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Grain size effect on martensitic transformation behavior in Fe-Ni invar alloys

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Abstract. We investigated the grain size effect on martensitic transformation behavior in Fe-30at.%Ni powder and ribbon specimens. The powder specimen with a particle size of 5 µm does not show an athermal martensitic transformation but does show an isothermal martensitic transformation after an incubation time of about 10⁴ s at 205 K. On the other hand, the powder specimen with a particle size of 20 µm shows an athermal martensitic transformation at 150 K. The value of \( M_s \) is much lower than that of the single crystal and of bulk specimens. However, the \( M_s \) temperature of a ribbon specimen with an average grain size of 15 µm is found to be almost identical to that of the single crystal and of bulk specimens. Considering these results, the athermal martensitic transformation is suppressed by the decrease in particle size if grains do not have grain boundaries.

1. Introduction

It is well known that grain size influences various physical properties. For example, nanosized particles of Au and Ag are found to have lower melting points than their respective bulk metals because of changes in surface energy [1,2]. Grain size is also known to have an influence on the martensitic transformation of \( \text{ZrO}_2 \) powder [3] and Fe-Ni powder [4]. However, detailed transformation behavior and the transformation mechanism are not clear yet.

For this paper, we investigated the effect of grain size on the martensitic transformation temperature and its kinetics using an Fe-30at.%Ni powder and ribbon specimens. As a result, we found that the behavior of the martensitic transformation with fine powder specimens is different from that of a ribbon and of coarse powder specimens.

2. Experimental Procedure

Powder and rapidly quenched ribbon specimens of Fe-30at%Ni were prepared. The powder was produced by an argon gas atomization process and classified to average particle sizes of 5 µm and 20 µm. Specimens of rapidly quenched ribbon were prepared by melt-spun methods with a copper roll at a rotation speed of 15.7 m/s.

The microstructure of each specimen was observed by optical microscope. To identify the phase of each specimen, an X-ray diffraction measurement was made. To investigate magnetic properties and martensitic transformation temperatures, magnetic susceptibility measurements were made using a SQUID magnetometer at temperatures from 300 K to 5 K.
3. Results and Discussion

3.1. Microstructure and crystal structure

Figure 1 shows a cross section of the microstructure for each specimen using an optical microscope after etching. The powder specimen with an average particle size of 5 μm had no grain boundary and the other powder specimen with an average particle size of 20 μm generally had no grain boundary either although a few powder specimens had boundaries. The ribbon specimen is a polycrystal with a grain size of about 15 μm.

Crystal structures of these specimens were confirmed by an X-ray diffraction measurement. Results are shown in Fig.2. As shown in the figure, only the fcc phase is apparent and the bcc phase is not observed in all the specimens.

![Fig.1 A series of optical microscope photograph of Fe-30at.%Ni.](image1)

![Fig.2. Powder X-ray diffraction patterns of Fe-30at.%Ni.](image2)

![Fig.3 Temperature dependence of magnetic susceptibility.](image3)

3.2. Magnetic susceptibility

To investigate magnetic properties and the martensitic transformation temperature, we measured the temperature dependence of the magnetic susceptibility for each specimen. Results are shown in Fig.3. It should be noted that the magnetic susceptibility of the powder specimen with a particle size of 5 μm
gradually increases as the temperature decreases. This indicates that the specimen does not show an athermal martensitic transformation. On the other hand, the powder specimen with a particle size of 20 \( \mu m \) shows a sudden increase at 150 K because of an athermal martensitic transformation. This value of \( M_s \) is much lower than those of the single crystal and bulk specimens as obtained in a previous study [5]. The susceptibility of the ribbon specimen, with grain boundaries, gradually increased as the temperature decreased and the sudden increase at 218 K is due to an athermal martensitic transformation. This \( M_s \) is almost identical to that in a previous study [5]. On the other hand, we investigated in previous work that the effect of quench speed, internal stress and deficiency on magnetic susceptibility with ribbon specimens by various rotation speed and found that these factors hardly influenced on the behaviour of magnetic susceptibility[6]. These results clearly suggest that the particle size of the specimen influences the transformation behavior but the presence of a boundary does not.

![Figure 4. X-ray diffraction patterns for each specimen before and after cooling to 5K.](image)

Figure 4 shows X-ray diffraction patterns of each specimen before and after cooling to 5K. The powder specimen with a particle size of 5 \( \mu m \) does not show a bcc phase even after cooling. The powder specimen with a particle size of 20 \( \mu m \) shows a small amount of bcc phase after cooling while the ribbon specimen shows a large amount of bcc phase after cooling. These results agree well with the magnetic susceptibility measurements shown in Fig. 3.

For the powder specimen with a particle size of 5 \( \mu m \) the martensitic transformation behavior is not athermal but is isothermal because a hysteresis is present in the susceptibility curves for the cooling and the heating process, as shown in Fig. 3. We thus measured the time dependent nature of magnetic susceptibility for the powder specimen with a particle size of 5 \( \mu m \) at 205 K. The result is shown in Fig. 5. As shown in this figure, the magnetic susceptibility increases slightly after an incubation time of about 10^4 s. This result indicates that the 5 \( \mu m \) powder certainly undergoes an isothermal martensitic transformation.

Considering the results described above, we can say that the athermal martensitic transformation is suppressed by a decrease in particle size if no grain boundary is present. Further experiments are required to study this phenomenon.

3.3. Spontaneous magnetization of the Fe-30at%Ni fcc phase

In this study, the athermal martensitic transformation was suppressed in the 5 \( \mu m \) grain size powder. We thus measured the spontaneous magnetization of the Fe-30at%Ni fcc phase at a very low
temperature using this fine grain size powder. A typical result at 5 K is shown in Fig. 6. As a result, the spontaneous magnetization of the fcc phase was found to be 114 emu/g (1.16 $\mu_B$/atom) by drawing a line from the curve at the higher magnetic field as shown with the dotted line in Fig. 6. The value of the spontaneous magnetization is almost identical to that obtained in a previous study (1.1 $\mu_B$/atom) [5].

![Fig. 5 Time dependence of magnetic susceptibility for the 5$\mu$m powder.](image1)

![Fig. 6. Spontaneous magnetization of the fcc phase at 5 K.](image2)

4. Conclusions

We investigated the effect of grain size on the martensitic transformation temperature and its kinetics using a Fe-30at.%Ni powder and ribbon specimens. As a result, a fine grain size powder of 5 $\mu$m with a single crystal shows an isothermal martensitic transformation. On the other hand, the sample with a grain size of about 20$\mu$m with a single crystal shows an athermal martensitic transformation but its $M_s$ is lower than that of the bulk specimen. The same result is obtained for the ribbon specimen with a grain size of about 15 $\mu$m, but its $M_s$ is almost at the same temperature as that of the bulk specimen. These results suggest that the particle size of the specimen influences the transformation behavior and the presence of a boundary does not.

The spontaneous magnetization of the fcc phase is found to be 114 emu/g (1.16 $\mu_B$/atom). The value of spontaneous magnetization is found to be almost identical to that found in a previous study [5].

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