

DOMAIN OBSERVATION OF A CoCr FILM BY THE COLLOID-SEM METHOD

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The domain structure of a CoCr film was studied in different magnetic fields using the method of a combination of the colloid technique and scanning electron microscopy (colloid-SEM method). The results obtained are presented and the advantages of this method are discussed.

1. Introduction

CoCr films are most promising as a perpendicular magnetic recording medium. Recently, Yamamoto et al. [1] have reported D50 values of over 250 kbp. In order to understand the recording performance in more detail, it is necessary to study the magnetization reversal. Observing the domain configuration can contribute to the solution of this problem.

A report on the domain structure of different CoCr layers was first published by Schmidt and Hubert [2] using a digital image processing system connected to a Kerr microscope. They observed mostly stripe domains at remanence, for all their samples. Using the same microscope Lodder et al. [3] came to the conclusion that it is useful to divide the CoCr layers into 3 categories namely low ($H_c/H_k \approx 2\%$), medium ($H_c/H_k \approx 5\%$) and high ($H_c/H_k \approx 10\%$) coercivity films. Their layers showed domain structures with long, short and a dot-like configuration, respectively. These structures are clearly connected with the macroscopical vibrating sample magnetometer (VSM) hysteresis loop via the coercive force and the so-called "shoulder" on the magnetizing curve. By studying the VSM loop as a function of the angle and

amplitude of the applied field the difference between low and high coercivity samples is also clearly shown [4].

In ref. [5] the Kooy and Enz model [6] is used for comparison with the experimental results obtained from the initial slope of the hysteresis loop and the domain period measured from the Kerr observations. For low H_c/H_k films the stripe theory is in very good agreement, but for the high coercivity films the measured and calculated values are not.

The aim of this paper is to introduce the combined colloid-SEM method for observing the domain structure in CoCr films. This method was first used by Goto et al. for the domain observation of hexagonal cobalt [7] and $\text{BaFe}_{12}\text{O}_{19}$ [8]. Later, Šimšová et al. [9] verified the possibility of observing submicron domains in garnet layers in this way. Goto et al. [10] also used this method for making visible high density perpendicular magnetic recording in CoCr films. The observation was performed on Bitter patterns dried in a magnetic field of 16 kA/m.

In this paper we report on the observations of the domain structure of a CoCr film on both the ascending (virginal) and the descending branch of the hysteresis loop. The drying of the colloid was

carried out in different magnetic fields up to 320 kA/m permitting us to measure the changes of the domain period as a function of the applied field.

2. Experimental

The CoCr layer is deposited on an Si substrate by rf magnetron sputtering using a 3" (7.6 cm) target, back pressure 1.5×10^{-7} mbar and $p(\text{Ar}) = 6 \times 10^{-3}$ mbar. The sample (22 at% Cr) is characterized by the thickness $h = 300$ nm (XRF), magnetization $M_s = 420$ kA/m (VSM) and a first-order uniaxial anisotropy constant $K_1 = 1.42 \times 10^5$ J/m³ (torque magnetometer).

The preparation of the sample for the domain observation consists of:

a) putting a small droplet of colloid into a 2 mm diameter ring and drying it in a magnetic field perpendicular to the sample surface, and

b) covering the dried Bitter patterns with an evaporated gold film to prevent charging of the surface in the scanning electron microscope. The observation of the dried Bitter patterns was performed using a JEOL JXA 733 SEM, operated at 15 kV.

For our observations various colloids were tested. Appropriate filtering and diluting with distilled water was necessary for observing submicron domains. Ultrasonic vibration applied to the colloid before use made some improvement to the quality of our photographs.

