ELECTRICAL AND MAGNETIC STUDIES ON GRAIN BOUNDARY SEGREGATION IN A Ni-3AT.%Sn ALLOY

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Abstract

The variations of the grain boundary precipitation, the electrical resistivity and the magnetization of a Ni-3at.%Sn alloy have been investigated as a function of annealing time and annealing temperature. The averaged growth rate of the size of the grain boundary precipitates for samples annealed at 773 K is roughly 7 μm per week. The residual electrical resistivity for samples annealed at 773 K decreases roughly 0.6 μΩ-cm per week. All the electrical and magnetic properties in the Ni-3at.%Sn samples annealed at 773 K vary monotonically with respect to the growth of the grain boundary precipitation and segregation.

Introduction

Much work concerning the morphology and growth kinetics of the grain boundary precipitation and segregation in binary nickel-based system has been studied through optical microscopy [1-5]. Comparatively little effort has been devoted to the relation between the magnetic property and the grain boundary precipitation and segregation of this binary nickel-based system. The magnetic properties such as magnetization and Curie temperature are very sensitive to the composition change due to grain boundary segregation and precipitation, and hence magnetic analyses are useful tool for identifying composition variations in magnetic alloy system. In this investigation, relations between magnetic properties and composition variations in solid solutions of a Ni-3at.%Sn alloy are discussed.

Experimental Considerations

The ingot with appropriate amounts of Ni and Sn was homogenized at 1325 K for two weeks to remove any microscopic segregation. Samples for this study were cut from the ingot and were homogenized again in vacuum at 1325 K for 4 hours and water quenched. These samples were cut in the form of rectangular parallelepipeds by a low speed saw. Typical sample dimensions were roughly 2x9x9 mm^3 for the microscopy study, 1x2x20 mm^3 for the electrical resistivity study and 1x2x6 mm^3 for the magnetization study.

The grain boundary precipitation was observed by using optical microscope. The size of the grain boundary precipitates was measured with 50 measurements being 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 which was used before [3]. The electrical resistivity of all samples was determined by using the conventional four-probe technique. Temperatures between 4 and 300 K were achieved in a Displex closed-cycle refrigeration system (Model CS-308, Air Produ.). Temperatures between 300 and 700 K were produced by a Marshall vacuum furnace. The furnace could be either evacuated or filled with helium gas. The magnetization was measured between 300 and 700 K by a vibrating sample magnetometer. We assume that the magnetization of inhomogeneous solid solution is given by a superposition of the magnetizations for small regions of different solute contents.

Results and Discussion

For samples homogenized at 1325 K, clear grain boundary, as shown in Fig. 1(a), and single Curie temperature were observed. Fig. 1(b) shows the grain boundary precipitates in the sample annealed at 773 K for two weeks. The averaged size of the cells of the grain boundary precipitates for this sample was roughly 14 μm. The growth of the cells of the grain boundary precipitates is a function of both annealing temperature and annealing time. Fig. 2 shows the averaged size of the cells of the grain boundary precipitates, L, as a function of annealing time, t, at both 773 K and 823 K. Manifestly, L increases monotonically with increasing annealing time and temperature.

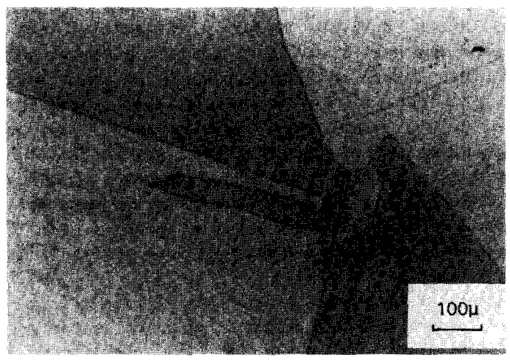

Fig. 1(a) The grain boundary of the homogeneous Ni-3at.%Sn sample.

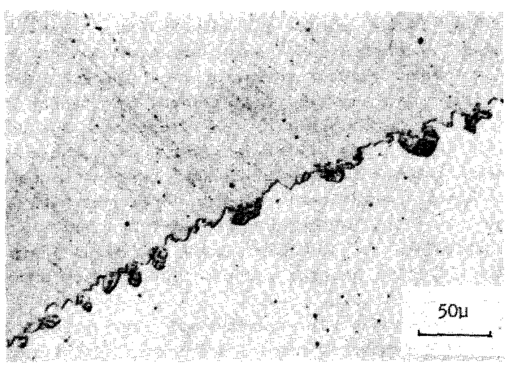

Fig. 1(b) The grain boundary precipitates in the sample annealed at 773 K for two weeks.

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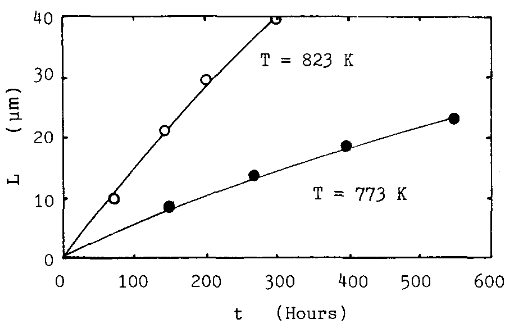

Fig. 2 The averaged size of the cells of the grain boundary precipitates as a function of annealing time at temperatures 773 K (•) and 823 K (o).

