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Domain evolution during the spin-reorientation transition in epitaxial NdCo₅ thin films

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Abstract. The domain structure and its changes with temperature were investigated for an epitaxial NdCo₅ thin film with in-plane texture in which a spin-reorientation transition takes place from the easy *c*-axis via the easy cone to the easy plane. Scanning electron microscopy with polarization analysis reveals a transition from a two-domain state at temperatures above 318 K via a four-domain state back to a 90°-rotated two-domain state at temperatures below 252 K. The transition temperatures correspond well to those determined by global magnetization measurements. The magnetization configuration at the three different regimes of magnetic anisotropy and its transition with temperature were analysed in detail. From the local measurements, the spin-reorientation angle and the magnetocrystalline anisotropy constants of first and second order were derived.

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1. Introduction

Alloys of cobalt and rare earth (RE) metals generally possess a strong magnetocrystalline anisotropy and usually a high Curie temperature. Furthermore, for several of such intermetallic phases the individual contributions of the cobalt- and the RE metal sublattices to the magnetocrystalline anisotropy lead to a temperature-driven spin-reorientation transition (SRT). This is due to the fact that the anisotropy of the RE sublattice decreases strongly with increasing temperature owing to the weak RE–RE exchange interaction, J_{RR} , whereas the Co sublattice anisotropy is maintained even at high temperatures ($J_{\text{CoCo}} \gg J_{\text{RR}}$). At higher temperatures, the uniaxial anisotropy of the Co sublattice (parallel to the crystallographic c -axis) will always dominate, while a possible basal plane anisotropy of the RE subsystem may take over at lower temperature. This leads to a temperature-driven change in the easy magnetization direction known as SRT. Upon cooling down, an SRT with an opening of a magnetic easy cone takes place, e.g. in PrCo_5 , where it was investigated in bulk [1–4] as well as in epitaxial thin film samples [5]. A complete SRT from a magnetic easy c -axis via a magnetic easy cone to a magnetic easy plane in the ab -plane of the hexagonal crystal is observed in NdCo_5 . Additionally, in the easy basal plane the a -axis is energetically preferred over the b -axis [2, 6–8]. This third-order anisotropy effect is, however, not investigated in the present study. Like in the case of PrCo_5 , for NdCo_5 the SRT was initially observed in bulk samples [2, 6, 7, 9–12], where it takes place between 280 and 240 K. We recently succeeded in preparing epitaxial NdCo_5 films on $\text{MgO}(110)$ and $\text{Al}_2\text{O}_3(0001)$ substrates with an in-plane and out-of-plane texture, respectively [13, 14]. For the in-plane samples the transition temperatures were determined as 310 and 255 K, while for the out-of-plane samples the transitions were qualitatively confirmed, but the exact temperatures were not accessible due to the influence of the shape anisotropy. For the in-plane textured film on $\text{MgO}(110)$ investigated here, a sketch of the temperature-dependent easy axis of magnetization and the alignment of the crystal structure with respect to the substrate is shown in figure 1. Due to the shape anisotropy of a thin film, the different possible orientations along the surface of the easy cone or within the easy plane are no longer energetically identical. Instead, the intersection of the surface of easy magnetization (magnetic easy cone or magnetic easy plane) with the film plane defines a set of new easy magnetization directions for each temperature. They nevertheless form the same spin-reorientation angle with the crystallographic c -axis as in the case of a bulk sample.

