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Three dimensional microstructural characterization of nanoscale precipitates in AA7075-T651 by focused ion beam (FIB) tomography

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ABSTRACT

The size and distribution of precipitates in Al 7075 alloys affects both the mechanical and corrosion behavior (including stress corrosion cracking and fatigue corrosion) of the alloy. Three dimensional (3D) quantitative microstructural analysis of Al 7075 in the peak aged condition (T651) allows for a better understanding of these behaviors. In this study, Focused ion beam (FIB) tomography was used to characterize the microstructure in three dimensions. Analysis of grains and precipitates was performed in terms of volume, size, and morphology. It was found that the precipitates at the grain boundaries are larger in size, higher in aspect ratios and maximum Feret diameter compared to the precipitates inside the grains, due to earlier nucleation of the precipitates at the grain boundaries. Our data on the precipitates at the interface between grains and Mg_2Si inclusion show that the surfaces of inclusion (impurity) particles can serve as a location for heterogeneous nucleation of precipitates.

1. Introduction

7075 aluminum alloys are used extensively due to their high strength-to-weight ratio [1,2]. The microstructure of the alloy at different length scales is shown in Fig. 1. It consists of a polycrystalline aggregate of grains as well as second phase particles. The second phase particles can essentially be divided into inclusions (also called constituent particles) and precipitates, whose dimensions are on the order of micrometers and nanometers, respectively. These second phase particles are known to affect the mechanical and corrosion properties of Al alloys [3,4]. Inclusions are known to be formed during casting from impurities. 3D microstructural characterization of inclusions in this alloy using x-ray synchrotron tomography has been performed in previous work [5].

The nanoscale precipitates are formed when the alloy is either naturally aged or artificially aged following a solution heat treatment. It has been widely accepted that the precipitation sequence in AA7075 is as follows: Supersaturated solid solution → GP zones → η' → η [6,7]. However, some have argued that the precipitation sequence follows this sequence: Supersaturated solid solution → GP zones → η' → (η_p →) η [8,9]. Although the yield strength is the highest for peak-aged temper, it is also more susceptible to stress corrosion cracking than over-aged temper [10]. The variation in mechanical and corrosion properties has been attributed to the size, shape, and distribution of precipitates in the alloy microstructure [11,12]. Therefore, in order to understand the monotonic and corrosion properties of the alloy, it is important to quantify the three dimensional (3D) structure of the precipitates in the alloy.

Several studies have been performed on the size and distribution of precipitates in Al alloys [10,13–20]. To the best of our knowledge, to date the characterization of large volumes of precipitates in AA7075 has been performed only in two dimensions (2D). Most of these studies have used transmission electron microscopy (TEM), which provides very high resolution images. However, due to the small area considered, the results usually cannot be considered a statistically representative sample of the materials. Birbills et al. [21] used 2D stereology along with TEM to characterize and quantify size of precipitates. Recently, other techniques such as amplitude-modulated atomic force microscopy (AM-AFM) along with TEM have been used to quantify the precipitate structure [13], while differential scanning calorimetry (DSC) [12, 22,23] and X-ray scattering techniques [24] have been used to study the formation and dissolution of precipitates in Al 7075 alloys. The shape of η' precipitates has been shown to be platelike [22,25–27], whereas η is formed in either plate or rod form [10,22,25,26]. The size of η and η' precipitates from all these studies falls in the range of approximately 10–300 nm.

Experimental techniques for 3D microstructural characterization include atom probe tomography (APT) [28], electron tomography [29], focused ion beam (FIB) tomography [30], serial sectioning followed by optical microscopy [31], and x-ray tomography [32]. Serial sectioning followed by optical microscopy and x-ray tomography can be used to study large volumes of material, resulting in statistically significant

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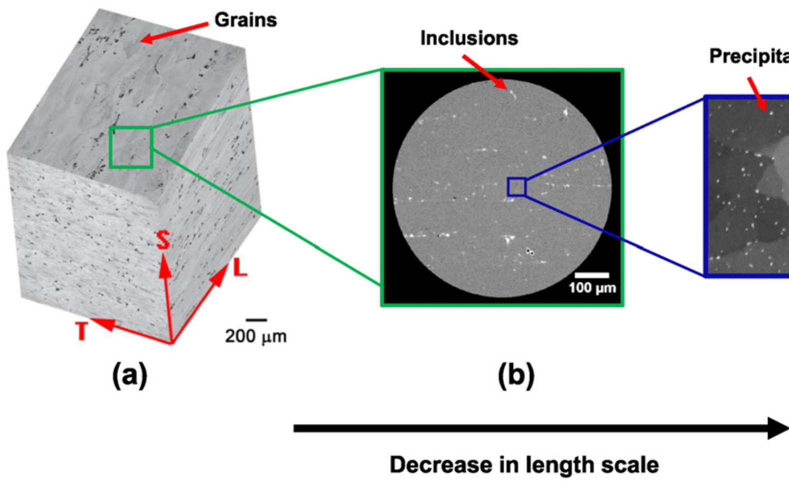

