MFM STUDY OF CORE/COVER INTERFACE
IN A MgB_2/Fe SUPERCONDUCTING WIRE

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1. Introduction

In 2001, it was found that MgB_2 material is superconducting [1]. Its temperature of the transition to the superconducting state is T_c = 39 K. Magnesium diboride belongs to the group of ceramic superconductors. So far, ceramic superconductors achieve the highest transition temperatures, among other types of superconducting materials.

For practical applications it is necessary to shape a brittle ceramic material into wires. The manufacturing process called powder-in-tube (PIT) serves for this purpose. According to the starting material we distinguish between the *in situ* and *ex situ* variant of the preparation procedure. In the first approach (*in situ*) the starting material is a powder in the form of a mixture of magnesium and boron elements with appropriate stoichiometric ratio. The powder is first filled into a metal tube. The tube is then drawn into wire of the required shape and dimensions. Finally, the wire is sintered to obtain the superconducting MgB_2 phase. In the second approach (*ex situ*) the starting material is also a powder, but here in the form of already reacted MgB_2 compound. MgB_2 compounds are prepared by powder metallurgy technology. From an economic perspective, the second approach is preferred because the final sintering of powder is not required.

Atomic force microscopy (AFM) [2] is a technique for studying surface properties of samples with high spatial resolution. This technique is based on the measurement of the probe-sample interaction. The probe consists of a sharp tip located at the free end of a flexible cantilever. The principle of measurement is as follows: The probe is brought into close proximity to a sample surface at the desired location. Depending on the tip-sample spacing, short-range or even long-range forces are acting on the tip. If the spacing between the tip and the sample is on the order of a few tenths of a nanometer at most, short-range forces of repulsive character are acting on the tip. This causes the cantilever to deflect or twist. Mechanical changes of the cantilever caused by the action of atomic forces are detected optically. If the tip is moved farther away from the surface, long-range forces (magnetic, electrostatic, etc.) will begin to dominate. Depending on which long-range forces are measured, we distinguish magnetic force microscopy [3, 4], electric force microscopy [5] and other scanning probe microscopy techniques.

Magnetic force microscopy (MFM) [3, 4] is a mode of operation of atomic force microscope in which magnetic force gradients above the surface of a sample can be mapped. To measure magnetic forces, a probes coated with a ferromagnetic thin film are used. The cantilever is excited to vibrate at a frequency, which is close or equal to resonance. Measurement in MFM mode is performed in two steps. In the first step, a probe with a magnetized tip scans in one line the surface topography. Then it rises from the surface into the pre-set distance, where it is assumed that the acting of magnetic forces dominates. In the second and last step, the probe moves along the path that follows the already recorded profile of the topography. This maintains a constant probe-surface separation. The distance of the tip will, however, change by the action of magnetic forces. During the measurement, variations in the phase of oscillation (the phase shift between the excitation force and the vibrations of cantilever) and/or the vibration amplitude of the cantilever are recorded, which both of them reflect the acting of magnetic forces. This procedure is repeated for each row of the raster pattern until the entire studied area is scanned. The results of measurements are to be plotted in two figures. One shows the surface topography. The second maps the magnetic stray fields above the surface. Both images describe the same area on the sample surface.

The measured material here was superconducting MgB_2/Fe wire. The aim of this work was to evaluate the quality of the Fe/MgB_2 interface by observing voids which arise as a result of imperfect contact of the two materials. We believe such voids could be observed by AFM in topography of the cross section.

2. Experimental Details

AFM is a technique for studying surfaces. The problem which we study is, however, in the volume. Therefore we had to design a sample preparation procedure by which we revealed the interface and prepared the sample for measurements performed using atomic force microscope. Sample preparation procedure consisted of the these steps: (1) cutting across (revealing the interface); (2) embedding in mechanically resistant polymeric substance - SPOFACRYL® (fixation in the vertical direction); (3) grinding (flattening the surface); (4) etching in Ar plasma (getting rid of particles that glazed the interface in the process of grinding). Immediately after the plasma etching, the wire sample was transported to a laboratory with atomic force microscope. To prevent oxidation of the etched surface during the transportation, the sample was packed into multiple reclosable polyethylene bags filled with argon gas.

