

Projekt TN01000008 Centrum elektronové a fotonové optiky

Identifikační číslo výsledku: TN01000008/10-005

Název výsledku: Nanoscale calibration sample for magnetic contrast imaging in SEM

Druh výsledku: Gfunk

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Veřejně popsán na:

<http://surfaces.fme.vutbr.cz/laboratories/developed-instruments/2022-calibration-set-for-magnetic-contrast-measurements-with-different-domain-sizes/>

Within the subproject a single multi-scale domain sample was planned. This idea turned out to be unfeasible, as single sample with appropriate magnetic field strength was evaluated extremely challenging after several experimental proof-of-concept trials. Hence, instead, a set of three distinct samples was prepared and characterized in order to fulfill the original aims without any compromising details.

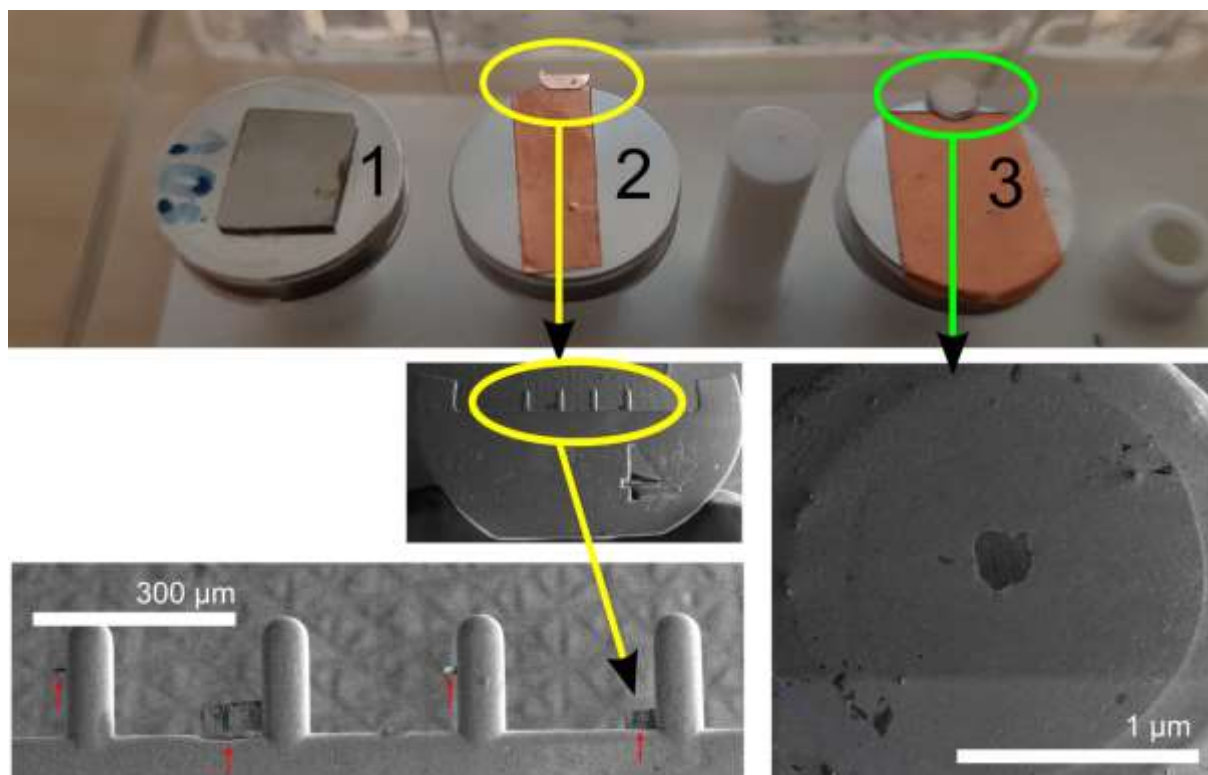


Fig. 1: Top: A set of three macroscopic samples for magnetic SE imaging. From left: bulk FeSi sample (1), wedge-shaped lamella (2), wedge shaped annular disc of cobalt (3). The TEM lamella (2) close up is shown at the bottom, with a yellow arrow pointing to the one shown in this report. The other arrows (red) point to other lamellas with different directions.

