Materials Letters 65 (2011) 2808–2811

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

STEM-HAADF tomography investigation of grain boundary precipitates in Al–Cu–Mg alloy

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ARTICLE INFO

Article history:
Received 7 April 2011
Accepted 31 May 2011
Available online 12 June 2011

Keywords:
Aluminum alloy
Grain boundary
Heterogeneous nucleation
STEM-HAADF tomography

ABSTRACT

The high angle annular dark field scanning transmission electron microscopy (STEM-HAADF) tomography technique was applied to understand the three dimensional (3D) morphology and distribution characteristic of grain boundary precipitate (GBP) in peak-aged Al–Cu–Mg alloy. The results indicate that GBPs show both spherical and lenticular shapes and triangularly distribute along grain boundary (GB). Based on 3D observations from various directions, the values of GBP relevant parameters such as GBP size, center to center distance, number of GBPs per unit GB area and area fraction of GB covered by GBPs are further determined. The 3D method for GBP relevant parameter determination seems an effective way to avoid misunderstandings in the conventional two dimensional (2D) methods induced by GBP overlapping and projection effect as well as curved GB surface.

1. Introduction

Grain boundary (GB) structures of high strength aluminum alloys, including grain boundary precipitate (GBP) and precipitate free zone (PFZ), were recognized as one of the most vital factors which affect the fracture toughness [1–15] and stress corrosion crack properties [16] especially in the peak- or over-aged conditions [4,5,11]. During loading process, strain localization occurs in the soft PFZs [2,7,11,14–16] and microvoid initiates to form, grow and coalesce near GBPs [2,8,12,16], which will finally cause intergranular fracture especially along high angle grain boundaries [9,10]. This, together with the coarse constitute particle induced fracture and microvoid-induced transgranular fracture, are considered to be three predominant fracture micromechanisms [7,9,10,14,15].

To quantitatively evaluate the effect of GBP on the fracture toughness, many models [2–4,7,9,10,12–15] were proposed with consideration of the microstructural parameters such as GBP size, center to center distance, number of GBPs per unit GB area and area fraction of GB covered by GBPs. As for a GB segment as illustrated in Fig. 1a, these parameters were determined by the following methods. The first one is suggested to tilt the GB surface to be parallel to the incident beam (edge-on) firstly in a transmission electron microscope (TEM) (Fig. 1b) and determine the average values of GBP size, center to center distance of GBPs according to the statistics results, then estimate the number of GBPs per unit GB area and area fraction of GB covered by GBPs using mathematical approximation methods [8,14–16]. However, due to the factor of sample thickness, while keeping GB edge-on GBPs in different depth will more or less overlap with each other especially when they are high in density and small in size. To solve this problem, Li and Reynolds [8] further suggested that TEM observations should be carried out near the thin foil edge, the thickness of which is of the order of the precipitate diameter (Fig. 1c). Nonetheless, this thickness requirement is actually hard to be satisfied or controlled. Accordingly, the thought of the second determination method aimed to take into consideration of the real distribution characteristic of GBPs on GB surface and make real-time measurement of the values of GBP relevant parameters from two or more observation directions [1,2,6] (Fig. 1d). Using this method, GB surface should be tilted to be parallel and inclined to the incident beam successively and meantime parallel to the tilting axis. Unfortunately, this requirement is still not very easy to be satisfied in TEM due to the limitation in tilting ranges of the sample holder, thus the misunderstanding induced by GBP overlapping cannot be effectively avoided. Above all, for both of these two methods, the GBP morphology was determined from observation of one or several two dimensional (2D) TEM images, which are actually projection(s) of GBPs in certain or several independent directions (Fig. 1b–d). Under such conditions, the observed 2D GBP morphology may vary as the observation direction changes and the relevant parameters such as GBP size, center to center distance may also deviate from the real values. Furthermore, GB surface is more often curved rather than planar, thus the determination of the values of GBP relevant parameters by 2D TEM methods may cause even larger errors. The larger the deviation of the measured values of parameters from the real ones, the harder to verify and evaluate a micromechanical model based on the intergranular fracture induced by GBPs. Therefore, a more effective and accurate method to observe the 3D morphology of GBP and further determine its relevant parameters is of

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doi:10.1016/j.matlet.2011.05.115