Measured MgB_2/Fe wire of square cross section was produced by the powder-in-tube technique and the *ex situ* approach. The core of the superconductor consists of MgB_2 phase; the sheath is made of iron. The dimensions of the core are 0.7 mm × 0.7 mm. The overall dimensions of the wire are 1 mm × 1 mm.

The topography of the surface and magnetic force gradients around the interface were obtained by Dimension Edge atomic force microscope (Veeco Instruments Inc., USA). In order to minimize the influence of ground vibration, the microscope was placed on a pneumatic anti-vibration table (Technical Manufacturing Corporation, USA). During the measurements, the microscope was covered by an acoustic enclosure. The measurements were carried out at room temperature. We used a probe with model name MESP from Veeco Company. The tip of the probe is coated with a layer of magnetic material based on Co. The nominal radius of the tip apex indicated by the manufacturer is 20 nm. The maximum permissible radius is 50 nm. The probe tip was magnetized before use in the microscope. The quality of the probe and the proper operation of the instrument were checked by measuring and evaluating the calibration grating. In the next step, by measuring a test videotape (Veeco Instruments Inc., USA) in MFM mode, we were determining whether the tip is well-magnetized. The images presented in this paper capture an area of 20 × 20 μm^2 and they were scanned at a rate of 4 μm/s. The resolution of the images is 512 × 512 pixels. The measured data were processed in NanoScope Analysis software (Veeco Instruments Inc., USA).

3. Results and Discussion

Fig. 1 shows the MgB_2/Fe interface measured in magnetic force microscopy mode. The development of topography across the interface is depicted in Fig. 1a. The size of the scanned area is 20 × 20 μm^2. Scratches caused by grinding can be seen on the surface, but they are less pronounced. Scratches usually do not pass into the polycrystalline MgB_2. Position of the interface can be estimated from the record of the phase shift (Fig. 1b). In the Fig. 1b, one can see three regions: a nonmagnetic region, magnetic region and a region of an interface. The nonmagnetic region is homogeneous and has a zero field. In the magnetic region we see domains. At the interface, we often see distinct direction of the magnetic field - black areas. Here it is important to note that the probe is sensitive to the *z* component of the magnetic induction. The phase image thus maps variations in magnetic induction in the vertical direction. By simulation of analogous case of the air/magnet interface it can be demonstrated that, due to the edge of the magnetic material there must exist a minimum in the phase shift. The minimum is always located in the nonmagnetic region behind the edge of a magnet. Black spots at the interface between the nonmagnetic and magnetic region are probably related to the fact that in the vicinity of such interface is a physical edge of a magnetic material.

