Magnetization study on grain-boundary precipitation in a Ni-8 at. % Sn alloy

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The variations of the grain-boundary precipitates, the electrical resistivity, and the magnetization of a Ni-8 at. % Sn alloy have been investigated as functions of annealing temperature and annealing time. For samples annealed at 773 K, the averaged growth rate of the size of the grain-boundary precipitates is roughly 0.42 μm/h for the first 24 h; the electrical resistivity at $T = 10$ K and the magnetization at $T = 10$ K and $H = 5$ kG vary monotonically with respect to the annealing time for the first 2 weeks, changing from 22.5 to 7 $\mu\Omega$ cm for the electrical resistivity and from 27 to 33 emu/g for the magnetization. A large tail section in the magnetization versus temperature curve was also observed in the aged samples. All these electrical and magnetic variations in the Ni-8 at. % Sn samples annealed at 773 K varied monotonically with respect to the growth of the grain-boundary precipitates.

I. INTRODUCTION

In recent years considerable attention has been drawn to the morphology and growth kinetics of the grain-boundary precipitates in various binary alloy systems.[1-5] However, relatively little research work has been devoted to the relation between the grain-boundary precipitation and the electrical and magnetic properties of these binary alloy systems.[6] It is well known that the electrical and magnetic properties such as the electrical resistivity and the magnetization are very sensitive to the composition change due to grain-boundary precipitation, and hence these analyses are a very useful tool for identifying composition variations in magnetic binary alloy systems.

II. EXPERIMENTAL PROCEDURE

The Ni-8 at. % Sn alloys 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 1325 K for 2 weeks to remove any microscopic segregation. Samples for this study were cut from these ingots and were strain-relief annealed again in vacuum at 1325 K for 6 h and water quenched. For heat treatment studies, samples in evacuated quartz tubes were annealed at 773, 823, and 1023 K for the microscopy study, and at 773 K only for the electrical and magnetic studies. Typical sample dimensions were roughly $3\times9\times9$ mm^3 for the microscopy study, $1\times2\times20$ mm^3 for the electrical resistivity study, and $1\times2\times6$ mm^3 for the magnetization study.

The grain-boundary precipitates were observed by using an optical microscope. The size of the 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 used by others previously.[2]

III. RESULTS AND DISCUSSION

For samples homogenized at 1325 K, clear grain boundaries, as shown in Fig. 1, were observed. Also, a single Curie temperature T_c was detected by the electrical resistivity and magnetization measurements. However, for the samples annealed at 773 K, we observed both grain-boundary precipitates and variations in the electrical and magnetic properties. As an example, Fig. 2 shows the grain-boundary precipitates in the sample annealed at 773 K for 24 h. The average size of the cells of the grain-boundary precipitates for this sample was found to be roughly 10 μm. The growth of the cells of the grain-boundary precipitates is a function of both annealing temperatures and annealing times. Figure 3 shows the averaged size of the cells of the grain-boundary precipitates, L as a function of annealing time t at 773, 823 and 1023 K. Manifestly, L increases monotonically with increasing annealing temperature and time. The growth rate of grain-boundary precipitates is roughly 0.42, 3.8, and 20 μm/h for samples annealed at 773, 823, and 1023 K, respectively.

The electrical resistivity ($H = 0$) and the magnetization at $H = 5$ kG as functions of temperatures between 10 and 800 K for Ni-8 at. % Sn samples homogenized at 1325 K

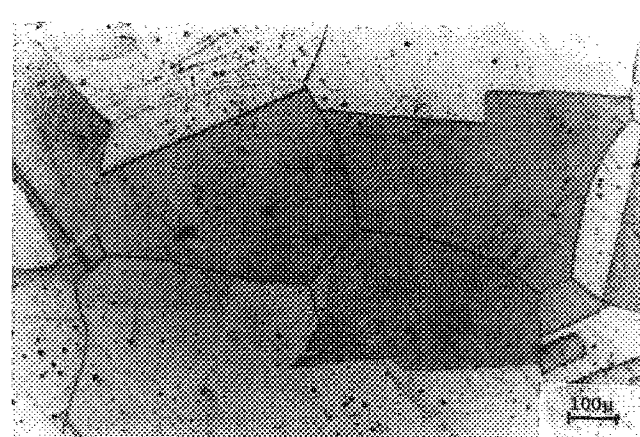

FIG. 1. Grain boundary of the homogenized Ni-8 at. % Sn sample.

