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ON TEMPERATURE DEPENDENCE OF DOMAIN STRUCTURE IN COBALT

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The type-I magnetic contrast in a scanning electron microscope is used to study the domain behaviour on the basal planes of cobalt monocrystals of different thicknesses during a heating cycle. Digital image processing is applied to the original scanning electron microscope images for their restoration, enhancement and analysis. The main reasons for the application of digital image processing are: low level of type-I magnetic contrast, particularly used at the higher temperatures, and the complex character of the magnetic domain structure. The changes in both domain structures and type-I magnetic contrast are due to the strong temperature dependence of magnetocrystalline anisotropy energy for the hcp phase of cobalt, which implies the rotation of magnetic easy axis from the c-axis to the basal plane. The temperature of the magnetic phase transition between an open-flux and a closed-flux domain configuration was found to be dependent on the specimen thickness. The changes in domain structure during the heating cycle were reversible under the condition that the specimen was not carried through the hcp-to-fcc phase transition. Otherwise, they were partially or completely irreversible and were caused by crystal imperfections originating from the transition. The paper shows the great advantage of using digital image processing system for data restoration, enhancement and analysis.

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1. Introduction

The domain structure of cobalt monocrystals can be observed in the scanning electron microscope (SEM) by means of the type-I magnetic contrast. This contrast arises when stray magnetic fields above the specimen surface are sufficient to influence the trajectories of the emerging low-energy secondary electrons, the latter being interaction product of the incident primary electron beam with the specimen [1, 2].

Because of the strong temperature dependence of the magnetic anisotropy energy in cobalt, a change of the magnetic domain structure occurs when the temperature is increased. This change can be treated as a magnetic phase transition between the open domain structure (i.e. with stray fields above the specimen surface) and the closed one (i.e. without stray fields). The purpose of the present paper is to study the temperature changes in the domain structure of cobalt monocrystals, by registering type-I magnetic contrast in the SEM.

2. Experimental

2.1. Digital image processing system

Because of the decrease in type-I magnetic contrast with increasing temperature and the complex character of the domain structure on the basal surface, a digital image processing system was built and applied to the SEM images for their restoration, enhancement and analysis [3–5]. The system consists of an analogue-to-digital converter (ADC), an IBM PC computer, and software. The ADC is used for acquisition of the analogue image from the SEM. The digital image consists of 256 × 256 image points and is displayed at 16 grey levels on the computer monitor.

2.2. Specimen preparation and experimental conditions

A few specimens of cobalt monocrystals were cut from the same sample, oriented by means of an X-ray diffractometer. The observation surface was smoothed in order to reduce topographic contrast, by mechanical polishing. We used polishing cloths, abrasive papers, diamond and alumina powders produced by PRESI. The specimen was placed on a bifilarly coiled heater with power 30 W produced by VARIAN. An iron–constantan (ANSI Symbol J) thermocouple was used to measure the specimen temperature.

Images were obtained using a Tesla BS 340 SEM at an accelerating voltage 20 kV and an electron beam current 10 nA. To improve the magnetic contrast the specimen was tilted by an angle 25° away from the collector as well as the long time of image recording (20 s) and "trapping" the pump were used.

3. Results and discussion

The specimens studied were heated up to a maximum temperature T_{max} and then cooled down to room temperature. T_{max} was in the range approximately 600–750 K. The results obtained can be divided into three groups related to the reversible, partially irreversible and completely irreversible changes in domain structure during the heating cycle, respectively [5].

Each SEM original image was transformed and analysed with a digital image processing system in the same manner as described in [4, 5]. This allowed us to obtain images with a very good contrast (Fig. 1) and calculate the corresponding statistical distributions of domain widths (Fig. 2).

As the temperature is increased, the first anisotropy coefficient K_1 becomes equal to zero at 518 K and then decreases to a negative value less than $-2K_2$ (K_2 is the second anisotropy coefficient) above 613 K [6]. This behaviour is responsible for the change of magnetocrystalline anisotropy from the uniaxial to the planar, and thus for the corresponding re-arrangement of the domain structure. In all the

