HAL open science

POSITION SENSITIVE ATOM PROBE STUDIES OF THE COMPOSITION OF Ω AND θ' PRECIPITATES IN Al-Cu-Mg-Ag ALLOYS

C. Grovenor, B. Shollock, K. Knowles

▶ To cite this version:

C. Grovenor, B. Shollock, K. Knowles. POSITION SENSITIVE ATOM PROBE STUDIES OF THE COMPOSITION OF Ω AND θ' PRECIPITATES IN Al-Cu-Mg-Ag ALLOYS. Journal de Physique Colloques, 1989, 50 (C8), pp.C8-377-C8-380. 10.1051/jphyscol:1989864 . jpa-00229962

HAL Id: jpa-00229962

https://hal.science/jpa-00229962

Submitted on 4 Feb 2008

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

COLLOQUE DE PHYSIQUE
Colloque C8, Supplément au n°11, Tome 50, novembre 1989

POSITION SENSITIVE ATOM PROBE STUDIES OF THE COMPOSITION OF Ω AND θ' PRECIPITATES IN Al-Cu-Mg-Ag ALLOYS

C.R.M. GROVENOR, B.A. SHOLLOCK and K.M. KNOWLES*

Department of Metallurgy and Science of Materials, Park Road, GB-Oxford OX1 3PH, Great-Britain
* *Department of Materials Science and Metallurgy, Pembroke Street, GB-Cambridge CB2 3QZ, Great-Britain*

Abstract Low concentration additions of silver to Al-Cu-Mg alloys can lead to the formation of platelike precipitates, designated Ω, is formed on {111} planes. Although the morphology of these precipitates has been extensively studied, little information exists about the chemistry of the Ω precipitates, and in particular, about local elemental segregation.
In this paper we report the results obtained in a series of experiments carried out in the Oxford atom probe laboratory on the microchemistry of both Ω and θ' precipitates in an Al-Cu-Mg-Ag alloy. The Ω precipitates are found to be richer in magnesium and silver than the θ' precipitates, but are found to have a lower copper content. Studies of coherent interfaces of the Ω precipitates suggest that there is no preferential segregation of magnesium or silver to these interfaces.

1 - INTRODUCTION.

It has been known for some time that the addition of low concentrations of silver to Al-Cu-Mg alloys can result in an increased hardening on ageing between 150 and 250°C (1, 2). It has also been shown that on ageing silver-containing alloys with high copper to magnesium ratios a fine distribution of platelike precipitates is formed on {111} planes, and it is conjectured that it is these precipitates which lead to the increased hardness of the alloys (3). This precipitated phase has been designated Ω by Chester and Polmear (4), and Polmear and Cooper (5) have recently described how Al-Cu-Mg alloys with compositions in the $\alpha + \theta + S$ or $\alpha + \theta$ regions of the phase diagram can be modified by addition of silver to give alloys which contain strengthening distributions of both Ω and θ'. These precipitated phases are easily distinguished in transmission electron microscope images because the well-known θ' phase forms preferentially as large plates on {100} planes (6, 7). Wrought aluminium alloys containing about 6% Cu, 0.5% Mg and 0.5% Ag can have an excellent range of mechanical and electrical properties as a result of the presence of these phases (5).

The structure of the Ω phase has been the subject of some discussion, but Knowles and Stobbs (8) have concluded on the basis of very detailed high resolution electron microscopy (HREM) and electron diffraction experiments that the phase has a face centred orthorhombic structure with a = 0.496nm, b = 0.859nm and c = 0.848nm. This structure is very similar to that proposed earlier by Auld (9, 10), and can be considered as a slight distortion of the Al_2Cu θ phase formed in overaged Al-Cu alloys. These Ω precipitates are almost coherent with the aluminium matrix in the {111} planes but are heavily misfitting in the [111] directions, and so grow as large but very thin plates on the {111} planes.

However, while the structure of the Ω phase is now well established, its composition is only poorly characterised. It has been generally assumed that these precipitates must contain both silver and magnesium, and Taylor et al (11) have suggested that they nucleate as Mg_3Ag Guinier Preston (GP) zones and grow by the collection of copper and aluminium atoms to attain an overall stoichiometry of approximately Al_2Cu. More recently Cousland and Tate (12) identified only GP zones of nominal composition MgAg in the early stages of decomposition of Al-Mg-Ag alloys, (although by analogy with data obtained by atom probe microanalysis on the composition of GP zones in Al-Cu and Al-Zn-Mg alloys (13, 14) it seems likely that the zones also contain significant amounts of aluminium). Muddle and Polmear (15) have used Energy Dispersive X-ray analysis (EDX) to study the composition of relatively large Ω precipitates, and observe silver in all cases but magnesium only when the experiment is carried out in a Scanning Electron Microscope on precipitates revealed by etching away the surrounding aluminium matrix. They suggest that Ω contains as much as 28at% Cu with much lower levels of silver and with no definite conclusions reached as to the magnesium content, but emphasise that it is difficult to obtain quantitative data on precipitate composition in a conventional EDX experiment. In addition, these authors show that silver may segregate to the interface between the Ω phase and the matrix, and analyse the θ' precipitates to contain neither silver nor magnesium.