3. Results and discussion

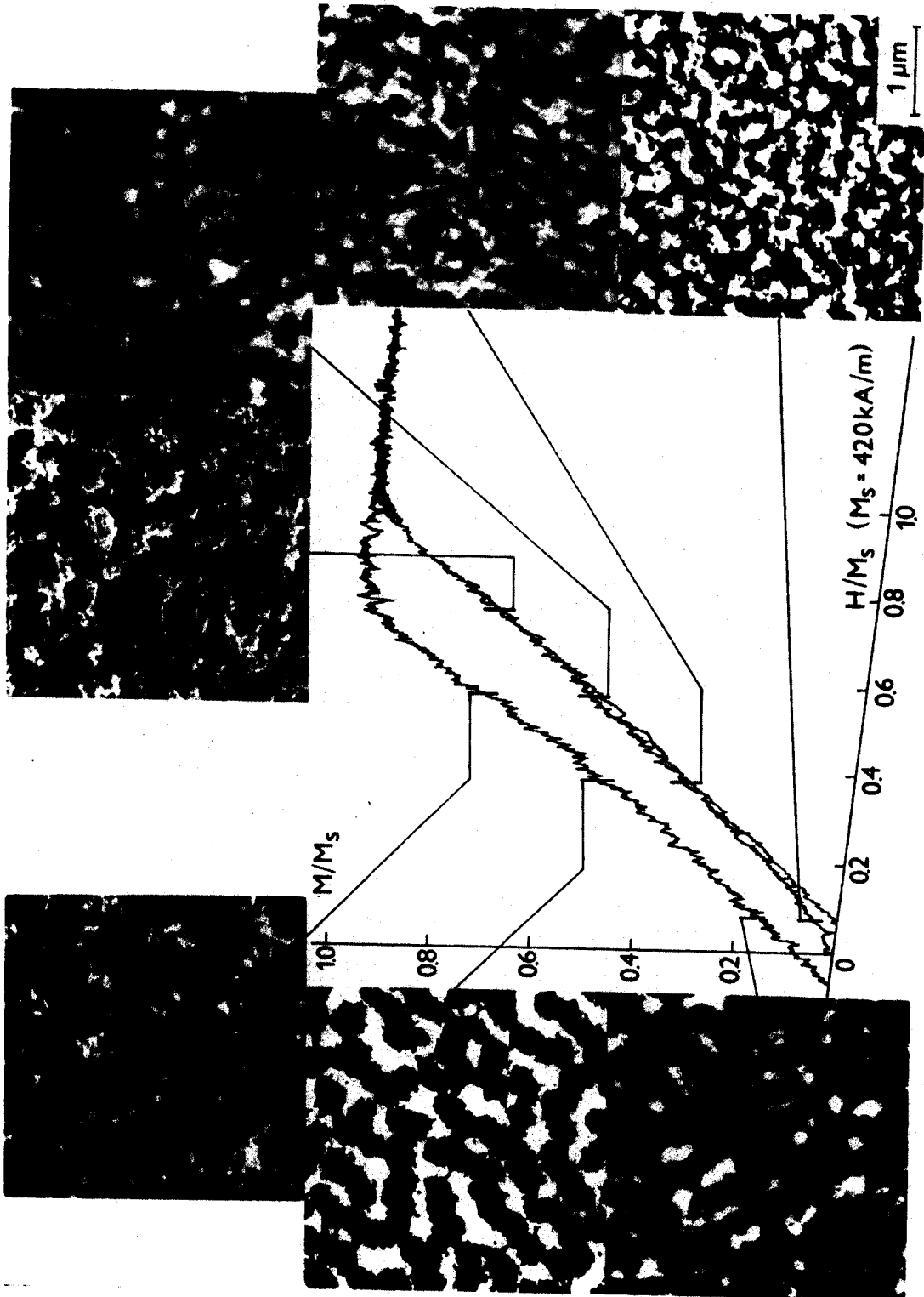
The domain structure of the sample was investigated for the dependence of its magnetic history (see table 1 and fig. 1). Table 1 contains the

experimental values $d_1 + d_2$ and the corresponding values normalized with respect of the extrapolated period in zero field $2d_0 = 285$ nm. When measuring on the ascending branch of the hysteresis loop the sample was first demagnetized in an ac field applied normal to its plane. Starting with an amplitude of 64 kA/m the field was slowly decreased to zero. The colloid was then placed on the surface and dried in a dc field having values of 32, 160, 240 and 320 kA/m also applied normal to the sample plane. To measure on the descending branch of the loop the sample was saturated in a normal magnetic field of 480 kA/m with a drop of colloid on the surface; the field was then reduced to 240, 160 and 32 kA/m successively allowing the colloid to dry in the respective field.