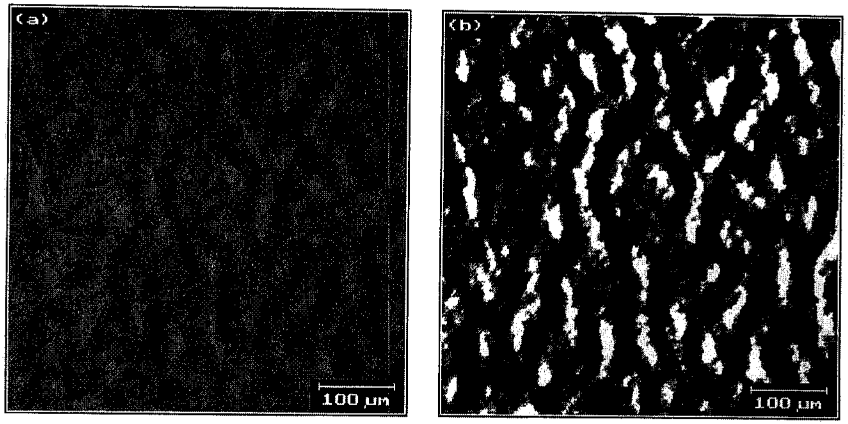

Fig. 1. The magnetic domain structure of a cobalt monocrystal at 464 K during the heating process. (a) SEM original image; (b) image with enhanced contrast obtained after applying a simple digital procedure to the original image.

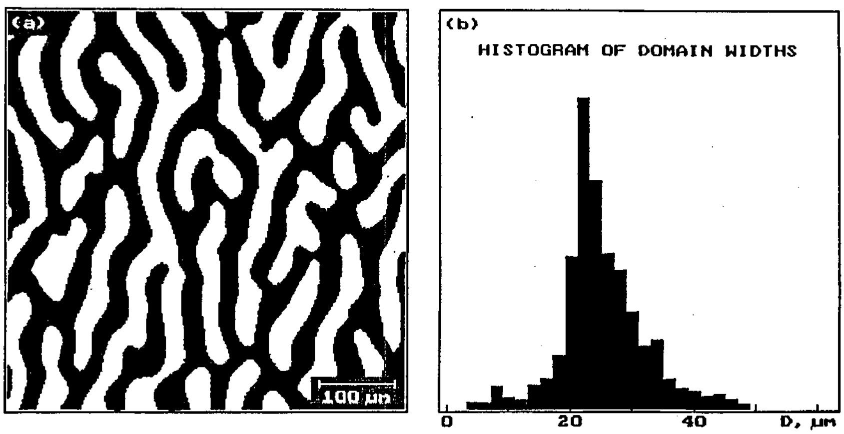

Fig. 2. (a) Image obtained by digital image processing of SEM image from Fig. 1a; (b) corresponding statistical distribution of magnetic domain widths.

cases studied the magnetic phase transition between an open-flux domain structure and a closed one was reversible during the heating cycle. The temperature of the transition was thickness dependent and decreased from about 530 K to 500 K as the crystal thickness increased from approximately 0.7 mm to 1.9 mm. Simple theoretical considerations show that for cobalt at room temperature the energies of Kittel parallel-plate domain structure and of Landau–Lifshitz closed domain configuration are nearly the same [7]. The magnetic phase transition is therefore expected to occur at temperature lower than that at which the process of the magnetization rotation from the hexagonal axis to the basal plane is finished. This prediction is confirmed by the obtained experimental results. As the temperature was increased, under the conditions of reduced anisotropy energy we observed a slight increase in the domain width and a decreasing number of reverse spikes followed by the formation of a closed domain structure. For specimen thicknesses greater than about 1.4 mm the magnetic phase transition took place at temperature lower than 518 K, at which the mentioned rotation of the magnetic easy direction only starts.

TABLE

The experimental results obtained for the specimens of different thicknesses during the heating cycle. (1), (2) and (3) mean the reversible, partially irreversible and completely irreversible changes in domain structure, respectively.

Specimen thickness [mm]	0.72	0.80	0.89	1.17	1.37	1.52	1.67	1.73
Character of changes in domain structure	(1)	(3)	(2)	(1)	(1)	(1)	(3)	(2)
Maximum temperature [K]	632	729	743	610	655	662	705	694

The reversible or irreversible character of the domain behaviour during the heating cycle is related to the martensitic transition between the hcp and the fcc phase of cobalt at about 690 K. This transition produces intrinsic lattice imperfections, i.e. dislocations and point defects. The dislocations, due to their strong magnetostrictive interactions with the domain walls, act as effective obstacles to domain wall mobility. A growth of lattice imperfections with each subsequent heating cycle was also observed [8]. On the basis of Table we can see that the domain behaviour during the heating cycle was reversible as the specimen was not carried through the hcp-to-fcc phase transition. Otherwise, the changes in domain structure were partially or completely irreversible.

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