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Experiment and modeling of ultrafast precipitation in an ultrafine-grained Al–Cu–Sc alloy

L. Jiang [a], J.K. Li [a], P.M. Cheng [a], G. Liu [a,*], R.H. Wang [b], B.A. Chen [a], J.Y. Zhang [a], J. Sun [a,*], M.X. Yang [c], G. Yang [c]

[a] State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, PR China
[b] School of Materials Science and Engineering, Xi'an University of Technology, Xi'an 710048, PR China
[c] Central Iron and Steel Research Institute, Beijing 100081, PR China

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ABSTRACT

Experimental results revealed that the aging precipitation behaviors were significantly enhanced in an ultrafine grained (UFG) Al–Cu–Sc alloy when compared with its coarse grained (CG) counterpart. In the UFG Al–Cu–Sc alloy aged at 398 K for 20 h, a large number of θ'-Al_2Cu particles with an average radius of about 38 nm were precipitated within the grain interior. While in the CG alloy aged at the same condition, no precipitations were found. The ultrafast precipitation behaviors observed in the UFG alloy is rationalized by developing a precipitation kinetics model. This model is based on the classical N-model framework of Kampmann and Wagner (KWN), but is modified to capture some precipitation features in the UFG regime, such as highly enhanced diffusion and greatly reduced nucleation energy barriers. The modified model yields predictions in good agreement with experimental data, which are several orders of magnitude faster than those predicted by the classical model. This work indicates that the classical N-model, after some modifications, is still applicable for the quantitative description of precipitation behaviors in UFG Al alloys.

1. Introduction

In order to improve the mechanical properties, there is considerable interest in the research of ultrafine grained (UFG) heat-treatable Al alloys processed by severe plastic deformation (SPD), which have been reported to exhibit enhanced strength [1–4]. However, the UFG Al alloys usually have disappointingly low ductility at ambient temperatures mainly due to insufficient strain hardening, which is related to their inability to accumulate dislocations [1–3,5–9]. Dispersing nanosized precipitations in the grain interior has been found [5–9] to be an effective approach to increase the strength and simultaneously improve the ductility of the UFG Al alloys, since the intragranular particles can generate, pin down and thus accumulate dislocations within the grains. Upon annealing treatment, this approach has been successfully utilized in the three kinds of heat-treatable Al systems, i.e., Al–Cu–Mg-based 2000 series [8,9], Al–Mg–Si-based 6000 series [10–12], and Al–Zn–Mg-based 7000 series [6,7,13,14] UFG Al alloys.

Precipitation is so important for heat-treatable Al alloys that any slight variation in precipitate parameters (e.g., volume fraction or size) will cause a visible change in mechanical response. The precipitation behaviors of UFG Al alloys have received growing attention, and some remarkable different precipitation characteristics (e.g., accelerated precipitation kinetics [15], changed precipitation morphology and sequence [14,16]) have been demonstrated in the UFG Al alloys compared with the coarse grained (CG) counterparts. Extensive studies [17–32] have been carried out on the precipitation behaviors in heat-treatable CG Al alloys, including modeling and simulation of precipitation thermodynamics and kinetics. In comparison, the precipitation behavior in UFG Al alloys is somewhat hard to be described in a quantitative manner [21–23,27,32].

The UFG Al alloys processed by SPD are characterized by two microstructural features: a large fraction of grain boundaries (GBs) and a high density of dislocations [1–4,6–8]. These characteristic microstructures are in metastable high-energy condition and should have remarkable effect on the precipitation behaviors. On the one hand, the numerous dislocations in grain interior are prone to assist the heterogeneous nucleation and growth of intragranular precipitates, which should accelerate the precipitation during aging treatment [17] and promote the precipitate dispersion within the grain interior, resulting in noticeable strengthening and ductilizing

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[*] Corresponding authors. Tel.: +86 29 82667143; fax: +86 29 82663453.
E-mail addresses: lgsammer@mail.xjtu.edu.cn (G. Liu), junsun@mail.xjtu.edu.cn (J. Sun).

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response [7–12]. On the other hand, the precipitations in UFG alloys preferentially occur at grain boundaries [15–17], due to the predominant effect of large angle grain boundaries. This not only makes the precipitation in UFG Al alloys much complicated and hard to control, but also inhibits the investigations of intragranular precipitation and quantitative modeling.

Limited previous investigations on the precipitation behavior of UFG Al alloys are mainly phenomenological and qualitative. However, quantitative understanding and modeling of the precipitation thermodynamics/kinetics are crucial to achieve the controlling of precipitation behaviors and mechanical responses. Especially in UFG Al alloys, a high volume of GBs and a large number of dislocations make the precipitation much distinct from their CG counterparts. Applying classic thermodynamic and kinetic models to UFG Al alloys becomes a challenge. Recently, Huang et al. [15] has made an attempt to model the intergranular θ precipitation in UFG Al–Cu binary alloy. The precipitation kinetics was shown to occur at many orders of magnitude higher than that predicted by classical nucleation and growth theory. In contrast, intragranular precipitation in the UFG alloy is far from clear, especially in a quantitative manner. Feasible nucleation and growth models at UFG condition are in urgent need in order to aid the further design of advanced UFG precipitation-hardened alloys.

