DOI: 10.1017/S1431927606068140
Microsc Microanal 12(Supp 2), 2006

Temporal evolution of GP zones in an Al-Ag alloy

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In the phase-separating Al-Ag system, intermediate metastable precipitates (Guinier Preston zones) are formed. The metastable miscibility gap is characterized by a large asymmetry with a gap extending to ~55 at.% Ag below 175°C and to ~30 at.% Ag above 220°C [1]. The exact structure of the GP zones (ordering, Ag concentration) and the existence of temperature-dependent phases (ordered at low T and disordered at high T) is still matter of debate and the experimental studies published on this system have yet to concur. Previous atom probe studies have confirmed the variations of GP zone concentration with temperature [2] and a more recent work by Al-Kassab et al. showed the coexistence of two types of precipitates [3]. Recent TEM observations have also indicated that in the initial stages of decomposition, GP zones form with a surprising morphology consisting of a Ag-rich shell surrounding an Al-rich inner core [4].

This paper discusses our recent transmission electron microscopy and atom probe tomography measurements of the three-dimensional Ag concentration in precipitates, including the temporal evolution.

An Al-2.7 at.% Ag alloy (prepared by arc melting of 99.999% purity Al and Ag) was homogenized at 580°C for 48 hours in Ar atmosphere. After iced water quenching, the alloy was aged in an oil bath at 130°C and 220°C for times varying from 30 minutes to 1000 hours. TEM and atom probe tips were prepared by electropolishing using a solution of 1:3 nitric acid in methanol. Field evaporation was performed on a LEAP microscope at 20K, with a pulse fraction between 15 and 20% and a repetition rate of 200kHz.

Quenching the alloy lead to phase separation with the appearance of Ag-rich domains. The domains do not have a precise shape at early times but evolve to spherical precipitates with faceting at long aging times. During aging at 130°C, the domain size coarsening kinetics follow a power law with an exponent close to 1/3. Surprisingly, the concentration of the precipitates is size dependent: the Ag concentration decreases with decreasing size, suggesting a strong role of the interfacial energy. T late times, the Ag concentrations in the GP zones follows that predicted by the metastable phase diagram: 38±4 at.% Ag at 220°C and 63±3 at.% Ag at 130°C.

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[5] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the United States Department of Energy National Nuclear Security Administration under contract DE-AC04-94AL85000. Special thanks to Mark Homer for his invaluable contribution to specimen preparation.

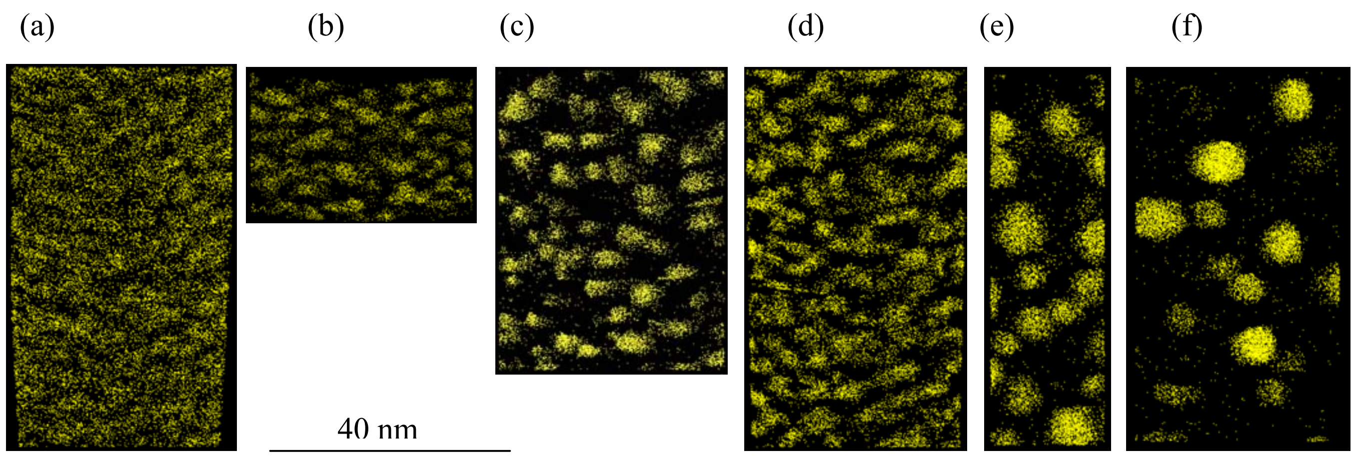

FIG. 1. 3D reconstructions from tips (a) in the as quenched condition and (b-f) after aging at 130°C for 30 minutes, 2, 8, 44 and 116 hours. Ag atoms are represented by yellow dots while the Al atoms are omitted for clarity of the image.

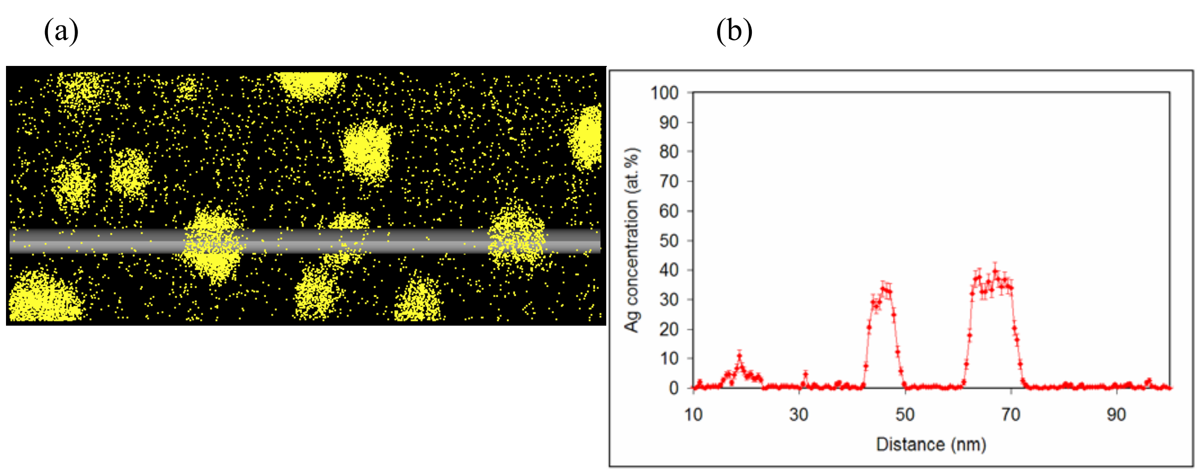

FIG. 2. (a) Slide from a 3D reconstruction obtained from a tip aged at 220°C for 1 hour. Ag atoms are represented by yellow dots while the Al atoms are omitted for clarity of the image. The GP zones are well defined and a concentration profile taken along the cylinder intercepting GP zones in shown in (b). The average concentration in the precipitates is 63±3 at.% Ag.

https://doi.org/10.1017/S1431927606068140 Published online by Cambridge University Press