Influence of Surface Properties on Determination of RQ Ribbon's Magnetic properties

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INTRODUCTION

The surfaces of thin metallic ribbons prepared by rapid quenching (planar flow casting) of the melt on air (RQMA) deserve a closer look. Due to its rather large ratio to the rest of ribbon's volume (="interior") is the effect of surfaces on the ribbon properties often significant. This fact influences measurements on nanocrystalline and the precursor ribbons as well. The surfaces of RQMA ribbons more or less differ from the interior as a rule. Oxidic, hydroxidic and more complex contaminations are often observed already on the as-cast surfaces by sensitive methods as XPS [1, 2].

This chemical surface-interior difference does not simply disappear during thermal treatment; it merely transforms variably and affects the interior too. The difference manifests most after a non-vacuum anneal and results in affecting the structural transformation (partial crystallization) - the effect is seen e.g. on MS [L10, this Conference] and best on combined MS+ CEMS spectra parameters:

Alloy id	1 hr Anneal	A_{cr} (% crystalline)		$\langle B_{hf} \rangle$ am [T]	
		MS	CEMS	MS	CEMS
$FeCo_{20}NbB$ Hitperm	Vac 600°C	13.5 + 1.5	25.5	25.3	24.8
	Ar 600°C	13.0 + 2.0	50.9	24.8	26.3
$FeNbCuBSi_{4.5}$ Finemet	Vac 540°C	21.0	41.0	23.3	-
	Ar 540°C	14.2	100	24.4	-

The Hitperm shows two crystalline „phases"; the minor one is closer to the ordered B2-type structure. Note its lack at surfaces – in the CEMS data. The Si-poor Finemet shows completely crystallized sufrace. $\langle B_{hf} \rangle$am stands for average hyperfine field of amorphous remainder (further Hitperm details in [3]).

Note the surface-integral difference being observed after vacuum anneal as well.

