PRECIPITATION BEHAVIOR IN HIGH PURITY AL-300 PPM FE ALLOY

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ABSTRACT Precipitation behaviors in high purity Al-300 ppm Fe alloy with and without pre-deformation were studied by resistivity measurement and transmission electron microscopy. Resistivity decreased in two stages with aging time in the not-deformed alloy, while it decreased in three stages in pre-deformed alloy. The first stage of resistivity in both alloys is attributed to the annihilation of quenched-in vacancies and the following stages are attributed to the precipitation of iron. TTP-diagrams in the not-deformed alloy indicates one C-curve, while that in the pre-deformed alloy indicates two C-curves. The lower curve is related to the precipitation of a metastable Al_6Fe, and the higher curve in the pre-deformed alloy and also the curve in not-deformed alloy is related to the precipitation of a stable Al_3Fe.

Keywords: *Al-Fe alloy, precipitation, Al_3Fe, Al_6Fe, resistivity measurement, TEM observation*

1. INTRODUCTION

Since iron is a common impurity in commercial aluminum alloys, effects of Fe on mechanical properties [1], recrystallization [2], texture [3], etc., have been extensively studied. However, iron contents in these studies are high in spite of low solid solubility of iron in aluminum, about 0.05 at%. Reports on precipitation phenomena in Al-Fe alloys having low iron contents, less than the solubility limit, are very few. Miki and Warlimont [4] reported that precipitation occurred more slowly and higher temperatures than other precipitates in aluminum alloys. Holm and Hornbogen [5] presented time-temperature-precipitation (TTP) curves consisting of one C-curve. Stickels and Bush [6] deduced the precipitation of a metastable phase from analyses of Mössbauer spectra.

In the present study, a high-purity Al-Fe alloy is used in order to avoid confusions due to impurities because the solubility limit of iron is comparable to impurity levels of other elements, and precipitation phenomena are investigated in detail.

2. EXPERIMENTAL PROCEDURES

The high-purity Al-300 mass ppm Fe alloy used was prepared using 99.999% Al and 99.99 % Fe. Chemical composition of the alloy is shown in Table 1.

Table 1 Chemical composition of the alloy (mass ppm).

Fe	Si	Cu	Mn	Mg	Cr	Ni	Zn	Ga	Ti	Al
299	2	1	1	0	0	1	1	1	0.3	bal.

Wires for resistivity measurements of no deformation specimens were prepared by drawing into 1.1 mm in diameter, solution heat treated at 913 K for 3.6 ks in argon atmosphere, quenched into iced water and then isothermally aged at various temperatures in the range of 393 to 848 K. While, wires for deformed specimens were drawn into 1.3, 1.8 and 2.0 mm in diameter, solution heat treated at the above mentioned condition, drawn again into 1.18 mm in diameter, which results in about 18, 38 and 65 % reduction in area, respectively, and then aged. The wires were cut into about 180 mm in length for resistivity measurements. Resistivity was measured dipping the specimen in liquid nitrogen. Specimens with deformation for TEM observations were deformed by cold rolling after solution heat treatment and then aged. TEM observations were carried out by JEM-200CX operating at 100 kV in accelerating voltage.

3. RESULTS

3.1 Not deformed alloy

Changes in resistivity in not-deformed specimens aged at various temperatures are shown in Fig. 1, in which ρ_0 and ρ are the resistivities at as quenched and aged conditions, respectively. Resistivity decreases with aging time at all the temperatures in this study. Small amount of decrease in resistivity occurs after beginning of aging (Stage I), which is followed by a large decrease (Stage II). Such a two-stage decrease occurred in the range of aging temperatures of 573 to 723 K. Decrease in the Stage I was not detected above 773 K, while the Stage II was not occurred below 523 K in the range of the present study.

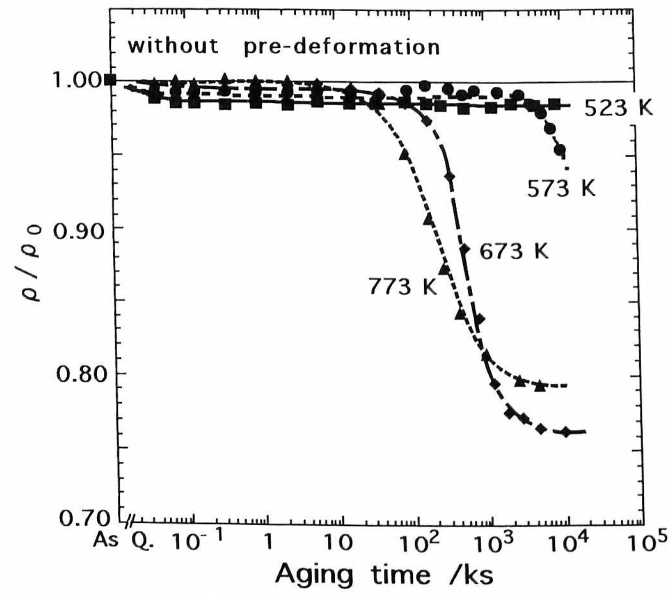

Fig. 1 Changes in resistivity in not-deformed alloy.

