Ernst Ruska-Centrum für Mikroskopie und Spektroskopie mit Elektronen ER-C

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Magnetic microstructure in stress-annealed FeSiNbBCu soft magnetic alloys studied using Lorentz microscopy and electron holography

A. Kovács[1], K.G. Pradeep[2], Z.-A. Li[3], G. Herzer[4], D. Raabe[2] and R.E. Dunin-Borkowski[1]

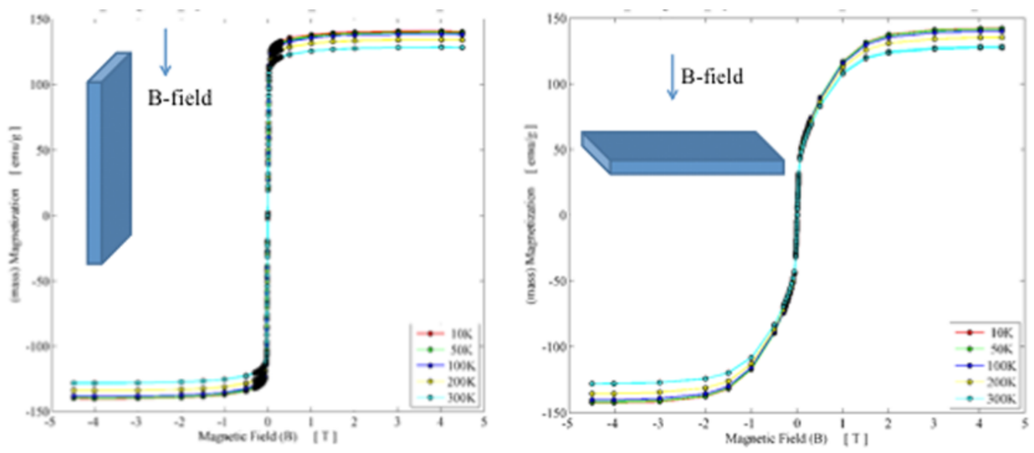
[1]Ernst Ruska-Centre (ER-C) and Peter Grünberg Institute, Forschungszentrum Jülich, Germany
[2]Max Planck Institut für Eisenforschung, Düsseldorf, Germany, [3]CeNIDE, University Duisburg-Essen, Duisburg, Germany,
[4]Vacuumschmelze GmbH & Co. KG, Hanau, Germany

Introduction

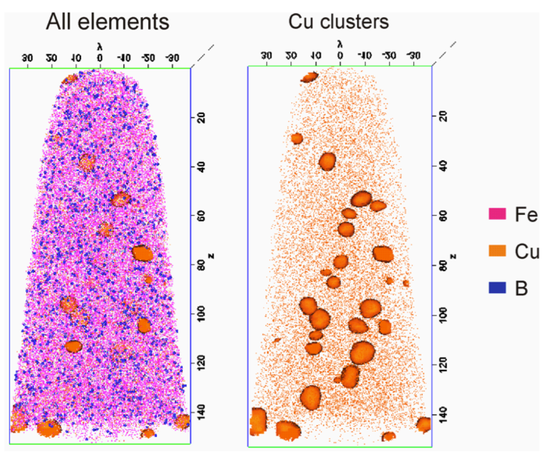
The unique physical and magnetic properties of FeSiNbCuB alloys, such as their low coercivity and high saturation magnetization combined with near-zero magnetostriction, make them attractive for high-frequency applications. Furthermore, their magnetic properties can be tailored by applying a magnetic field or stress during annealing, resulting in uniaxial anisotropy. Here, we study the magnetic domain wall structures in stress-annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ alloy using both the Fresnel mode of Lorentz microscopy and off-axis electron holography in the transmission electron microscope.

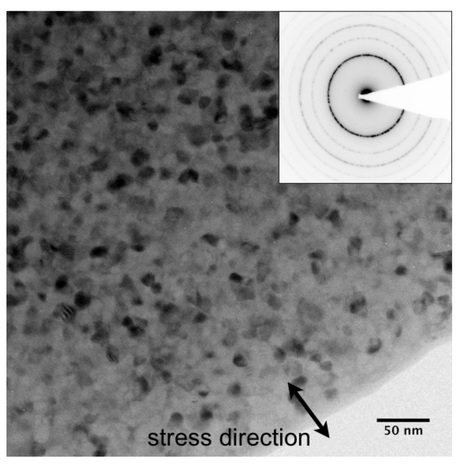
Magnetometry

A 600 MPa stress was applied to selected samples during a rapid 4 s annealing, resulting in strong uniaxial anisotropy perpendicular to the stress direction, as confirmed by SQUID measurements performed both at room temperature and at 10 K. The coercivity values of the material were measured to be 17 and 14 Oe at room temperature for a magnetic field applied parallel to the in-plane and out-of-plane directions, respectively.



