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The Influence of 0.5 wt % Ag on Defect Structures in an Al/3.2 wt % Cu/1.5 wt % Mg Alloy

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A quantitative study has been made by electron microscopy of the defect structures in an Al/3.3 wt % Cu/1.6 wt % Mg alloy. The main defects observed in the quenched and lightly aged alloy were prismatic dislocation loops, while in an alloy of similar copper and magnesium contents, but containing also 0.5 wt % Ag, fewer defects were observed, these being mainly dislocation helices. In the quenched condition the average loop and helix diameters increased with increase in quenching bath temperature, and on ageing the loop and helix size increased with ageing time. These observations have been interpreted on the basis of silver increasing the solute/vacancy interaction, possibly with the formation of complex clusters of Cu, Mg and Ag atoms with vacancies.

1. Introduction

The defect structures of quenched binary aluminium alloys have been widely studied by electron microscopy to determine quantitative features such as the numbers and sizes of dislocation loops and the concentration of vacancies, and hence to determine the factors controlling the structures, e.g. [1-3]; however, relatively little work of this type appears to have been done on more complex systems, such as high strength alloys containing several alloy elements, e.g. Al/Cu/Mg, Al/Cu/Mg/Si. The solute atom/vacancy binding energy is an important factor in relation to the behaviour of vacancies on quenching; if several types of solute atom are present there is the possibility of complex interactions between these and the vacancies. The quenched-in vacancy concentration can influence the kinetics of zone formation on ageing; in addition, the defects such as dislocation loops and helices formed by vacancy condensation can affect the kinetics of precipitate nucleation, e.g. of S' phase nucleation in the Al/Cu/Mg system. It has been shown that the quenched-in defect structure of Al/Cu/Mg alloys is significantly modified by the additions of small amounts of Si [4] or Ag [5], but quantitative observations are lacking. The present paper concerns the effect of the addition of 0.5 wt % Ag on an Al/3.2 wt % Cu/1.5 wt % Mg alloy; electron microscopical observations have been made on structures produced by various quenching rates, and also on subsequently aged structures.

2. Experimental Procedure

A ternary alloy of Al/3.3 wt % Cu/1.6 wt % Mg and a quaternary alloy of Al/3.2 wt % Cu/1.5 wt % Mg/0.5 wt % Ag were prepared from high purity materials by the British Aluminium Company Ltd.* The silicon and iron contents of the alloys were each $\sim$0.005 wt %.

The alloys were semi-continuously cast as $2\frac{5}{8}$ in. (1.0 in. = 2.5 cm) diameter billets, homogenised, hot-extruded and cold-rolled with inter stage annealing to 0.02 in. thick strip. Strip specimens were solution treated in a salt bath for 16 h at 500 $\pm$ 5° C for the ternary alloy and 495 $\pm$ 5° C for the silver-containing alloy. The standard quenching treatment was into water at 20° C, the specimens being transferred manually to the quenching bath. Quenching treatments into boiling water, and into a mixture of liquid nitrogen and petroleum ether at -100° C were also used. Ageing was done at

*Address: Research Laboratories, Chalfont Park, Gerrards Cross, Bucks, UK room temperature and also, using an oil bath, at 130, 160 and 190° C. Thin foils were prepared from 0.02 in. thick discs in a PTFE holder [6] using an acetic/phosphoric/nitric acid/water solution at 0 ± 5° C.

The foils used were generally of (001) orientation. Measurements of numbers of loops, and of dislocation loop and helix diameters were made from micrographs from duplicate specimens. In estimating the number of loops/cm^3 and the concentration of "precipitated" vacancies, a foil thickness of 2000 Å was assumed, and no corrections were made for invisibility of loops in certain contrast conditions or for loss of loops to the foil surface. Although as-quenched specimens were examined as soon as possible after quenching (i.e. within about 1 h), they were in effect lightly aged, since some hardening is observed within this time at room temperature.

3. Results

In both quenched and lightly aged samples of the ternary alloy the main defects were prismatic dislocation loops [7, 8], $a/2\langle 110\rangle\{110\}$ e.g. fig. 1; these were usually circular, but were sometimes diamond or hexagonal shaped. There were a few sessile loops and helical dislocations present. Zones free of loops were present adjacent to grain boundaries.