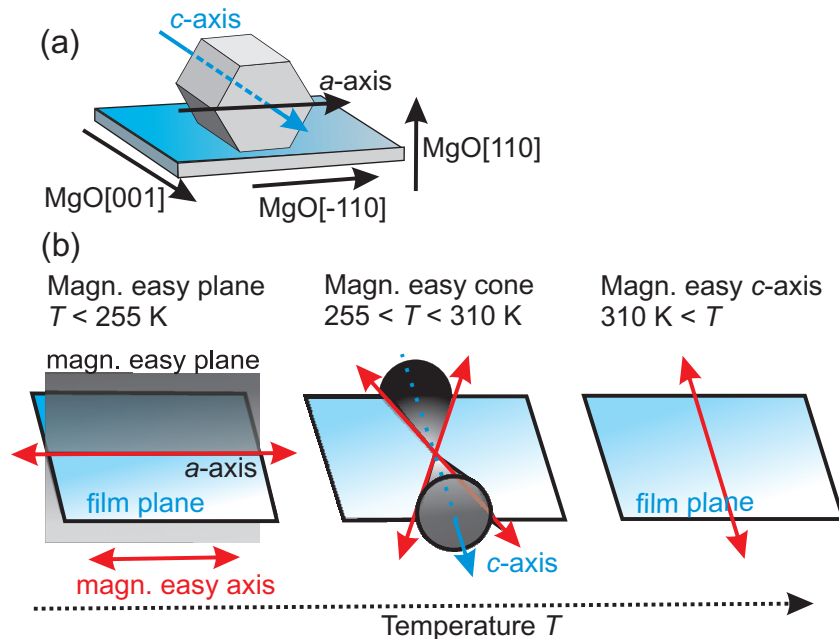


Figure 1. (a) Sketch of the texture relation NdCo₅–MgO[110] substrate. (b) Magnetic easy directions as a function of temperature.

Despite this clear picture, which is derived from global magnetization measurements [13], there are open questions concerning the domain structure in the three regimes of magneto-crystalline anisotropy and concerning the changes of the domain pattern when cooling down through the spin-reorientation regime. The case of complex magnetocrystalline anisotropies such as the easy cone or the easy plane is much less investigated than domain processes in uniaxial materials. There are no domain observations of the SRT on NdCo₅ or other RE–transition metal (TM) films, and microscopic SRT studies on bulk RE–TM samples are rare [15, 16]. The question of first- and second-order phase transition is tackled by global magnetization measurements on Nd_{1–x}Y_xCo₅ single crystals, but is not unambiguously answered [17]. All this calls for a direct observation. A very useful technique to investigate the domain structure at the surface of magnetic samples is scanning electron microscopy with polarization analysis (SEMPA), which we have previously applied to study the stray-field-driven SRT in Co/Au(111) [18] and Co/Pt multilayer films [19]. With an appropriate detector geometry it allows determining the in-plane direction of magnetization at the film surface. Since with our particular instrument the temperature of the sample can be set between 370 and 40 K, it is well suited for the investigation of the domain pattern and its changes during the SRT in in-plane textured NdCo₅ films.

2. Experiment

A NdCo₅ thin film has been fabricated by alternating pulsed laser deposition from pure neodymium and cobalt targets. The film with a thickness of 60 nm was grown on a heated MgO(110) single crystal which was covered with a 15 nm thick chromium buffer layer. On top, a 10 nm chromium cap layer was deposited to prevent oxidation. The deposition temperature

of 500°C is below the Curie temperature of the material, so that the magnetic domain structure already starts forming during deposition and relaxes into a thermally demagnetized state during cooldown to room temperature. X-ray diffraction measurements and pole figure measurements confirm a hexagonal crystal structure and a single epitaxial orientation of the NdCo₅ crystals with respect to the substrate. The NdCo₅ *c*-axis ([0001]) is aligned in-plane along the MgO[001] direction, the *a*-axis ([10 $\bar{1}$ 0]) is parallel to the MgO[-110] direction and the *b*-axis [1 $\bar{1}$ 00] is oriented out-of-plane (figure 1). An analysis of characteristic Bragg reflections in tilted geometry and an evaluation of the corresponding lattice constants show that the nominal NdCo₅ film is actually composed of a Co-rich Nd_{1-y}Co_{5+2y} and an Nd₂Co₇ phase, both with hexagonal crystal structure and the above-stated epitaxial relation to the substrate [13]. The global magnetic properties were investigated with vibrating sample magnetometry (VSM) in a temperature range between 400 and 20 K. The measurements were carried out parallel to the crystallographic *c*- and in-plane *a*-axis.

The magnetic domain pattern is investigated by means of a SEMPA (or spin-SEM) [20]. The microscope is operated under ultra-high vacuum conditions with a base pressure < 10⁻¹⁰ mbar. Due to the high surface sensitivity of spin-SEM the capping layer has to be removed. This is done *in situ* via Ar ion milling at 1 keV. The polarization asymmetry that is attained after sputtering is about 7%, which is close to the asymmetry value we obtain for a clean Co surface in our microscope. Correcting for the detector sensitivity this indicates an average spin polarization of the low-energy secondary electrons from Co of the order of 32%. Despite of the good vacuum and especially when the sample is at low temperatures, the asymmetry drops relatively fast down to $\approx 3.5\%$ within less than 60 min. The fast decrease indicates a high reactivity of the surface although the polarization signal indicates that Co is dominant in the surface. Apparently, small fractions of a monolayer of adsorbed residual gases have a strong impact on the electronic states in the topmost layers. This has important implications for the investigation as the cleaning of the surface has to be performed during the analysis frequently. Depending on the focus of the study, ion milling has to be performed every 30 min.