The magnetizations at 8 kG as a function of temperature between 300 and 700 K for samples homogenized at 1325 K, and annealed at 773 K for 2 and 4 weeks are plotted in Fig. 3. The magnetization of the homogenized sample decreases much faster than that of those annealed samples near 530 K (this is roughly the Curie temperature of the homogeneous Ni-3at.%Sn alloy). For those annealed samples, we have observed quite large tail section above 530 K. This tail of magnetization goes to zero near 625 K (the Curie temperature of pure Ni is near 625 K). The magnetization at 300 K also increases with aging times. These results suggests that Ni-rich phase is precipitated into the grain boundary.

Following the similar analysis of the morphology and growth kinetics of the grain boundary precipitation [5], we know that both nickel-rich phase and Ni_3Sn intermetallic compound are precipitated into the lamellar structure of the grain boundary at all aging temperatures. One of the results of this growth effect of the grain boundary is the formation of solute-poor regions in the matrix which deforms strongly the "tails" of the superimposed magnetization curves, as shown in Fig. 3.

From the electrical resistivity measurements, we observed that the electrical resistivity at 4 K decreases monotonically from 8.26 μΩ-cm for homogeneous sample, to 6.02 μΩ-cm for sample annealed at 773 K for 4 weeks. This means that the residual electrical resistivity of the Ni-3at.%Sn alloy annealed at 773 K decreases roughly 0.6 μΩ-cm per week.

Fig.s 4 (a), (b) and (c) show both the electrical resistivity, ρ, and the derivative of the electrical resistivity, dρ/dT, as functions of temperatures between 500 and 650 K. If the Curie temperature is determined from the starting point of the abrupt drop in the dρ/dT vs. T curves. It is clear that double Curie temperatures occur in samples annealed at 773 K.

In conclusion, this is the first time that the relation between the growth of grain boundary precipitates and the electrical and magnetic properties in the Ni-Sn alloy system is reported. We have found that the averaged growth rate of the size of the grain boundary precipitates for samples annealed at 773 K is roughly 7 μm per week, and the growth rate for samples

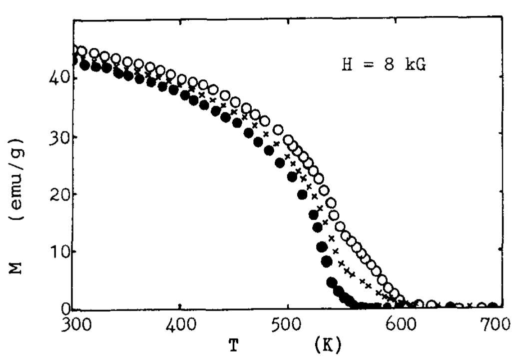

Fig. 3 The magnetization at 8 kG as a function of temperature between 300 K and 700 K for samples homogenized at 1325 K (•), and annealed at 773 K for 2 weeks (x) and 4 weeks (o).

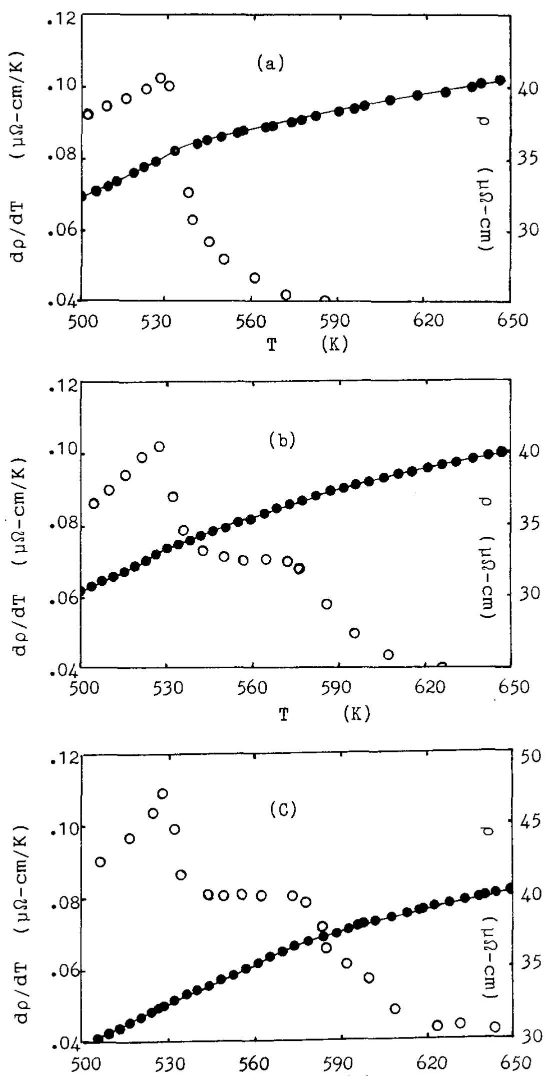

Fig. 4 The electrical resistivity (•) and the derivative of the electrical resistivity (o) as functions of temperatures between 500 K and 650 K.
(a) for sample homogenized at 1325 K.
(b) for sample annealed at 773 K for 2 weeks.
(c) for sample annealed at 773 K for 4 weeks.

annealed at higher temperature should be much higher; for example, the growth rate of grain boundary precipitates for samples annealed at 823 K is roughly 23 μm per week. The residual electrical resistivity of the Ni-3at.%Sn sample system annealed at 773 K decreases roughly 0.6 μΩ-cm per week. Double Curie temperatures were observed in the annealed samples by means of the electrical resistivity studies. Strong tail section in the magnetization curve has been measured in the aged Ni-3at.%Sn samples. Finally all the electrical and magnetic properties in the Ni-3at.%Sn samples annealed at 773 K varied monotonically with respect to the growth of the grain boundary precipitates.

Acknowledgements

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