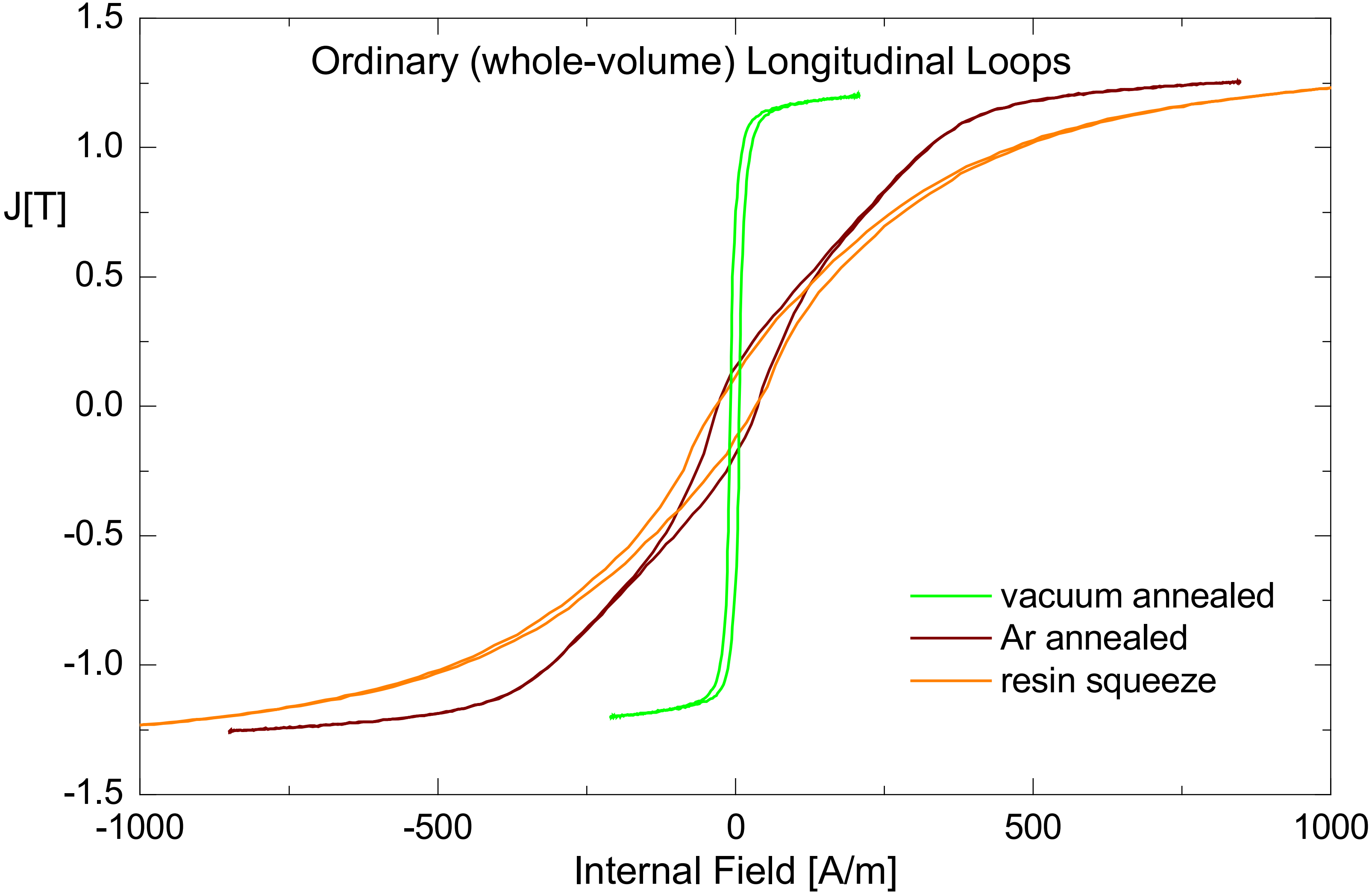
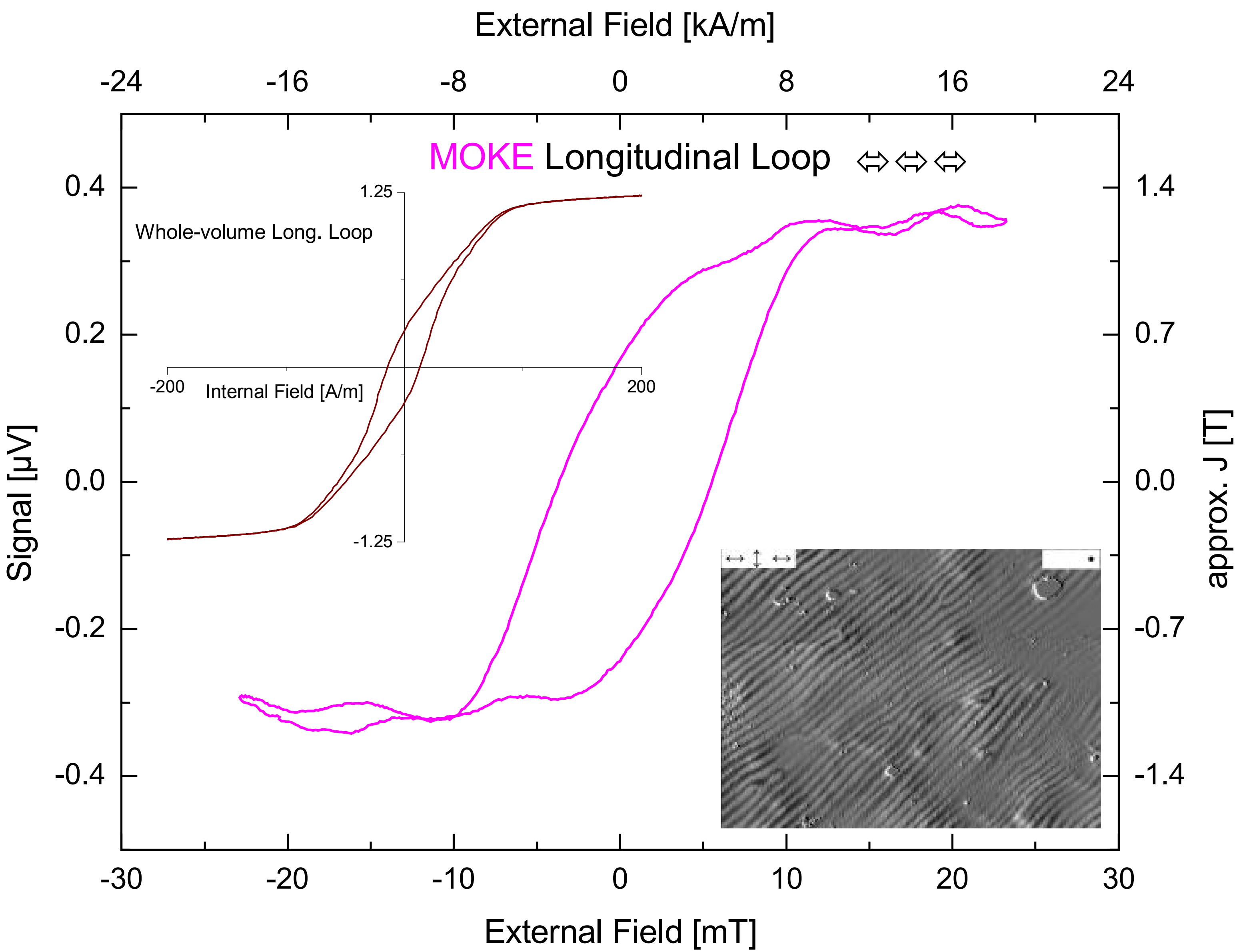
