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On the thermal stability of ultrafine-grained Al stabilized by *in-situ* amorphous Al_2O_3 network

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ABSTRACT

Bulk Al materials with average grain sizes of 0.47 and 2.4 μm, were fabricated by quasi-isostatic forging consolidation of two types of Al powders with average particle sizes of 1.3 and 8.9 μm, respectively. By utilizing the native amorphous Al_2O_3 (am-Al_2O_3) film on the Al powders surfaces, a continuous, ~7 nm thick, am-Al_2O_3 network was formed *in situ* in the Al specimens. Systematic investigation of the changes to the am-Al_2O_3 network embedded in the Al matrix upon heating and annealing up to 600 °C was performed by transmission electron microscopy (TEM). At the same time, the stability of the Al grain structure was studied by transmission Kikuchi diffraction (TKD), electron back-scatter diffraction (EBSD), and TEM. The am-Al_2O_3 network remained stable after annealing at 400 °C for 24 h. *In-situ* TEM studies revealed that at temperatures $\geq$ 450 °C, phase transformation of the am-Al_2O_3 network to crystalline γ-Al_2O_3 particles occurred. After annealing at 600 °C for 24 h the transformation was completed, whereby only nanometric γ-Al_2O_3 particles with an average size of 28 nm resided on the high angle grain boundaries of Al. Due to the pinning effect of γ-Al_2O_3, the Al grain and subgrain structures remained unchanged during annealing up to 600 °C for 24 h. The effect of the am-Al_2O_3→γ-Al_2O_3 transformation on the mechanical properties of ultrafine- and fine-grained Al is discussed from the standpoint of the underlying mechanisms.

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1. Introduction

Traditionally, Al-based components are fabricated either by casting or deformation and/or machining of wrought feedstock. Compared to traditional techniques, powder metallurgy (PM) offers various advantages, including: (i) microstructural refinement, increased solubility of alloying elements, and a homogenous microstructure, resulting in enhanced mechanical properties; (ii) the flexibility to produce metal matrix composites (MMCs) by incorporating strengthening phases with various matrix chemistries; and (iii) cost effective production of (near) net-shape complex parts. Typically, Al PM parts are fabricated from gas atomized feedstock powder material.

The exceptionally high thermodynamic stability of the Al_2O_3 phase leads to the formation of a continuous protective Al_2O_3 film covering all of the gas atomized Al powder surfaces, even under high vacuum conditions [1]. Moreover, because of the high cooling rates that are typical of atomization, and the resulting fine powder sizes and structure, the native Al_2O_3 films on the surface of as-atomized powders (especially those with finer size) are amorphous (am-Al_2O_3) [2–4], with a thickness of ~2–3 nm [2,4–7]. The amorphous state is preferred below the critical film thickness, because the higher bulk Gibbs free energy of am-Al_2O_3 film, compared to crystalline γ-Al_2O_3 film, is overcompensated for by the low value of the combined am-Al_2O_3 surface and am-Al_2O_3/Al interfacial energies [8]. The am-Al_2O_3 remains thermodynamically stable up to the critical thickness of ~5 nm [4,8] depending on the crystallographic orientation of the Al substrate and temperature [4,9]. Once this critical thickness is exceeded, am-Al_2O_3 will transform to crystalline γ-Al_2O_3 (or transitional γ`-Al_2O_3) in the range 450–550 °C [4,10]. The γ-Al_2O_3 crystals grow epitaxially in respect to an Al substrate on am-Al_2O_3/Al interface [11] and later they develop preferentially along the grain boundaries [12]. The growth of γ-Al_2O_3 films is limited by the rate of inward oxygen diffusion through the films. The γ-Al_2O_3 on the surface of loose powder remains thermodynamically stable during thermal exposures to temperatures normally associated with Al PM

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processing, i.e., up to the melting point of Al (T_m=660 °C) [4]. Consequently, any am- or γ-Al_2O_3 phases present on Al particle surfaces behave as a dispersed second phase during powder processing and consolidation. This finding is universal and applies for a wide range of PM processing/consolidation techniques including: ball milling [13,14], hot working (e.g., forging, extrusion) [15,16], sintering (e.g., conventional, spark plasma sintering (SPS)) [17,18], hot isostatic pressing (HIP) [15,19] and severe plastic deformation [20]. Typically, the volume fraction of *in-situ* Al_2O_3 phase in PM parts fabricated from gas atomized powders varies between ~0.1 and ~3 vol% [7]. Depending on the processing and parameters used during consolidation, the dispersed Al_2O_3 phase either persists in the form of an amorphous, nanometric, continuous network or discrete platelets [3]. Otherwise, it crystallizes into nanometric γ-Al_2O_3 particles [15]. Similar to the crystallization of the native am-Al_2O_3 films on as-atomized powders under an oxidizing environment, the nanometric am-Al_2O_3 dispersed phase (network or platelets) in Al matrix remains thermodynamically stable upon annealing to ~450 °C, where it transforms into nanometric γ-Al_2O_3 particles [3,19]. However, the transformation mechanism, the nature of Al_2O_3 phases (specifically in terms of size, crystallinity and distribution) and their thermal stability in an absence of direct diffusion of gaseous oxygen, all remain open questions.