In this paper we report the results obtained in a series of experiments carried out in the Oxford atom probe laboratory on the microchemistry of both Ω and θ' precipitates in an Al-Cu-Mg-Ag alloy.

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jphyscol:1989864

2 - EXPERIMENTAL DETAILS.

An alloy of composition Al 4% Cu, 0.3% Mg, 0.4%Ag by weight was solution treated at 520°C followed by quenching and ageing at 170°C for 24 hours. This treatment produces a very fine distribution of Ω precipitates with some θ' particles as well (4). Specimens for Field Ion Microscope (FIM)/atom probe experiments were prepared by electropolishing in the standard solutions for aluminium alloys. These specimens were examined in the Oxford FIM 100 atom probe. Further information on the procedures that have to be followed successfully to image and analyse FIM specimens of aluminium alloys are presented in an earlier publication (13).

3 - RESULTS.

Figure 1 presents a FIM micrograph of the heat treated alloy formed in argon at approximately 60K. Two kinds of brightly imaging precipitates are visible, θ' and Ω. The Ω precipitates are smaller and appear brighter in the image, the θ' larger and less bright because they have an effective voltage in excess of the best image voltage. The precipitates appear as long rows of bright spots, thus implying that we are looking along the edge of the disc-shaped precipitates. As copper and silver are expected to image brightly in Al, these precipitates must be rich in at least one of these two solute elements. As magnesium does not image brightly in Al (16), no conclusions concerning the segregation of magnesium can be made from the FIM images.

The POSAP has proved to be a valuable tool in evaluating the three dimensional microchemistry of the two types of precipitates in this alloy. Figure 2(a) presents a POSAP copper distribution map from a typical θ' precipitate. The geometry of the precipitate is similar to that observed in the FIM image in Figure 1, and the three dimensional morphology and orientation of the precipitate can be clearly identified in the plan view and cross-sectional images. The overall composition of this precipitate is 77.5±1at% Al, 21.9±2at% Cu, 0.2±0.2at% Mg and 0.4±0.2at% Ag. The magnesium and silver contents of the θ' precipitates are not in excess of the alloy composition, and the θ' precipitates have a composition approaching $CuAl_2$. Figure 2(b) presents a cross-sectional POSAP solute distribution map from this precipitate. There is no evidence of magnesium or silver segregation to the coherent precipitate/matrix interfaces.

Figure 3 presents POSAP data from an Ω precipitate, with Figures 3(a), 3(b) and 4(c) showing the plan view distribution maps of copper, magnesium and silver. The precipitate geometry is similar to that observed in the FIM image shown in Figure 1. The overall composition of the Ω precipitate is 85±1at% Al, 13±2at% Cu, 1±0.2at% Mg and 1±0.2at% Ag. Figure 4 presents a cross-sectional POSAP solute distribution map from this precipitate. No evidence of segregation of magnesium or silver to the coherent precipitate/matrix interfaces was found.

In this study we have investigated the composition and morphology of some thirty θ' and Ω precipitates. In all cases the precipitate compositions fell in a narrow range around the figures quoted above. The average composition of the θ' precipitates was 77.8±1at% Al, 21.3±5at% Cu, 0.4±0.2at% Mg and 0.5±0.2at% Ag while the average composition for the Ω particles was 85.2±1at% Al, 12.3±3at% Cu, 1.4±0.5at% Mg and 1.1±0.3at% Ag. We therefore conclude that these compositions can be taken to be characteristic of these two precipitate types at this stage of annealing in this alloy.

4 - DISCUSSION.

The present work has shown that θ' and Ω precipitates can co-exist in an alloy of composition Al 4% Cu, 0.3% Mg, 0.4%Ag by weight after ageing at 170°C for 24 hours. The compositional information obtained from POSAP analysis of θ' precipitates in this alloy shows no partitioning of silver or magnesium, which is in agreement with EDX studies (15). The composition as determined by POSAP analyses is approaching $CuAl_2$, as expected (17, 18).