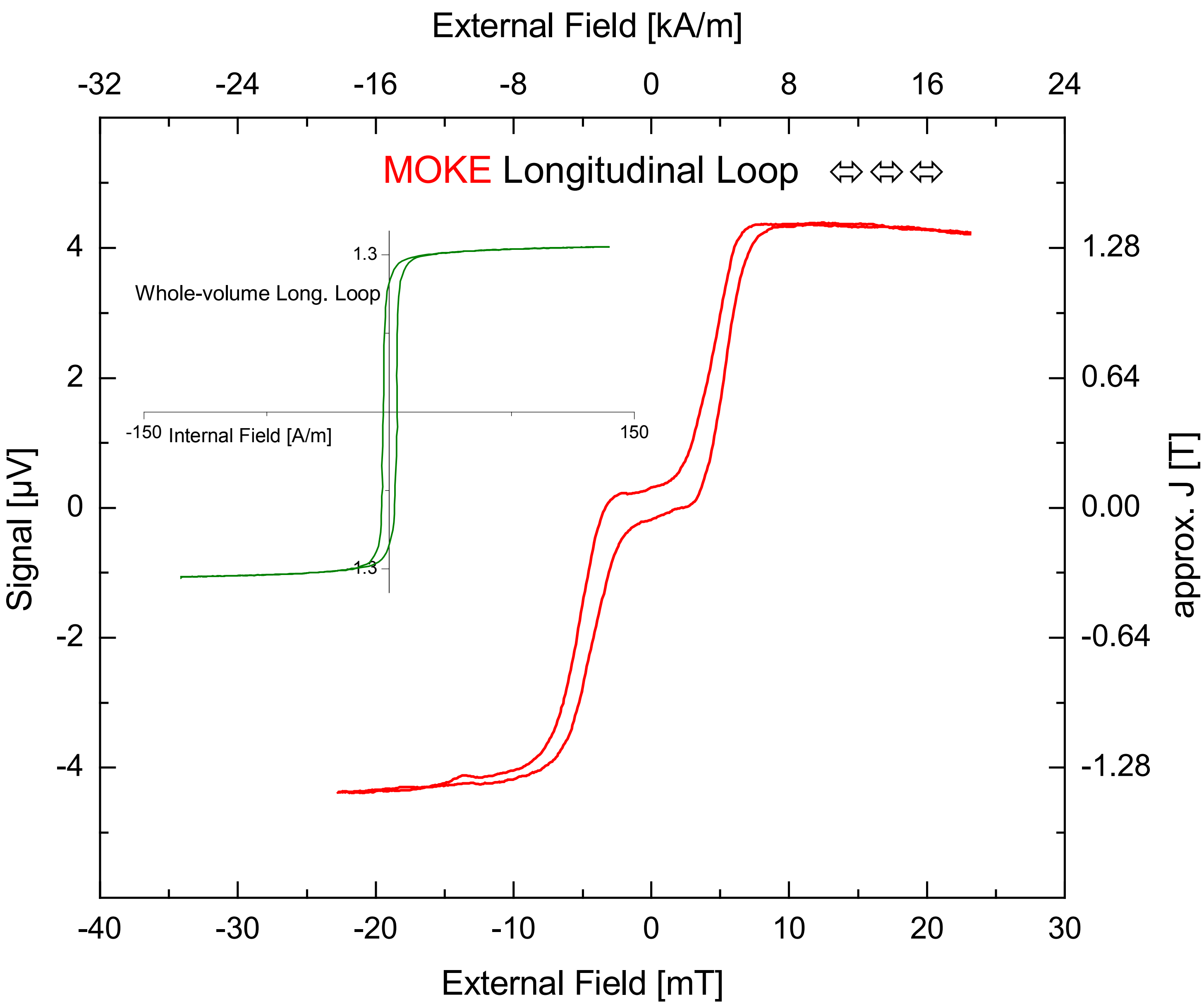

