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Experimental research on electric field jump in low magnetic fields: Detection of damage in new *ex-situ* MgB_2 barriers in MgB_2 wires

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ABSTRACT

We explored the incorporation of field sweep (constant current and rapidly increasing magnetic field) into the four-probe method as a new technique to detect defects in barrier layers in superconducting MgB_2 wires. This method allows us to observe jumps in the electric field in low magnetic fields. The scanning electron microscopy results indicate that such a jump originates from cracks in Nb barriers and *ex-situ* MgB_2 barriers. Our research indicates that the field sweep allows us to detect damage to barriers that are made of superconducting materials. This method can be the basis for an industrial method for detecting damages in MgB_2 wires. These defects reduce the critical current of MgB_2 wire. Detection and removal of these defects will allow us to produce MgB_2 wires with *ex-situ* MgB_2 and Nb barriers that will have improved critical current density. Manufacturing of MgB_2 wires with new *ex-situ* MgB_2 barriers is a new technological concept. This type of barrier is cheaper and easier to manufacture, leading to cheaper MgB_2 wires. Moreover, we show that critical current can be measured by two methods: current sweep (constant magnetic field and quickly increasing current) and field sweep.

1. Introduction

The *ex-situ* MgB_2 barrier was invented in 2006 by A. Morawski of the Institute of High Pressure Research in Warsaw (Poland) and B Głowacki of the Department of Materials Science and Metallurgy, University of Cambridge (United Kingdom) [1]. MgB_2 wires with *ex-situ* MgB_2 barriers are fabricated by the powder-in-tube (PIT) method. This type of barrier is also cheaper and easier to use in the PIT treatment than Nb, Ti, Fe, and Ta barriers. The energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD) results of Kario et al. [2] indicate that the *ex-situ* MgB_2 barrier allows us to obtain high-purity *in-situ* MgB_2 material in the core or filaments of the wire. This is because the *ex-situ* MgB_2 barrier reduces the reactions of Mg and the sheaths of the wires. Moreover, the *ex-situ* MgB_2 barrier does not increase the hardness after cold drawing as in the case of an Nb barrier. This might indicate that cold drawing will create less damage in *ex-situ* MgB_2 barriers.

Scanning electron microscope (SEM) studies show that the *ex-situ* MgB_2 barrier is uniformly distributed along the wire and creates good contact with the wire sheath and the *in-situ* MgB_2 material because it has low shrinkage [2–5]. Measurements of the critical current for MgB_2 wires with *ex-situ* MgB_2 barriers, and iron and copper sheaths show that these wires have very low critical current density (J_c) anisotropy of about 2–4 % (with only a small difference between the J_c in perpendicular and parallel magnetic fields) [4,5]. These advantages demonstrate that this barrier can also be applied in other superconducting wires. Further studies are needed to improve the barrier properties. The research showed by Häßler [6], Maeda [7], Susner [8], Adamczyk [9], Dou [10] and Zhou [11] for MgB_2 wires with Nb barrier and MgB_2 materials indicate that these wires have great potential for application.

SEM images of MgB_2 wires [12] show that a Nb barrier might be damaged after cold drawing. This damage to the barrier causes the formation of phases such as Cu_2Mg [13] and pure B. In addition, it reduces the amount of superconducting material and decreases the critical current density. Eikin presents results on the U–I characteristic for low-temperature superconductor (LTS) wires [14]. These measurements suggest that this method makes it possible to detect

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a fault in the structure of NbTi wires. When the current sweep type is used in MgB_2 wires, we cannot identify reasons for the appearance of an electric field. Similar electric fields occur for short wires [15] and NbTi wires with damaged microstructure [14]. Studies indicate that the critical current density J_c on short and long MgB_2 wires is similar [16,17]. The J_c in these wires is dependent on parameters of the annealing process. In contrast, J_c in the wires with the damaged microstructure depends on the number and size of defects. Currently, there are no reports of a measurement method that makes it possible to detect damage in Nb and *ex-situ* MgB_2 barriers. The lack of such a method impedes study on increasing J_c in MgB_2 wires.

In this paper, we present the results obtained with the field sweep method for MgB_2 wires with Nb and *ex-situ* MgB_2 barriers. These measurements indicate that the field sweep method allows us to detect damage in Nb and *ex-situ* MgB_2 barriers and will allow us to eliminate these defects in the future.

