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Accelerated precipitation and growth of phases in an Al-Zn-Mg-Cu alloy processed by surface abrasion

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ABSTRACT

The surface microstructure and its evolution during long-term room-temperature storage were studied using transmission electron microscopy for an Al-Zn-Mg-Cu alloy processed by surface abrasion with grinding paper. An altered surface layer (ASL) with thickness of 0.4–0.8 μm was present on the alloy after abrasion. Ultrafine subgrains with width of about 50–120 nm and a high density of dislocations were observed in the ASL. The pre-existing aging-induced η′ and η precipitates dissolved during surface abrasion. During room-temperature aging, relatively pure Zn, Al_2Cu and AlCu phases were observed to precipitate at the extreme surface and subgrain boundaries in the ASL. These phases are very unusual in that they are typically not formed in Al-Zn-Mg-Cu alloys. Mg was not found in these particles, as it remained dissolved in the solid solution of the ASL. Al_2Cu and AlCu phases also precipitated at the grain boundaries in the underlying substrate right below the ASL, as far as 6 μm in depth from the extreme surface. Considerable growth and coarsening of these phases occurred during natural aging over a period of 42 months. The enhanced diffusion accelerated by vacancies, dislocations, and subgrain/grain boundaries was considered to be mainly responsible for the accelerated precipitation and growth of these atypical phases.

1. Introduction

High-strength age-hardenable aluminum alloys are commonly used for structural components of aircraft. Many Al alloys components experience processes including cold rolling, machining, grinding and polishing that can introduce heavy plastic deformation on the alloy surface. The heavy deformation can create deformed surface layers with thickness ranging from hundreds of nanometers to a few micrometers. Such deformed surface layers contain ultrafine grains/subgrains and other defects, which can influence the service performance of the components, in particular the corrosion resistance [1–7]. Corrosion fatigue, one of the most significant damage mechanisms in aging aircraft structures, is often initiated by localized corrosion pits, which can readily form if the surface is susceptible to attack [8,9]. Liu et al. reported that a deformed surface layer with fine grains was produced on AA6011 by mechanical grinding [3]. During subsequent aging at 180 °C for 30 min, Q phase ($Cu_2Mg_8Si_7Al_4$) precipitated at the grain boundaries in the surface layer, while no such phase was observed in the underlying bulk matrix. The enhanced phase precipitation in the surface layer made the alloy susceptible to filiform corrosion [3]. Surface abrasion and polishing of Al alloys, similar to what might be performed during test sample preparation or in surface finishing of a manufactured part, also can result in the formation of an altered surface layer (ASL) with microstructure that is different than the underlying bulk material [10–16]. Such ASLs in Al alloys have been shown to exhibit a different corrosion resistance than the underlying matrix, and can be either more susceptible or more resistant to corrosion [10–15].

Relevant to the formation of micro- and nano-scale microstructures is the development of severe plastic deformation (SPD) processing techniques like equal-channel angular pressing (ECAP) and high-pressure torsion (HPT), which have received a lot of attention recently [5,17,18]. In particular, the effects of SPD on the nucleation, growth and coarsening of precipitates in Al alloys have

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been extensively studied [19–31]. In general, dynamic precipitation during SPD or precipitation after SPD occurs much faster than precipitation in an undeformed Al alloy at elevated temperature [23,29,32]. A remarkable observation in Al-Cu alloys is the copious precipitation of θ phase at the grain boundaries in a sample processed by ECAP at room temperature followed by several months of natural aging, while no evidence of Guinier-Preston (GP) zones or metastable transition phases like θ″ and θ′ was observed within the grains [26]. Accelerated θ phase precipitation was also observed at the grain boundaries in an Al-Cu sample after room-temperature ECAP followed by aging at 100 °C for 24 h, while only GP zones were found for the same aging treatment on an undeformed sample [28]. On the other hand, only GP zones were detected in an Al-Zn-Mg-Cu alloy after room-temperature HPT followed by several months of natural aging [23]. Precipitation was accelerated during artificial aging of the same material at elevated temperature, but the precipitation sequence was not changed by SPD [23]. Similar results were also observed in ECAP-processed AA7075 at 473 K [32]. It is still not very clear why the precipitation kinetics of these two alloy systems respond differently to SPD. When an SPD process like surface mechanical attrition treatment (SMAT) is applied to a material surface, a thick surface layer with ultrafine grains is achieved on the coarse-grained bulk material, which can improve the mechanical and tribological properties of the material [17].

In the present work, the surface microstructure on an Al-Zn-Mg-Cu alloy (AA7055) subjected to surface abrasion with grinding paper and its evolution during long-term natural aging were investigated by scanning transmission electron microscopy (STEM). Atypical phases precipitated both at the subgrain boundaries in the ASL and at the grain boundaries in the underlying substrate adjacent to the ASL. Furthermore, over a period of 42 months at room temperature, these phases grew and coarsened to an extent that would not be expected. The precipitation of these phases and their accelerated growth at room temperature have never been reported in an Al-Zn-Mg-Cu alloy. Thus, the present paper focuses on studying the precipitation behavior in surface microstructure on AA7055 processed by surface abrasion, with the aim of understanding these unusual and interesting phenomena.