1. Bulk FeSi sample

Bulk samples were prepared from FeSi alloy, to ensure large magnetic intensity and also large domain size. The samples were prepared by electropolishing (95% acetic acid, 5% perchloric acid). Characterization was done by Kerr microscopy (with resolution around 500 nm which is enough for this domain size) and SEM imaging (see Fig. 2).

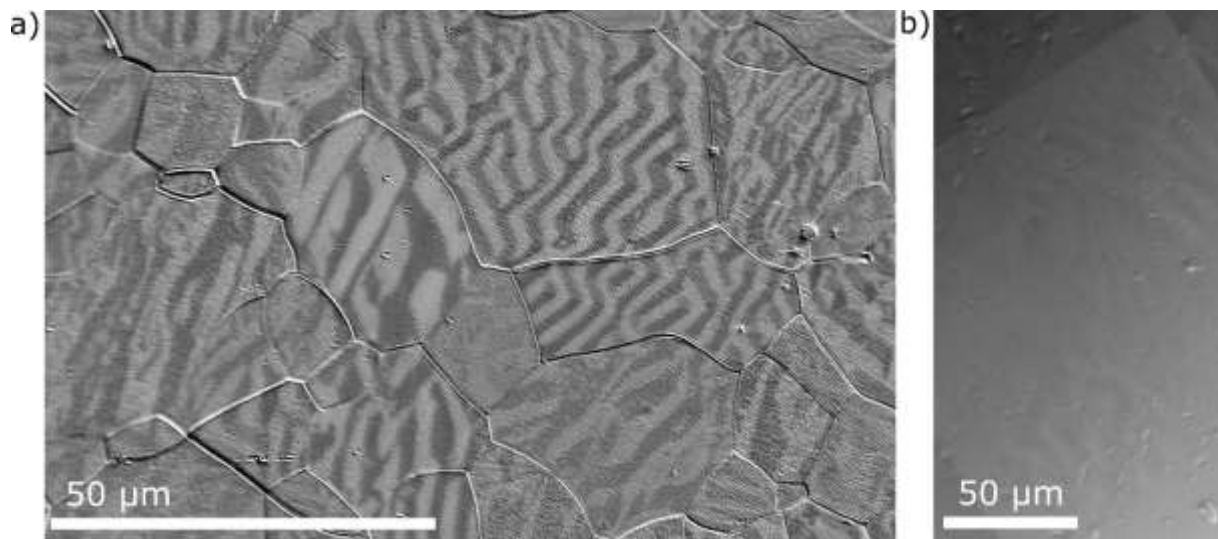


Fig. 2: a) Kerr microscopy image of electrochemically polished polycrystalline FeSi sample, showing different magnetization domains within each grain. The domain size is grain dependent, but on average it is in the range of micrometers. b) SEM SE image taken on TFS Apreo system, showing faint magnetic contrast on the same sample. Imaging of domains of these sizes is rather simple and serves as a benchmark for magnetic contrast imaging in SEM.

2. Wedge shaped TEM lamella

In order to prepare magnetic domains of smaller size (as compared to 1) we have fabricated wedge-shaped TEM lamella. First, a common TEM lamella preparation procedure was followed (Fig. 3a, utilizing Xe Plasma-FIB tool (PFIB, to avoid Ga contamination). However, the last polishing step was different, in order to achieve the wedge shape (Fig. 3b). The aim was to achieve constantly varying lamella thickness h over a relatively large lamella size (hence the PFIB usage).