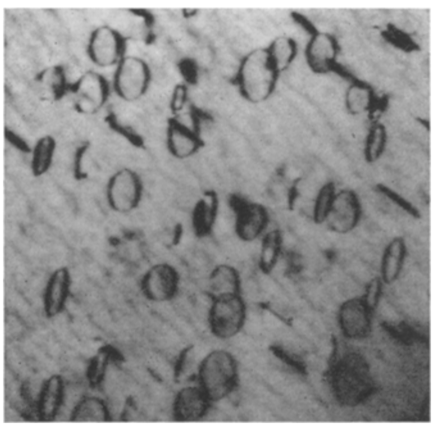

Figure 1 Al/3.3 wt % Cu/1.6 wt % Mg alloy quenched to 100° C (001) foil (×50000).

The average loop diameter in the as-quenched structure increased slightly as the quenching bath temperature was increased from −100° C to +100° C (table I and fig. 2); also the diameter increased with ageing time on ageing at 130, 160, and 190° C, the increase occurring more rapidly with increasing ageing temperature. The average number of loops was hardly changed by changing the severity of the quench, but a significant decrease in the numbers occurred as the ageing time increased. The concentration of "precipitated" vacancies increased only slightly as the bath temperature was raised, while it increased by a factor of 5 to 10 times as ageing time increased. There was evidence of loop nucleation occurring during ageing, in that, after 7 days ageing at room temperature small loops were present, about 500 Å in diameter, which were not observed in as-quenched samples. Similar loops, about 125 Å in diameter were observed in the silver-containing alloy (fig. 3).

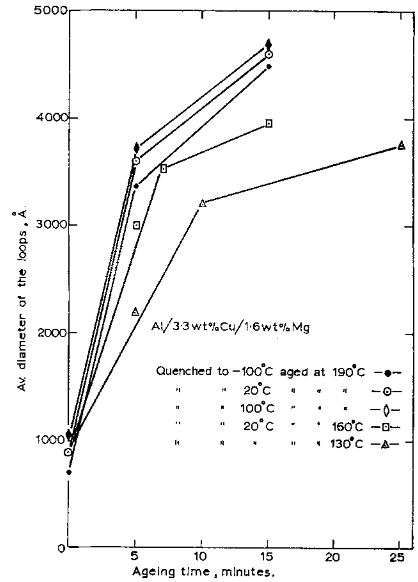

Figure 2 Al/3.3 wt % Cu/1.6 wt % Mg alloy. Average diameter of dislocation loops after various quenching and ageing treatments.

In the silver-containing alloy, the total numbers of defects were much less than in the ternary alloy, and those present were predominantly dislocation helices; occasionally prismatic loops were seen. Also, there were some relatively straight dislocations in the as-quenched samples which, on ageing for 7 days at room temperature, tended to transform to helices by climb.

Because the distribution of helices was non-uniform, vacancy concentrations were not calculated, but data for average helix diameter are shown in fig. 4. In the as-quenched condition the diameter increased with increase in quenching bath temperature, the effect being particularly marked when the temperature was changed

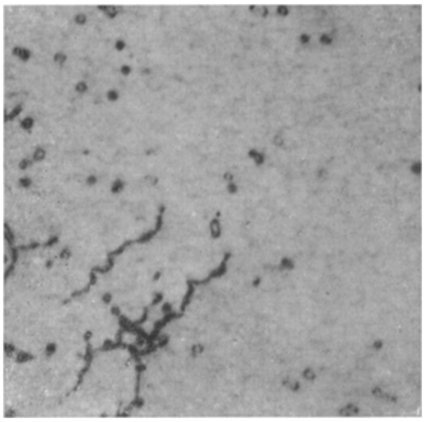

Figure 3 Al/3.2 wt % Cu/1.5 wt % Mg/0.5 wt % Ag alloy, quenched to 20° C and aged for 7 days at room temperature (×80000).

from 20 to 100° C. On ageing at 130, 160, and 190° C the diameter increased with ageing time, reaching an approximately constant value after a certain time; the initial rate of increase in diameter became greater with increasing ageing temperature.