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FIG. 2. Grain-boundary precipitates in the Ni-8 at. % Sn sample annealed at 773 K for 1 day.

and annealed at 773 K for 1, 2, 7, and 14 days are plotted in Figs. 4 and 5, respectively. Each curve is indicated by a number which reflects the annealing time (days) at 773 K. The electrical resistivity at 10 K decreases monotonically with increasing annealing time from 22.5 $\mu\Omega$ cm for the homogenized sample, to 7.0 $\mu\Omega$ cm for the sample annealed at 773 K (14 days) as shown in Fig. 6. We have observed that the electrical resistivity at 10 K of the Ni-8 at. % Sn alloy annealed at 773 K decreases very fast at the beginning (roughly 4.54 $\mu\Omega$ cm/day); and its decrease gradually slows down to roughly 0.43 $\mu\Omega$ cm/day after 7 days of annealing. This phenomenon suggests that the low-resistivity Ni-rich phase is easy to decompose from the binary solid solution at the beginning; and it may be gradually saturated for samples annealed at 773 K longer than 2 weeks. Another interpretation is that this is a grain-boundary phase size effect. As the grain-boundary phase coarsens, ρ would be expected to decrease since there will also be fewer scattering centers.

The magnetization at 5 kG of the homogenized sample decreases very fast with increasing temperature near T_c ($\simeq 370$ K), and becomes zero above T_c. However, for the annealed samples at 773 K, we have observed (Fig. 5) quite a large tail section (extra magnetization above T_c compared with the homogenized sample) above roughly 370 K; and the area of this tail section becomes very large for samples annealed at 773 K longer than 2 days. These tail sections of the magnetization go to zero roughly above 600 K. Generally speaking, the magnetization of an inhomogeneous solid solution is given by a superposition of the magnetizations for small regions of different solute concentrations. Following a similar analysis of the morphology and growth kinetics of the grain-boundary precipitates as in Ref. 5, we conclude that both the nickel-rich phase and the Ni_3Sn intermetallic

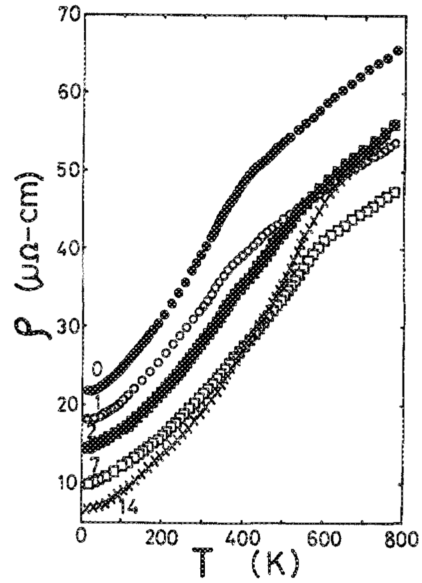

FIG. 4. Electrical resistivity as a function of temperature between 10 and 800 K for Ni-8 at. % Sn samples homogenized at 1325 K (●), and annealed at 773 K for 1 day (○), 2 days (■), 7 days (□), and 14 days (×).

