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Mechanical behavior and strengthening mechanisms in ultrafine grain precipitation-strengthened aluminum alloy

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Abstract

To provide insight into the relationships between precipitation phenomena, grain size and mechanical behavior in a complex precipitation-strengthened alloy system, Al 7075 alloy, a commonly used aluminum alloy, was selected as a model system in the present study. Ultrafine-grained (UFG) bulk materials were fabricated through cryomilling, degassing, hot isostatic pressing and extrusion, followed by a subsequent heat treatment. The mechanical behavior and microstructure of the materials were analyzed and compared directly to the coarse-grained (CG) counterpart. Three-dimensional atom-probe tomography was utilized to investigate the intermetallic precipitates and oxide dispersoids formed in the as-extruded UFG material. UFG 7075 exhibits higher strength than the CG 7075 alloy for each equivalent condition. After a T6 temper, the yield strength (YS) and ultimate tensile strength (UTS) of UFG 7075 achieved 734 and 774 MPa, respectively, which are ~120 MPa higher than those of the CG equivalent. The strength of as-extruded UFG 7075 (YS: 583 MPa, UTS: 631 MPa) is even higher than that of commercial 7075-T6. More importantly, the strengthening mechanisms in each material were established quantitatively for the first time for this complex precipitation-strengthened system, accounting for grainboundary, dislocation, solid-solution, precipitation and oxide dispersoid strengthening contributions. Grain-boundary strengthening was the predominant mechanism in as-extruded UFG 7075, contributing a strength increment estimated to be 242 MPa, whereas Orowan precipitation strengthening was predominant in the as-extruded CG 7075 (~102 MPa) and in the T6-tempered materials, and was estimated to contribute 472 and 414 MPa for CG-T6 and UFG-T6, respectively. © 2013 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Al alloys; Precipitation; Strengthening mechanism; Ultrafine-grained materials; Atom-probe tomography

1. Introduction

Conventional coarse-grained (CG) precipitationstrengthened 7000 series aluminum (Al) alloys have been widely used for aerospace and transportation applications because of their high strength and heat treatability [1,2]. In this class of precipitation-strengthened alloys, extremely small and uniformly dispersed precipitates, which act as obstacles to dislocation movement, form within the Al matrix upon heat treatment and thus strengthen the materials [3]. This phenomenon is generally referred to as precipitation strengthening. Natural aging, or precipitate formation at room temperature, occurs in most 7000 series alloys [4,5]. It has been generally accepted that the precipitation starts with the formation of Guinier–Preston (GP) zones, which may be regarded as coherent metastable precipitates [3,6-8]. The typical diameter of GP zones is of the order of a few nanometers [8]. Subsequent evolution of the precipitates involves the replacement of the metastable GP zones with a metastable semicoherent phase, η' -MgZn₂ [9]. This occurs primarily because GP zones are isostructural

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with the matrix and, therefore, have a lower interfacial energy than intermediate or equilibrium precipitate phases that possess a different crystal structure. As a result, the nucleation barrier for GP zones is significantly smaller [1]. The incoherent equilibrium hexagonal phase, η -MgZn₂, forms from η' -phase precipitates at higher aging temperatures and longer aging times. These n-phase precipitates are generally larger in size (diameter > 50 nm) and are preferentially located at grain boundaries. The precipitation sequence can be summarized as follows: supersaturated solid solution \rightarrow GP zones $\rightarrow \eta'$ (MgZn₂) $\rightarrow \eta$ $(MgZn_2)$ [3,8]. Basically, the presence of a high number density of GP zones and fine n'-phase precipitates is responsible for the strengthening of the material [6,10]. In an effort to accelerate aging kinetics, artificial aging is performed at higher temperatures, during which the strength achieves a maximum value. After long aging times or higher aging temperatures, the strength begins to decrease and the alloy becomes over-aged [3,11]. The commonly used artificial aging treatment, called a T6 temper, that results in peak microhardness for these alloys with conventional coarse grains, is performed at 120 °C for 24 h [5].