3. Results and discussion

3.1. Global magnetization behaviour

Figure 2(a) exemplifies the SRT in the NdCo₅ film by showing the course of the continuously measured temperature-dependent remanent magnetic moment m_r together with discrete values of m_r obtained from hysteresis loops measured at fixed temperatures. The result is taken from [13]. The measurement started at 400 K with the field axis and the sensitivity axis of the magnetometer parallel to the *c*-axis. The magnetization in the remanent state follows the easy magnetization direction and thus the measured values quantify the projection of the easy magnetization direction onto the *c*-axis. The spin-reorientation angle is calculated as $\theta_{SR} = \arccos \frac{m_r}{m_s}$, with m_s being the saturation magnetic moment at a given temperature. Anisotropy constants of first and second order (K_1 , K_2) are derived from additional hysteresis measurements perpendicular to the easy magnetization direction. They have been performed along the in-plane *a*-axis of the NdCo₅ film above 320 K and along the *c*-axis below 240 K. A Sucksmith–Thompson analysis [21], i.e. the derivation of magnetocrystalline anisotropy constants from a hard-axis magnetization loop of a uniaxial material, results in K_1 and K_2 , as seen exemplarily in figure 2(b) for the easy plane case at 200 K. Note that even small deviations

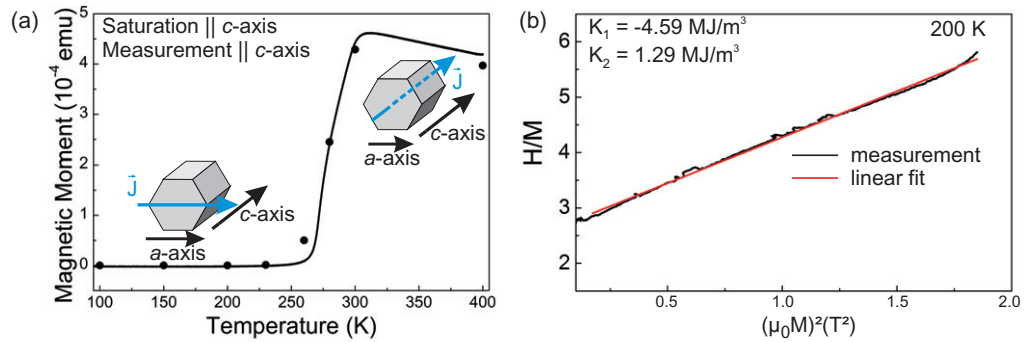


Figure 2. (a) Temperature-dependent magnetization of a NdCo_5 thin film measured in zero field parallel to the c -axis after saturation parallel to this axis at 400 K. The circles represent values of the remanent magnetization taken from hysteresis loops measured at the respective temperatures. The inset sketches illustrate the easy direction of magnetization (see also [13]). (b) Sucksmith–Thompson plot for the measurement at 200 K parallel to the c -axis together with the linear fit ($M = m/V$ with V the volume of the magnetic sample).

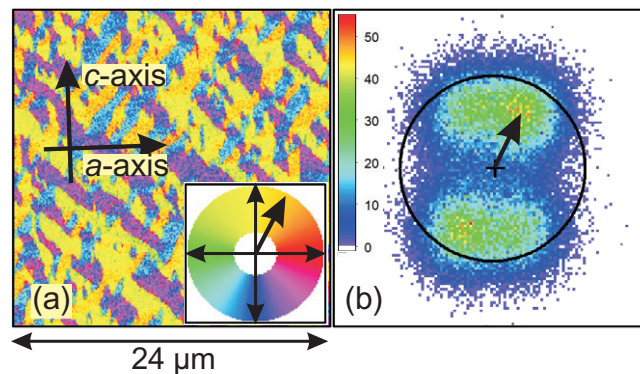


Figure 3. (a) Overview SEMPA image taken of a NdCo_5 thin film at 296 K. The direction of local magnetization is colour coded as given by the colour wheel in the inset. (b) Two-dimensional (2D) histogram of the measured polarization vectors. The black circle corresponds to an asymmetry of 8%.

from a perfect texture can lead to rather large errors in K_1 and K_2 [22]. For the present sample with a texture spread of about $2\text{--}3^\circ$ we estimate an error of up to 15%.

