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MICROSTRUCTURE AND MAGNETIC PROPERTIES OF Fe-Cr-Co-V ALLOYS

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ABSTRACT

The relationship between the microstructure and magnetic properties of heat treated Fe-23wt%Cr-15wt%Co-5wt%V has been studied by transmission electron microscopy and Lorentz microscopy. Three different heat-treatments were adopted for the present investigations, viz., 1) isothermal aging, 2) thermomagnetic treatment (TMT) + step-aging, 3) continuous cooling. It has been found that the magnetic properties of the alloy are very sensitive to the temperature of the TMT. Step-aging gave the best magnetic properties, producing an elongated ferromagnetic phase, 300Å in diameter and 1200Å in length. Lorentz microscopy revealed domain walls and these lie within the Cr-rich phase and pinned by the Fe-rich phase in the isothermally aged alloy at 650°C. Magnetic domains of optimally step-aged alloys, 0.5μm in width, are elongated along the direction of the applied magnetic field. The results suggest that the magnetic anistropy is introduced parallel to the direction of the applied magnetic field during TMT and step-aging treatments.

INTRODUCTION

Fe-Cr-Co-V alloys are potential ductile permanent magnets with properties comparable to those of Alnico 5, which can be easily heat treated to produce optimum properties (1). Previous work has concentrated on the base ternary Fe-Cr-Co alloy in which the microstructures and phase relationships are well characterized(2,3). The present investigation describes the microstructural changes of an Fe-23wt%Cr-15wt%Co-5wt%V alloy with various heat treatments, (isothermal aging, thermomagnetic treatment, step-aging and continuous cooling) using electron microscopy. Since magnetic hardening of the alloy occurs on a very fine scale (3), transmission Lorentz electron microscopy has been used to study the magnetic domain structures, in an attempt to understand the magnetization reversal process of the alloy.

EXPERIMENTAL PROCEDURES

An Fe-23wt%Cr-15wt%Co-5wt%V alloy was homogenized at 1000°C for 1 hour in an argon atmosphere and quenched in ice water. The specimens were given various heat-treatments, and the magnetic properties of the specimens were measured with an automatic flux meter. Specimens for electron microscopy were thinned in an automatic jet polisher using an electrolytic solution of 23% perchloric acid and 77% acetic acid. Magnetic domain walls and domain configuration of the specimens were observed by the out-of-focus and displaced aperture methods (4,5).

RESULTS AND DISCUSSION

A. Microstructure

(1) Isothermal aging. The bright field micrographs shown in Fig.1 are taken from the alloy aged for 1 hour at 660°C, 650°C, 640°C and 630°C, respectively. The phase with bright contrast is the Fe-rich phase (α_1) and the one with dark contrast is the Cr-rich phase (α_2)(3). These micrographs suggest that the morphology of the microstructure and the volume fractions of the two phases are very sensitive to the aging temperature, i.e., the lower the aging temperatures, the finer the α_1 phase. The α_1 phase appears as rod shaped particles which become interconnected after aging at 640°C. These results are important in understanding the effect of thermomagnetic treatment (TMT) of the alloy at these temperatures.

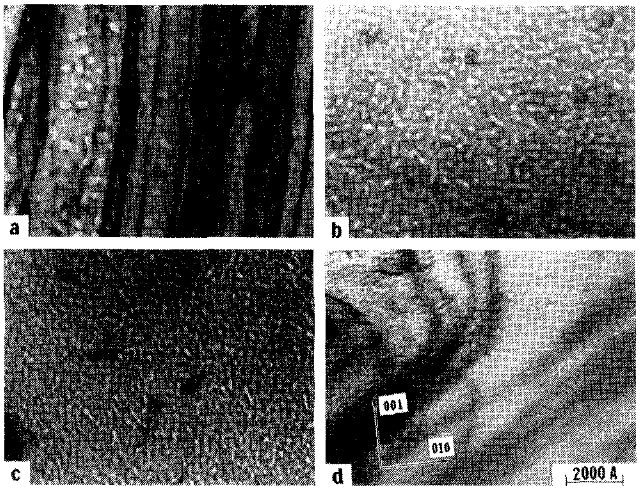

Fig. 1. Bright field (B.F.) micrographs taken from the isothermally aged alloy for 1 hour at, (a) 660°C, (b) 650°C, (c) 640°C and (d) 630°C.

