Vacancy enhancement of µ-phase precipitation in Ag–10 at % AI alloy

M. R. NAGY, G. H. DEAF, R. KAMEL*

Physics Department, Faculty of Education, Ain Shams University, Cairo, Egypt

Locally prepared and homogenized Ag–10 at % Al wires were quenched from 400° C to room temperature followed by different ageing treatments at temperatures in the precipitation zone to obtain samples with different amounts of μ -phase precipitation. Room temperature steady state creep and microhardness tests were performed in order to assess the degree of precipitation in the samples. Increased precipitation of the μ -phase was found to decrease the strain rate and to increase the hardness. The precipitation process was possibly enhanced by the quenched vacancies. X-ray analysis after creep fracture revealed a remarkable drop in the integral intensities, indicating the destruction of the ordered structure of the μ -phase, i.e. partial redissolution of it.

1. Introduction

Ag-10 at % Al alloy is characterized by the formation of the precipitation hardening compound Ag₃Al, which has an ordered cubic structure below a temperature of 250° C [1-3].

Hardening in Ag–Al binary alloy arises from several interactions [4-10]: such as (1) elastic or Cottrel interaction, (2) electronic interaction, (3) short- and long-range order interaction, and (4) chemical or Suzuki interaction. These interactions play an important role in creep resistance and hardening of the Ag–Al alloy. The steady state creep of Ag–Al alloy [11-13] is affected by particle size, distribution, volume fraction, mass fraction of the second phase, and coherency stresses between particles and matrix.

It is known that when a dislocation is moved during creep deformation through the precipitated lattice at low temperature, it destroys the local order of the precipitates across the dislocation slip plane.

The aim of the present work is to study the effect of the growth of the ordered μ -phase in Ag-10at% Al alloy on its microhardness and steady creep characterization.

2. Experimental technique

Locally prepared Ag-10at% Al alloy was cold-

drawn to wires of 0.4 mm in diameter. Samples were divided into two groups.

The first group was annealed under vacuum at 400° C for 3 h. The samples were then quenched to room temperature to produce a short-range α single phase. The short-range ordered samples were aged for different times (5, 15, 30, 45, 60, 90 and 120 min) at different ageing temperatures (85, 100, 120, 150, 160, 170, and 190° C), in order to obtain samples having different fractions of ordered cubic μ -precipitation phase exhibiting a spherical morphology.

The second group was also annealed for 3 h under vacuum at 400° C followed by slow cooling to obtain completely precipitated ordered samples. The previous pre-heat treatment was carried out before performing the microhardness and creep tests at room temperature.

A D 500 X-ray diffractometer (Siemens) was used to investigate the structural variations in samples, quenched and aged at 120° C for the ageing times given above, and in slowly cooled samples before and after creep tests.

3. Experimental results

Microhardness and creep tests were performed at room temperature, immediately after each isothermal ageing pulse. Results of Vickers hard-

*Physics Department, Faculty of Science, Cairo University, Egypt.



Figure 1 Effect of μ -phase precipitation on hardness behaviour of Ag-10 at % Al alloy. The precipitation temperature is given for each curve.

ness number (VHN) observations are shown in Fig. 1, which shows that the hardness decreased initially with ageing time, and then increased to a peak value at an ageing time of about 90 min. The peak height was greatly affected by changing the ageing temperature but its position on the ageing-time axis was constant.

Creep tests were made on slowly cooled samples and on quenched samples, Fig. 2. The creep runs were also made on previously aged samples under a constant stress (29.5 kg mm⁻²) at RT.



Figure 2 Creep curves taken at RT under a constant applied stress ($\sigma = 29.5 \text{ kg mm}^{-2}$) for slowly cooled and quenched Ag-10 at % Al alloy.

The temperature dependence of steady creep rate at any precipitation time is shown in Fig. 3. The initial decrease in the steady state strain rate was achieved at an ageing temperature in the range 85 to 190° C. Increasing the precipitation temperature decreased the strain rate, $\dot{e}_{\rm st}$, to a minimum value by increasing the precipitation time. The formation of the ordered precipitated phase in this alloy hardened the samples and the steady creep rate subsequently diminished or even stopped.

The dependence of precipitation parameter, X on isothermal precipitation time is shown in Fig. 4, for samples having different mass fractions of ordered μ -precipitation phase. This parameter is given by $X = [(\dot{\epsilon}_0 - \dot{\epsilon}_t)/\dot{\epsilon}_0]_{T=\text{constant}}$ where $\dot{\epsilon}_0$ and $\dot{\epsilon}_t$ are the steady creep rates at isothermal precipitation time zero and at t, respectively, T being the precipitation temperature. X increased appreciably with increase in both the precipitation time and temperature. The increase in the mass fraction of the μ -phase, X, was more pronounced until the isothermal precipitation time reached 90 min, and after further ageing it remained constant.

The nature of the experimental results of VHN led to the use of the Johnson-Mehl-Avrami (JMA) equation [14, 15]. The creep, microhardness and X-ray results have been correlated by assuming that the change of creep rates, VHN and



Figure 3 The effect of precipitation temperature on the steady state creep rate at RT for different precipitation times.

X-ray integral intensities are directly proportional to the mass fraction of μ -precipitates. The equilibrium mass fraction of μ -phase in the matrix at different precipitation times and temperatures is shown in Fig. 5.