Ordinary (whole-volume) Longitudinal Loops
J[T]
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-1000
-500
0
500
1000
Internal Field [A/m]
vacuum annealed
Ar annealed
resin squeeze

Fig.1. Length x width = 10 x 1 cm stripe samples. Exciting field corrected for demagnetization. Being different as for the density and/or thermal expansion, the surfaces can exert force on the interior (and vice versa). This is specifically significant for magnetostrictive materials (λ_s ~ 8E-6 for $FeNbCuBSi_{10}$ Finemet shown), thus it concerns the majority of compositions used to prepare magnetic ribbons. As the tensile strength of the former majority amounts at least 200 MPa even after nanocrystallization, so some 2μm thick surface layers can exert 50 MPa on the interior of a 20 μm thick ribbon without breaking the surfaces. This is by far enough to induce a hard-ribbon-axis anisotropy by magnetoelastic interaction in a positively magnetostrictive ribbon whose surfaces squeeze the interior. Although not necessarily isotropic, the compressive stress is a bi-axial in-plane one. It is seen that the loop slanted by resin jacket squeeze (the vacuum-annealed sample is potted) resembles rather closely the loop after equivalent Ar annealing (540°C, 1 hour).



External Field [kA/m]
-24
-16
-8
0
8
16
24
MOKE Longitudinal Loop ⇔⇔⇔
Whole-volume Long. Loop
1.25
-1.25
-200
Internal Field [A/m]
200
0.4
0.2
0.0
-0.2
-0.4
Signal [µV]
1.4
0.7
0.0
-0.7
-1.4
approx. J [T]
-30
-20
-10
0
10
20
30
External Field [mT]