(2) Thermomagnetic treatment and step-aging. It is reported that thermomagnetic treatment and step-aging have a beneficial effect on improving the magnetic properties of the system (1,3,6). Four different temperatures of thermomagnetic treatment (TMT) were chosen to investigate the effect of the TMT temperature on the magnetic properties and their microstructures. After TMT in a magnetic field of 2 KOe, the alloy was step-aged at 620°C, 600°C, 580°C and 560°C for 1 hour and subsequently aged at 540°C for 5 hours.

Fig. 2 illustrates the B.F. micrographs of the step-aged alloy after different TMT at, (a) 660°C (Hc∿420 Oe), (b) 650°C (Hc∿520 Oe), (c) 640°C (Hc∿370 Oe) and (d) 630°C (Hc∿80 Oe), respectively. The coercive force of the alloy is remarkably affected by the temperature of the TMT. For example, Fig. 2(a) shows two morphologies of the α_1 phase, viz., elongated α_1 particles, 300Å in diameter, and spherical α_1 particles, 135Å in diameter. Since the larger α_1 particles are elongated, they should be formed during TMT, whilst the small α_1 phase must be nucleated after TMT. These morphologies are produced when the step-aging interval (ΔT) between $T_{step(n)} - T_{step(n-1)}$ is large (3). Fig. 2(b) corresponding to the optimum properties shows the α_1 phase, approximately 300Å in diameter and 1200Å in length, giving an aspect ratio of 4. In Fig. 2(c) and 2(d), the rod diameter is about 200Å and 140Å, and the length about 400Å and 220Å, respectively. The fine spherical particles are absent in Fig. 2(c) and 2(d). This is believed to be due to the fact that the step-aging interval is small.

It is concluded that the morphology of microstructure and the shape or size of the ferromagnetic phase is very sensitive to the TMT temperature, resulting in different magnetic properties. This emphasizes that careful temperature control is needed to produce good magnetic properties.

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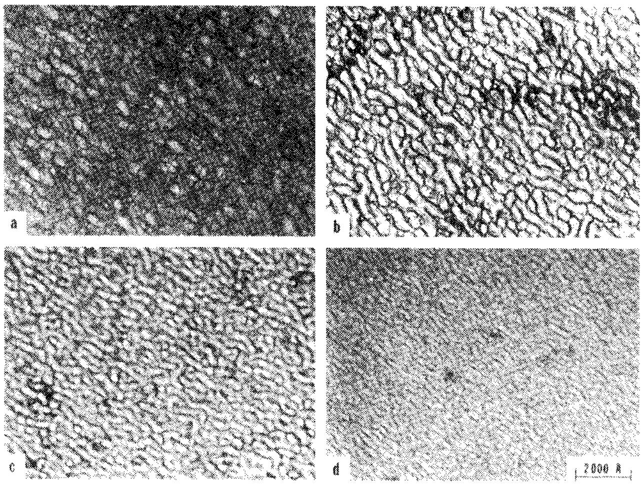

Fig. 2. B.F. micrographs taken from the step-aged alloy after thermomagnetic treatment for 1 hour at, (a) 660°C ($H_c \sim 420$ Oe), (b) 650°C ($H_c \sim 520$ Oe), (c) 640°C ($H_c \sim 380$ Oe) and (d) 630°C ($H_c \sim 80$ Oe).

(3) Continuous cooling. The step-aging process can be facilitated by continuous cooling, giving optimum magnetic properties (1). In order to study the effect of continuous cooling rate on the magnetic properties and their microstructures, the alloy was thermomagnetically treated at 650°C for 1 hour since 650°C is the best temperature for TMT, and then continuously cooled to 540°C.