Our recent research results [4] showed that minor Sc addition into UFG Al–Cu alloys can result in a full intragranular precipitation in the 12-passes ECAP processed alloys. This provides possibility to study the intragranular precipitation at UFG conditions and develop related precipitation models. The present paper will thus focus on quantifying the precipitation kinetics during the aging process of an SPD-processed UFG Al–Cu–Sc alloy, with a view to explaining these unusual aging behaviors and finally manipulating the microstructure and mechanical property of UFG precipitation- hardened Al alloys. This should be helpful for microstructural tailoring and material design of advanced UFG Al-based alloys with higher mechanical properties. The choice of UFG Al–Cu–Sc as studied materials is because that the Al–Cu–(X) alloys are typical heat-treatable aluminum alloys, which have attracted extensive theoretical studies on precipitation thermodynamics, kinetics, and strengthening in CG condition [19–32]. They are also ideal model materials for investigating the precipitation behaviors at UFG condition, especially for quantitative purpose.

2. Experimental procedures

2.1. Material preparation and heat treatments

Alloys with composition of Al–2.5 wt% Cu–0.3 wt% Sc (abbreviated Al–Cu–Sc alloys) were melted and cast in a stream argon. No impurities were detected, considering the experimental accuracy. After homogenization treatment at 723 K for 4 h, billets 100 mm × ∅ 10 mm were machined from the cast ingots, then solute treated in vacuum for 3 h at 853 K and immediately water-quenched to room temperature. Subsequently, the billets were immediately (within half an hour) subjected to 12 passes ECAP by route B_c [1,3], at room temperature in which the work piece was rotated 90° along its longitudinal axis in the same sense between each pass. After ECAP processing, the samples were immediately artificially aged at 398 K for 10 h, 20 h, 30 h and 40 h in oil bath. The maximum error of all the temperature measurements in present experiments was ±1 K.

2.2. Microstructural characterizations

Microstructures of the ECAP-processed samples were characterized by backscatter electron imaging (BSE) and electron backscattered diffraction (EBSD), which was performed on a high-resolution JSM 7001F fitted with a Pegasus XM2–EBSD system, operating at 20 kV. Specimens for SEM-EBSD observation were prepared by electro-polishing using an electrolyte of 25% nitric acid and 75% methanol at 253 K (−20 °C) at an operation voltage of 15 V. To study the microstructure at nanoscale, transmission electron microscopy (TEM) was carried out using a JEOL 2100F operating at 200 kV. Quantitative evaluations of the number density and size of precipitates were reported as an average value of more than 600 measurements. Volume fraction of the second phase particles was determined using the corrected projection method [27,33]. Details about the measurements can be referred to previous publications [27–29].

The dislocation density in a grain was first calculated by counting the number of dislocations in the high-resolution TEM images of the grain and dividing the number by the overall area in the images. The dislocation densities in 60 grains were measured to obtain the statistical data. Meanwhile, according to the Williamson-Hall method [34], microstrain and dislocation density also can be calculated from the XRD peak broadening, following others' work [13,34,35]. Quantitative X-ray Diffraction (XRD) measurements were performed with a Rigaku D/max-RB X-ray diffractometer equipped with a Cu target and a graphite monochromator.

2.3. Measurements of mechanical properties

Tensile testing was used to measure yield strength and ductility (elongation to failure) of the samples before and after artificial aging treatment. Smooth tensile specimens have a gauge size of 1 mm in thickness, 2 mm in width, and 10 mm in length, with axis along the extrusion direction. The testing was performed using an MTS-C43Tester at a constant strain rate of $2\times10^{-4}\ \mathrm{s}^{-1}$ with the load direction parallel to the specimen axis. Engineering stress–strain curves were automatically recorded and the yield strength was determined as the 0.2% offset.

3. Results

3.1. Deformation structures

Fig. 1 presents the EBSD orientation map (Fig. 1(a)) and transmission electron microscopy (Fig. 1(c)) of the Al–Cu–Sc alloys processed by 12 passes of ECAP. Statistical results give average grain size of approximately 340 nm. EBSD analyses results show that the grain boundaries are predominantly high angle grain boundaries (HAGBs ≥ 15°) in the UFG Al–Cu–Sc alloys (Fig. 1(b)). There have been different reports on the dislocation distribution in UFG metals [1–3,6–8,13,17,35–41]. Some claimed [37] that the grain interiors were devoid of dislocations since the dislocations were easily trapped at the GBs, while others observed a high density of dislocations in the grain interior [7,8,13,35,36,38–41]. Hu et al. [17] found a grain size dependent dislocation distribution in UFG 7075 alloy in which dislocations were absent with grain size smaller than about 200 nm. In other's experimental results [7,41], however, dislocations were greatly observed within grains with grain size of several tens of nanometers. It seems that the dislocation distribution in UFG metals should depend not only on the grain size, but also on the processing method/procedures, material characteristics, etc. In present ECAP-processed Al–Cu–Sc alloys with grain size of above ~300 nm, a larger number of dislocations were truly detected in the grain interior. Zhao et al. [7,13] have drawn a conclusion that the dislocation density measured by the XRD method was close to that determined by TEM examinations. Similar results have been also repeated in the present work, as typically shown in Fig. 1(c) for the as-deformed