Influence of annealing temperature and atmosphere on surface microstructure and magnetism in FINEMET-type FeSiNbCuB ribbons

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AIM: Detection of quadratic magneto-optical effects and their relation with surface microstructure in $Fe_{73,5}Si_{13,5}Nb_3Cu_1B_9$ ribbons

Sample preparation

Material: $Fe_{73,5}Si_{13,5}Nb_3Cu_1B_9$

Thermal treatment: annealing in vacuum (10^{-5} Pa) and hydrogen at 733 K, 743 K, 823 K, 923 K

Sample dimensions: 20 μm thick and 6 mm wide

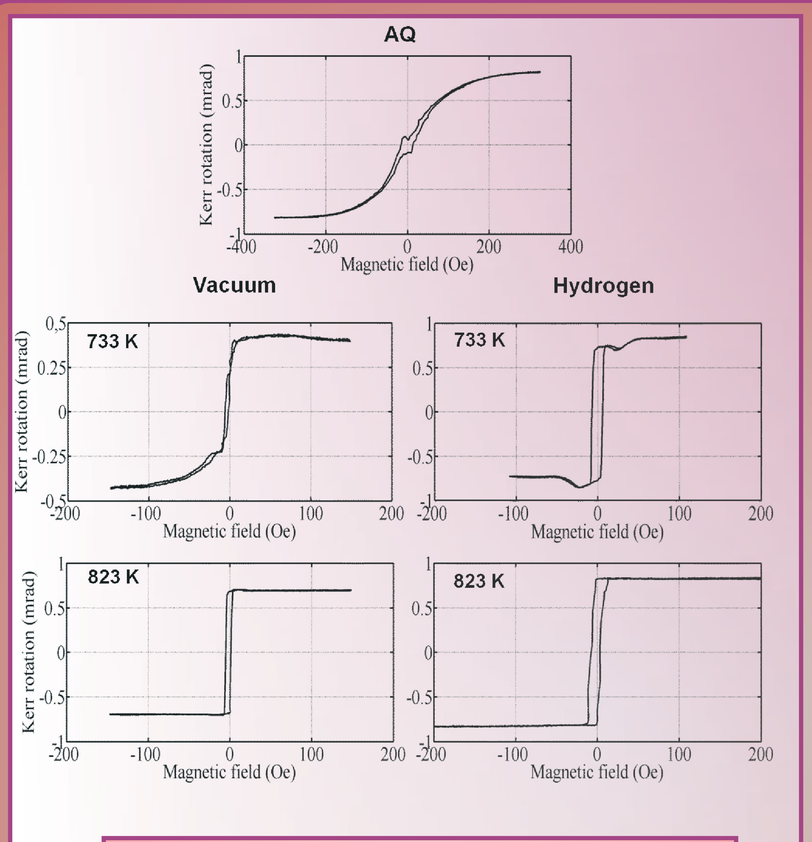
Investigated side: shiny - side in contact with air during rapid quenching

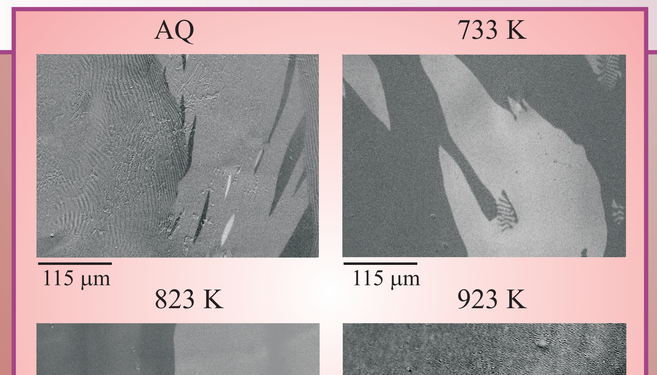
Experimental methods

Magneto-optical Kerr effect (MOKE): penetration depth PD ≈ 20 nm, measured longitudinal magnetization component (in the plane of the sample and incident light), wavelength: 670 nm, angle of light incidence: 50°, *s* - polarized light
XRD: CoKα radiation in Bragg-Brentano geometry, PD ≈ 10 μm, λ = 0,17902 nm
Grazing incidence XRD (GIXRD): grazing angle ≈ 1,5°, PD lower than 100 nm
TEM: samples prepared in a form of thin lamellas by focused ion beam (FIB) technique in scanning electron microscope

Experimental results

Surface magnetism (MOKE)





AQ sample

- amorphous as confirmed by TEM and XRD detection of two magnetically different phases - presence of clusters