4. Discussion

The magnitude of the binding energy, E_B, between solute atoms and vacancies can strongly influence the nature of the defect structure in quenched aluminium alloys [2]. The vacancy mobility decreases as the binding energy increases and the present results can be interpreted on this basis, and suggest that the addition of silver to the Al/Cu/Mg system increases the solute atom/vacancy interaction.

In the ternary alloy, the mobility of vacancies is sufficiently high for dislocation loops to nucleate and grow during quenching. The value of $5 \times 10^{13}/cm^3$ for the density of loops is in reasonable agreement with values of 10^{13} to $10^{16}/cm^3$ reported for aluminium and its dilute alloys [9]. As previously reported for an Al/1.2 wt % Si alloy [2], the density of loops varies only slightly with the severity of quench (table I), indicating that loop nucleation is not much affected by quenching rate within the range studied. However, with increasing quenching bath temperature the diffusion time is increased, so that the loop size is increased, and the fraction of vacancies precipitated is increased.

The increase of average loop size during ageing at 130 to 190° C also depends on vacancy mobility, while cluster and zone formation will involve vacancy-assisted diffusion of Cu and Mg atoms. The sum of the atomic diameter of Cu (2.556 Å) and of Mg (3.196 Å) is approximately double the atomic diameter of Al (2.862 Å), so that the association of Cu and Mg atoms in a cluster should reduce the lattice strain and result in the release of vacancies from the cluster.

In the silver-containing alloy, the scarcity of dislocation loops, and the relatively small

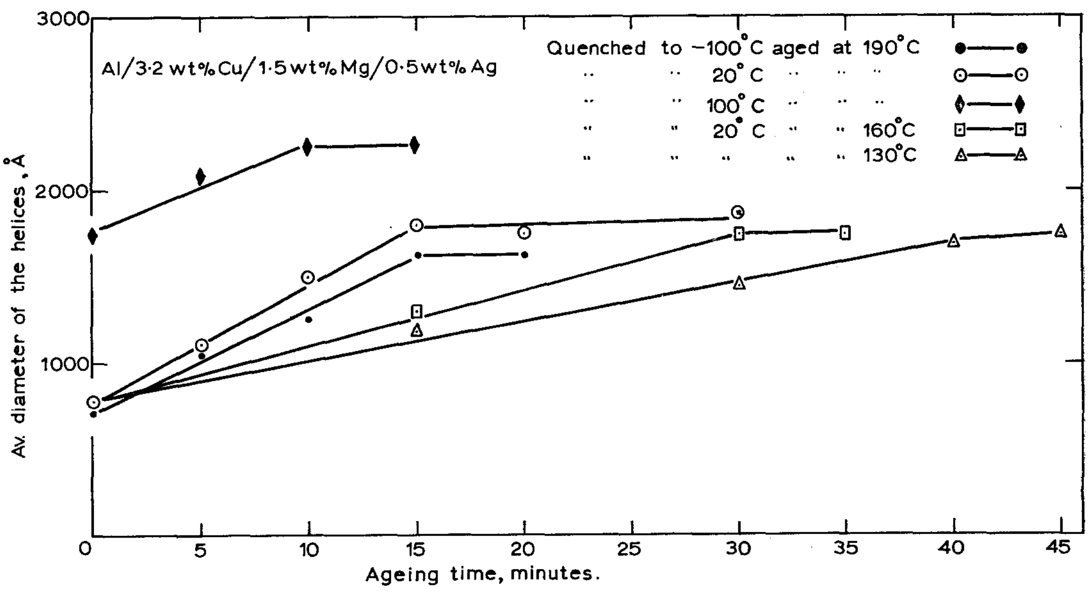

Figure 4 Al/3.2 wt % Cu/1.5 wt % Mg/0.5 wt % Ag alloy. Average diameter of dislocation helices after various quenching and ageing treatments.

