HYSTERESIS MEASUREMENTS ON RCo₅ MICRO-PARTICLES

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Résumé. — On discute des courbes d'hystérésis magnétique, mesurées sur des particules isolées de quelques microns de dimension des composés RCo₅, avec R = Sm, Pr ou La. Les champs critiques nécessaires pour la nucléation et la mobilisation des parois sont mesurés séparément, et l'influence du vieillissement est observée. L'accrochage des parois semble décisif pour la détermination de la coercivité.

Abstract. — Magnetic hysteresis loops measured on isolated single particles of a few microns size are shown for RCo₅ compounds with R = Sm, Pr and La, respectively. The critical fields at which walls nucleate and propagate are measured separately, and the influence of ageing is observed. Pinning of domain walls seems to be significant in determining the coercive force.

The magnetization process of RCo₅ compounds (R for rare-earth metal) takes place at field strengths far below the anisotropy field, which indicates that it must be interpreted in terms of nucleation and displacement of Bloch walls. The coercivity depends largely on the field strength $H_c$ at which a reverse domain is nucleated and on the field strength $H_p$ at which an already existing domain wall is propagated.

It is interesting to know whether $H_c$ or $H_p$ is determining coercivity in SmCo₅, how these critical fields are affected by ageing and how they behave in other RCo₅ compounds.

A wall-nucleation model devised by Becker [1] considers the nucleation field $H_n$ as mainly determining the coercivity. However, other measurements make it likely that in certain cases wall pinning can be predominant, leading to a wall-displacement model [2, 3].

These discussions are hampered by the fact that hysteresis measurements are made on samples that contain a large number of crystals giving the average of many wall processes. The nucleation and displacement of single walls cannot be observed individually and manifest themselves merely as noise. However, if measurements are made on samples small enough to display one elementary process at a time, more information on the magnetization process might be gathered. Such samples should have a size of a few microns. Hysteresis measurements on these samples are possible with a new type of magnetometer, the « Vibrating Reed Magnetometer » [4]. We have used this instrument to observe wall processes in a single particle taken from a SmCo₅ powder having a coercivity of 14 kOe. The hysteresis loop measured along the easy axis of the particle is shown in figure 1a. The powder was ground in a vibration mill with steel balls under hydrogen. After grinding it was handled under exclusion of air until a few hours before the measurement.

The measurement starts at 21.5 kOe, this being the highest field we could make in the magnetometer. The magnetization remains constant until at about zero field a small jump occurs. As this jump is reversible on this field scale it is ascribed to the magnetization reversal of some small amount of iron possibly abraded from the steel balls. It will be ignored in the following discussion.

At $-11.4$ kOe a wall is nucleated, which jumps through a large part of the crystal until it becomes stuck somewhere. Upon further increase of the counterfield it creeps slightly until at $-14.8$ kOe another jump occurs after which the magnetization of about threequarters of the crystal volume is reversed.

By creep is meant a continuous but irreversible displacement of the wall due to field variation. No time effects are observed.

The remaining part of the magnetic moment is reversed by a creep process, which we have found in all hysteresis loops measured on RCo₅ single particles up to now. Evidently the wall is reluctant to leave the crystal. As this portion of the loop is irreversible there must be some concentration of pinning sites preventing the wall from crossing the surface layer. This is in
agreement with the wall-pinning model set forth in ref. [2] and [3], where a SmCo₅ crystal is considered as consisting of soft regions in which walls are free to move, separated by hard layers where walls are strongly pinned. The nature of the pinning sites is still unclear. The other branch of the loop shows roughly the same picture, apart from slightly different values of the critical fields.

The crystal was measured again after 12 days, during which it was exposed to air at room temperature. The loop is shown in figure 1b. The field strength at which the first big jump occurs is reduced to 9 000 Oe, perhaps by surface corrosion making nucleation of a reverse domain easier. Creep no longer occurs between this jump and the next. This is strange because the position of the wall must be about the same. Evidently pinning has become stronger. The next jump occurs at about the same field as in figure 1a. This degradation or ageing of the magnetic hardness has also been observed in powders and magnets of the same material [5].

The ageing process can be accelerated by heating the sample in air. After 30 minutes at 100 °C the sample was measured again (Fig. 1c). The small jump at zero field has vanished. The presumed iron contamination has probably been oxidized.

At about − 6 000 Oe a kink is visible, which is interpreted as the nucleation of a wall that remains pinned close to its nucleation site. Upon increase of the counterfield it creeps inward until at 9 000 Oe it becomes detached and jumps to its favourite position, where it remains until, at 11 000 Oe, it jumps through the crystal into the hard surface layer and vanishes reluctantly from the crystal. The other branch of the loop shows no such behaviour; nucleation of reverse domains is different in opposite directions. This is probably connected to the moderate field of about 21 kOe not being strong enough to drive all walls out.

Figure 1d shows the loop after 17 ½ hours at 100 °C. Nucleation has become much easier and the coercivity is entirely determined by the pinning which keeps the wall near its nucleation site until − 9 000 Oe. The jump at 14 kOe has vanished. Apart from making nucleation easier the heat treatment also seems to influence the pinning. This is more pronounced in figure 1e, where the loop is shown after 600 hours at 100 °C. The wall nucleated at zero field is detached from its pinning sites at 6 000 Oe and 8 200 Oe. It is worth noting that the loops of figures 1d and 1e are symmetric.

The picture is somewhat different with other RCo₅ compounds. The hysteresis loop of PrCo₅ is shown in figure 2. Nucleation is seen to be easy. The wall penetrates deeply into the crystal where it becomes pinned. But at the relatively low field strength of 1 500 Oe it is detached and jumps through the crystal. The average slope of the curved parts corresponds again with the demagnetization factor of a sphere. However, the reluctant approach to saturation is still visible, indicating that pinning, however relatively weak, is effective here.

With LaCo₅ no appreciable pinning could be observed (Fig. 3). The hysteresis loop extends in smooth curves from saturation in one direction to that in the other. Also nucleation is easy.

Resuming we may say that in the high-coercivity SmCo₅ particles immediately after grinding nucleation is determining the coercivity in accordance with Becker's nucleation model. Ageing at 100 °C primarily promotes nucleation but also affects the pinning. In this case the wall-pinning model is valid.

In PrCo₅ the wall-pinning model is applicable, whereas LaCo₅ cannot be described with either model, as no Barkhausen jumps are observed here. The question remains : why is the pinning force so different in these highly anisotropic compounds of isomorphous crystal structure.

References