Fig. 1. Microstructure of Al 7075 alloys in decreasing length scales (a) optical surface image showing grains, (b) X-ray tomography image (2D virtual slice of 3D volume) showing inclusions, and (c) grains and nanoscale precipitates visible in FIB-SEM slice.

information, but due to limited resolution, these techniques cannot be used for precipitates. 3D atom probe tomography and electron tomography provide very high resolution images, but only very small volumes can be analyzed. Although destructive in nature, focused ion beam (FIB) tomography is well suited to study the precipitates in Al alloys. It provides high resolution images (on the order of 10 nm) along with a large enough volume to provide statistically accurate information of nano-scale precipitates. FIB tomography has already been successfully utilized to characterize the microstructure in Al-SiC nanolaminates [33], Pb-free solders [34,35], and superalloys [36,37].

The present work is a follow up to our recent work on correlative tomography [38]. Non-destructive XRM (X-ray microscopy) data acquisition was used to identify inclusions (larger in size as shown in Fig. 1) and associated pores, and then to inform and automate FIB-SEM (Focused ion beam-scanning electron microscopy) tomography in the same area to visualize precipitate structures (at much smaller length-scale). This approach may be extended to multiple FIB-SEM tomography sites, greatly expanding FIB utilization via the navigation workflow of Atlas 5 and XRM data [38,39]. Furthermore, the preceding non-destructive XRM analysis may, importantly, be extended to time-dependent (4D) or in situ conditions to quantify the evolution of a structure (processing, aging, corrosion, etc.) prior to inspection by FIB-SEM tomography at defined locations. Recently, we have visualized and quantified the microstructure of inclusions and pores in 3D and have also obtained their mechanical and corrosion properties from the same plate of AA7075 [5,40,41].

In this study, we have obtained the 3D microstructure of precipitates in an aluminum alloy AA7075-T651. The volume, size, and distribution of precipitates were visualized and quantified both in the grain boundaries and inside the grains. For the first time, a large volume was studied using FIB tomography to obtain quantitative, statistical information on the 3D microstructure of the precipitates. We also show that large inclusion particles can serve as heterogeneous nucleation sites for precipitate formation, thus localizing precipitate formation. A knowledge of the 3D microstructure will facilitate a better understanding of the mechanical and corrosion properties of the alloy.

2. Experimental procedure

The material used in this study was a commercially available 7075-T651 aluminum alloy (5.63 Zn, 2.45 Mg, 1.55 Cu, 0.045 Si, 0.18 Fe, 0.008 Mn, 0.19 Cr, 0.004 Ni, 0.049 Ti, and rest Al) rolled to a 2.5 cm thickness (Alcan rolled product, supplied by Dix Metals Inc). A small specimen (~3 mm × 3 mm × 4 mm) was cut near the surface of the rolled plate. The specimen was polished to a 1 µm diamond finish and then to a final finish of 0.05 µm colloidal silica.

Correlative tomography using a combination of X-ray microscopy (XRM) and FIB-tomography was performed on the polished sample. The 3D XRM datasets were loaded into ZEISS Atlas 5 for alignment and registration with the 2D surface SEM images (online acquisition), upon which the XRM dataset was used to navigate the FIB-SEM to the precise region for FIB-tomography. The details of data acquisition using X-ray microscopy are provided elsewhere [38].

Atlas FIB-SEM tomography was acquired on a ZEISS Crossbeam 540 utilizing 10 nm isotropic voxel size and an EsB detector with SEM voltage of 1.5 kV. The collected FIB-SEM volume was approximately 18 µm × 8 µm × 7 µm. The sampled volume (~1000 μm^3) was chosen to obtain a good statistics on the size and shape of the precipitates and

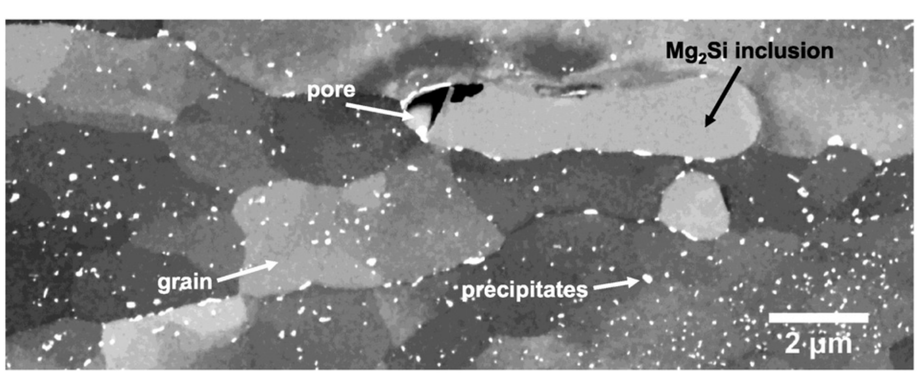

Fig. 2. 2D slice of AA7075 obtained from FIB tomography showing grains, an inclusion, a pore associated with the inclusion, and precipitates.