3.2. Microscopic domain investigation of the spin-reorientation transition

The SEMPA image (figure 3(a)) taken at the temperature of 296 K shows an overview of the domain structure. It displays the domain pattern of a NdCo_5 film in the as-grown state by means of a colour-coded in-plane magnetization (see the colour wheel). Magnetic domains are found with a typical size in the range of micrometres. A statistical analysis of the measured asymmetry vectors gives the 2D histogram shown in figure 3(b). In this plot four accumulation

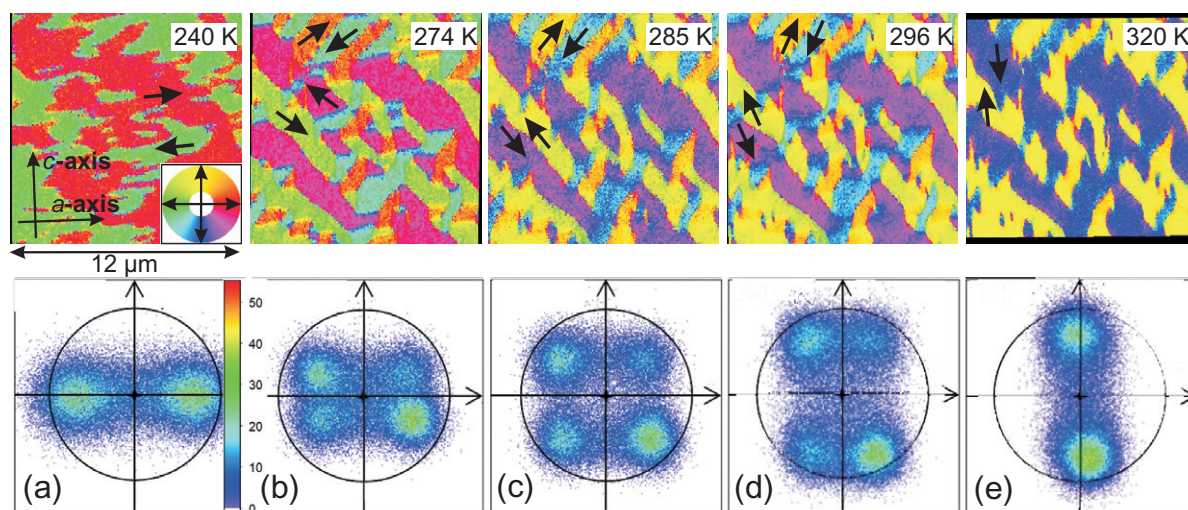


Figure 4. Evolution of the domain pattern in the temperature regime between 240 and 320 K and the corresponding 2D histograms of the measured polarization vectors. The black circle corresponds to an asymmetry of 8%.

points are found, which indicates that the image consists basically of four domain phases with different orientations of magnetization. The vector from the origin (cross) to the position of the accumulation points gives the direction of magnetization, respectively. The black circle corresponds to an asymmetry of 8%.

The evolution of the domain pattern as a function of temperature is presented in figure 4. All images show the same area of the film. A slight lateral shift of about $1\ \mu\text{m}$ has been corrected. At 240 K (figure 4(a)), the magnetization is predominantly aligned along the a -axis. The 2D histogram of the in-plane polarization components exhibits two pronounced maxima indicating two domain phases with opposite directions of magnetization. A similar result is found for 320 K; however, the direction of magnetization is rotated by 90° in the film plane (figure 4(e)). The magnetization is now aligned along the c -axis. (Close observation actually yields a small deviation of about -4° with respect to the c -axis at 320 K. There is also indication of a small fraction of domains remaining at $+4^\circ$, which would hint at the persistence of a tiny splitting at higher temperature. If this proves to be a real effect its origin is unclear at present.) The predominant directions of magnetization above and below the transition interval agree with the global magnetization measurements by vibrating sample magnetometry. In the intermediate temperature range, the magnetization changes its orientation as expected [13]. Exemplarily for the easy cone regime, the domain patterns for selected temperatures (274, 285 and 296 K) are shown in figures 4(b)–(d). The two accumulation points in the 2D histogram at 240 K split into four when the temperature is raised. With increasing temperature they move on a circle until they coalesce again into two accumulation points at 320 K. The histograms demonstrate clearly that the magnetic properties change from uniaxial through biaxial to uniaxial behaviour during the spin-reorientation. Interestingly, the general orientation of the domain walls is preserved during the whole SRT. Although the walls change locally and may even disappear when two domain orientations coalesce into one, in the next heating or cooling cycle they reappear. This localization of the domain walls is most likely caused by a dense network of pinning sites. Such pinning sites are known for nanograined RE-Co films [23, 24] and their presence