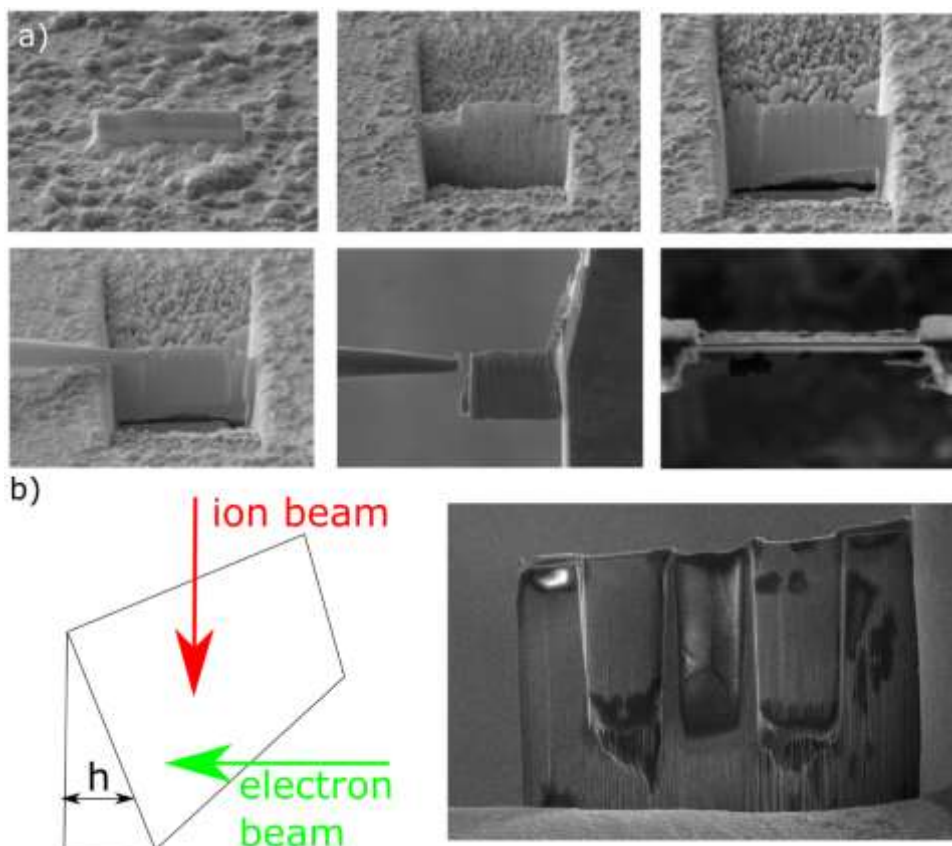


Fig. 3: a) Standard TEM lamella preparation procedure (adapted from CEITEC Nano Lyra3 TEM lamella preparation procedure). b) Wedge-shape lamella schematic (left, showing the direction of ion beam during fabrication – red; and electron beam during imaging -green) and overview image from SEM (right).

The sample was then characterized by magnetic force microscopy (MFM), Fig. 4, and TEM (Fig.5). The domains in this case have one of the dimensions smaller than $1 \mu\text{m}$. The use of Kerr microscope for characterization was pointless due to the domain size.

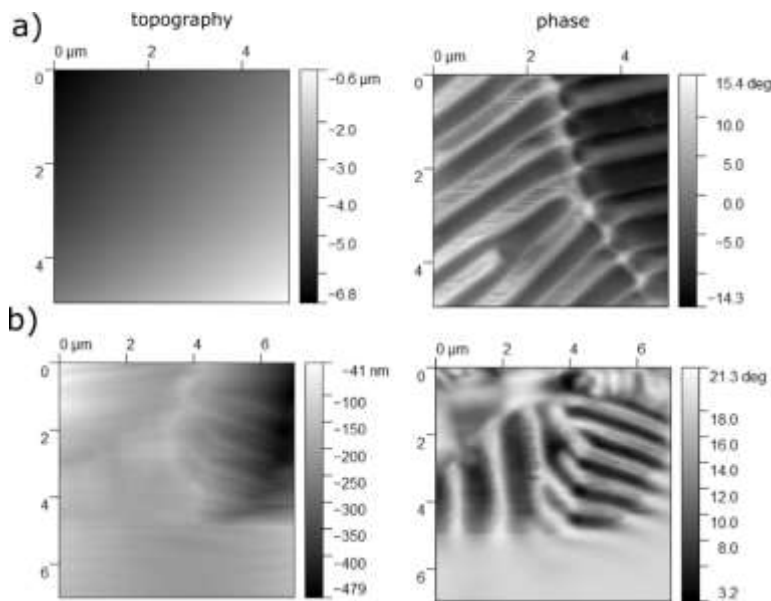


Fig. 4: Two areas (a,b) measured using MFM. The images show sample topography (left column) and phase (right column). The latter images represent a mixture of electrostatic and magnetic force (note that electrostatic forces cannot be excluded from the measurement). The imaged domains are of submicrometer size.