Fig. 3 shows the micrographs of the alloys continuously cooled at the rate of (a) 1°C/min ($Hc \sim 220$ Oe) and of (b) 0.25°C/min ($Hc \sim 520$ Oe). These micrographs suggest that the morphology of the microstructure appears to be very similar, almost independently of the cooling rate. Since both specimens have the same TMT, the morphology of the microstructure must be established during TMT. This observation is similar to those observed in Alnico alloys (7).

The coercive force of the fast cooled (1°C/min) specimens can be remarkably increased from 220 Oe to 520 Oe by isothermal aging at 540°C for 14.5 hours after continuous cooling. But the coercive force of optimally cooled specimen (0.25°C/min) increases from 520 Oe to only 590 Oe by the same treatment. It has been observed that there is no noticeable difference in morphology of the microstructures between continuously cooled and low temperature aged alloys after continuous cooling. These results imply that the composition of the two phases differs depending on the continuous cooling rate, giving different coercive forces.

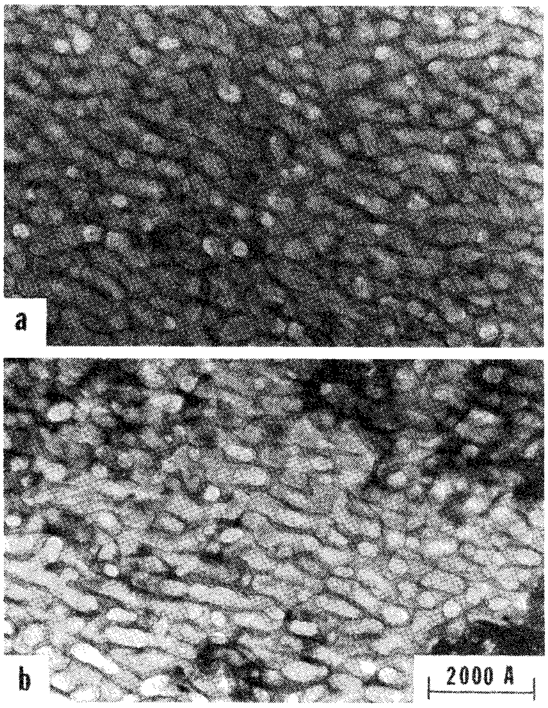

Fig. 3. B.F. micrographs taken from the continuous cooled alloys after TMT at 650°C for 1 hour at a rate of, (a) 1°C/min ($H_c \sim 220$ Oe) and (b) 0.25°C/min ($H_c \sim 520$ Oe).

Therefore, there are two alternative methods to produce the optimum properties in this alloy. One is by continuous cooling at a rate of 0.25°C/min. The other is by cooling at a rate of 1°C/min and subsequently aging at low temperatures for long times.

E. Domain Structures

Fig. 4 shows the domain wall of the isothermally aged alloy at 650°C for 1 hour, imaged by the defocus method. The domain walls appear to be straight. From this micrograph it is uncertain where the domain wall exactly lies. To solve these uncertainties, the alloy was further aged at 650°C for 50 hours, growing the α_1 particle from 150Å (in Fig. 4) to 900Å in diameter.

Fig. 5 shows the Fresnel micrographs of the overaged alloy. The domain wall with black contrast (divergent wall) lies within the α_2 matrix phase. This stems from the fact that the domain wall energy of the α_2 phase is lower than that of the α_1 phase. After photographing Fig.5(a), the specimen was taken out from the microscope and was put in a magnetic field. Then it was observed that the domain wall changed its position before (Fig.5(a)), and after (Fig.5(b)) applying the magnetic field. In Fig.5(b), the domain wall exists in the