The lattice parameter, X-ray integral intensity and line width of the ordered cubic μ -precipitation phase increased from 0.407 to 0.409 nm and from 0 to 600 mm² and from 5 to 14 mm, respectively, with increase in the precipitation time from 0 to



Figure 4 The relationship between the mass fraction of μ -precipitates, X, is deduced from creep tests and the precipitation time for different precipitation temperatures.

120 min at an ageing temperature 120° C , as shown in Fig. 6.

After creep deformation was performed on aged samples at 120° C, the strong intensity of filtered X-ray diffracted lines having indices (111, 200, 220, 311, 222) greatly diminished. An associated sharp decrease in X-ray integral intensity, in lattice parameter, and in the X-ray line width of the crept samples was also observed with the increase in strain rate, Fig. 7.

4. Discussion

The microhardness and creep rate of samples with μ -precipitates depended mainly on the interaction between the lattice defects and these precipitates. This relatively high interaction may be responsible for the increase in the microhardness, and would therefore reduce the creep rate according to the size, distribution and mass fraction of the μ precipitates (Ag₃Al) in the quenched and in the annealed samples. For partially precipitated samples (aged samples), the observed increase in Vickers hardness number might be due to partial precipitation of ordered μ -phase. The initial decrease in microhardness might be attributed to annihilation of quenched vacancies, after which the μ -precipitation centres enrich the silvermatrix so that the Vickers hardness increases to a maximum value. The samples' hardness reached a peak at a precipitation time of about 90 min, corresponding to some equilibrium mass fraction of ordered μ -precipitates in the Ag-10 at % Al



Figure 5 The time and temperature dependences of the mass fractions of μ -precipitates are deduced from X-ray, creep and Vickers microhardness tests, respectively.

alloy at temperatures in the precipitation zone. On the other hand, the subsequent drop in the samples' hardness after 90 min (annealing time) might be attributed to the formation of larger and widely spaced spherical μ -precipitates.

The results of creep tests on these samples imply that the observed general decrease in the steady creep rate may be due to an increase in the volume and/or the mass fraction of the ordered μ -precipitates with the increase in the ageing time or ageing temperature. The steady creep rate decreased markedly as the precipitation time increased, but it approached its minimum steady value after a period of about 90 min.

Because of these results, it was the intention to test changes in the mass fraction of μ -precipitates of quenched samples after different ageing times ranging from 0 to 120 min. It was found that the differences in the mass fraction of μ -precipitates, at a precipitation time of 90 min, were due to the



Figure 6 Relation between precipitation time and (a) lattice parameter, (b) relative change in lattice parameter, (c) X-ray integral intensity, and (d) X-ray line width, for quenched samples aged at 120° C.



differences in the precipitation temperatures. The high value of X was attributed to the formation of fully ordered μ -precipitates with maximum concentration in the silver-matrix.

The observed peaks of microhardness and the lowest isothermal creep rates seemed to be due to enhancement of the precipitation process activated by the existing quenched vacancies.

The relationship between the measured lattice parameter, X-ray line width, and X-ray integral intensity were found to increase with increase in the precipitation time of μ -precipitates at an ageing temperature of 120° C. This behaviour was thought to be due to an increase in the mass fraction and the size of μ -precipitates in the test matrix until it became fully precipitated.

After creep deformation X-ray investigations showed that the integral intensity, line width and lattice parameter of ordered cubic μ -precipitates decreased with increase in the creep rates. This decrease was attributed to partial dissolution and to the associated destruction of the ordered μ -precipitate centres with increasing creep rate.



Figure 7 Relation between (a) lattice parameter, (b) X-ray integral intensity, (c) X-ray line width and strain rate for quenched samples were previously aged at 120° C and crept at RT.

References

- 1. T. B. MASSALSKI, in "Phase Transformation" (American Society for Metals, Metals Park, Ohio, 1970).
- 2. J. D. AYERS and T. B. MASSALSKI, Met. Trans. 13 (1972) 261.
- 3. M. KOGACHI, J. Phys. Chem. Solid 35 (1974) 109.
- V. S. POSTNIKOV, I. M. SHARSHAKOV and V. A. YEVSUKOV, Fiz. Metal. Metalloved. 32 (1971) 195.
- 5. Idem, 32 (1971) 431.
- 6. R. M. BONESTEEL and O. D. SHERBY, Acta. Metall. 14 (1966) 385.
- 7. M. PAHUTOVA, J. CADEK and P. RYS, *ibid.* 17 (1969) 745.
- 8. J. C. FISHER, *ibid.* 2 (1954) 9.
- 9. J. C. FISHER, in "Mechanical Behaviour of Materials at Elevated Temperature", edited by J. E. Dorn (McGraw Hill, New York, 1961) p. 45.
- 10. O. D. SHERBY, R. A. ANDERSON and J. E. DORN, *Trans. Met. AIME* 191 (1951) 643.
- 11. E. M. HOWARD, W. L. BARMORE, J. D. MOTE and J. E. DORN, *Trans. Met. AIME* **227** (1963) 1061.
- 12. H. R. PEIFFER, Trans. Met. AIME 224 (1962) 1247.
- 13. L. CAMBAL and E. B. HAWBOLT, Met. Trans. 4 (1973) pp. 1389, 1417.
- J. W. CHRISTIAN, "The Theory of Transformation in Metals and Alloys" (Pergamon Press, New York, 1965) p. 471.
- 15. R. W. CAHN, Acta Metall. 4 (1956) 449.

Received 18 February and accepted 19 May 1983