder surfaces almost always cannot be broken [9,16,17]. It then follows that the disruption of oxide films during UHP consolidation in the present study can be presumably attributed to the relatively larger difference in $\Delta V/V$ between Al and Al_2O_3.

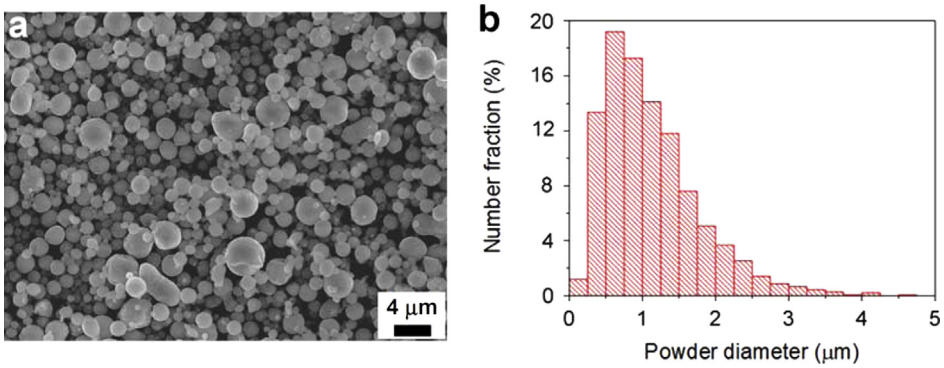

Fig. 1. The microstructure of as-received Al powders: a) SEM image showing typical morphology of Al powders, and b) the statistical distribution of powder diameters.