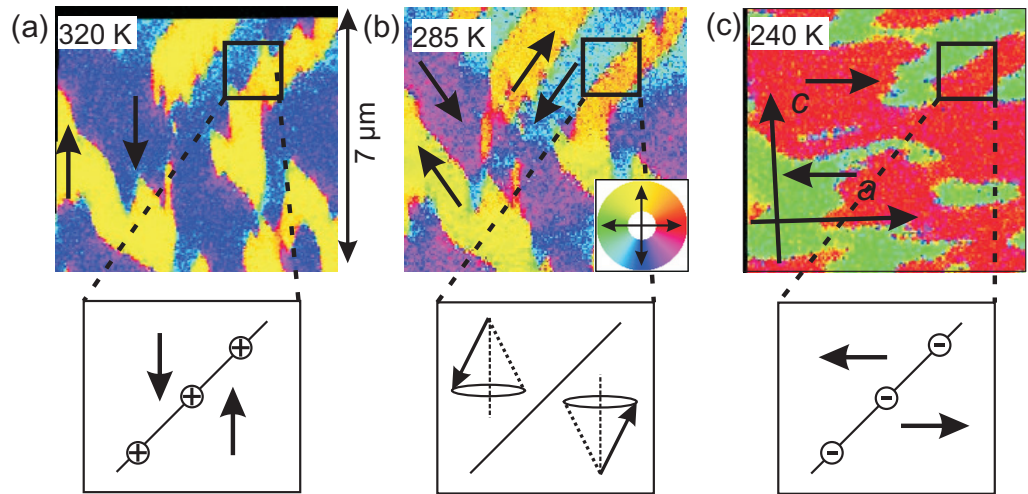


Figure 5. Magnetization processes taking place at 45° -oriented domain walls during the spin-reorientation.

in the studied film is established from a pinning dominated coercivity mechanism determined via angular-dependent hysteresis measurements [13]. Analysing the orientation of the domain walls, one finds three predominant directions. In the easy c -axis regime, walls parallel to the c -axis dominate. Analogously, at low temperatures most walls are oriented parallel to the a -axis, which is now the easy magnetization direction. In addition to these two types of walls, walls oriented along both 45° directions are visible. They are predominantly present in the easy cone regime but are also found in the easy a - and easy c -axis state with lower preference.

Comparing the images taken at 285 and 320 K (figures 4(c) and (e)), a general behaviour concerning the rotation sense of magnetization through the SRT becomes obvious. For a given domain boundary in one of the easy axis states, the rotation of magnetization occurs in such a way that it aligns more parallel to the existing domain boundary when entering the easy cone regime. The driving force for this behaviour is an initial reduction of stray field energy, which will become clearer in the following examples.

A close-up of the domain state at 320, 285 and 240 K is shown in figure 5 together with a sketch of the magnetization processes which take place at a domain wall oriented at 45° . At 320 K these walls are magnetically charged and therefore energetically not favoured (figure 5(a)). The tilted domain walls present a kind of zigzag wall which forms to reduce the charge density where two domains meet head-on [25]. Upon lowering the temperature, the magnetization seeks to reduce these magnetic charges by choosing a rotation sense which brings the moments more parallel to the domain boundary. Once the rotation sense is selected, the magnetostatic energy associated with the charges at the domain boundary decreases with decreasing temperature until the spin-reorientation angle θ_{SR} exceeds the domain orientation angle of 45° (figure 5(b)). Lowering the temperature further leads to a charged wall in the regime of the easy a -axis, now with opposite polarity (figure 5(c)). When the sample is heated up again, the magnetic charges are once again the driving forces for the selection of the direction of rotation and the process runs in the opposite direction.