- inhomogeneous magnetization behavior responsible for random clusters anisotropy

- wide - curved and fingerprint domains - consequence of stresses during preparation

Annealing at 733 K and 743 K

- relaxation of the stresses - vanishing of fingerprint domains

- measured asymmetric hysteresis loops - explanation based on quadratic magneto-optical effects (QMOKE)

- excellent soft magnetic properties with coercive field not higher than 4 Oe

Annealing at 823 K

- marked decrease of QMOKE amplitudes (symmetrization of the loops)

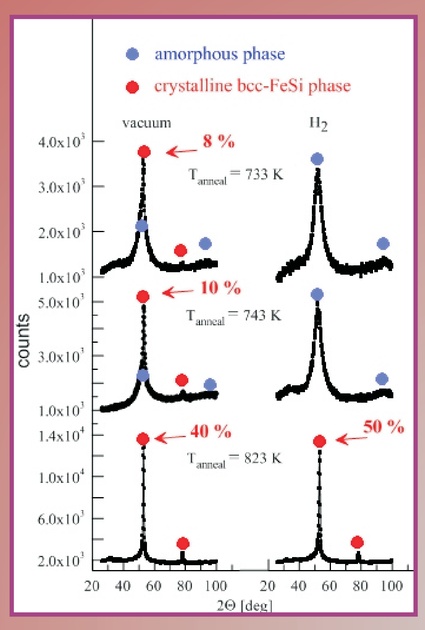
Annealing at 923 K

- increase of coercive field (up to 100 Oe) indicating strong surface crystallization

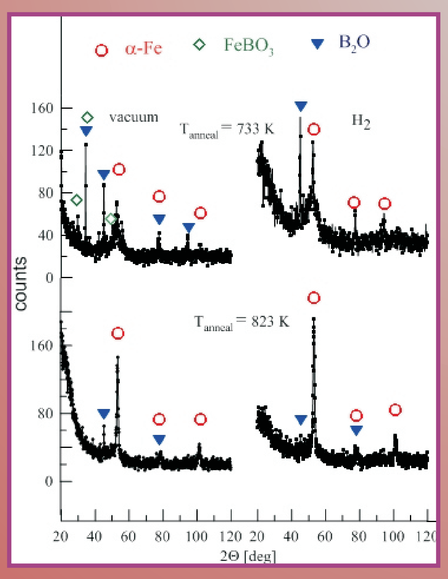
- response of crystallites clearly visible on magnetic domain patterns

Surface microstructure

XRD (PD = 10 μm)



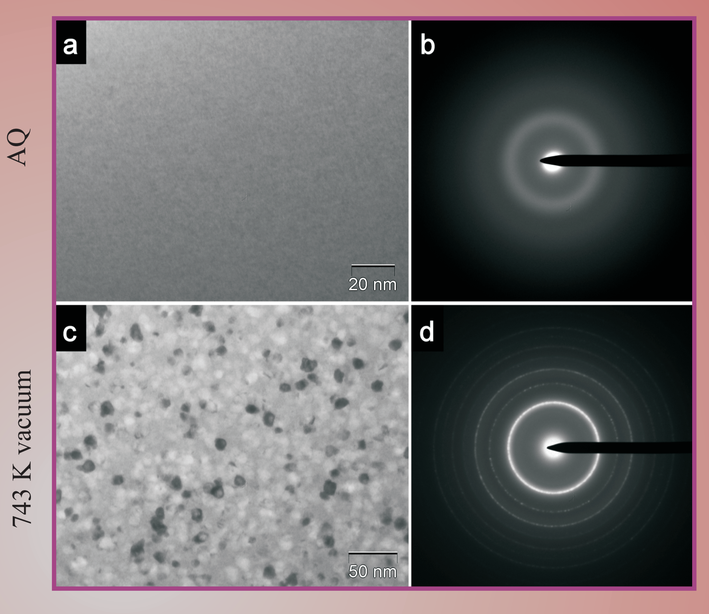
GIXRD (PD < 100 nm)



TEM

a) Featureless microstructure with contrast fluctuations of the size below 1-2 nm.

b) Diffuse ring typical of amorphous materials .



c) Distinct nanocrystals size about 5-15 nm.

d) Sharp dense rings indexing bcc structure with a ~ 0,28 nm and weak diffuse ring of amorphous phase .

GIXRD detected the close surface oxidation and nanocrystallization already at 733 K for both vacuum and hydrogen annealed sample.

