Electrical resistivity, magnetization, and grain-boundary precipitate in nickel-rich nickel-indium alloys

Y. D. Yao, Y. Y. Chen, T. H. Chuang,[a] C. Kung, and C. J. Lin
Institute of Physics, Academia Sinica, Taipei, Taiwan, Republic of China

The variations of the electrical resistivity, the magnetization, and the grain-boundary precipitates of a Ni-rich Ni-In alloy system with In concentration up to 7.5 at. % have been investigated as functions of annealing time at 773 K. For samples homogenized at 1225 K, clear grain boundaries are observed. However, for these aged samples, we observed both grain-boundary precipitates and variations of the electrical resistivity and the magnetization; and the binary alloy with higher In concentration has the higher variation rate in the decrease of the electrical resistivity, the increase of the magnetization, and the growth of the grain-boundary precipitates.

INTRODUCTION

There has been considerable progress in the understanding of the morphology and growth kinetics of the grain-boundary precipitates in binary-alloy systems in the last few decades.[1–3] Little progress, however, has been made in the comprehension of the relationship between the electrical resistivity, the magnetization, and the averaged diameter of grain-boundary precipitates of the binary-alloy systems.[4–6] It is well known that the electrical resistivity and the magnetization are very sensitive to the composition change due to grain-boundary precipitates, and hence these analyses are a very useful tool for identifying the variation of grain-boundary precipitates in many magnetic binary-alloy systems. For the Ni-In alloy system, the resistivity measurements have been predominately confined to the very dilute Ni-In alloys.[7,8] Therefore, we are motivated to study the electrical resistivity,[9] the magnetization, and the relation of these properties with grain-boundary precipitates in the nickel-indium alloy system.

In this study, we report investigations of the variation of the electrical resistivity, the magnetization, and grain-boundary precipitates of nickel-rich nickel-indium alloys containing In up to 7.5 at. % as functions of temperature between 4 and 800 K, as well as the annealing time at 773 K.

EXPERIMENTAL PROCEDURE

The nickel-indium alloy samples containing In up to 7.5 at. % of In, were prepared by melting in an induction melter under a positive pressure of argon and casting into 10-mm-diam molds. These ingots were homogenized at 1225 K for two weeks to remove any microscopic segregation. Samples for this study were cut from these ingots and were strain-relief annealed again in vacuum at 1225 K for 6 h and water quenched. For heat-treatment studies, samples in evacuated quartz tubes were annealed at 773 K for the electrical, magnetic, and scanning electron microscopy (SEM) studies. Typical sample dimensions were roughly $1\times2\times20$ mm^3 for the electrical resistivity study, $1\times2\times6$ mm^3 for the magnetization study, and $3\times9\times9$ mm^3 for the SEM study.

The averaged diameter of grain-boundary precipitates was determined from 50 measurements made from different regions of the sample. The data were averaged and multiplied by a factor of $\pi/4$ to be consistent with the bulk averaging technique previously used by others.[2] The electrical resistivity of all samples was determined by using the conventional four-probe technique. The magnetization was measured by a vibrating sample magnetometer (VSM) above room temperature, and by both VSM and SQUID magnetometers below room temperature.

RESULTS AND DISCUSSION

Clear grain boundaries were observed for samples homogenized at 1225 K. However, for samples annealed at 773 K, the cells of the grain-boundary precipitates grew as a function of annealing time. As an example, Fig. 1(a) shows the clear grain boundary of a homogenized Ni-4.6 at. % In sample; Figs. 1(b) and 1(c) present the grain-boundary precipitates of two Ni-4.6 at. % In samples which have annealed at 773 K for 8 and 20 h, respectively. The growth of the cells of the grain-boundary precipitates is a function of both the In concentration and the annealing time at 773 K. Figure 2, as an example, shows the averaged diameter of the cells of the grain-boundary precipitates L as a function of annealing time t at 773 K for Ni-1.4 at. % In, Ni-2.3 at. % In, and Ni-4.6 at. % In samples. The growth rate of grain-boundary precipitates is roughly 2 μm/day, 16 μm/day, and 46 μm/day for Ni-1.4 at. % In, Ni-2.3 at. % In, and Ni-4.6 at. % In samples, respectively. Manifestly, Fig. 2 tells us that in the case of the Ni-In alloys for a given annealing temperature which is far below the annealing temperature for homogenity, the alloy with higher solute concentration has the higher growth rate of grain-boundary precipitates.