The specimen aged at 673 K for 10^4 ks, at which the Stage II had been completed, was examined by TEM. Precipitates were observed, an example of which is shown in Fig.2. Tilting experiments showed that the precipitate had a plate-like shape. Analysis of diffraction pattern (b) shows that the precipitate is a stable phase, Al_3Fe, which has a monoclinic structure with a=1.549, b=0.808, c=1.248 and β=107.72° [7]. The decrease in the Stage II is attributed to precipitation of Al_3Fe.

Time for start of the Stage II is determined in ρ/ρ_0-t curves at various temperatures and a TTP diagram is plotted as shown in Fig. 3, which forms a C-curve with the nose temperature of about 800 K.

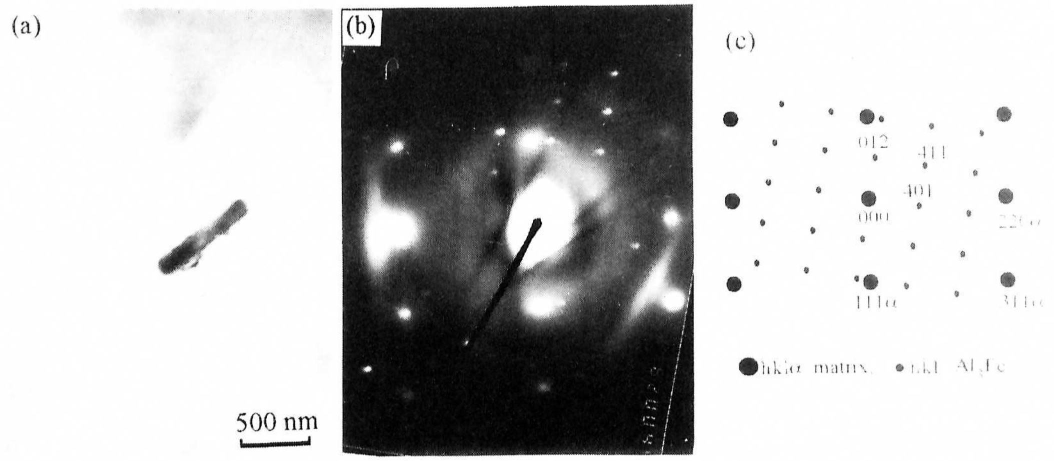

Fig. 2 Precipitate in the not-deformed alloy aged at 673 K for 10^4 ks. (a) bright field image, (b) SADP and (c) key diagram for (b).

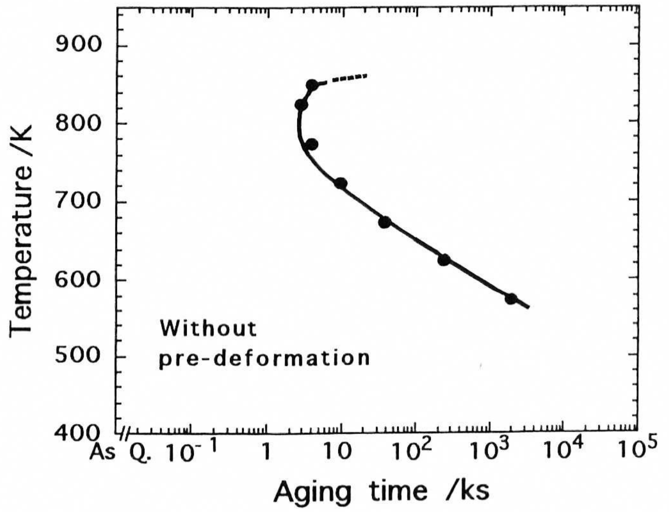

Fig. 3 TTP-curve for the alloy without pre-deformation.

3.2 Pre-deformed alloy

Changes in resistivity of the specimens pre-deformed at 65 % and aged at various temperatures are shown in Fig. 4. Resistivity decreases with aging like as the not-deformed alloy. However, the time for start of decrease became short compared with that in the not-deformed alloy aged at the same temperature (Fig.1). Moreover, three-stage decrease can be seen in the curve of 673 K aging as indicated in the figure. Decrease of the Stage III can bee also seen in the curve of 698 K aging as indicated by the arrow. The time for beginning the Stage III at 698 K aging became short. At the temperatures ranging 648 to 698 K, three stages were conspicuous on the ρ/ρ_0-t curves. Above this temperature range, resistivity decreases in two stage again.