The composition of the Ω phase is of importance, and as discussed previously, poorly characterised. This study has found the composition of these precipitates to be 85at% Al, 13at% Cu, 1at% Mg and 1at% Ag, showing that they are richer in magnesium and silver than the θ' precipitates, but contain less copper. The copper content as measured in the present study is less that the 28at% reported by Muddle and Polmear (15). Their results were obtained using EDX techniques, and it is difficult to quantitatively determine precipitate compositions using conventional EDX techniques. The values for the magnesium and silver concentrations represent the first quantitative measurements of these solute contents in Ω precipitates. No conclusive evidence for magnesium or silver-rich precursor zones to the Ω precipitates was found.

The question of interfacial segregation of magnesium and silver with respect to θ' and Ω precipitates still remains. Muddle and Polmear (15) showed that silver may segregate to the interface between the Ω precipitate and the matrix. In the present work, no evidence for segregation of magnesium or silver to interfaces of θ' and Ω precipitates was identified. It is important to note, however, that the POSAP data shown relate only to the coherent interfaces of the precipitates. Work is presently underway to evaluate the segregation at the semi-coherent interfaces. The POSAP provides a unique opportunity to study such problems since three-dimensional data are readily obtained.

5 - CONCLUSIONS.

The POSAP has shown to be a valuable tool in the analysis of θ' and Ω precipitates in an Al-Cu-Mg-Ag alloy by providing three-dimensional atomic scale compositional information. The θ' precipitates were found to be close to

$CuAl_2$ with no significant enhancement of magnesium or silver content. The Ω precipitates contained less copper and much higher magnesium (1at%) and silver contents (1at%) than the θ' phase. The magnesium and copper were evenly distributed in the precipitates; no segregation to the coherent matrix/precipitate interfaces or conclusive evidence for solute rich precursor zones was found.

REFERENCES.

(1) I. J. Polmear, Trans. Met. Soc. 230 (1964) 1331.
(2) J. T. Vietz and I. J. Polmear, J. Inst. Metals 94 (1966) 410.
(3) J. H. Auld and J. T. Vietz in "The Mechanism of Phase Transformations in Crystalline Solids", Monograph and Report Series No. 33, Institute of Metals London (1969) 77.
(4) R. J. Chester and I. J. Polmear in "Metallurgy of Light Alloys", Institute of Metals London (1983) 75.
(5) I. J. Polmear and M. J. Couper, Met. Trans. 19A (1988) 1027.
(6) C. Laird and H. I. Aaronson, Trans. Met. Soc. AIME 242 (1968) 1393.
(7) C. Laird and H. I. Aaronson, Acta Metall. 14 (1966) 171.
(8) K. M Knowles and W. M. Stobbs, in press Acta Cryst. B.
(9) J. H. Auld, Mat. Sci. and Technology 2 (1986) 784.
(10) J. H. Auld, Acta Cryst. A28 suppl. (1972) S98.
(11) J. A. Taylor, B. A. Parker and I. J. Polmear, Met. Sci. 12 (1978) 478.
(12) S. McK. Cousland and G. R. Tate, J. Appl. Cryst. 19 (1986) 174.
(13) S.R. Ortner, C. R. M. Grovenor and B. A. Shollock, Scripta Metall. 22 (1988) 839.
(14) K. Hono, T. Hashizume, Y. Hasegawa, K. Hirano and T. Sakurai, Scripta Metall. 20 (1986) 487.
(15) B. C. Muddle and I. J. Polmear, Acta Metall. 37 (1989) 777.
(16) E. D. Boyes, A. R. Waugh, P. J. Turner, P. F. Mills and M. J. Southon, Int. Phys. Conf. Ser. No. 36 (1977) 343.
(17) G. C. Weatherly, Acta Metall. (1970) 15.
(18) L. F. Mondolfo, "Aluminium Alloys: Structure and Properties", Butterworths London (1979).

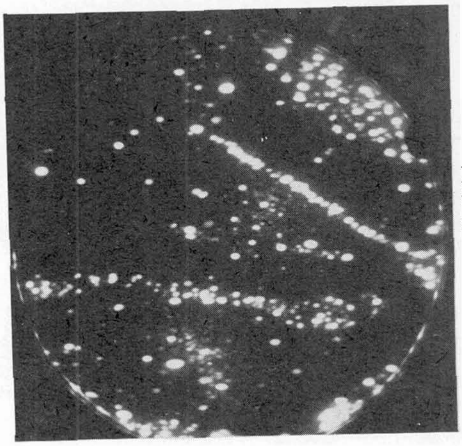

Figure 1: FIM micrograph of aged Al-Cu-Mg-Ag alloy taken in argon imaging gas at about 60K. Brightly imaging θ' and Ω precipitates can be seen.