One has to remark that the domain boundary does not remain completely unaltered upon this change in temperature. Both, small tilts of the overall orientation of the domain wall

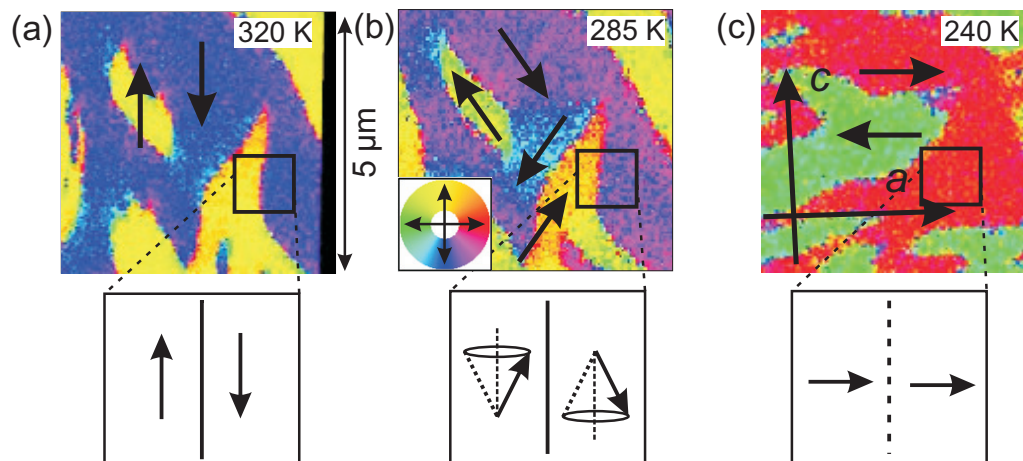


Figure 6. Magnetization processes taking place at a domain wall parallel to the c -axis during the spin-reorientation.

(compare figures 5(b) and (c)) and a further formation of the zigzag patterns, which are already visible in figure 5(a), are observed (compare figures 5(a) and (b)). The described energy considerations allow understanding most of the observed walls. Of course, due to local impurities and because of frustration effects with neighbouring domains at a few points, irreversible domain processes such as the formation or disappearance of domain walls can also take place.

In contrast to these 45° -oriented domain walls, the walls parallel to the a - or c -axis only exist in a certain temperature range. The magnetization processes that occur at a wall parallel to the c -axis are sketched in figures 6(a)–(c). Such walls do not carry any charges in the easy c -axis regime and are therefore energetically favoured at high temperatures (figure 6(a)). When the temperature is lowered, the rotation of the magnetization in the neighbouring domains occurs in such a way that magnetic charges are avoided—in both domains the magnetization rotates into the same direction (figure 6(b)). Consequently, the domain wall disappears in the regime of the magnetic easy a -axis and a new single domain has formed (figure 6(c)). An equivalent process takes place at the domain walls which are parallel to the a -axis at low temperatures (figures 7(a)–(c)). Like the walls parallel to the c -axis at high temperatures, these walls are magnetically uncharged. When the temperature is raised, the magnetic moments in the neighbouring domains rotate in the same direction, which leads to the formation of one domain at high temperatures. Why the 45° walls are favoured among the walls that are not parallel to the c - or a -axis cannot be answered at present. Possibly they have a lower energy due to the high symmetry but this needs to be proven e.g. by micromagnetic calculations.

3.3. Temperature-dependent magnetocrystalline anisotropy

While the above-discussed considerations were derived from a purely qualitative analysis of the domain configuration, the measured asymmetry distribution also allows for additional quantitative conclusions on the SRT. From the fact that the accumulation points in the 2D histograms are positioned on a circle with a constant radius—within the experimental uncertainty—it follows that the magnetization is lying in the film plane at all temperatures

