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

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The unique physical and magnetic properties of FeSiNbCuB 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 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 (TEM). A 600 MPa stress was applied to selected samples during a rapid 4 s annealing [2], resulting in strong uniaxial anisotropy perpendicular to the stress direction, as confirmed using bulk measurements performed using a superconductive quantum interference device magnetometer 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. X-ray diffraction and atom probe tomography studies revealed that the samples comprised 80 % of a crystalline Fe_3Si phase with a DO_3 structure and 20 % of an amorphous matrix that was enriched in B and Nb. 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. The stress-annealed specimen was prepared with the applied stress direction in the plane of the FIB lamellae. Structural studies of the samples using TEM revealed a polycrystalline microstructure without any detectable crystallographic texture. 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, on a 2k x 2k charged-couple device camera. Off-axis electron holograms were recorded using an electrostatic biprism located close to the selected area aperture plane of the microscope. The biprism voltage and holographic interference fringe spacing were 110 V and ~ 4.3 nm, respectively.
Figure 1 (a) shows a random arrangement of magnetic domain walls (DWs) in an $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ sample that had been annealed in the absence of an applied stress, as shown in Fig. 1 (a). In contrast, a regular DW pattern consisting of near-perfect 180° and 90° DWs was observed in a stress-annealed sample, as shown in Fig. 1 (b). The application of successive in-plane magnetic fields of 1.4 T applied to the specimen resulted in switching of the magnetization directions of the domains, but the same DW pattern formed at remanence each time. Figure 2 shows Lorentz TEM and off-axis electron holography results obtained from the intersection of a 180° DW with two 90° DWs. 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 (b). Figure 2 (c) shows a corresponding phase image reconstructed from an off-axis electron hologram recorded from the same region of the specimen. Although the mean inner potential contribution to the recorded phase has not been removed from the recorded signal here, the sample thickness is relatively homogenous in this region. Direct measurements from the corresponding phase profiles provided upper limits of (49±3) and (94±3) nm for the widths of the 180° and 90° DWs in Fig. 2(c), respectively. Figure 2 (d) shows approximately equally-spaced phase contours around the intersection of the DWs, generated by displaying the cosine of Fig. 2 (c).

References

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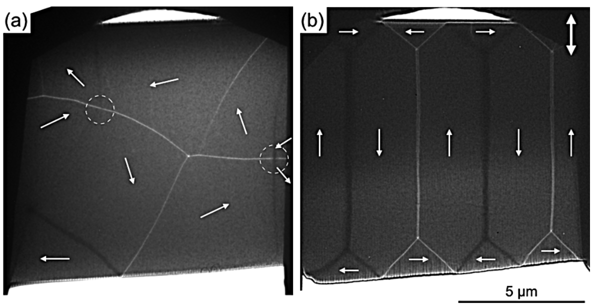

Figure 1. Fresnel defocus images recorded from $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ samples that had been (a) annealed at 695 °C for 10 s in the absence of an applied stress and (b) annealed at 690 °C for 10 s in the presence of a stress of 600 MPa. The Fresnel defocus images were recorded (a) 1 mm and (b) 0.4 mm overfocus. The nominal thicknesses of the FIB lamellae were ~100 nm. White arrows mark the possible magnetization directions in the samples. The dashed circles in (a) mark the positions of cross-tie DWs. The double-headed white arrow in (b) marks the stress direction applied during annealing.

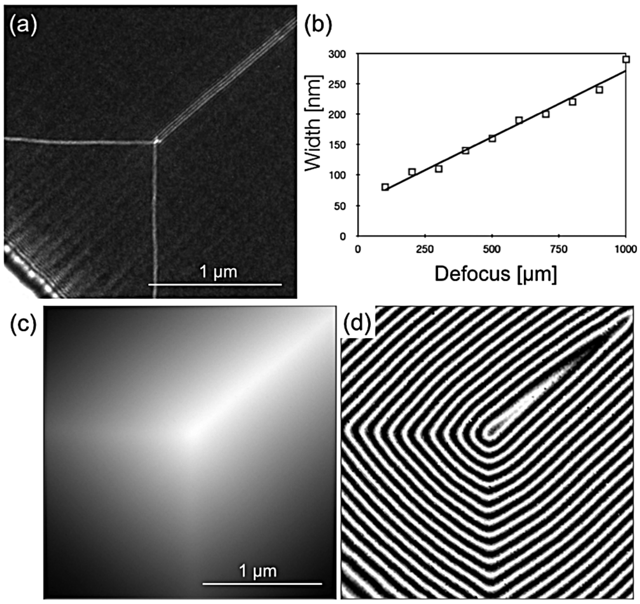

Figure 2. (a) Fresnel defocus image recorded 1 mm overfocus from the intersection of a 180 DW with two 90 DWs in the stress-annealed $Fe_{73.5}Si_{15.5}B_7Nb_3Cu_1$ sample. (b) Width of the divergent contrast arising from the 180 DW measured as a function of defocus. The width of the contrast extrapolated to zero defocus is (5310) nm. (c) Total (magnetic and mean inner potential) contribution to the phase shift recorded from the junction of the DWs using in-focus image-plane off-axis electron holography. (d) Cosine of the phase image (1× amplification), generated from (c), corresponding to a step in phase of 2 radians between adjacent contours.