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A multi-scale Al–Mg alloy containing ultra-fine lamellar structure

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ABSTRACT

This paper reports a study on multi-scale metallic materials containing ultra-fine lamellar structure, which have received very limited attention in the published literature. In the present study, a multi-scale Al–Mg alloy, which comprises ultra-fine lamellar structure with a high density of dislocations, as well as nearly dislocation-free equiaxed ultra-fine, fine and coarse grains, was produced via partial recrystallization of the heavily cold-rolled coarse-grained (CG) counterpart. The multi-scale Al–Mg alloy achieves both high strength (~320 MPa in 0.2% offset yield strength in comparison with 145 MPa in 0.2% offset yield strength for the annealed CG counterpart) and high ductility (~11% in uniform elongation). Our results show that the ultra-fine lamellar structure takes a volume fraction as high as 42.5% and presents a high strength (~450 MPa in 0.2% offset yield strength), enabling it to make a substantial contribution to the high overall strength of the multi-scale alloy. Interestingly, the ultra-fine lamellar structure concurrently exhibits a reasonably high ductility (~6% in uniform elongation), which benefits for the multi-scale alloy to attain the high ductility.

1. Introduction

As two of the most important mechanical properties for structural materials, strength and ductility usually exclude each other, i.e., strength increases with a decrease in ductility and vice versa. This has been extensively reported in the published scientific literature [1–8] albeit the fact that some engineering structural materials exhibit both high strength and ductility [9–11]. More recently, in an effort to achieve a desired combination of strength and ductility, the concept of multi-scale microstructure has attracted considerable interest [12–25]. In a multi-scale microstructure [26], nano-sized (< 100 nm) and/or ultra-fine (100 nm to 1 μm) grains on the small side of the size distribution exhibits higher strength but lower ductility; in contrast, fine (1–10 μm) and coarse (> 10 μm) grains on the large side of the size distribution present lower strength but higher ductility. Hence, it can be anticipated that a material comprising multi-scale microstructure would possess a good combination of high strength and ductility as compared to singular unimodal nanostructured (NS, grain size < 100 nm), ultra-fine grained (UFG) or coarse grained (CG) materials. Inspection of the published studies indicates that the multi-scale microstructures consisting of equiaxed nano-sized/ultra-fine and coarse grains in Al [12–14] and Al alloys [15–18], Cu [19–21], Fe [22] and steels [23], Ni [24], and Ti [25] have been extensively investigated.

The multi-scale microstructures containing nano-sized/ultra-fine lamellar structures (i.e., nano-sized/ultra-fine dimensions in lamellar width), however, have been reported only in few studies (e.g., a multi-scale Cu containing nanoscale twin bundles in Ref. [27]). Nano-sized/ultra-fine lamellar structures are usually generated via severe plastic deformation (SPD) [28,29], yielding a high density of dislocations; hence, this type of structures can impart significantly high strength as a result of both Hall–Patch effect and dislocation strengthening. Indeed, Lu and colleagues [30] most recently reported that a nano-lamellar structure (~20 nm in average lamellar width) with an extremely high density of dislocations (on the order of 10^{16} m^{-2}) in pure Ni exhibits a hardness value (6.4 GPa) much higher than that in pure Ni processed by various SPD approaches. The ultra-high hardness is primarily attributable to the high density of dislocations in the nano-lamellar structure [30]. Given the lack of the studies on the multi-scale materials containing nano-sized/ultra-fine lamellar structures, it is the objective of the present study to provide insight into the microstructure and mechanical properties of this class of materials. In the present study, a multi-scale Al–Mg alloy with the ultra-fine lamellar structure was selected as a model material, which was processed via partial recrystallization of the heavily cold-rolled CG counterpart.

2. Experimental

A commercial Al–Mg alloy, 5083 Al alloy (Al–4.4Mg–0.7Mn–0.15Cr–0.2Fe, wt%) was purchased in the form of as-rolled plates. A 5 mm thick piece sectioned from the as-received plates was first

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annealed at 540 °C for 2 h followed by water quenching, in order to obtain fully annealed CG 5083 Al alloy. Then, the piece was cold-rolled to a total thickness reduction ratio of 90% in multiple passes (the thickness reduction ratio from 5% to 10% during each pass). Finally, the 90% cold-rolled piece was annealed at 250 °C for 1 h.

The microstructure of the annealed 5083 Al alloy was studied in the longitudinal planes (i.e., the planes determined by rolling direction and normal direction) using an optical microscope (OM, model: Axiovert 200 MAT) and transmission electron microscope (TEM, model: JEOL JEM 2010) operated at 200 kV. In both OM and TEM micrographs, the equiaxed grain size was denoted by an area-equivalent circle diameter and the average grain size ($\overline{d}$) was evaluated by fitting the statistical distribution of grain sizes using a lognormal probability function [31,32]. In TEM micrographs, the intercept lengths along lines perpendicular (d_T) and parallel (d_L) to the lamellar boundaries were measured and their average values ($\overline{d}_T$ and $\overline{d}_L$) determined by using the same fitting approach for the average equiaxed grain size as described above were implemented to calculate the average boundary spacing of the lamellar structure ($\overline{d}_R$) by the equation [30,33]: $\overline{d}_R = 2/(1/\overline{d}_T + \pi/2\overline{d}_L)$.

