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Martensitic Transformation of γ-FeN in High Magnetic Field Generated by a Hybrid Magnet

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High magnetic filed was applied to γ-FeN foils in order to assist the fcc -> bct martensitic transformation. The molar fraction of martensite monotonically increased with increasing the magnetic filed at 4.2 K, and spontaneous magnetization at 280 K reached 214 emu/g. The amount of retained austenite was reduced to 10 % at 350 kOe from 36 % at zero magnetic field.
Key words: Iron-nitrides, γ-FeN, Martensitic Transformation, High magnetic Field, Hybrid Magnet.

1. INTRODUCTION

Iron nitrides in the bct structure, α'[1] and α"[2] phases, are expected as new magnetic materials characterized by large magnetization than pure iron[3-7]. Thin film methods, such as MBE[4], sputtering[5,6], and ion implantation[7], have succeeded to produce single-phase or single-crystal samples of these. But single-phase sample is not obtained in bulk form so far.

The nitrogen atoms in these phases occupy the octahedral interstitial sites in the bct matrix of Fe, as same as carbon atoms do in the Fe-C steel[1]. And, just like Fe-C system, α' phase is formed as martensite phase in Fe-N steel by quenching the austenite (γ) phase[1]. If all of the γ phase is successfully transformed into the α' phase by some means, bulk sample would be produced and opens up variety of application.

The γ phase spontaneously undergoes the fcc -> bct martensitic transformation when it is cooled below the Ms temperature. A large volume change accompanied with the transformation suppresses further development of the α' phase, and the retained austenite always remains in the sample as far as high nitrogen γ phase is concerned. Therefore, single phase sample of α' can not be obtained when one just cooled the γ phase below its Ms temperature.

The γ phase is paramagnetic even at 1.4 K[8] and considerably decreases gross magnetization of sample when it remains. Therefore, it is of necessity to exploit the method to reduce or remove the retained austenite in order to pursue the high magnetization material, even if the α' phase possesses higher magnetization than pure iron.

Kakeshita et al. have carried out systematic research on effects of hydrostatic pressure and magnetic field on martensitic transformation[9]. They clearly demonstrated external magnetic field lowers Gibbs free energy of ferromagnetic martensite phase, and drives the transformation. Present authors have applied high magnetic field up to 280 kOe to γ-FeN, and found higher magnetic filed turns out higher molar fraction of α' phase[10,11]. In this report, we have used a hybrid magnet generating 350 kOe of steady field to reduce the retained austenite further.

2. EXPERIMENTAL PROCEDURE

Commercial iron foils (0.01 mm in thickness, 10 mm x 10 mm in size, and 99.8 % in purity) were annealed at 1000 ℃ in dry hydrogen gas to decaburize for 1 hour, and successively nitrified with a mixture of NH_3 and H_2 gas at 650 ℃ for 1 hour. Foil thickness and annealing time were optimized to assure composition homogeneity in samples. Nitrified samples were subsequently quenched into distilled water without having contact with the air, and single phase sample of γ was obtained. The surface of the samples was shiny silver and no surface treatment was applied through out present research.

X-ray diffraction spectra were measured with a two-circle goniometer equipped with a graphite monochrometer. Mo Kα radiation was used for precise determination of lattice parameter and longer penetration depth than Cu or Co Kα radiation, which is estimated to be 5.9 μm at $2\theta = 20$ °, i.e. more than a half of the foil thickness. Lattice parameter was determined using the Nelson-Riley plot with 21 diffraction lines after deconvoluting the $K\alpha_1$ and $K\alpha_2$ lines. Samples with lattice parameter of $3.646\ \text{A} \leqq a_0 \leqq 3.647\ \text{A}$ were selected and used for following experiments. According to composition dependence of lattice parameter, corresponding nitrogen concentration is 9.6 at %N[12].

The γ phase samples were cooled to 4.2 K in zero magnetic field, and external magnetic field was applied in the foil plane as to minimize the demagnetizing factor. A Bitter type magnet was used to generate magnetic field of $0\ \text{kOe} < H \leqq 230\ \text{kOe}$. For experiments above 230 kOe, a hybrid magnet consisting of 140 kOe superconducting and 210 kOe Bitter type magnet was employed. After removing the field, the sample was

heated to room temperature. A SQUID magnetometer (Quantum Design MPMS5.5) was used to measure magnetization curves in the field range of -40 kOe ≦ H ≦ +40 kOe. Sample weight was determined with an ultra-micro balance (Mettler UMT-2).