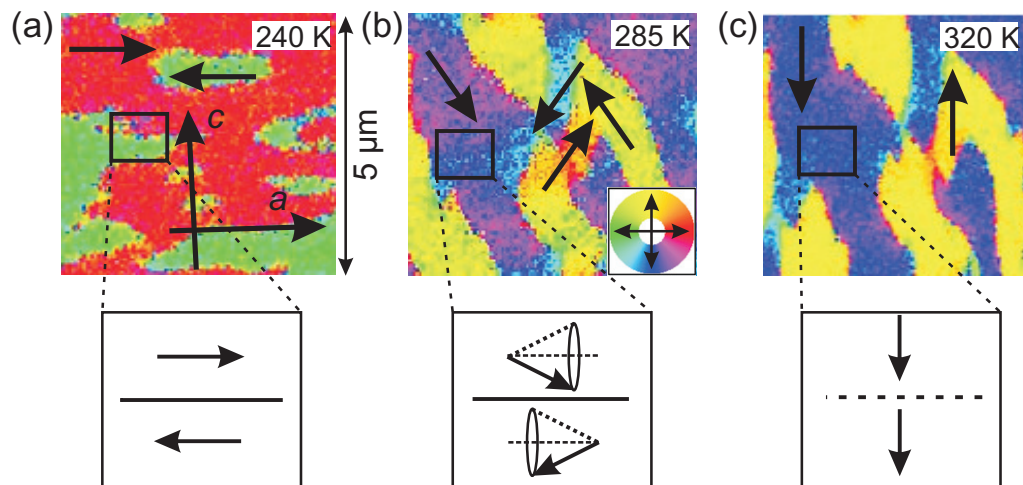


Figure 7. Magnetization processes taking place at a domain wall parallel to the a -axis during the spin-reorientation.

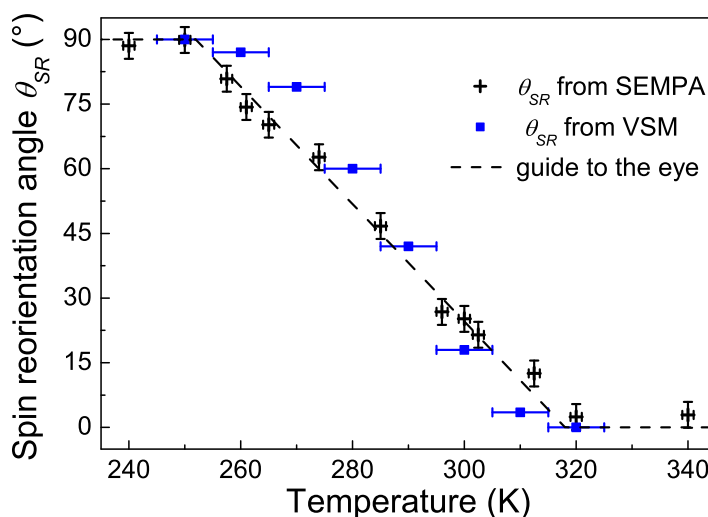


Figure 8. Spin-reorientation angle θ_{SR} derived from SEMPA measurements. The black dotted line is a guide to the eye. For comparison, the results of VSM measurement are also plotted.

and an out-of-plane component within the domains is absent. An out-of-plane component of magnetization may occur (and is even expected) for some domain walls; the resolution of the present images can, however, only hint at it. The interesting question of domain wall structure during spin-reorientation will be answered in an upcoming paper by means of micromagnetic calculations [26]. In the present measurements, the orientation of the domain magnetization can be unambiguously correlated with the orientation of the crystal axes of the film. In particular, the spin-SEM measurements allow for an exact determination of the magnetization angle with respect to the c -axis. The analysis of the data is presented in figure 8 as a function of temperature. It appears that the angle changes linearly with temperature from an alignment parallel to the a -axis to parallel to the c -axis. These data were compared with the spin-reorientation

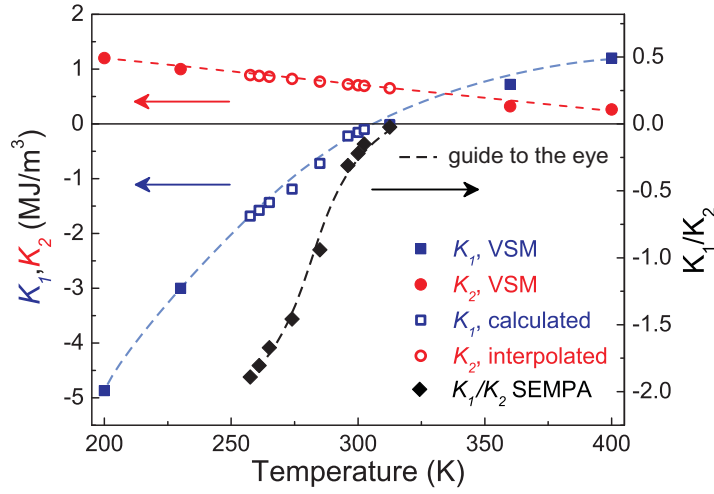


Figure 9. Ratios K_1/K_2 (black diamonds, right axis) and the measured (VSM, solid squares/circles) and calculated (open squares/circles) values of K_1 and K_2 (left axis).