The specimens for OM observations were prepared by mechanical grinding and polishing. The microstructure was revealed by etching using 10 vol% H_3PO_4 in deionized water as the reagent at 70 °C for 90 s. The specimens for TEM analysis were prepared by means of mechanical grinding to 30–40 μm in thickness and twin-jet polishing using a solution of 25 vol% nitric acid and 75 vol% methanol at −30 °C. X-ray diffraction (XRD) analysis was also performed on the annealed 5083 Al in a Rigaku D/MAX-2500 diffractometer equipped with a Cu target using the step mode at the speed of 0.02° and the count time of 3 s per step.

Tensile tests on both the annealed and the as cold-rolled 5083 Al were conducted at room temperature using an Instron-5948 Micro-Tester (2 kN) 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 for each type of materials. The dog-bone-shaped tensile specimens with 10 mm in gauge length and 2.5 mm × 0.5 mm in cross-section were sectioned with the axes of the tensile specimens being parallel to the rolling direction via electro-discharge machining and then mechanically polished. During tensile tests, the strains of the gauge sections were accurately measured using a video extensometer. Following tensile tests, the fractured surfaces of the annealed 5083 Al tensile specimens were studied using field emission gun scanning electron microscopy (SEM) Hitachi S-4800.

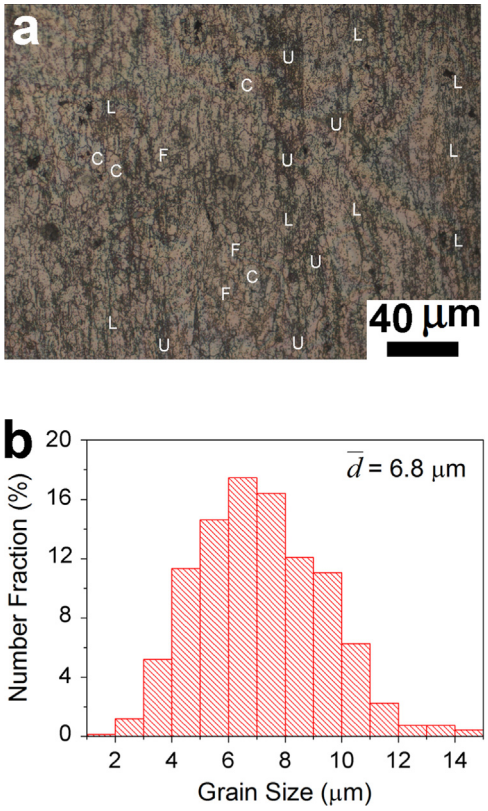

Fig. 1. (a) The typical OM microstructure of the annealed 5083 Al; (b) the statistical distribution of fine and coarse grain sizes.

3. Results

3.1. Microstructure

3.1.1. Overall OM microstructure

The OM micrograph in Fig. 1a shows the typical overall microstructure of the annealed 5083 Al, and it is characterized by a multi-scale microstructural feature that comprises the following components: lamellar structure with ultra-fine dimensions in width as marked by "L" and multi-scale equiaxed grains, including ultra-fine, fine and coarse ones as marked by "U", "F" and "C", respectively. These microstructural components exhibit a reasonably homogeneous distribution. Based on a large number of OM micrographs, the area fractions of the aforementioned four components are measured using software "Image-Pro Plus" [34]: first, an OM micrograph is opened under software "Image-Pro Plus" and the correct scale bar is set in the opened micrograph; then, for a specific region with one of the four microstructural features, the boundary between the region and other regions with different microstructural features is manually delineated; finally, the area of the region is directly measured by the software. Using the aforementioned procedure, areas of the four components in a large number of OM micrographs are determined and summed. The area/volume fractions of the four components are then estimated to be ~40%, ~10%, ~35% and ~15%, corresponding to "L", "U", "F" and "C", respectively. By measuring 670 randomly selected grains, the statistical distribution of fine and coarse grain sizes was determined as shown in Fig. 1b, and the average grain size was evaluated to be ~6.8 μm. In order to provide in-depth understanding of the aforementioned microstructural features, the results of TEM analysis are presented as follows.

3.1.2. TEM microstructure of ultra-fine lamellar structure

TEM bright field (BF) image as shown in Fig. 2a reveals dimensions of the lamellar structure (i.e., regions marked by "L" in Fig. 1a): ~100 to ~500 nm in width and ~0.5 to ~3 μm in length. Fringes and/or network structure are evident inside the lamellae, indicative of the presence of a high density of dislocations [31,35,36]. This observation suggests that the lamellar structure is actually the retained heavily deformed structure that did not undergo recrystallization. Based on the analysis of more than 200 lamellar boundaries, Fig. 2b and c displays the statistical distributions of the intercept lengths along lines perpendicular (d_T) and parallel (d_L) to the lamellar boundaries, respectively. The average values of d_T and d_L were assessed to be $\overline{d}_T = 261$ nm and $\overline{d}_L = 1.4\ \mu m$. The average boundary spacing $\overline{d}_R$ is calculated as ~404 nm.

In order to determine the misorientation of the boundary between two neighboring lamellae, their selected area electron diffraction (SAED) patterns were acquired under the identical global orientation of the TEM specimen and compared with each other. Fig. 3a–c presents the comparison of the SAED patterns between lamellae I and II, between lamellae II and III, and between