Magnetization Process in Mechanically Alloyed Fe-Ni Invar

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By mechanically alloying and successive heat treatment, Fe-Ni Invar alloys around 35 at% Ni have been made with wide variety of concentration fluctuation. Observed magnetization curve changed significantly by annealing. The saturation magnetization decreased first, took a minimum at 500°C annealing and then increased with further increasing annealing temperature. By assuming Gaussian-type fluctuation, observed magnetization curves could be explained.

Key words: invar alloys, iron-nickel alloys, magnetization curve, mechanical alloying, concentration fluctuation

1. Introduction

Fe-Ni alloys around the Invar composition of 35 at% Ni are known to show various anomalies in both mechanical and magnetic properties. Anomalies in physical properties such as low thermal expansion coefficient were interpreted to be due to the large positive value of the magnetovolume coupling constant.

Anomalies in magnetic properties have been understood as a result of the instability of the 3d-band ferromagnetism in fcc lattice. Due to the existence of a large sharp peak at the top of the 3d-electron band of the fcc phase, the ferromagnetic state becomes energetically unstable when the number of the outer electrons is decreased by decreasing the Ni concentration beyond the Invar region. According to this model, a sudden disappearance of the magnetic moment was expected at a critical concentration. However, observed anomalous decrease of the saturation magnetization from the Slater-Pauling curve is not so sharp as expected from the model of 3d-band ferromagnetism.

To interpret the actual curvature of the decrease of the saturation magnetization from the Slater-Pauling curve, several models have been proposed taking into account the existence of fluctuation of alloy concentration. Kachi et al. considered a Gaussian distribution of the concentration fluctuation and explained the experimental curve. Komura and Takeda counted an effective local concentration around a Fe atom and also succeeded in explaining their results of small angle neutron scattering experiments.

The importance of the concentration fluctuation was also pointed out in the appearance of premartensitic lattice distortion in Invar alloys. Thus the concentration fluctuation is expected to play an important role in the magnetic properties of Invar alloys. However, no such investigation has been made as to modulate artificially the actual concentration fluctuation and then, to see changes in the magnetic properties.

In the present paper we report a result of magnetization measurements in Fe-Ni Invar alloys of which the concentration was modulated by mechanical alloying.

2. Experiment

Fe and Ni powders of the particle size of 100 mesh and the purity of 99.9% were milled for 75 hours in a planetary ball mill in argon atmosphere using stainless steel balls and containers. The intensity of the milling expressed by the ball velocity hitting was 5 m/s. As the milling proceeded, both the balls and the containers came to be covered with the powders, and the products finally obtained in the form of powders were almost half as much in weight as the initial powders. Therefore, no contamination such as an inclusion of Cr can be expected from the balls and the containers by using the present method. The products were annealed at various temperatures between 400 and 1000°C for 1 hour in shielded vacuum and then quenched to room temperature.

The products were examined by X-ray diffraction, and proved to consist mainly of fcc phase. Measurements of magnetization curves were made for as-milled and annealed specimens using a subtracting-sample type magnetometer at room temperature.

![Fig.1 X-ray diffraction pattern for as-milled Fe-Ni Invar alloy.](image-url)
3. Results and Discussion

Observed X-ray diffraction pattern for as milled specimen was shown in Fig.1, where it is seen that the products were mainly in an fcc phase. However, a small amount of bcc phase was also detected. It is seen in this figure that the Bragg peaks are very weak. The intensity of the fcc 111-peak was as weak as 300 c/s. After annealed at 500°C, the weak peaks corresponding to the bcc phase disappeared completely, and the intensity of the Bragg peaks of the fcc phase increased significantly as seen in Fig. 2. The intensity of the fcc 111-peak was 1500 c/s. After annealed at 1000°C, the peak intensity increased further increasing annealing temperature to 95 emu/g. Judging from the present results of the X-ray diffraction, i.e. the peak intensity came to saturate after 1000°C annealing, and also from the results of the work made by Hays et al., this value of the saturation magnetization can be considered to be the bulk value.

The large value of Ms for the as-milled specimen was due to the inclusion of the bcc phase. The decrease at 500°C annealing is due to the disappearance of this phase. The gradual increase of Ms after annealing at higher temperatures, on the contrary, cannot be attributed to existence of other phases than fcc. This gradual increase of Ms can be explained by considering that the range of concentration fluctuation decreases with increasing the annealing temperature, and then it reaches to the saturated value after annealing at 1000°C.

By assuming the same Gaussian-type concentration fluctuation as adopted by Kachi et al., the half-width of the distribution is estimated for each stage of annealing. For annealed specimen at 500°C the half width of the concentration fluctuation is estimated to be 60 times wider than the value adopted by Kachi et al. for the bulk specimen. This value decreases with annealing at higher temperature to the bulk value, which corresponds to the value of the specimen annealed at 1000°C. Thus, it becomes possible to control the range of the concentration fluctuation in Fe-Ni Invar alloys in a wide range.
range by mechanically alloying and successive annealing.

4. Conclusion

In Fe-Ni Invar alloys wide variety of concentration fluctuation was introduced by mechanical alloying and successive annealing at temperatures between 500 and 1000°C. Observed magnetization curves and the initial decrease followed by a gradual increase of Ms with increasing annealing temperature above were explained by considering the disappearance of small amount of a bcc phase and then the decrease of the width of the Gaussian type concentration fluctuation.

References