

Silver segregation to θ' (Al_2Cu)–Al interfaces in Al–Cu–Ag alloys

Julian M. Rosalie^{a,*}, Laure Bourgeois^{b,c,d}

^a National Institute for Materials Science, Sengen, Tsukuba, Ibaraki 305-0047, Japan

^b Monash Centre for Electron Microscopy, Monash University, Vic. 3800, Australia

^c Department of Materials Engineering, Vic. 3800, Australia

^d ARC Centre of Excellence for Design in Light Metals, Vic. 3800, Australia

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Abstract

θ' (Al_2Cu) precipitates in Al–Cu–Ag alloys were examined using high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM). The precipitates nucleated on dislocation loops on which assemblies of γ' (AlAg_2) precipitates were present. These dislocation loops were enriched in silver prior to θ' precipitation. Coherent, planar interfaces between the aluminium matrix and θ' precipitates were decorated by a layer of silver two atomic layers in thickness. It is proposed that this layer lowers the chemical component of the Al– θ' interfacial energy. The lateral growth of the θ' precipitates was accompanied by the extension of this silver bilayer, resulting in the loss of silver from neighbouring γ' precipitates and contributing to the deterioration of the γ' precipitate assemblies.

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1. Introduction

Aluminium–copper (Al–Cu) alloys are one of the most-studied precipitation-strengthened alloy systems, in part due to their historic connection to the first precipitation-hardened Al alloys (Al–Cu–Mg) [1,2]. Although Al–Cu alloys are amongst the earliest examples of precipitation-strengthened Al systems, alloys derived from these compositions, such as alloy designations 2014 and 2024, are still used in aerospace applications [3].

Al–Cu alloys derive a significant measure of their strength from the controlled precipitation of the θ' phase on the {100} planes of the Al matrix. These precipitates impede dislocation glide, enhancing the resistance to plastic deformation and hence increasing the yield strength. The Al–Cu system is also of great practical interest since the precipitation of the θ' phase can be manipulated through

deformation and by the presence of various solutes, some of which (e.g. In, Sn and Cd) are potent even at trace levels [4–6].

Industrial variants of these alloys have considerably more complex compositions than simple, “model” alloys. Compositions are frequently chosen so as to improve mechanical properties through the formation of multiple precipitate phases. In Al–Cu–Mg alloys, for example, laths of S phase (Al_2CuMg) can form in addition to θ' plates. Multiple phase precipitation and the presence of additional elements in solution pose severe difficulties in clarifying the effect of any given element on θ' precipitation. Consequently, despite research spanning the past half-century, the mechanism(s) by which solute elements affect the precipitation of the θ' phase remain poorly understood.

The precipitate–matrix interface plays an important role in precipitation strengthening. A well-known example of this involves additions of Ag to Al–Cu–Mg alloys, which results in the precipitation of a phase termed Ω rather than θ' . Although the Ω phase shares the same bulk composition as θ' , it forms with {111} habit instead of {100} and

* Corresponding author. Tel.: +81 29 859 3343x6288; fax: +81 29 859 2101.

E-mail address: rosalie.julianmark@nims.go.jp (J.M. Rosalie).

displays remarkable coarsening resistance. Drift-corrected energy dispersive X-ray analysis [7] and 3-D atom probe studies [8] have shown that Ag and Mg both segregate to the Ω -Al matrix interface. The Ω phase has a distinctive interface structure, with a bilayer of Ag atoms at the coherent interface with the matrix [9]. Density functional theory calculations indicated that while substitution of either Mg or Ag alone was energetically unfavourable, a combination of both elements provided a lower-energy interface [10].

Recent work has shown that compositional changes at the θ' -matrix interface occur in other alloys. Si was found to segregate to the coherent {100} interfaces of the θ' phase in Al-Cu-Si alloys, substituting for Cu sites in the precipitate [11]. It has also been demonstrated that the interface composition and structure of θ' in binary Al-Cu differs substantially from the bulk structure, with additional Cu atoms occupying octahedral interstices in the precipitate [12]. Despite these studies a detailed understanding of precipitate-matrix interfaces has yet to be developed in the majority of Al alloys.