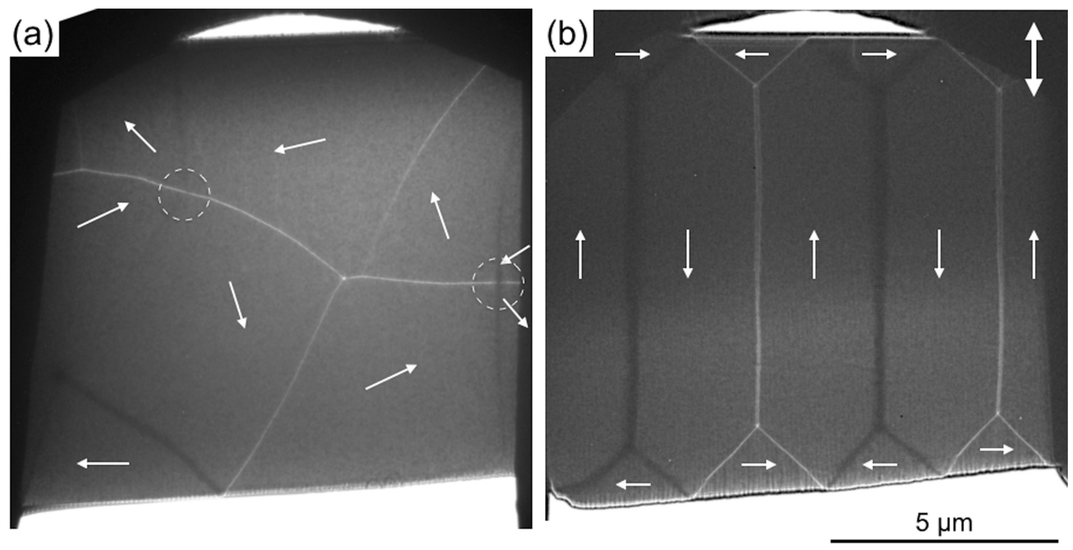
Atom probe tomography and transmission electron microscopy





APT studies revealed 80 vol.% of crystalline Fe_3Si phase with a DO_3 structure and 20 vol.% of an amorphous matrix that was enriched in B and Nb. The Fe_3Si grain size was measured to be 10 nm while Cu clusters were observed to form with sizes of ~6 nm. The structure of the sample is polycrystalline with randomly oriented grains.
TEM images, Fresnel defocus images and off-axis electron holograms were recorded using an FEI Titan TEM operated at 300 kV in magnetic-field-free conditions (< 0.5 mT) using a non-immersion Lorentz lens, with the conventional microscope objective lens switched off.

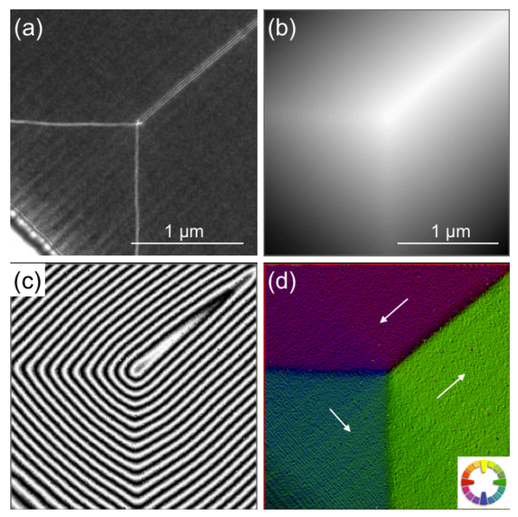
Domain structure



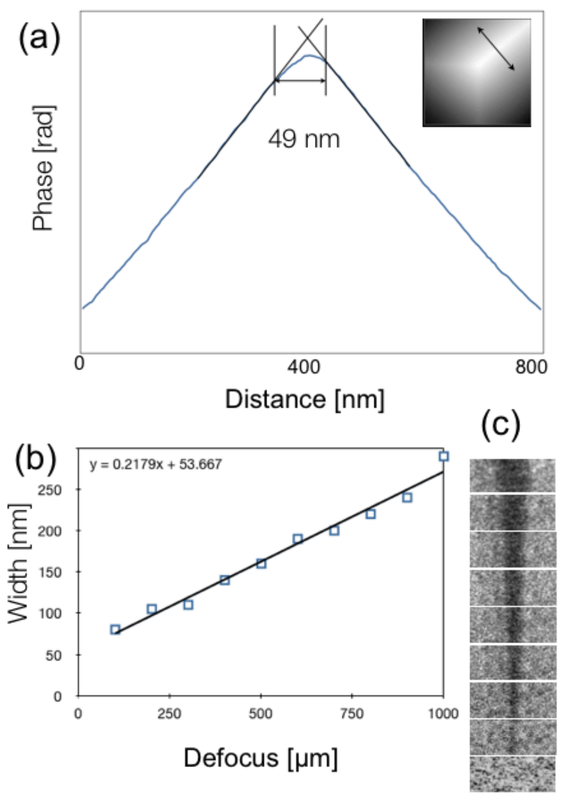
Fresnel defocus images recorded from $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ samples that had been (a) annealed at 695 °C for 10 s in the absence of an applied stress and (b) annealed at 690 °C for 10 s in the presence of a stress of 600 MPa.

Domain wall thickness

DW thicknesses were determined using a focal series of Fresnel images and phase shift maps recorded by off-axis electron holography.



Intersection of a 180º and two 90º DWs in (a) a Fresnel image and (b-d) electron holograms.
The width is determined as 53±10 nm and 49±3 nm for the 180º DW, and 94±3 nm for the 90º DWs in the stress annealed alloy.



Summary

- Strong uniaxial magnetic anisotropy was induced in rapid annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ alloy; the coercivity is <20 Oe in the annealed alloy;
- A regular magnetic domain pattern was observed;
- DW widths are ~50 and ~95 nm for 180º and 90º walls;

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