2. Experiment and sample preparation

Critical current was measured using the four-probe resistive method at 4.2 K by two different methods: the current sweep type - constant magnetic field and increasing current [14,18] - and the field sweep type - constant current and increasing magnetic field. Studies were made for specially selected samples with low critical current value. The length of wires was 20 mm in perpendicular magnetic field and 70 mm for measurements in parallel magnetic field. The critical current (I_c) was determined on the basis of the 1 μV/cm criterion. Critical current measurements were conducted in the International Laboratory of High Magnetic Fields and Low Temperatures in Wroclaw [16]. The research was carried out on MgB_2 wires with *ex-situ* MgB_2 barriers obtained from the Institute of High Pressure, Polish Academy of Sciences (PAS), and MgB_2 wires with Nb barriers fabricated by Hyper Tech Research. The wires were annealed under pressure [19,20] at the Institute of High Pressure in Warsaw. Parameters of the measured samples are presented in Tables 1 and 2 (with Nb wire in copper also studied for comparison). The magnetic field was generated by a Bitter magnet. Its maximum magnetic field of about 14 T was obtained over a time of 170 s. Measurements of critical temperature were conducted by using a physical properties measurement system (PPMS Model 7100, Quantum Design, AC current – frequency of 15 Hz and 100 mA) with fields ranging from 0 up to 14 T at the International Laboratory of High Magnetic Fields and Low Temperatures. Microstructure analysis was performed on a FEI Nova Nano SEM 230 in the Institute of Low Temperature and Structure Research (ILT&SR), PAS in Wroclaw and with the help of a Carl Zeiss microscope in the Institute of High Pressure (IHP), PAS in Warsaw.

The value of the critical current in the current sweep type was determined on the basis of 1 μV/cm. The same criterion was used in the field sweep type. Fig. 1 indicates that a quick increase in the magnetic field from 0 T to 14 T creates a loop of electric field ($t = 3$ min – t the times in which the perpendicular magnetic field increases from 0 T to 14 T, and from 14 T returns to 0 T). A slow increase of the magnetic field does not create this loop, however. These studies have proved that the electromotive force creates a changeable flux of magnetic field. This theory indicates that the loop in Fig. 1 is generated by the electromotive force. In Fig. 1, for quick increase of the magnetic field, we can determine I_c on the basis of 1 μV/cm because the electric field of 2 μV/cm comes from the electromotive force. The measurements showed that the transition to the normal state in the wires during slow and quick increase of the magnetic field is at the same point (blue line A – Fig. 1). The quick increase in the magnetic field allows measurements of I_c for large values of current and does not destroy the samples. We carried out a study of the critical current for undoped and doped MgB_2 wires with Nb, Fe (Hyper Tech Research, Inc) sheaths and *ex-situ* MgB_2 barriers (Institute of High Pressure Physics), NbTi wires (Donetsk Institute for Physic and Engineering, National Academy of Sciences of Ukraine), and the commercial bismuth strontium calcium copper oxide (BSCCO – SuperPower) tape in perpendicular and parallel magnetic fields. These results showed that the value of critical current from both measurement methods (current and field sweep method) is the same.

3. Experimental results

SEM studies showed that most of the MgB_2 wires have good Nb and *ex-situ* MgB_2 barriers, although we sometimes see damage in Nb and *ex situ* MgB_2 barriers. This damage is created during cold drawing of MgB_2 wires. SEM images were collected for samples and at places where there was a jump in the electric field in low magnetic fields. The SEM image in Fig. 2 shows breakage in the Nb barrier for SiC doped MgB_2 wires with GlidCop® sheath. This breakage reduces the amount of MgB_2 superconducting material and creates intermetallic phases inside and outside of the fiber. SEM images and EDX maps (Fig. 3) of cross-sections of MgB_2 wires with *ex-situ* MgB_2 barriers show the places where there this barrier is damaged. In MgB_2 wires with *ex-situ* MgB_2 barriers, cold drawing occasionally creates the damage in the *ex-situ* MgB_2 material. This damage in the *ex-situ* MgB_2 material allows diffusion of Cu from the sheath to react with Mg as well as the *in-situ* MgB_2 material. This migration through the *ex-situ* MgB_2 barrier creates phases of Cu_2Mg and $CuMg_2$ [13]. These phases are very deleterious, because they reduce the number of connections between grains in the *ex-situ* MgB_2 barrier. A smaller number of connections between grains can significantly reduce the transport critical parameters for the *ex-situ* MgB_2 barrier. This process reduces the amount of MgB_2 material in the fiber, and may create a higher amount of boride phase in the fiber.

Our studies indicate that most samples have the characteristics shown in Fig. 1. Sometimes, however, we see a jump in the electric field in low magnetic fields (Fig. 4(a) – black curve). We will hereafter call this magnetic field range–the transition region (TR). Fig. 4(a) shows the results obtained by the field sweep type and a comparison of MgB_2 wires with Nb barrier in a GlidCop® sheath with and without a transition region. The transition region in MgB_2 wires with Nb barrier is shown in low magnetic fields from 0.1 T to 1 T (sample A1 – black curve (Fig. 4(a)). This range of magnetic field indicates that it comes from the Nb barrier, because pure Nb has upper critical field close to 1 T [21]. We might see that the transition region decreases the critical magnetic field (B_K) from 8.5 T to 7 T

Table 1
Composition and construction of MgB_2 wires.

Samples identifier	Mg:B additive	Barrier	Mono sheath	Multi sheath	Fill factor (%)	Diameter (mm)	Multi filament
A1 (damaged)	1.10:2 10%SiC	Nb	Cu (20%)	Glidcop (30%)	16.6	0.83	6
A2	1.10:2 10%SiC	Nb	Cu (20%)	Glidcop (30%)	16.6	0.83	6
B1 (damaged)	1:2	*ex situ* MgB_2		Glidcop (50%)	50	1.2	1
B2	1:2	*ex situ* MgB_2		Glidcop(50%)	50	1.2	1