2. Experimental

Commercial AA7055-T77 with chemical composition of Al-7.76Zn-1.94Mg-2.35Cu was investigated. Some of the samples were solution-treated at 475 °C for 1 h, quenched in water to room temperature to achieve supersaturated solid solution, and then artificially aged at 120 °C for 6 h followed by aging for 24 h at 163 °C to achieve the T73 over-aged state.

After the artificial aging process, the samples were ground using SiC grinding paper from 180 to 320 or 1200 grit. Ethyl alcohol was used instead of water to minimize corrosion during abrasion. The original surface layer produced by rolling or machining was completely removed by surface abrasion. The ASLs observed in the present work were generated by surface abrasion, in particular the final abrasion step, because of continual material removal during the abrasion process.

Two methods were used for preparing TEM foils based on the different microstructures studied. To examine the surface microstructure, the focused ion beam (FIB) technique was used to prepare the cross-sectional TEM samples. These samples were cut parallel or perpendicular to the final abrasion direction as indicated by the scratches on the surface. Before FIB cutting, platinum was deposited on the sample surface to avoid possible surface damage during ion milling. To examine the bulk material far from the abraded surface, TEM foils were prepared by twin-jet electropolishing at 30 V using a solution of 30% nitric acid and 70% methanol cooled to −30 °C.

The TEM foils prepared by FIB were kept at room temperature in a storage case that was exposed to lab air over a period of 42 months. TEM characterization of the microstructure was performed on these TEM foils after periods of natural aging for 2, 12 and 42 months. TEM characterization of the microstructure was conducted with an FEI G2 Tecnai F30 microscope operated at 300 kV and an FEI Talos F200X operated at 200 kV equipped with Super-X energy dispersive spectroscopy (EDS). High-angle annular dark-field (HAADF) imaging, EDS line profiles and maps were performed in STEM mode. The probe size for EDS analysis was less than 3 nm and the step size of the beam scan was around 1 nm.

3. Results

3.1. Microstructural features in the ASL and ASL-affected zone

Fig. 1 shows cross-sectional bright-field TEM and HAADF images of the AA7055-T73 sample after grinding to 320 grit. A unique ASL with thickness of around 400 nm was located between the protective Pt layer and the underlying substrate. The extreme surface close to the Pt layer was wavy, because the TEM foil was cut by FIB sectioning perpendicular to the final abrasion direction. The topmost part of the ASL contained nearly equiaxed subgrains with size of around 50–100 nm as shown Fig. 1b. The black and gray wavy bands at a distance of ~0.4–1.4 μm from the top surface in Fig. 1a are likely bend contours.

HAADF images were acquired in STEM mode to provide strong contrast associated with differences in chemical composition. Fig. 1c shows the presence of the ASL and two grain boundaries in the underlying substrate that are marked with double-headed arrows. One grain boundary was as far as 5 μm in depth from the extreme surface. The other was in contact with the ASL. These two grain boundaries in the underlying substrate were different than the subgrain boundaries in the ASL produced by abrasion. It can be reasonably speculated that these grain boundaries were original, not produced during abrasion. Precipitates were observed on both grain boundaries. The bright contrast associated with the ultrafine subgrains in the high-magnification HAADF image shown in Fig. 1d clearly reveals the segregation of high atomic number elements at these boundaries. Many coarse blocky precipitates with size of about 50–100 nm were also evident at the subgrain boundaries in the ASL and are marked with triangles in Fig. 1d. However, the pre-existing aging-induced η′ precipitates were absent in the ASL.

Fig. 2 shows EDS line profiles measured along the lines marked as I-III in Fig. 1d and IV-VI in Fig. 1c. The relatively small precipitate crossed by the line labeled I contained high contents of Mg and Zn, with slight enrichment of Cu. This precipitate should be η phase. The large blocky precipitate crossed by the line labeled II and elliptical precipitates crossed by the lines labeled IV-VI contained high contents of Cu, with little Mg and Zn. Diffraction patterns of the precipitate crossed by the line labeled IV confirmed that this precipitate was consistent with Al_2Cu (θ) phase with body-centered tetragonal structure (*I*4/*mcm*, No.140, $a = b = 0.6063$ nm, $c = 0.4872$ nm), as shown in Fig. 3. The other precipitates with high Cu content are likely also θ phases. It is surprising that precipitation of θ phase occurred in an Al-Zn-Cu-Mg alloy, because θ phase is not commonly present in this alloy; η phase is the common grain boundary precipitate. The EDS line scan profiles in Fig. 2c, which is along the line labeled III in Fig. 1d, show that Al oxide was present in localized areas of the ASL.

Fig. S1 in the supplementary material shows an HAADF image and EDS line profiles of grain boundary precipitates in the bulk of the AA7055-T73 sample, far below the surface. The TEM foil of the