Figure 3 presents the electrical resistivity as a function of temperature between 4 and 800 K for both Ni-1.4 at. % In and Ni-7.5 at. % In. The dots represent the homogenized samples, and circles show data for samples annealed at 773 K for one day. Here, after one day's annealing, the

[a]Graduate Institute of Material Engineering, National Taiwan University, Taipei, Taiwan, Republic of China

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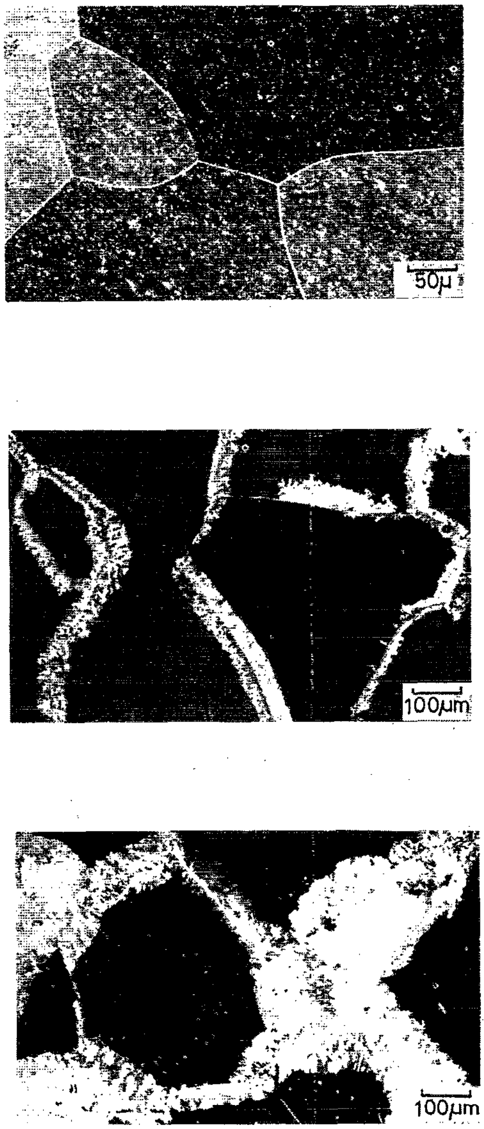

FIG. 1. (a) The grain boundary of a homogeneous Ni-4.6 at. % In sample. (b) The grain-boundary precipitates in a Ni-4.6 at. % In sample annealed at 773 K for 8 h. (c) The grain-boundary precipitates in a Ni-4.6 at. % In sample annealed at 773 K for 20 h.

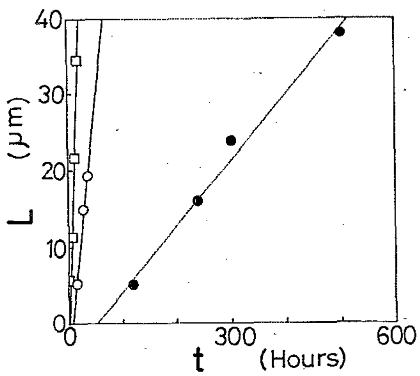

FIG. 2. The averaged size of the cells of the grain-boundary precipitates as a function of annealing time at 773 K for Ni-1.4 at. % In (●), Ni-2.3 at. % In (○), and Ni-4.6 at. % In (□) samples.

