

Clusters in Low-Concentrated Al-Mg Alloy

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SYNOPSIS

The state above the solubility temperature of GP zones of Al-3mass%Mg alloy, which has a tendency for precipitation and preprecipitation at low temperature, was studied by resistivity measurement. Homogenization treatment at high temperature reduced Mg atoms in the surface layer. After quenching from 623K, the specimen was annealed sequentially at various temperatures above the GP zone solvus. The stationary resistivity obtained in annealing at a temperature was the same irrespective of the starting state and increased with decreasing annealing temperature. No precipitation was observed in the annealing. The results are not in favor of the segregation of Mg atoms to the dislocation loops but of the short range clustering.

1. INTRODUCTION

In the alloy in which heterogeneous precipitation takes place at low temperature, there is a preference for like neighbors above the solubility temperature. Rudman and Averbach⁽¹⁾ studied X-ray scattering of the Al-Zn and Al-Ag alloys above the solubility temperature and found the excess of like neighbors in these alloys. Al-Mg alloy, as important a base alloy for practical use as Al-Zn alloy, has not been studied enough because of the disadvantage for X-ray measurement due to the small difference in the atomic scattering factors be-

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tween Al and Mg atom. Electrical resistivity, on the other hand, is sensitive to the state of the alloy and has been used to investigate preprecipitation and defect in the alloy. Ohta et al.⁽²⁾ measured carefully the electrical resistivity of the Al-Zn alloys during annealing above the GP zone solvus temperature and revealed the increase of resistivity caused by the short range clustering. Osamura and Ogura⁽³⁾ measured also the resistivity change during isothermal annealing of several Al-Mg alloys and ascribed the decrease in resistivity above the GP zone solvus to the depletion of solute atoms in the matrix which was induced by the segregation of solute atoms to the dislocation loops. But the resistivity arrived at during the annealing was found to be lower as the annealing temperature was higher, which was similar to the trend observed for the short range clustering in Al-Zn alloy⁽²⁾. In this paper, resistivity change during annealing at several temperatures above GP zone solvus was measured to clarify the clustering tendency in the Al-3mass%Mg alloy.

2. EXPERIMENTAL PROCEDURES

Al-3mass%Mg alloy was prepared by melting 99.99% aluminum and 99.99% magnesium in air. The ingot, 15mm in diameter and 150mm in length, was homogenized for 180ks at 648K and hot-forged to a plate about 5mm in thickness. A strip, 0.4mm in thickness, was formed by cold rolling with intermediate annealing at 648K and the specimen for the resistometry, $0.4 \times 0.7 \text{ mm}^2$ in cross section and 100mm in length having four leads, was cut out of the strip. Specimens for the

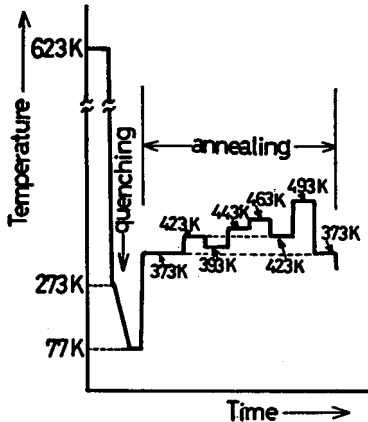


Fig.1 Schematic diagram of the heat treatment. Cooling to the liquid nitrogen temperature for every resistivity measurement is not shown.

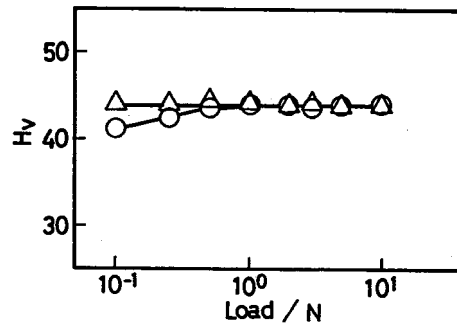


Fig.2 Dependence of the hardness on the load for the specimen held for 3.6ks at 773K, after the surface layer $10 \mu\text{m}$ (○) and $40 \mu\text{m}$ (△) thick was removed by electropolishing.

transmission electron microscopy (TEM) were prepared from the same strip by further rolling to 0.1mm in thickness. After the heat-treatment they were electropolished for TEM.