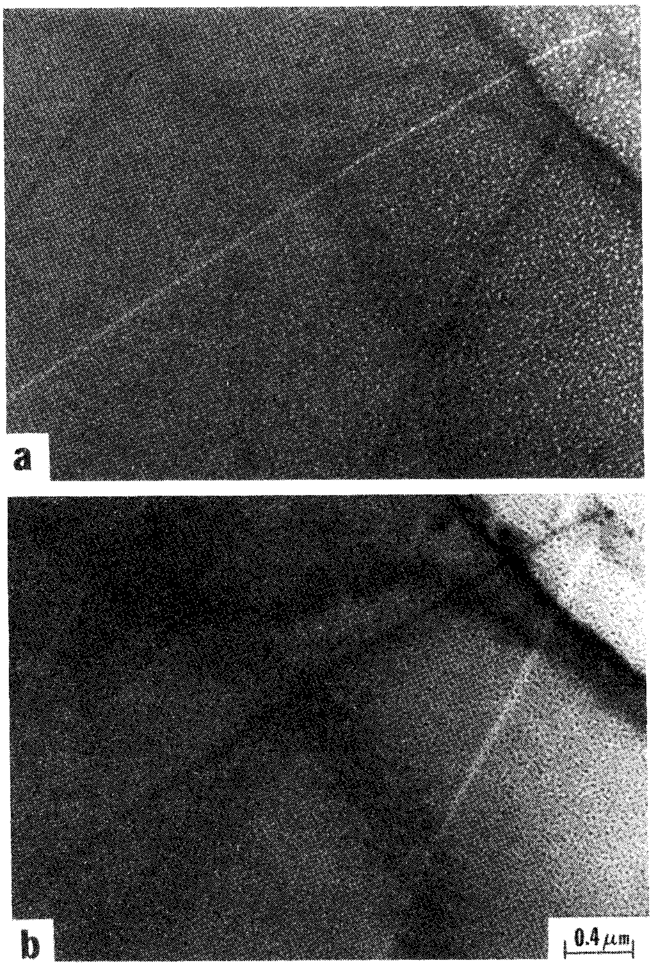

Fig. 4. Fresnel micrographs taken from the alloy aged at 650°C for 1 hour.

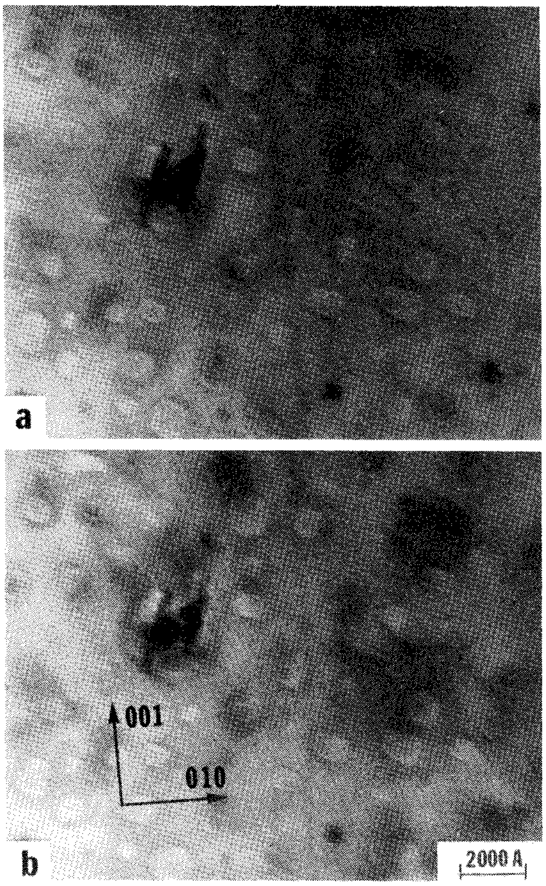

Fig. 5. Fresnel micrographs taken from the alloy aged at 650°C for 50 hours, showing the domain wall is pinned by the α_1 particle ($H_c \sim 50$ Oe).