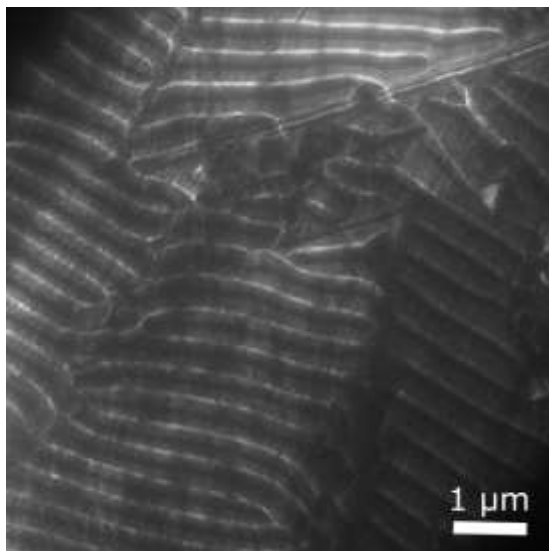
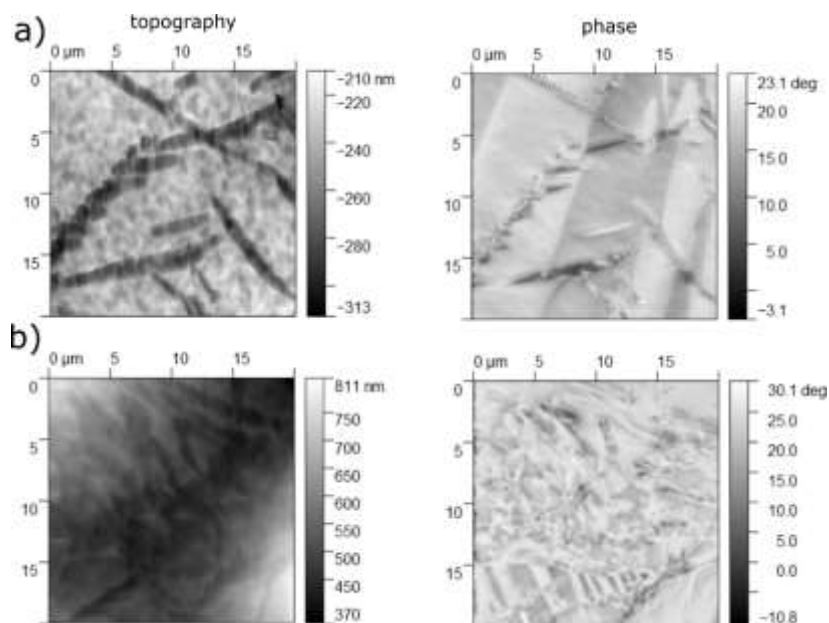


Fig. 5: TEM image of wedge-shaped lamella taken in the Lorentz microscopy imaging mode. The black-and-white contrast exhibits the same pattern as MFM images, justifying the conclusion of prevalent magnetic force in Fig. 4.

3. Annular magnetic wedge shaped disc

Similar effect as achieved on wedge-shaped TEM lamella can be achieved in bulk. The third sample prepared is a wedge-shaped cobalt annular disc. The MFM analysis (Fig. 6) shows again magnetic domain sizes below 1 micrometer.



The samples represent a complementary set of testing specimens for magnetic contrast imaging in SEM. The first, bulk sample, serves as a proof-of-concept test bed. The other two samples exhibit a wedge shape, based on a fact that reducing thickness results in change of the domain size (and magnetic field strength as well). These samples can thus be utilized for SE magnetic contrast resolution testing. Cobalt disc (sample 3) is easier to manipulate compared to sample 2 for bulk SEM imaging (however, indeed it does not allow comparative imaging in TEM). As demonstrated, altogether the samples provide a testing set of magnetic domains imaging with distinct range of domain sizes.