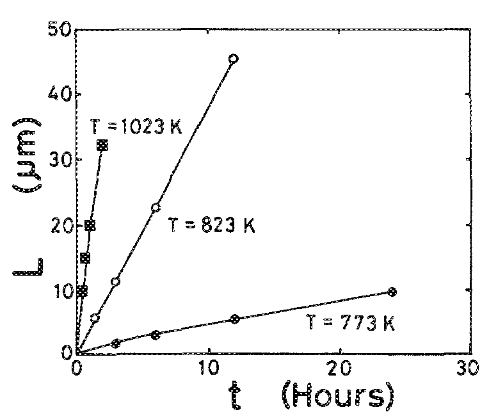

FIG. 3. Averaged size of the cells of the grain-boundary precipitates as a function of annealing time at temperatures of 773 K (●), 823 K (○), and 1023 K (■).

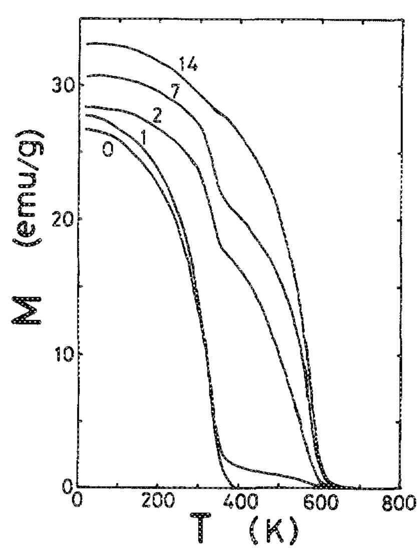

FIG. 5. Magnetization at $H = 5$ kG as a function of temperature between 10 K and 800 K for Ni-8 at. % Sn samples homogenized at 1325 K (No.0), and annealed at 773 K for 1 day (No.1), 2 days (No.2), 7 days (No.7), and 14 days (No.14).

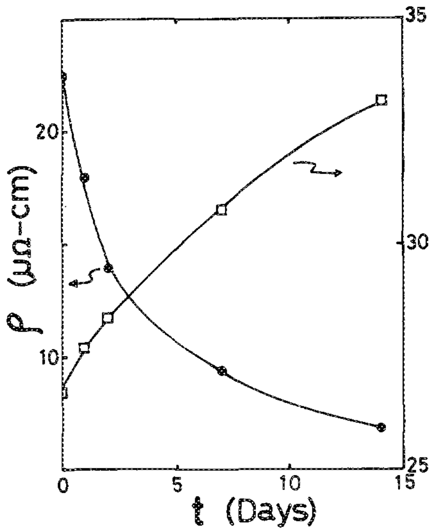

FIG. 6. Electrical resistivity (●) at $T = 10$ K and the magnetization (□) at $H = 5$ kG and $T = 10$ K as functions of annealing time for Ni-8 at. % Sn samples annealed at 773 K.

compound are precipitated into the lamellar structure of the grain boundaries at all aging temperatures. One of the results of this precipitation and growth effect in the grain boundaries is the formation of solute-poor regions in the matrix which affect the appearance of the tail section of the superimposed magnetization curves shown in Fig. 5. In Fig. 6, the magnetization at $T = 10$ K and $H = 5$ kG for samples annealed at 773 K is plotted as a function of annealing time. The value of the magnetization increases monotonically with increasing annealing time from 27 emu/g for a homogenized sample, to 33 emu/g for the sample annealed for 14 days. However, the slope of this curve changes smoothly from roughly 1.1 emu/g/day at the beginning to 0.2 emu/g/day after annealing 14 days.

In conclusion, we have reported the results of an experimental study of the variations in the grain-boundary precipitates, the electrical resistivity, and the magnetization for a Ni-8 at. % Sn alloy as functions of annealing temperature and annealing time. We have found that, for samples annealed at 773 K for at least 24 h, the average growth rate of the size of the grain-boundary precipitates is roughly 0.42 μm/h; the electrical resistivity at $T = 10$ K decreases roughly 0.19 $\mu\Omega$ cm/h; and the magnetization at $H = 5$ kG and $T = 10$ K increases roughly 0.046 emu/g/h. A large tail section in the magnetization versus temperature curve has been observed also in the aged Ni-8 at. % Sn samples. All the electrical and magnetic variations studied vary as monotonic functions of the grain-boundary precipitate size.

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