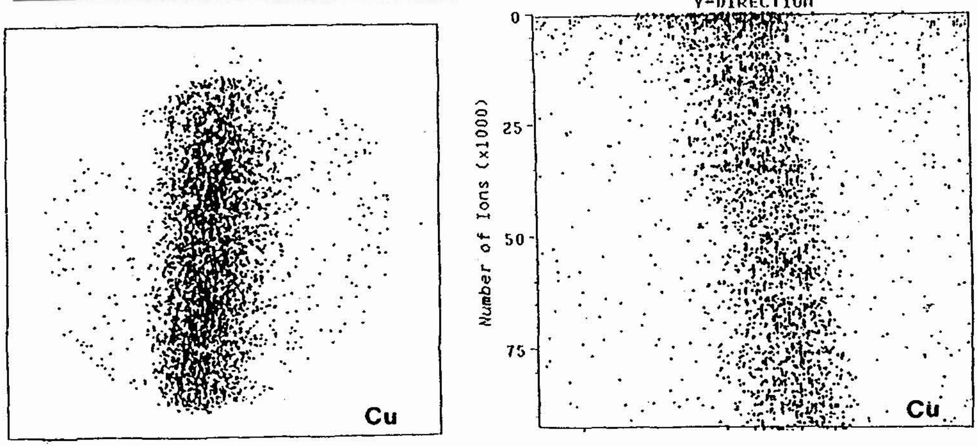

Figure 2: POSAP Cu distribution map from a θ' precipitate in an Al-Cu-Mg-Ag alloy. (a) plan view and (b) cross-sectional maps from the same precipitate.

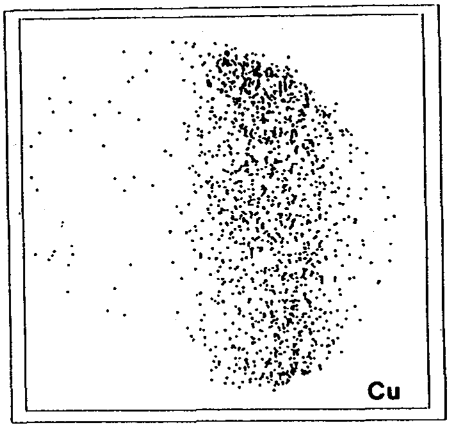

Figure 3a: POSAP Cu distribution map from an Ω precipitate. The diameter of the image is approximately 20nm.

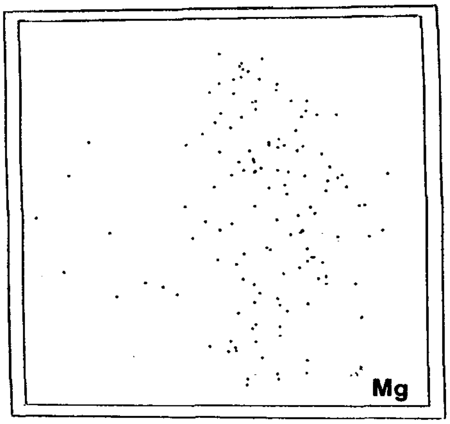

Figure 3b: POSAP Mg distribution map from an Ω precipitate. The diameter of the image is approximately 20nm.

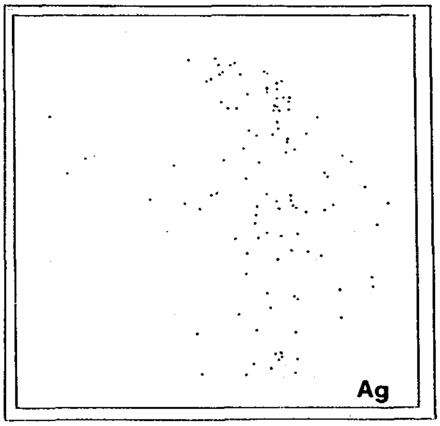

Figure 3c: POSAP Ag distribution map from an Ω precipitate. The diameter of the image is approximately 20nm.

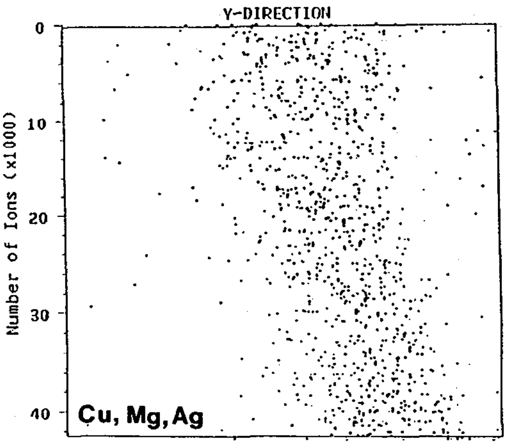

Figure 4: Cross-sectional POSAP solute distribution map from the Ω precipitate shown in Figure 3.