TABLE I Al/3.3 wt % Cu/1.6 wt % Mg alloys

Quenching temp. (° C)	Ageing temp. (° C)	Time (min) at ageing temp.	Average diameter of loop (Å)	Average number of loops ($cm^{-3} \times 10^{-13}$)	Vacancy concentration (precipitated) ($\times 10^{-4}$)
−100	As-quenched	—	700	5.2	0.3
−100	190	5	3360	2.3	2.9
−100	190	15	4500	1.5	3.5
20	As-quenched	—	900	5.0	0.5
20	190	5	3600	2.1	3.0
20	190	15	4600	1.5	3.5
100	As-quenched	—	1040	4.9	0.6
100	190	5	3720	2.0	3.1
100	190	15	4680	1.4	3.5
20	As-quenched	—	900	5.0	0.5
20	160	5	3000	2.6	2.6
20	160	7	3560	2.2	3.1
20	160	15	3960	1.9	3.3
20	As-quenched	—	900	5.0	0.5
20	130	5	2200	3.2	1.7
20	130	10	3200	2.2	2.5
20	130	25	3750	2.0	3.1

numbers of helices indicate that a much greater fraction of vacancies is retained in solution than in the ternary alloy. A similar effect results from the addition of Si to Al/Cu/Mg alloys [4]. The behaviour suggests a lower vacancy mobility and stronger solute atom/vacancy binding effects. Further support for this interpretation is provided by the low rate of helix growth during ageing, and the fact that the loops that appear on ageing for 7 days at room temperature are much smaller in the silver-containing alloy than in the ternary alloy.

Data on E_B determined in binary or ternary alloys, indicate that the value for silver is 0.08 eV [10] and that for copper is ~0.2 eV [11]. In the case of Mg there is some controversy, and values of E_B ranging between 0.01 to 0.05 eV have been reported [3, 12-14]. It appears likely that the strong interaction effect in the Al/Cu/Mg/Ag alloy is not attributable simply to silver atom/vacancy binding, but could involve a complex cluster of vacancies with atoms of copper, magnesium and silver. The effects of complex clusters on zone formation have been discussed for other systems, e.g. Al/Mg [8] and Al/Mg/Si [15].

The increase of helix diameter in the quaternary alloy brought about by reducing the severity of the quench is attributable to the greater diffusion time and also to the reduction in quenching stresses which leads to a reduction in the number of dislocations introduced by quenching.

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References

1. G. THOMAS, *Phil. Mag.* **4** (1959) 1213.
2. K. H. WESTMACOTT, R. S. BARNES, D. HULL, and R. E. SMALLMAN, *ibid* **6** (1961) 929.
3. S. KRITZINGER, P. S. DOBSON, and R. E. SMALLMAN, *ibid* **16** (1967) 217.
4. R. N. WILSON, D. M. MOORE, and P. J. R. FORSYTH, *J. Inst. Metals* **95** (1967) 177.
5. J. T. VIETZ and I. J. POLMEAR, *ibid* **94** (1966) 410.
6. G. W. BRIERS, D. W. DAWE, M. A. P. DEWEY, and I. S. BRAMMAR, *ibid* **93** (1964-65) 77.
7. D. VAUGHAN, unpublished work.
8. R. E. SMALLMAN and A. EIKUM, "Lattice Defects in Metals," edited by R. M. J. Cotterill *et al* (Academic Press, New York, 1965) p. 592.
9. A. KELLY and R. B. NICHOLSON, *Prog. Matls. Sci.* **10** (1963) 189.
10. D. R. BEAMAN, R. W. BALLUFFI, and R. O. SIMONS, *Phys. Rev.* **134** (1964) A532.

11. H. KIMURA, A. KIMURA, and R. R. HASIGUTI, *Acta Met.* **10** (1962) 607.
12. J. TAKAMURA, K. OKAZAKI, and I. G. GREENFIELD, *J. Phys. Soc. Japan* **18** *Suppl. III* (1963) 78.
13. A. EIKUM and G. THOMAS, *ibid* p. 98.
14. K. N. ENTWISTLE, J. H. FELL, and KANG IL KOO, *J. Inst. Metals* **91** (1962-63) 84.
15. D. W. PASHLEY, J. W. RHODES, and A. SENDOREK, *ibid* **94** (1966) 48.