The domain structure was thus studied at different points of the hysteresis loop as shown in fig. 1. Since all the magnetic fields applied were large enough for the colloid particles to settle on those domains in which the orientation of the magnetization is the same as that of the applied field (see, for example, ref. [10]) the value of domain period $d_1 + d_2$ was determined as the distance between two neighbouring Bitter patterns. The values of the domain period determined directly from the photographs are summarized in table 1. The error in determining the period is above all affected by the quality of the colloid used. It is, however, larger on the descending branch of the hysteresis loop. In order to exclude a subjective error in the determination of the value of the domain period, Fourier analysis of the digitalized photographs was carried out using a Pericolor image analyser. The Fourier frequencies were determined from two or three areas (128 × 128 pixels). The shape of the spectra reflects the Fourier frequencies from which the dominant period $d_1 + d_2$ in the picture can be calculated as $d_1 + d_2$ (μm) = 128/aL. Here

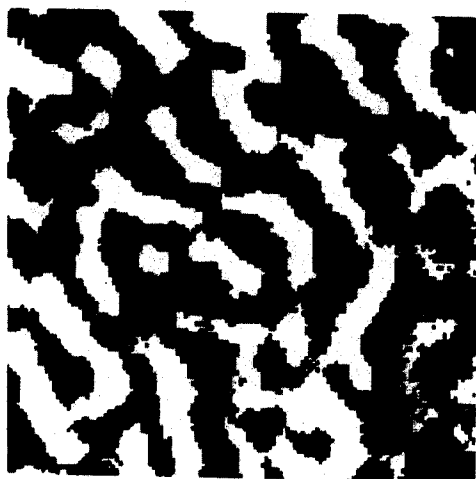
Table 1
Experimental values for the ascending and descending branch

	Ascending branch				Descending branch			
	32	160	240	320	240	160	32	
H (kA/m)	32	160	240	320	240	160	32	
H/M_s	0.076	0.38	0.57	0.76	0.57	0.38	0.076	
$d_1 + d_2$ (nm)	290 ± 30	280 ± 30	310 ± 30	410 ± 30	660 ± 50	530 ± 50	430 ± 50	
$(d_1 + d_2)/2d_0$	1.02 ± 0.11	0.98 ± 0.11	1.09 ± 0.11	1.44 ± 0.11	2.32 ± 0.18	1.86 ± 0.18	1.51 ± 0.18	



a is the distance of the dominant Fourier frequency from the centre of the spectra in pixels (larger semi-axis of the ellipse) and L is the length of 1 μm in pixels. Agreement with the direct measurements of $d_1 + d_2$ is obtained when assuming that the larger semi-axis of the ellipse corresponds to the domain period (see fig. 2). Based on preliminary experiments, it is expected that this will be a useful method for determining the domain period. The possibilities and accuracy of this method will be discussed in a future paper.

Fig. 3 shows the experimental dependence of the domain period $d_1 + d_2$ on the applied field H in reduced coordinates for both the ascending and descending branches of the hysteresis loop. It also



1 μm

47 pixels

Fig. 2. The digitalized printed photograph of the domain structure of a CoCr film at $H = 240 \text{ kA/m}$ (ascending branch) and the visualized Fourier spectra (not to scale).