angles determined from the temperature-dependent remanent polarization of the previously saturated sample (section 3.1). The agreement seen is remarkably good, considering the strongly complementary methods. It suggests that the domain configuration seen with SEMPA at the surface directly follows the direction of easy magnetization and is representative of the full depth of the film. From the plot of the SEMPA results, we can determine accurately the temperatures where the spin-reorientation sets in and ends, namely 252 and 318 K. The values are close to the temperatures, 255 and 310 K, found previously [13]. The experimental uncertainties of angle and temperature are $\pm 3^\circ$ and ± 1 K, respectively.

From the measured domain pattern, the magnetocrystalline anisotropy constants cannot be derived directly; it nevertheless provides a quantitative measure of the ratio $K_1(T)/K_2(T)$. For a hexagonal crystal, a Taylor expansion of the magnetocrystalline anisotropy energy density, E_{ani} , with respect to the c -axis is described in second-order approximation as

$$E_{\text{ani}} = K_1 \sin^2 \theta + K_2 \sin^4 \theta, \quad (1)$$

where θ is the angle between magnetization and the c -axis (see, e.g., [27]). A positive K_1 describes an easy axis anisotropy along the c -axis and a large negative K_1 an easy plane anisotropy in the basal plane of the crystallite. In the present epitaxial sample, the c -axis lies in the film plane, and due to shape anisotropy the magnetization is confined to this plane. Thus, the easy basal plane becomes an effective easy axis parallel to the a -axis for large negative K_1 . For a certain combination of negative K_1 and positive K_2 , i.e. $0 > K_1/K_2 > -2$, the anisotropy energy is minimized for a magnetization direction given by the spin-reorientation angle θ_{SR} with respect to the c -axis, with

$$\frac{K_1(T)}{K_2(T)} = -2 \sin^2(\theta_{\text{SR}}(T)). \quad (2)$$

Hence, the ratio K_1/K_2 can be derived within the range of validity from the spin-reorientation angle measured by SEMPA (figure 8). The result is plotted as black solid diamonds in figure 9 (right axis).

At 200, 230, 360 and 400 K, i.e. outside the spin-reorientation regime, the absolute values for K_1 and K_2 were taken from the previous Sucksmith–Thompson analysis and are plotted as solid blue squares and red circles (left axis). As can be seen, K_1 varies strongly with temperature and undergoes the expected sign change, while K_2 remains positive. Within 200 and 400 K it appears that K_2 can reasonably well be approximated by a linear interpolation of the VSM data (see red open circles). Doing so, $K_1(T)$ is calculated via equation (2) from the K_1/K_2 ratio based on the spin-SEM investigation. The results are plotted as blue open squares. These values fit very well with the K_1 values (solid blue) determined from the Sucksmith–Thompson analysis and also agree reasonably well with earlier measurements on single crystals [2].

4. Conclusion

This paper presents the first local magnetization studies on epitaxial NdCo₅ thin films. The transition from a two-domain state in the regime of the magnetic easy c -axis to a four-domain state in the regime of the magnetic easy cone to a two-domain state in the regime of the magnetic easy a -axis was imaged with SEMPA.

The orientation of magnetization follows the easy magnetization directions given by the temperature-dependent magnetocrystalline anisotropy and the shape anisotropy of the thin film. The actual domain configuration, however, and especially the behaviour of differently oriented domain walls is additionally influenced by the magnetostatic energy associated with magnetic charges along the wall.

The first conclusion is derived from the good agreement between the spin-reorientation angle determined by SEMPA and the magnetocrystalline anisotropy data derived from global VSM data. This also suggests that the domain pattern imaged at the surface is characteristic of the depth of the thin film. Indeed, first results of micromagnetic calculations support this finding [26]. The second conclusion is derived from a detailed analysis of differently oriented domains and domain walls through the SRT. The magnetization on either side of a domain wall initially rotates such that it lies more parallel to the existing wall in order to reduce magnetic charges. Due to the continuously rotating direction of easy magnetization through the spin-reorientation transition such charges can however never be fully avoided, which adds to the complexity of the domain pattern.

Acknowledgments

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