Quenching method was the same as previously reported.⁽⁴⁾ Annealing was carried out in an oil bath. The sequence of heat treatment is shown schematically in Fig.1. Only one specimen was used in this experiment to ensure the precise measurement of the resistivity change. Quenching procedure was limited to once to avoid missing of magnesium from the specimen surface during solutionization at high temperature⁽⁵⁾. The effect of holding the specimen at high temperature is shown in Fig.2. A specimen held for 3.6ks at 773K was hardness tested at various penetrating loads after electropolishing 10 μ m thickness to remove oxidized layer. The hardness decreased with decreasing load below 0.49N, while the same hardness number was obtained at 0.98N or more of load. This indicates the existence of soft surface layer, which may be due to the missing of solute magnesium from the surface. After the surface layer 40 μ m thick was removed, the load dependence of hardness was no longer observed.

Resistivity measurement was carried out by a conventional four-points method, the specimen being immersed in liquid nitrogen whose temperature was calibrated using a dummy specimen of the same alloy. TEM observation was carried out at 200kV with JEM-2000EX.

3. RESULTS AND DISCUSSION

Figs. 3 and 4 show the resistivity change during annealing at various temperatures shown in Fig.1. As quenched resistivity, 16.30n Ω m, decreased during annealing at 373K owing to the vacancy decay and arrived at a stationary

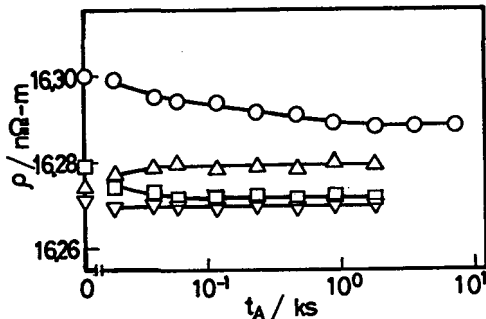


Fig.3 Resistivity change during annealing at various temperatures in the sequence shown in Fig.1. \circ 373K, \triangle 393K, \square 443K, ∇ 463K.

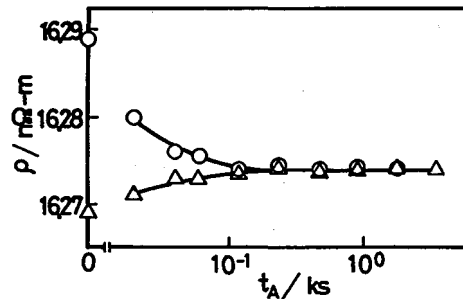


Fig.4 Resistivity change during annealing at 423K, after annealing at 373K (\circ) and after annealing at 463K (\triangle).

value, 16.289nΩm. In the subsequent annealing at 423K (o in Fig.4), resistivity decreased further and arrived at another stationary value, 16.274nΩm. In each annealing of the sequence its respective stationary resistivity was obtained. Fig.4 shows annealing curves at a temperature, 423K, but from different starting states, one after annealing at 463K and the other at 373K. The curves came to coincide with each other and gave a single stationary value of resistivity. The value obtained at 373K after annealing at 493K

also coincided with that obtained in annealing at 373K after the quench. From these results that the stationary value after annealing at a temperature was the same irrespective of the history of the heat treatment, the stationary value of resistivity can be considered characteristic of the annealing temperature.

The dependence of the stationary values on the annealing temperature was shown in Fig.5. Increasing resistivity with decreasing temperature is very similar to the result obtained in Al-Zn alloy⁽²⁾.

The precipitation, such as the segregation to the dislocation suggested by Osamura and Ogura⁽³⁾, would cause the decrease of resistivity with decreasing annealing temperature according to the depletion of more solute atoms from the matrix because the amount of precipitate should be larger at a lower temperature. No precipitates were observed by TEM in the specimen annealed for 3.6ks at 373K after quenching from 623K. A kind of reversibility shown in Fig.4 is also favorable for the clustering.

From the results and discussion above, it is concluded that the short range clustering exists and is responsible for the increase of resistivity at temperatures above the GP zone solvus.

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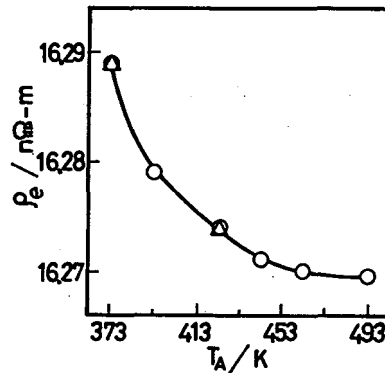


Fig.5 Dependence of the stationary value of resistivity on the annealing temperature.