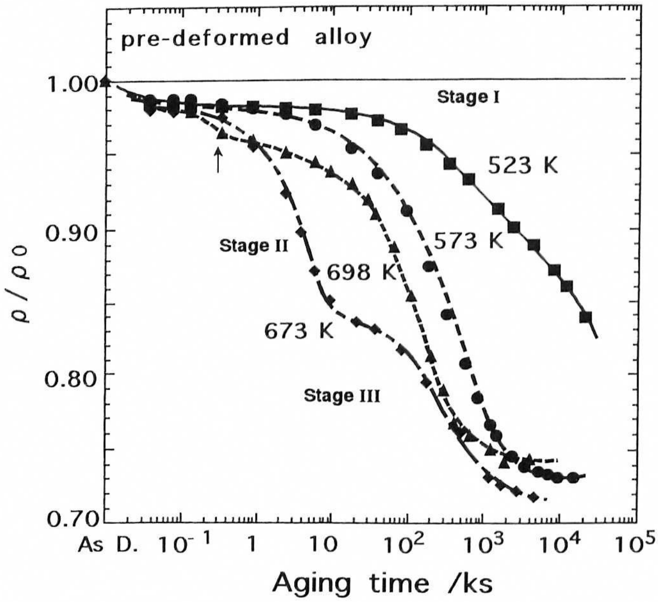

Fig. 4 Changes in resistivity in pre-deformed alloy.

Time for start the Stage II were determined on ρ/ρ_0-t curves at various temperatures and TTP-curve was plotted as shown in Fig. 5. Two C-curves can be drawn, nose temperatures of which are about 680 and 800 K. The C-curve for the not-deformed alloy (Fig. 3) is also shown in Fig. 5 by the dotted line, which coincides with the upper C-curve of the pre-deformed alloy.

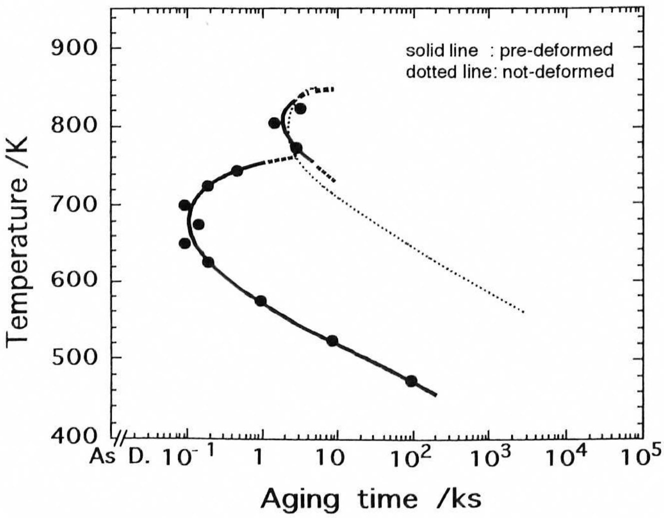

Fig. 5 TTP-curves for pre-deformed alloy (solid curves) and not-deformed alloy (dotted line).

Precipitates in the specimens aged at 673 K for 10 ks and 803 K for 4×10^2 ks, which correspond to the ends of the Stage II and III, were shown in Fig. 6 and 7, respectively. The SADP in Fig. 6 (b) is not interpreted as Al_3Fe but can indexed as Al_6Fe which is a metastable phase having an orthorhombic crystal structure with a=0.6464, b=0.7440 and c=0.8779 nm [8]. While, the SADP in Fig. 7 (b) is indexed as Al_3Fe like (c).

