Type of presentation: Oral

MS-12-O-2532 Magnetic microstructure in stress-annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ soft magnetic alloys

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

[1]Ernst-Ruska Centre for Microscopy and Spectroscopy with Electrons and Peter Grünberg Institute, Forschungszentrum Jülich, D-52425 Jülich, Germany, [2]Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany, [3]Faculty of Physics and Center for Nanointegration (CeNIDE), University of Duisburg-Essen, D-48047, Duisburg, Germany, [4]Vacuumschmelze GmbH & Co. KG, Hanau, Germany

Email of the presenting author: a.kovacs@fz-juelich.de

The unique physical and magnetic properties of Fe-Si-Nb-Cu-B alloys [1], 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 nanostructure and magnetic domain state of stress-annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ using atom probe tomography (APT) and transmission electron microscopy (TEM). A 600 MPa stress was applied to selected samples during rapid annealing for 4 s [2], resulting in strong uniaxial anisotropy perpendicular to the stress direction, as confirmed using bulk measurements performed using a superconductive quantum interference device magnetometer. X-ray diffraction and APT studies revealed that the samples comprised 80 vol.% of a crystalline Fe_3Si phase with a DO3 structure and 20 vol.% of an amorphous matrix that was enriched in B and Nb, as shown in Fig. 1 [3]. The Fe_3Si grain size in the present samples was measured to be (10±3) nm, while Cu clusters were observed to form with sizes of ~6 nm. Specimens were prepared for TEM examination from rapid-annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ ribbons using an FEI Helios Nanolab 600i dual-beam focused ion beam (FIB) workstation. Structural studies of the samples using TEM revealed a polycrystalline microstructure without any detectable crystallographic texture, as shown in Fig. 1 (b). 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) with the conventional microscope objective lens switched off. Off-axis electron holograms were recorded using an electrostatic biprism located close to the selected area aperture plane of the microscope. Figure 2 (a) shows a magnetic domain wall (DW) pattern comprising near-perfect 180° and 90° DWs in stress-annealed sample. Figure 2 (b) shows a Fresnel defocus image and off-axis electron holography results obtained from the intersection of a 180° DW with two 90° DWs. The insets show equally-spaced cosine phase contours around the intersection of the DWs in reconstructed phase images recorded from the same region of the specimen. The width (full-width-at-half-minimum) of the divergent contrast arising from a 180° DW in a defocus series of images, extrapolated to zero defocus, provides a value of (53±10) nm, as shown in Fig. 2 (c). Direct measurements from the corresponding phase profiles in electron holograms provided upper limits of (49±3) and (94±3) nm for the widths of the 180° and 90° DWs (not shown).

Acknowledgement: The authors acknowledge financial support from the German Research Foundation, the Helmholtz Association and the European Research Council.