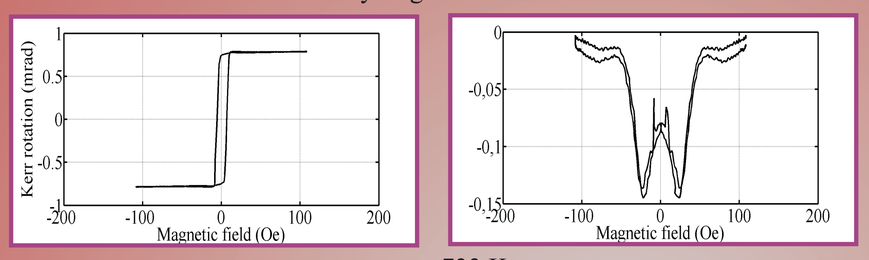
	vacuum			hydrogen		
Ta [K]	α-Fe [%]	B_2O+ $FeBO_3$ [%]	d [nm] Fe grains	α-Fe [%]	B_2O [%]	d [nm] Fe grains
733	35	65	5	53	47	4
823	60	40	13	94	6	10

XRD detected nanocrystallization by the vacuum annealed samples already at 733 K while by the hydrogen annealed samples as late as at 823 K.

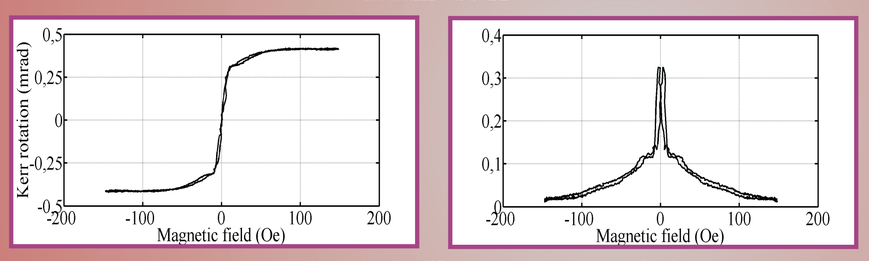
Quadratic magneto-optical effect (QMOKE)

Symmetric **Antisymmetric**

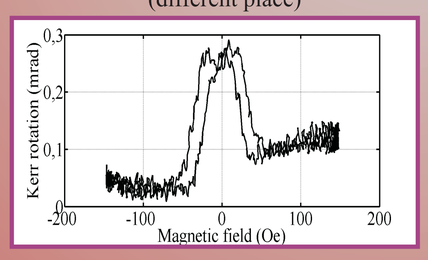
hydrogen 733 K



vacuum 733 K



Directly measured quadratic effect at almost normal incidence (different place)



- even dependence on applied magnetic field

- **origin**: small α-Fe and/or α-Fe(Si) nanocrystals dispersed in oxides as confirmed by GIXRD

- random orientation of nanocrystals responsible for changing the size and sign of QMOKE, when light is focused into the different places on the ribbon surface

- no polar (out-of-plane) component of magnetization detected → signal of QMOKE proportional to the mixed in-plane terms of $M_L M_T$ and M_L^2 - M_T^2 with prevailing contribution of $M_L M_T$

- annealing at 743 K and 823 K evoked changes in the surface and bulk microstructure probably responsible for loop symmetrization

- QMOKE fully dissapeared at 923 K

Conclusions

The main contribution of present investigations of the relatively frequently studied FINEMET type alloy in a ribbon form is the asymmetric reversal of longitudinal magnetization observed for the first time on the surface of an alloy prepared by planar flow casting process. This phenomenon termed as the quadratic magneto-optical effect was experimentally detected and described only in several well-defined bcc structures like Fe/MgO [1] or Co_2-based Heusler compounds [2].

The reason for this new phenomenon is probably a presence of small α-Fe nanocrystals dispersed in the thin oxide formed on the surface after annealing at low temperatures sufficiently below the initiation of the bulk crystallization which is responsible for hysteresis loop symmetrization and QMOKE diminishing.

Deeper understanding of this effect can bring some new aspects important for the sensor applications of this material.

[1] K. Postava, H. Jaffres, A. Schuhl, F. Nguyen Van Dau, M. Goiran, A. R. Fert, "Linear and quadratic magneto-optical measurements of the spin reorientation in epitaxial Fe films on MgO," *J. Magn. Magn. Mater*., **172** (1997) 199.

[2] J. Hamrle, S. Blomeier, O. Gaier, B. Hillebrands, H. Schneider, G. Jakob, K. Postava, C. Felser, "Huge quadratic magneto-optical Kerr effect and magnetization reversal in the Co_2FeSi Heusler compound," *J. Phys. D: Appl. Phys*. **40** (2007) 1563.

Acknowledgement
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