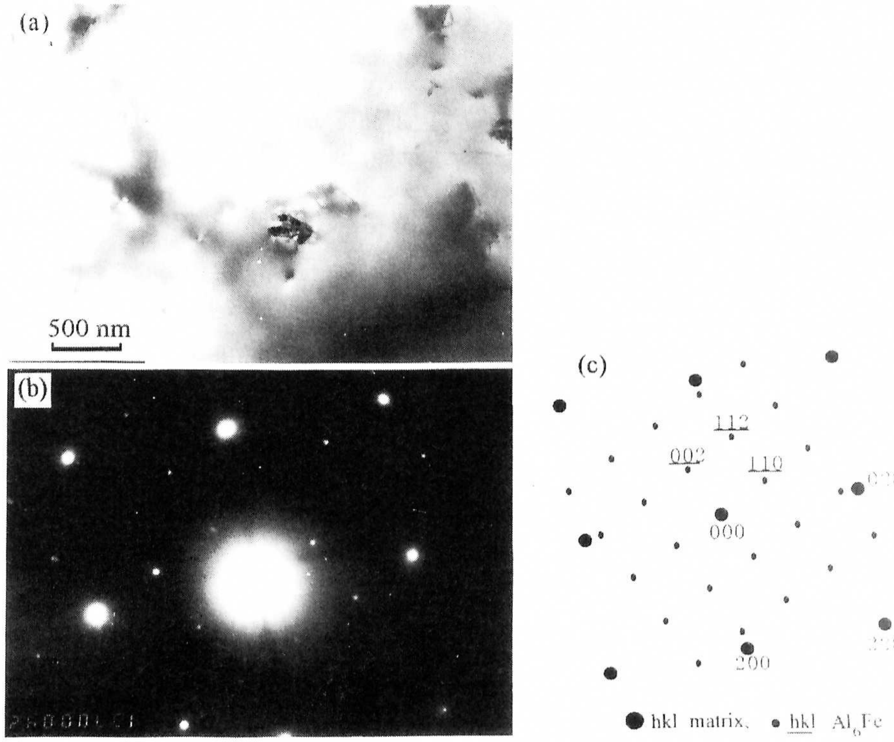

Fig. 6 Precipitate in the pre-deformed alloy aged at 673 K for 10 ks. (a) bright field image, (b) SADP and (c) key diagram for (b).

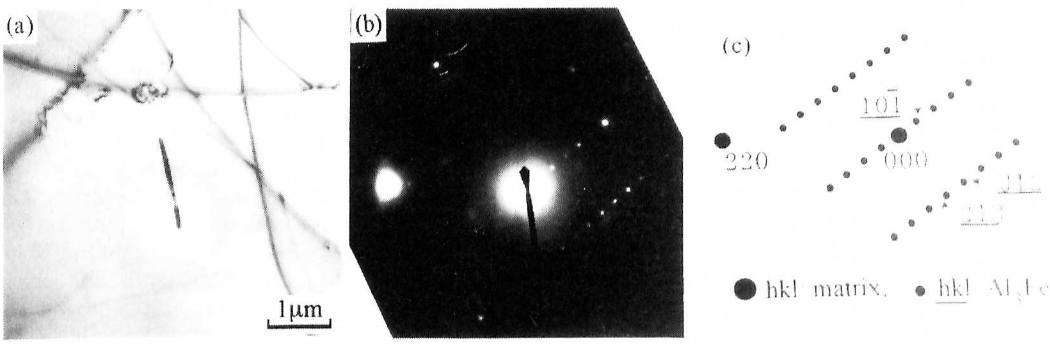

Fig. 7 Precipitate in the pre-deformed alloy aged at 803 K for 4×10^2 ks. (a) bright field image, (b) SADP and (c) key diagram for (b).

4. DISCUSSION

The amount of decrease in ρ/ρ_0 in the Stage I, about 0.02 % is comparable to that in an Al-Ni alloy reported by the same authors [9], in which the decrease is attributed to the annihilation of quenched-in vacancies. Disappearance of the Stage I in higher aging temperature is due to low densities of quenched-in vacancies because the aging temperature is close to that of the solution heat treatment. The Stage II in the not-deformed alloy is due to the precipitation of the stable Al_3Fe phase (Fig.2). The C-curve in Fig. 3 indicates the beginning of precipitation of the stable phase. Coincide of this curve with the upper C-curve for the pre-deformed alloy (Fig. 5) shows that the upper curve corresponds to the precipitation of the stable phase, which agrees with the TEM observation (Fig. 7). The lower curve, therefore, is considered to be attributed to the precipitation of the metastable Al_6Fe, which is confirmed by TEM observation (Fig. 6). Disappearance of the Stage III at high aging temperatures shows that the stable phase is directly formed, that is , the Stage I is followed by the Stage II without the Stage II.

Holm and Hornborgen [5], and Stickels and Bush [6] reported one C-curve for pre-deformed alloys. However, they did not carry out aging above 773 K.

5. SUMMARY

Precipitation behaviors in high purity Al-300 ppm Fe alloy are summarized as follows:

(1) Resistivity decreases in two stages with aging time in not-deformed alloy, while it decreases in three stages in the pre-deformed alloy.

(2) The Stage I in both the alloys is attributed to the annihilation of quenched-in vacancies.

(3) The Stage II in the not-deformed alloy and the Stage III in the pre-deformed alloy are due to the precipitation of the stable Al_3Fe phase. The Stage II in the pre-deformed alloy is due to the precipitation of the metastable Al_6Fe phase.

(4) TTP-diagram in the not-deformed alloy shows one C-curve, while that in the pre-deformed alloy shows two C-curves, which indicates the formation of Al_3Fe and Al_6Fe.

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