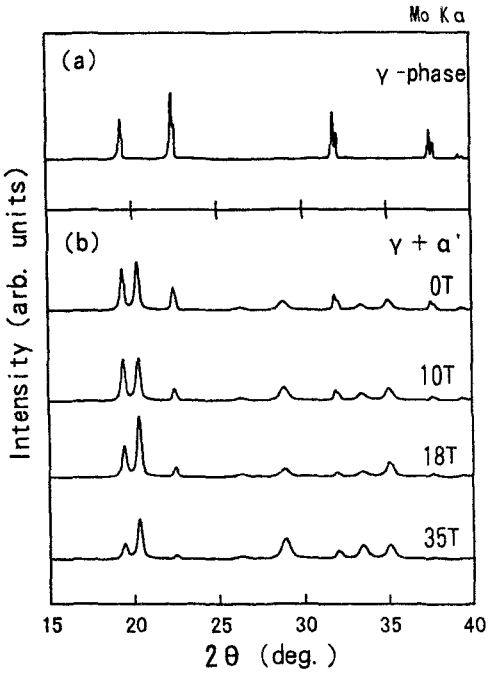

Fig. 1 X-ray diffraction spectra for FeN foils. (a) γ-FeN, (b) γ-FeN cooled to 4.2 K and subjected to various magnetic field at that temperature.

3. RESULTS AND DISCUSSION

Figure 1 shows X-ray diffraction profiles of the γ-FeN (Fig. 1(a)) and γ-FeN cooled to 4.2 K and subjected to magnetic field at that temperature (Fig. 1(b)). The Ms temperature for present samples is 230 K, and some additional diffraction lines are recorded in Fig. 1(b). All new lines are indexed in terms of single bct structure. With increasing the magnetic field at 4.2 K, intensity of lines from the α' phase increases and those of γ phase become weakened. The γ phase lines are still observable in spectrum of the 350 kOe sample. Higher magnetic field is found to be necessary for producing the single phase sample of α'.

Spontaneous magnetization of samples are plotted as a function of applied field at 4.2 K in Fig. 2. According to ref. 13, magnetization of pure Fe is 218 emu/g and plotted as a dashed line in the figure. In good accordance with Fig. 1(b), magnetization monotonically increases with increasing the magnetic field, and reaches 214 emu/g at 350 kOe. As can be seen in the figure, magnetization does not saturate even at 350 kOe, and would exceed 214 emu/g when higher magnetic field were available.

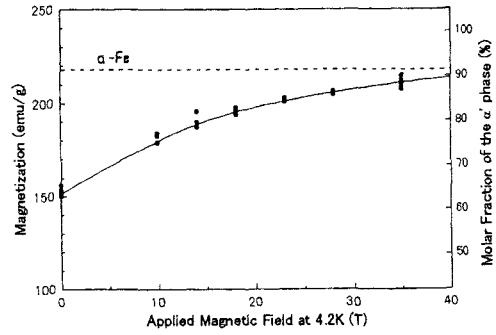

Fig. 2 Spontaneous magnetization of α'+γ phase samples at 280 K plotted as a function of applied magnetic field at 4.2 K. Dashed line indicates magnetization of pure iron. Molar fraction of martensite (right) is estimated assuming 238 emu/g for the α' phase.

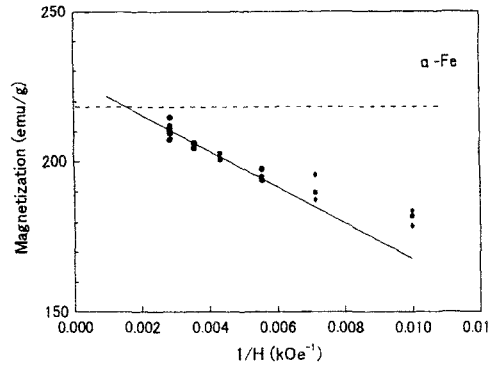

Fig. 3 Spontaneous magnetization of α'+γ phase samples at 280 K plotted as a function of 1/H.

Assuming the magnetization of the α' phase at present composition as 238 emu/g[11], the highest magnetization and zero field data in Fig. 2 correspond to molar fraction of 90 % and 64 % for the α' phase, respectively. In other words, the 350 kOe of field reduced the amount of retained austenite to less than one third of the value at zero field.

It would be worthwhile to deduce the intensity of magnetic field needed to reach magnetization of pure iron. Obviously, magnetization or volume fraction is not a linear function of applied magnetic field as shown in Fig. 2, and it makes almost impossible to extrapolate the data for estimation. Moreover, we do not have appropriate equation describing molar fraction of martensite as a function of magnetic field. Here, we tentatively plot the data in Fig. 2 against 1/H in Fig. 3 for trial, and found high magnetic field data above 140 kOe fall on a straight line. Extrapolating this linear relation to 218 emu/g, 700 kOe of steady field is estimated to be needed for present Fe-N samples. Unfortunately, this extreme high magnetic field is far above the capacity of present hybrid magnet. A combination of magnetic field and some other method should be examined for this particular interest.

4. Conclusion

The steady magnetic field of 350 kOe was found to reduce the amount of the retained austenite to less than one third of that for zero magnetic field. High magnetic field beyond the capacity of present hybrid magnet is needed to realize a large magnetization exceeding pure iron.

Acknowledgment

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