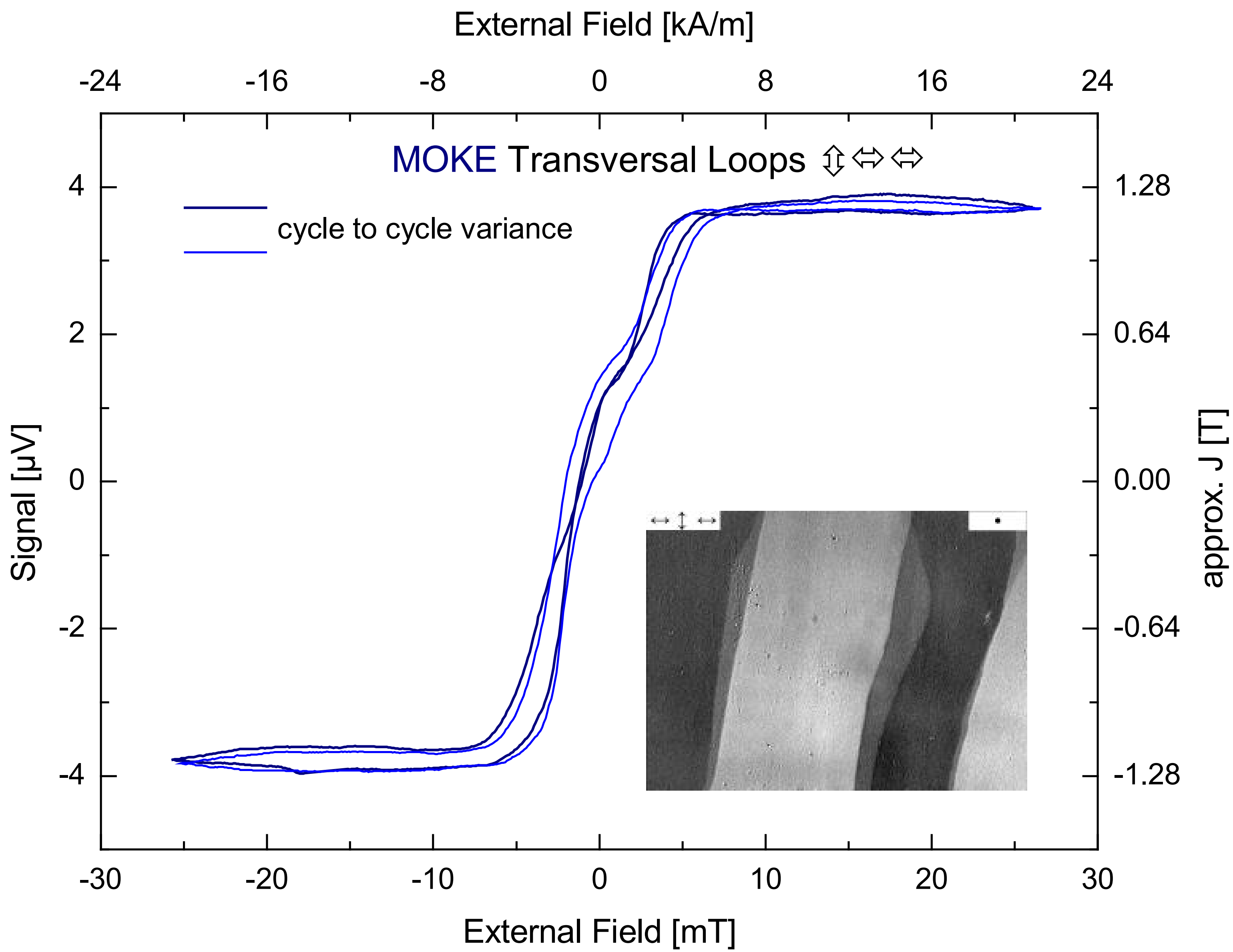
Fig. 2. The samples for the MOKE tests are discs of 5 mm diameter; exciting field is not corrected for demagnetization. The surfaces are rather rough (1÷2 μm on a 20 μm thick ribbon) and the more, the opaque one differs from the shiny one [4]. This feature hardly changes during a standard annealing, be it performed in vacuum or inert gas. Apart from influencing the effective magnetic anisotropy by stray fields, the roughness sometimes challenges optical methods used to see just the surface properties – e.g. Magneto-Optical Kerr Effect (MOKE). It works fine with specular reflection but the diffuse reflection from a rough surface cuts down the intensity which reaches the detector. Less of a problem if the ratio incident to detector-captured intensity holds constant, intricate problem if it does not. The surface texture is often directional and so the detector-captured light can vary its intensity as well as the particular location where it was reflected (e.g. if sample is rotated). The MOKE loops are acquired without a focusing lens. The light intensity is further diminished by a surface color cast often seen on Ar-annealed Finemets as this $FeNbCuBSi_{10}$ one. The surface domain structure (DS) shows rather fine closure domains which re-magnetize mostly by magnetization rotation at the used arrangement. The MOKE loop thus can be unexpectedly little different from the volume loop if the experiment hits such a "lucky" location on the surface. Generally, the necessity to observe many enough sites is the drawback of using local methods with heterogeneous samples.