α_2 phase and is slightly bent around the α_1 particles. These figures substantiate that domain walls are pinned by the α_1 phase. It is concluded that the magnetization process of the isothermally aged alloys is due to domain wall pinning. Thus the coercive force would be given by the difference in wall energy of the two phases; $H_c \propto (W\alpha_1 - W\alpha_2)$ where $W\alpha_1$ is the wall energy of the α_1 phase and $W\alpha_2$ that of the α_2 phase. This mechanism has also been proposed for Sm(Co, Cu, Fe)$_7$ magnets(8).

Fig. 6(a), 6(b) are the Fresnel micrographs of the optimally step-aged alloy, showing a domain wall, and Fig. 6(c), 6(d) are the Foucault micrographs (displaced aperture method). The observed domain wall is not straight. The domains are approximately 0.5μm wide, elongated along the direction of the applied magnetic field, and their size is smaller than that of the isothermally-aged alloy. The Foucault micrograph with higher magnification, as shown in Fig. 7 illustrates these features more clearly. The domain with black contrast is almost 1500Å wide and is elongated in the direction of the applied magnetic field (the direction in which the phase is elongated). These observations suggest that the magnetic anisotropy is introduced parallel to the direction of the applied magnetic field after TMT and step-aging. Since the contrast mechanism of the magnetically inhomogeneous material (α_1 phase and α_2 phase) in Lorentz microscopy is complex, more experiments are needed to interpret the nature of the imaged domain walls in detail.

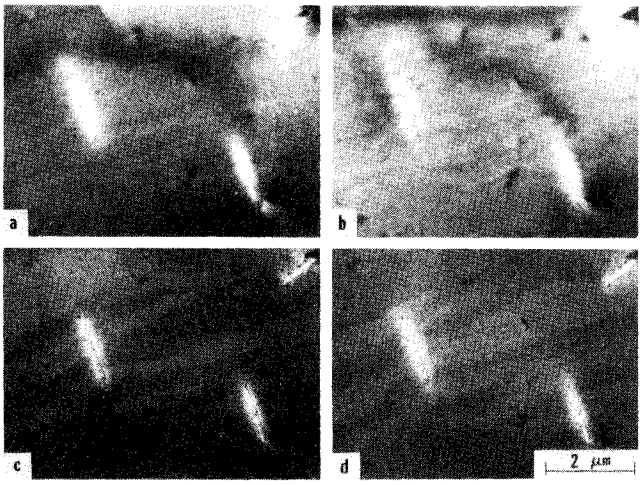

Fig. 6. Fresnel [(a), (b)] and Foucault micrographs [(c), (d)] of the step-aged alloy after TMT at 650°C for 1 hour ($H_c \sim 520$ Oe).

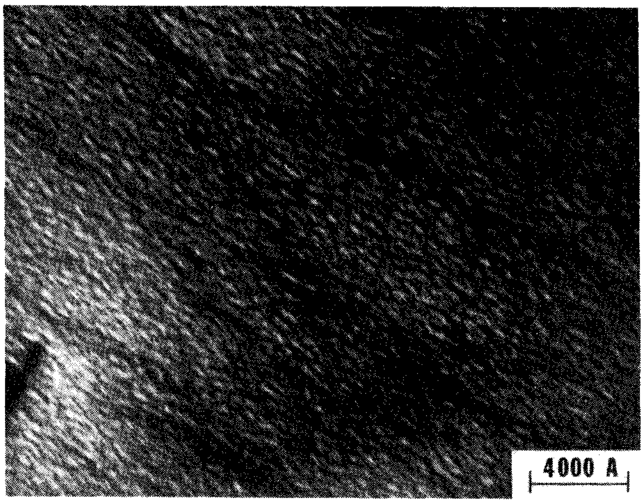

Fig. 7. Foucault micrographs of the step-aged alloy after TMT at 650°C for 1 hour.

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* This research is sponsored by the United States Department of Energy through the Lawrence Berkeley Laboratory.

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