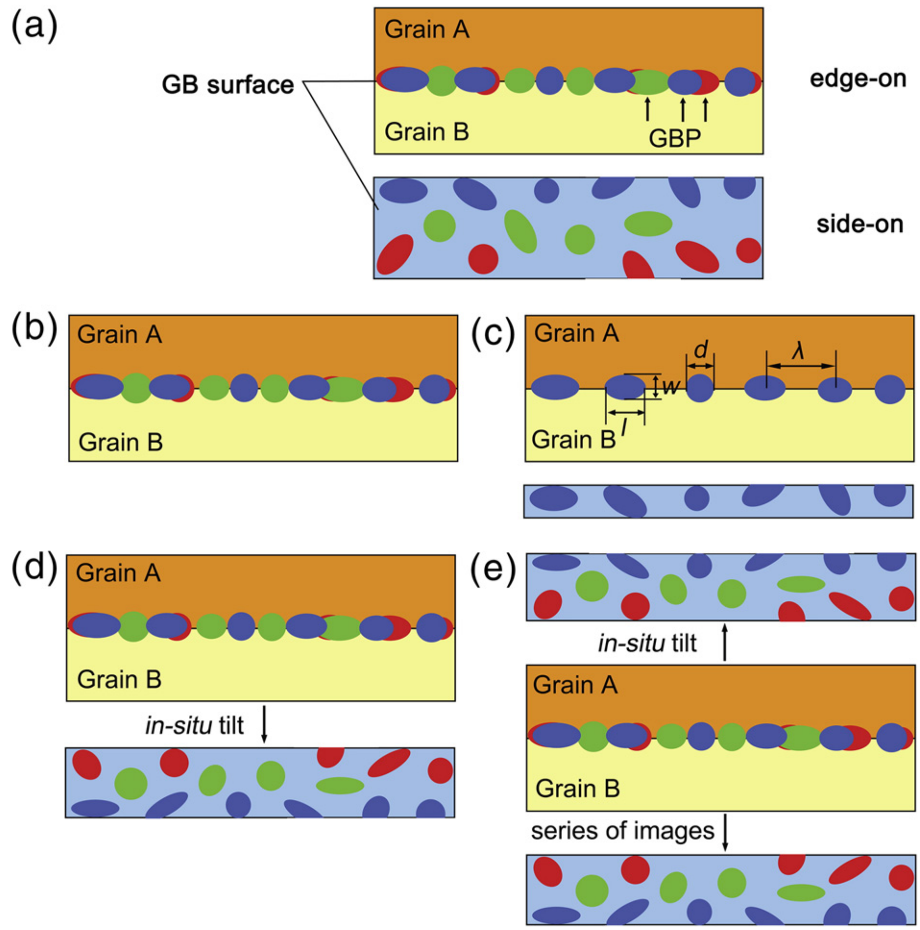

Fig. 1. Schematic illustrations of (a) the real distribution of GBPs along GB when keeping the GB surface edge-on and side-on; (b)–(d) the 2D methods for GBP relevant parameter determination proposed by Refs. [8,14–16], Ref. [8] and Refs. [1,2,6], respectively; (e) the data acquisition of 3D tomography in the present work. Note that GBPs with spherical and lenticular shapes are also represented in blue, green and red respectively, which indicates that these GBPs are in different depth of the sample.

overwhelming importance to fully understand the effect of GB structure on the final mechanical properties in Al–Cu–Mg alloys.

The high angle annular dark field scanning transmission electron microscopy (STEM-HAADF) tomography has been widely used nowadays in various investigation fields [17–19]. Using this method, a tilt series of STEM-HAADF images was acquired, aligned and finally reconstructed to give the real 3D morphology and distribution characteristics of the research objects (Fig. 1e). To effectively reduce measurement errors induced by overlapping and projection effect, the present work is an attempt to use this method to obtain 3D reconstruction of GBPs for fully understanding of their morphologies and distribution characteristics. Based on 3D observations, the values of GBP relevant parameters mentioned above are further determined.

2. Experimental

2024 aluminum alloy with nominal composition of Al–4.2Cu–1.5 Mg–0.6Mn–0.5Fe–0.5Si (wt%) was chosen for investigation. The cast ingot of the alloy was homogenized at 460 °C for 16 h and hot rolled to a 2 mm thin sheet, then solution treated at 505 °C for 45 min, water quenched and aged at 195 °C for 9 h to peak-aged condition. Specimen for TEM was prepared by twin-jet electro-polishing with a solution of 30% nitric acid and 70% methanol below −25 °C at 15 V. STEM observations were performed on a 300 kV field emission TEM, Tecnai F30G^2, equipped with fully automated STEM tomography system. A tilt series of STEM-HAADF images of a GB segment was acquired using Xplore3D software (FEI, Einthoven) and a single-tilt holder (Fischione model 2020) from −70° to +70°, with an increment of 2° at low angle range (<50°) and of 1° at high angle range (>50°). The tomography data was aligned and reconstructed using Inspect3D software (FEI, Einthoven) by a weighted back-projection method. Visualization was finally performed using AMIRA 5.2.

3. Results and discussion

The microstructure of 2024 alloy after aging at 195 °C for 9 h is shown in Fig. 2a and b. Many rod-like $Al_{20}Cu_2Mn_3$ dispersoids [20] can be observed throughout the microstructure of 2024 alloy. Meantime, S (Al_2CuMg) precipitates, the main strengthening phase of 2024 alloy, can be observed both in Al matrix and at GB. S precipitates in grain interiors always take on needle-like shapes, while those at GB (i.e. GBPs) show spherical or lenticular shapes and have dense distribution. To quantitatively investigate the effect of GBP on fracture toughness, the conventional 2D TEM methods aforementioned were adopted to determine the values of GBP relevant parameters. As for GB segment shown in Fig. 2a and b, it can be found that the GB surface is actually bowed rather than planar for the whole visible segment (labeled by arrows). When keeping the central GB part edge-on (Fig. 2a), GBPs cannot be distinguished one by one due to the overlap of the particles located in different depth of the sample. After in situ tilting the GB segment to another angle, GBPs can be clearly distinguished (Fig. 2b) but the values of GBP size and center to center distance still cannot be directly determined since the whole visible GB surface is not sure being kept perpendicular to the incident beam.