Interest in nanocrystalline or nanostructured (NS; grain diameter < 100 nm) and ultrafine-grained (UFG; grain diameter > 100 nm, but less than 1000 nm) materials, originally motivated by reports of novel deformation mechanisms as well as by the potential to attain notable enhancements in mechanical properties [12–16], has gradually moved from pure metals and simple alloys to more complex precipitation-strengthened alloys with many alloying components. Prior studies of Al 7075, a representative precipitation-hardenable aluminum alloy, have revealed that these alloys can be further strengthened by incorporating grain refinement. Zhao et al. [4,9] fabricated UFG 7075 with a grain diameter of ~ 400 nm utilizing commercial 7075 rod (grain diameter $\sim 40 \,\mu\text{m}$) through equal channel angular pressing (ECAP). They reported that the yield strength and the tensile strength of the UFG 7075 were 650 and 720 MPa, respectively, with natural aging for a month after ECAP, which represent strength increases of 103% and 35%, respectively, over its commercial Al 7075 counterpart. The improvement in the strength was ascribed to grain refinement and higher number densities of both GP zones and dislocations in the UFG material. In a related study, Zhao et al. also documented a simultaneous increase in both ductility and strength for NS 7075 (average grain diameter ~ 100 nm, processed by cryorolling) with subsequent artificial aging compared with the unaged condition [17]. In addition to the fine GP zones, η' - and η phase precipitates were introduced in the nanograins through aging, increasing the dislocation density. The increased dislocation density led to an improvement in the work-hardening rate and consequently contributed to the enhanced uniform elongation. It was concluded that the high dislocation density and fine grain size of the NS sample were primarily responsible for its improved strength over the CG sample, while the high density of second-phase

precipitates was responsible for its improved ductility over as-processed NS 7075 without aging [17]. In a related study. Panigrahi and Javaganthan [18] applied cryorolling to produce an UFG 7075 material with high-angle grain boundaries that exhibited improved strength due to the Hall-Petch effect and a higher dislocation density. It was documented that the microhardness and tensile strength of the cryorolled UFG 7075 was reduced after annealing at temperatures of 150-250 °C and subsequently remained constant when the annealing temperature was increased [19]. More recently, a NS 7075 material exhibiting an extremely high vield strength of ~ 1 GPa, combined with a uniform elongation of \sim 5%, was successfully produced by high-pressure torsion (HPT) [20]. It was suggested that the formation of a nanostructured architecture, which comprised a solid solution including a high number density of dislocations, sub-nanometer intragranular solute clusters, nanometer-scale intergranular solute clusters and grains of tens of nanometers in diameter, contributed to the dramatic increase in strength.

Despite ample evidence that grain refinement further improves the strength of precipitation-strengthened Al alloys, precise determination of the underlying strengthening mechanisms has been hindered by the complexity of the possible mechanisms, including: grain-boundary strengthening (Hall-Petch effect), solid-solution strengthening, dislocation strengthening and precipitation strengthening. Accordingly, the goal of the present study is to formulate a quantitative insight into strengthening mechanisms by providing a direct comparison of an UFG precipitationstrengthened material with an otherwise equivalent powder metallurgy (PM)-derived CG material. It is noted that unlike CG materials made by casting, the PM-derived CG material is expected to exhibit a relatively fine grain diameter, in the range of $1-5 \,\mu m$ [21]. To the best of our knowledge, this is the first time a direct comparison between UFG and CG materials, consolidated and heattreated using identical processing steps, has been documented for such a complex precipitation-strengthened Al alloy. More importantly, fundamental insights into the interrelationships between grain refinement, precipitation characteristics and mechanical behavior are provided.

2. Experimental procedure

Al 7075 was chosen as a representative alloy for this investigation, partly because the precipitation sequence and kinetics in this system have been extensively studied. Cryomilling, a mechanical attrition technique in a cryogenic environment [14,15,22–24], was utilized in our study to obtain NS 7075 powder. This technique takes advantage of the low boiling temperature, 77.2 K, of liquid nitrogen, which suppresses recovery and recrystallization in the powder, and leads to nanocrystalline grain structures and rapid grain refinement. Cryomilling also results in a high number density of dislocations in the material through severe plastic deformation (SPD) [14,15]. Additionally, fine oxide/

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