External Field [kA/m]
-32
-24
-16
-8
0
8
16
24
MOKE Longitudinal Loop ⇔⇔⇔
1.3
Whole-volume Long. Loop
-150 Internal Field [A/m]
150
-1.3
Signal [µV]
4
2
0
-2
-4
approx. J [T]
1.28
0.64
0.00
-0.64
-1.28
-40
-30
-20
-10
0
10
20
30
External Field [mT]

Fig. 3. Again somewhat surprisingly, the vacuum-annealed sample of $FeNbCuBSi_{10}$ Finemet shows pronounced difference between surface and volume longitudinal loop. At low fields, the surface magnetization evidently lags behind the volume magnetization. Although the surfaces of RQMA ribbons are always (despite the annealing ambience) suspect of being at variance from the interior, the above comparison presents one of the most pronounced *magnetic* differences seen so far on vacuum annealed samples. However it should be stressed once again, one should not forget about spot-to-spot variation frequently observed when inspecting small portion of a relatively much larger surface area. Thus the explanation drawn from DS (next figure) is merely a possibility so far.

---As in other magneto-optical results, **the three arrows** indicate the direction of (left to right): ribbon long axis, maximum magneto-optical sensitivity, external field. While the laser-exposed area is ~1 mm^2 (reflection is diffuse!) at MOKE loops recording, the DS images display even smaller real area of 0.32 x 0.44 mm.



External Field [kA/m]
-24
-16
-8
0
8
16
24
MOKE Transversal Loops ⇳⇔⇔
cycle to cycle variance
4
2
0
-2
-4
Signal [µV]
1.28
0.64
0.00
-0.64
-1.28
approx. J [T]
-30
-20
-10
0
10
20
30
External Field [mT]

Fig. 4. The same sample as formerly seen longitudinally, this time in-plane rotated by right angle. This MOKE *transversal* loop is more alike to the volume *longitudinal* loop. Thus it looks like the surface shows a transversal easy axis and just this is shown by DS. Not only the walls run almost transverse but they move along (!) the applied (weak) field as seen by the "shadows" induced by increasing the field during image averaging. The walls move rather irreproducibly and this appears to show also in the MOKE loop. Another fact to note is the intersection at the loop center which reminds of "partially inverted loops". However in the situation presented, it seems straightforward that the irreproducible wall movement *at the observed site* can be the source. While it is for sure that the integral – whole-volume – easy axis is longitudinal, from as yet unclear reason the surface (shiny one) shows the easy direction at an large angle. It cannot be ruled out that after a vacuum annealing, the surfaces can longitudinally tense the interior, contrary to Ar annealing – see Fig. 1. The effect does not come from an unintended stress; here it was tested that thin (~0.1 mm) epoxy layer used to fix the sample does not significantly influence the measured response. Still other explanations abound and should be thoroughly tested. Nevertheless, neither vacuum annealing of a RQMA ribbon seems to prevent or dispose of the surface-interior differences.

CONCLUSIONS

The advance in surface-sensitive experimental technology driven mainly by thin film research is at least equally useful also for thicker materials. This concerns specifically thin RQMA ribbons as these show surface-to-interior differences capable of affecting the material as a whole. Though optical methods like MOKE meet additional difficulties when applied to rough-surfaced ribbons, MOKE is *the* powerful method to gain knowledge about surface magnetic properties. In turn, these properties compared to integral (whole-volume) magnetic properties show again that and how much the surfaces differ from the interior.

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References

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