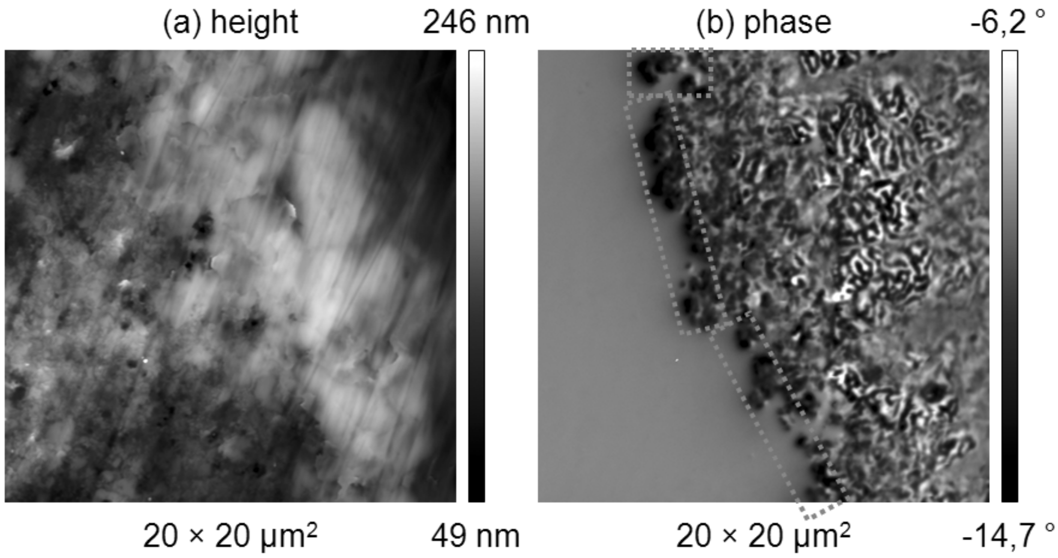

Fig.1: *(a) Topography and (b) phase image of the MgB_2/Fe interface.*

Fig. 2 shows a detailed development of topography and phase shift of the selected section. From the surface morphology (Fig. 2a) we can see where the metal physically ends. From the phase shift (Fig. 2b) we can see where the magnetic part ends. Comparing the two dependencies we can notice, that the minimum of the phase shift is located in the iron region (which is in contradiction to the simulation). We therefore propose the following hypothesis which explains the observed phenomenon: At the MgB_2/Fe interface, there exists a narrow surface layer of iron, which exhibits inferior magnetic properties, because it is damaged. This proves that the minimum of the phase shift moves toward the inside of the iron.

Several voids were observed in two other records of the topography (not shown here). They were located at the boundary of MgB_2/Fe contact. The voids were about 200 to 250 nm deep. One should realize, however, since it is a cross section of the material from the whole void we see only its part.

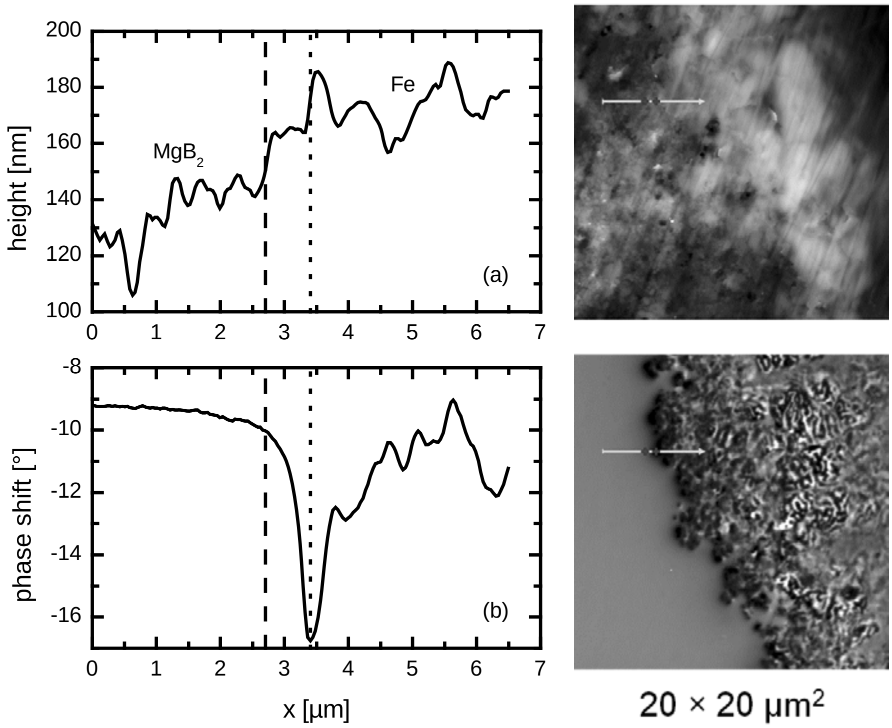

Fig.2: *Cross section through the (a) topography and (b) phase image.*

4. Conclusions

We have developed a procedure by which it is possible to prepare a superconducting wire for measurements on an atomic force microscope. We located the interface between the MgB_2 core and the iron cover. The results of measurements indicate that at the MgB_2/Fe interface, there exists a narrow surface layer of iron, which is damaged. The minimum of the phase shift thus moves toward the inside of the iron.

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