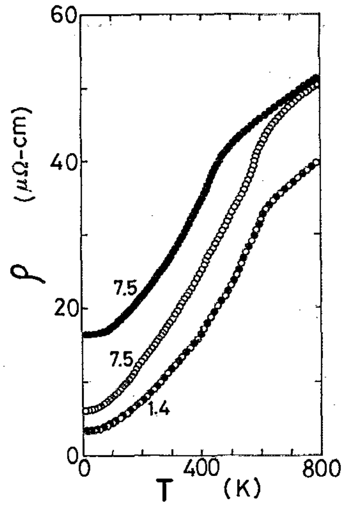

FIG. 3. The electrical resistivity as a function of temperatures between 4 and 800 K for Ni-1.4 at. % In and Ni-7.5 at. % In samples (●: homogeneous samples; ○: samples annealed at 773 K for one day).

decrease of the electrical resistivity at 4 K is roughly 10.7 $\mu\Omega$ cm for Ni-7.5 at. % In, but only 0.16 $\mu\Omega$ cm for Ni-1.4 at. % In. Generally speaking, for all the Ni-In samples which were thermally annealed at 773 K, the electrical resistivity at 4 K decreases monotonically with annealing time; and the rate of decrease is a monotonical function of indium concentration. According to scanning electron microscopy and x-ray diffraction,[10] the Ni-In alloys at all aging temperatures will decompose completely by discontinuous precipitation into a fine lamellar structure of nickel-rich solid solution and Ni_3In precipitate phase. This lamellar structure will then decompose at all aging temperatures by a discontinuous coarsening reaction. One of the results of this effect is the decrease of the residual electrical resistivity.

The magnetization at 5 kG as a function of temperature between 5 and 700 K for homogenized samples (dots) and aged samples (circles) at 773 K for one day are plotted in Fig. 4 for both Ni-1.4 at. % In and Ni-7.5 at. % In. At least, two facts are evident from this plot. First, the mag-

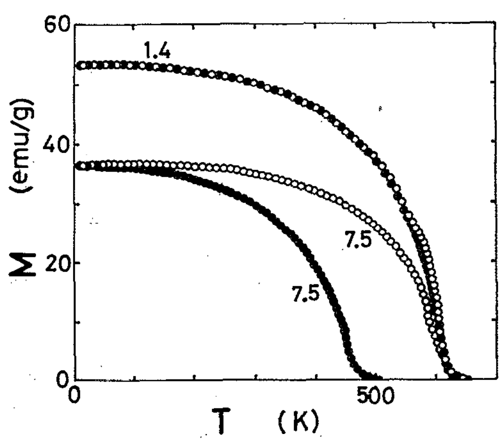

FIG. 4. The magnetization at $H = 5$ kG as a function of temperature between 5 and 800 K for Ni-1.4 at. % In and Ni-7.5 at. % In samples. (●: homogeneous samples; ○: samples annealed at 773 K for one day).

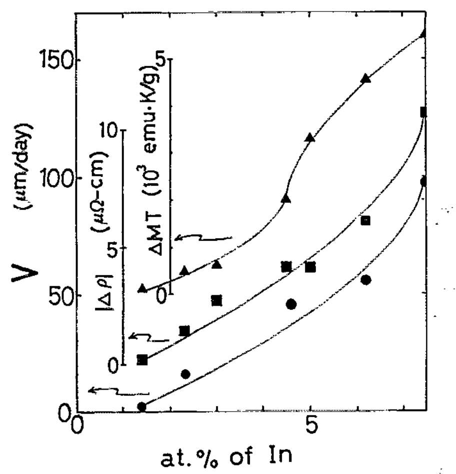

FIG. 5. V, $|\Delta\rho|$, and ΔMT as functions of the In concentration in the Ni-In alloy system (●: V, ■: $|\Delta\rho|$, and ▲: ΔMT).

netization at 5 K is not varied significantly with the aging times. Second, the area under the M-vs-T curves seems to increase with aging times.

Finally, in Fig. 5, we present the rate of the averaged size of the cells of the grain boundary precipitates, $V = dL/dt$ (μm/day), the decrease of the electrical resistivity at 4 K after one day of annealing at 773 K, $|\Delta\rho|$, and the increase of the area under the M versus T curve after one day of annealing at 773 K, ΔMT, as functions of the indium concentration for the Ni-rich Ni-In system. We conclude that the values of V, $|\Delta\rho|$, and ΔMT increase roughly monotonically with the increase of the indium concentration in the Ni-rich Ni-In alloys.

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