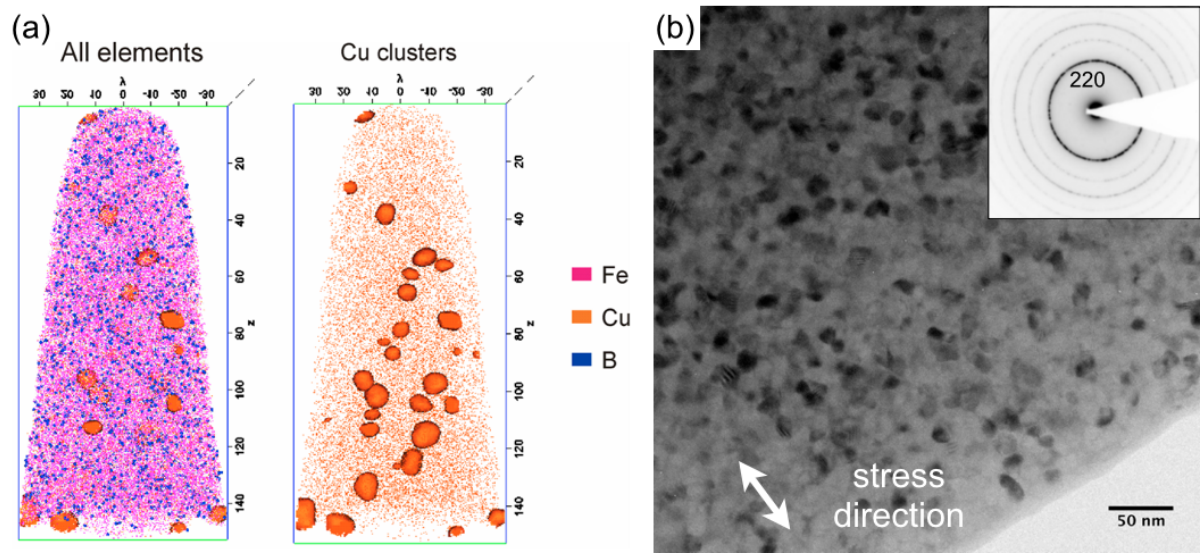

Fig. 1: Figure 1. (a) APT spatial distribution map and (b) BF TEM image of $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ that had been annealed at 695 °C for 10 s in the presence of a stress of 600 MPa. The electron diffraction pattern in (b) shows that the grains have random orientations.

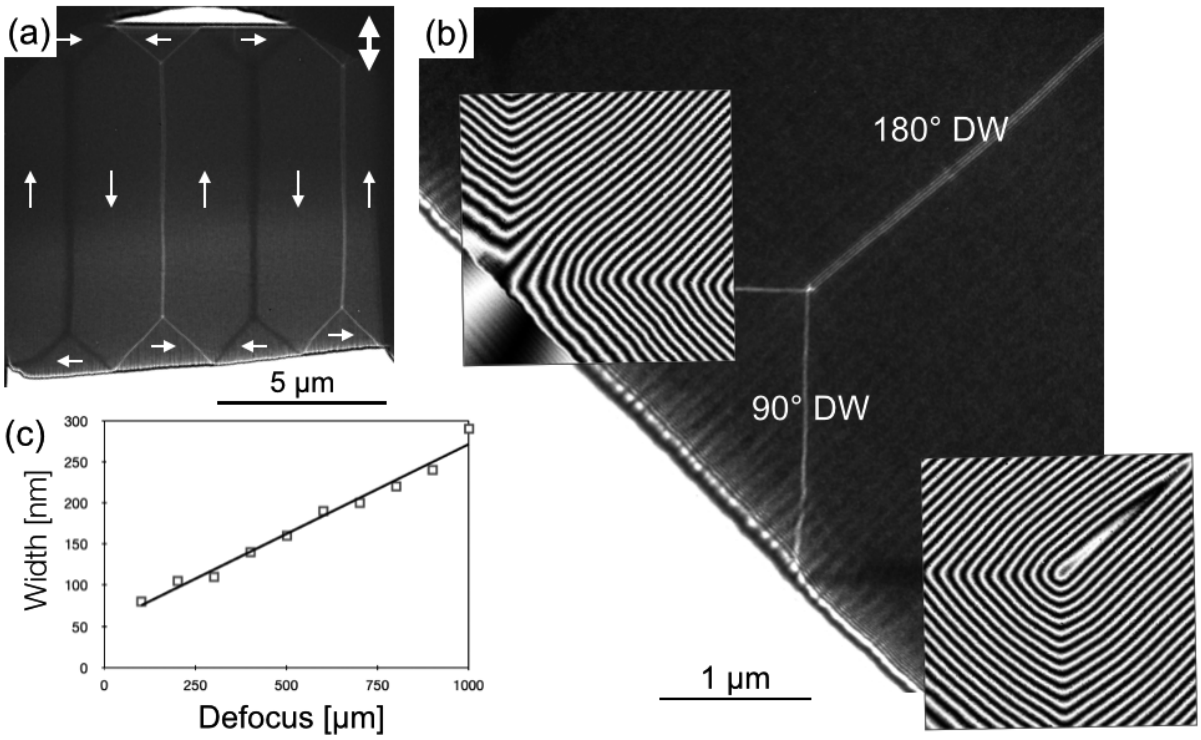

Fig. 2: Figure 2. (a, b) Fresnel defocus image recorded 1 mm overfocus. The double-headed white arrow in (a) indicates the applied stress direction. The insets are reconstructed phase images. The step in phase between adjacent contours is 2π radians. (c) Width of the divergent contrast arising from the 180° DW measured as a function of defocus.