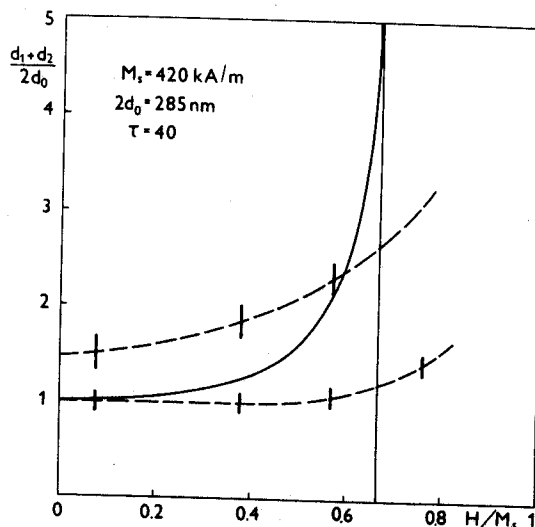


Fig. 3. The dependence of the domain period $d_1 + d_2$ on the external field H for $\tau = 40$ in reduced coordinates. The curve in full line is theoretical, calculated according to refs. [6,11]. The vertical bars are our measurement as shown in table 1.

gives a plot of the theoretical dependence for the reduced thickness $\tau = 9.38(h/d_0)^2 \doteq 40$ (which is the value for our film) calculated on the basis of the theory of Kooy and Enz [6] and its mathematical solution by Duijvestijn and Boonstra [11]. As can be seen the experimental points do not quite follow the theoretical curve, the period values on the ascending and the descending branches of the hysteresis loop are shifted vertically with respect to each other and, in addition, the saturation field as obtained from the measured hysteresis loop (see fig. 1) is much higher than the theoretical one ($H_s/M_s = 0.666$).

A similar and even more pronounced behaviour is shown in a recent paper by Lodder et al. [5] where the high coercivity specimen no. 6 does not show a tendency to saturate even at fields around $0.8M_s$, while the low coercivity specimen no. 1 (with long stripes) was in fair agreement with the theoretical curve. This behaviour suggested the possibility that the saturation field depended on the domain shape. We therefore calculated the collapse field for an isolated infinite stripe domain, H_{cl} , and an isolated bubble domain, H_{cb} , according to ref. [6] and obtained (in relative units) $H_{cl}/M_s = 0.64$ ($H_{cl} = 270 \text{ kA/m}$) and

$H_{cb}/M_s = 0.72$ ($H_{cb} = 300$ kA/m), respectively. Although the bubble domain has a higher collapse field than the stripe domain it is still too low to explain the difference in the behaviour of the long and short stripes or dot-like domains [5] quantitatively.

We may conclude that the domain shape is not the primary reason for the disagreement. This might be caused by the coercive force which in turn will be conditioned by structural defects.

4. Conclusion

The combined colloid-SEM method was used for the first time to study the submicron domain structure in CoCr films as a function of the applied field. Compared to Lorentz electron microscopy [12] the colloid-SEM method is not limited by the thickness of the film. On the other hand, however, it does not achieve the resolution of the former technique. Compared to the digitally enhanced Kerr microscope [2] the colloid-SEM method simplifies the observation of the domain pattern belonging to the initial magnetization curve. This is problematic with the Kerr microscope because it depends on the correct use of the necessary reference image. Also the somewhat puzzling dependence of the observed domain period on the wavelength of the light used (see ref. [2], fig. 11) sets a limit to the use of the Kerr method. The resolution of the colloid-SEM method is comparable or better than that of the digitally enhanced Kerr method. The measured dependence of the domain period proved the expected qualitative agreement with the theoretical predictions of Kooy and Enz [6]. The quantitative agreement is rather poor, however, the measured saturation field is much higher than that calculated for stripe or cylindrically shaped domains. The coercivity which pins domain walls in the material and which is not included in the theory [6] certainly contributes to

the above disagreement. It also gives rise to the observed hysteresis of the domain period.

Observations of the in-plane hysteresis loop shows a very clear jump around zero field which can be attributed to the initial layer of the CoCr film with the substrate. It has already been shown that this layer influences the reversal mechanism [13]. The extent to which this can be seen in the observed domain period has not yet been determined.

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