The presence of the *in-situ* Al_2O_3 dispersed phase, especially for those PM parts fabricated from coarse powders, is often neglected despite the findings that the *in-situ* Al_2O_3 dispersoids can restrict grain growth up to high homologous temperatures [7,13]. Moreover, the Al_2O_3 phase may contribute to strengthening, stiffening and creep resistance of the Al matrix in a broad temperature range [3,7,21]. Initial mechanical behavior data indicate that am-Al_2O_3 is more efficient in strengthening and stiffening of the Al matrix in comparison to γ-Al_2O_3 [3]. In addition, the materials with am-Al_2O_3 phase, either in the form of network or platelets, show superior creep performance [15,21,22]. However, fundamental information related to the strengthening and grain pinning effects of am-Al_2O_3 and γ-Al_2O_3 phases in Al PM structures has yet to be established. Motivated by these factors, the objective of the present work is threefold and it aims to investigate:

i. the changes of am-Al_2O_3 phase dispersed in PM Al upon heating;
ii. the effect of the am-Al_2O_3→γ-Al_2O_3 transformation on the mechanical properties of PM Al; and
iii. the stabilizing effect of am- and γ-Al_2O_3 phases on Al grain structure.

For this purpose, bulk Al MMCs reinforced and stabilized with a nanometric am-Al_2O_3 network with the commercial designation HITEMAL® (high temperature aluminum), were fabricated *in situ* by quasi-isostatic forging (QIF) compaction of fine gas-atomized commercial purity (CP) Al powders with particle sizes of 1–10 μm [3]. In these composites, a nearly continuous am-Al_2O_3 network, with a relatively large volume fraction (up to 2.7 vol%), forms during QIF as result of subtly sheared Al powders with only limited fracturing of native am-Al_2O_3 films. The grain structure of QIF HITEMAL is characterized by a mixture of high and low angle grain boundaries (HAGBs, LAGBs), where am-Al_2O_3 resides at HAGBs, and LAGBs are free of Al_2O_3 [3]. Ultrafine-grained (UFG) HITEMAL virtually free of LAGBs and fine-grained (FG) HITEMAL with developed subgrain structures were fabricated from two types of fine CP Al powders of different particle sizes in order to elucidate the stabilizing effect of Al_2O_3 on LAGBs. At the same time, the effect of different grain size on susceptibility to grain coarsening was studied. Moreover, the effect of the am-Al_2O_3→γ-Al_2O_3 transformation on UFG and FG materials with distinctively different deformation behavior was studied.

2. Experimental

Two different types of commercially available nitrogen atomized Al powders (Al ≥ 99.8 wt%) with median particle sizes (d_{50}) of 1.4 and 8.9 μm were used for this study. The specific powder surface area of the powders, determined by physical adsorption with multipoint analysis using nitrogen gas according to the Brunauer-Emmett-Teller (BET) principle, was 3.1 and 0.9 $m^2 g^{-1}$, respectively. The loose powders were pressed by cold isostatic pressing (CIP) at 20 MPa. Blanks 35 mm in diameter and 50 mm in length were machined from CIPed green compacts. The blanks were subjected to 5 Pa vacuum degassing (VD) performed at 425 °C for 12 h. VDed blanks were evacuated for 12 h in a vacuum chamber prior to vacuum hot pressing (VHP) performed at 425 °C and 370 MPa. VHPed blanks were consolidated by QIF with a limited amount of shear deformation. QIF was carried out at 420 °C and impact energy of 14 kJ using a screw press FICEP PVX160, and a semi closed forging die. The materials forged from Al powders of d_{50}=1.3 and 8.9 μm were labeled as A and B, respectively. To eliminate the work hardening effects induced during processing, as-forged materials were annealed at 400 °C for 24 h (hereafter referred to as the "as-processed" material). In order to execute the am-Al_2O_3→γ-Al_2O_3 transformation, as-forged materials were annealed in an Ar atmosphere at 500 and 600 °C for 24 h prior to characterization and testing. The designation for all samples and annealing conditions is summarized in Table 1.

The materials' densities were measured by using the Archimedes' principle. Samples were characterized by using a JEOL 2500SE TEM equipped with an *in-situ* hot stage holder and a Phillips XL-30 scanning electron microscope (SEM) equipped with an Oxford NordlysNano electron back-scatter diffraction (EBSD) detector. The heating rate during *in-situ* TEM was set to be 5 °C min^{-1} with 30 min dwells at 400, 450, 500, 550 and 600 °C, respectively. In order to maintain uniform thermal exposure conditions, the same heating sequence was applied for all *in-situ* TEM. Energy dispersive X-ray spectrometry (EDS), EBSD and transmission Kikuchi diffraction (TKD) [23,24] were used to assist in the analysis. For FG B materials the average grain size and the misorientation of neighboring grains were determined by SEM equipped with EBSD using $2 \times 2 \times 2$ mm^3 samples. In order to improve the index rate, the average grain size and the misorientation of neighboring grains of UFG A materials were determined by SEM equipped with EBSD using thin foils (i.e., by TKD). Thin foils for TEM and TKD were prepared by electric discharge machining (EDM), mechanical grinding and dimpling followed by the final thinning carried out by Ar ion milling at 4 kV and cooled by liquid nitrogen. The specimens for EBSD analyses

Table 1
The designation of as-forged and annealed materials.

Material	d_{50} (μm)	BET ($m^2 g^{-1}$)	As-forged	Annealed at 400 °C for 24 h (as-processed)	Annealed at 500 °C for 24 h	Annealed at 600 °C for 24 h
A	1.4	3.1	A0	A400	A500	A600
B	8.9	0.9	B0	B400	B500	B600

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