Aluminium-copper-silver (Al-Cu-Ag) alloys are ideal for studying the effect of a third element on the precipitation of the θ' phase. Unlike many ternary systems (e.g. Al-Cu-Mg) the Al-rich region of the phase field contains only binary phases [13], eliminating the complexities associated with competing ternary phase precipitation. The interatomic spacing in pure Ag also corresponds very closely to that of pure Al, and substantial additions of Ag have little effect on the lattice parameter of Al [14,15]. Solute misfit can therefore be regarded as minimal, and solute-induced strain can be largely neglected. In addition, the chemical affinity between Ag and Cu atoms is weak and the strong co-clustering behaviour exhibited by systems such as Al-Si-Mg is not observed.

Early studies on Al-Cu-Ag alloys established that for compositions of ~ 1 at.%(Ag,Cu) both γ' (AlAg₂) and θ' precipitates were formed [16] and it been shown that both phases can form on dislocation loops, with θ' and γ' platelets observed at different regions of the same dislocation loop [17,18].

We have recently shown that there is significant Ag segregation around the dislocation loops at the site where θ' precipitates nucleate and that the precipitates are later surrounded by an atmosphere of Ag [18]. This work examines the segregation of Ag to θ' precipitates in an Al-Cu-Ag alloy with an emphasis on the atomic structure of the Al- θ' coherent interfaces.

2. Experimental details

Alloys containing Al-0.90 at.% Ag-0.90 at.% Cu (Al-3.45 wt.% Ag-2.05 wt.% Cu) were cast from high-purity elements in air at 700 °C. The composition was verified via inductively coupled plasma-mass spectroscopy. The largest single impurity was Fe (0.02 wt.%), with most other impurities (including Mg) present at <0.005 wt.%. The cast ingots were homogenized (525 °C, 168 h) before being

hot-rolled (to 2 mm thickness), followed by cold-rolling to 0.5 mm thickness. Further external deformation was avoided from this point onwards. Discs (3 mm diameter) were punched from the sheet, solution-treated (525 °C, 0.5 h) in a nitrate/nitrite salt pot and quenched to room temperature in water. Isothermal ageing of these discs was conducted in an oil bath at 200° for between 2 and 4 h.

Transmission electron microscopy foils were prepared by manually grinding the aged discs, followed by thinning to perforation by twin-jet electropolishing in a nitric acid/methanol solution (~ 13 V, -20 °C in 33% HNO₃/67% CH₃OH v/v). Discs were plasma-cleaned prior to examination.

High-angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) images were obtained using a FEI Titan³ 80–300 microscope operating at 300 kV, with convergence semi-angle of 15 mrad, thus resulting in a spatial resolution of ≈ 1.2 Å and with an inner collection angle of 40 mrad. By default, images are presented adjusted only for brightness and contrast. Image analysis was performed using ImageJ software, version 1.44. Energy-dispersive X-ray analysis (EDX) maps were obtained in STEM mode using a JEOL Si(Li) detector installed on a JEOL 2100F microscope operating at 200 kV with a probe size of 0.5 nm. The maps were 256 × 256 pixels in size, with an acquisition time of 0.2 ms per pixel over ≈ 100 frames.

3. Results

3.1. Microstructural overview

In foils aged for 0.16 – 2 h at 200 °C the microstructure contains only γ' (AlAg₂) precipitates and Ag-rich Guinier–Preston zones (GPZs). The γ' precipitates are arranged in elliptical loops, each comprised of alternating variants, with the loop normal close to a $\langle 011 \rangle$ direction. The morphology and formation of these precipitate assemblies have been described previously [17] and are therefore not discussed in detail.

θ' precipitates are observed in samples aged for a minimum of 2 h at 200 °C. Fig. 1(a) presents a low-magnification HAADF-STEM image obtained after 4 h ageing and shows several γ' precipitate assemblies, some of which also include θ' precipitates. Spheroidal Ag-rich GPZ are also present. Most γ' assemblies have been truncated during foil preparation; however, one complete example is present in the centre of the image.

θ' precipitates are frequently observed in pairs at opposite ends of a precipitate assembly, as is shown in Fig. 1(b). Both θ' precipitates in Fig. 1(b) show enhanced contrast on the coherent interfaces with the matrix, which, as will be demonstrated, is due to Ag segregation.

EDX maps show segregation of Ag to residual dislocations at the ends of the precipitate assemblies. Fig. 2 shows HAADF-STEM images and EDX maps of θ' and γ' precipitates at the end of an assembly. Both precipitates display

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