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Use of amorphous boron and amorphous nano boron powder mixture in fabrication of long in-situ MgB_2/Fe wires

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ABSTRACT

We report a study on the structural and transport properties of long in situ MgB_2/Fe wires that are prepared by use of amorphous boron and nano amorphous boron powders with 50–50% weight ratio. The wire samples are fabricated by means of a standard Powder-in-Tube (PIT) method. Transport measurements are applied under high magnetic fields, of up to 9 T, obtained in a Bitter magnet. We find that use of a mixture of the amorphous boron and amorphous nano boron precursor powders at equal amounts is very promising way to fabricate long wires without any degradation in transport engineering J_{ce} values in the presence of low and moderate magnetic fields.

1. Introduction

Suitable superconducting materials for industrial applications must have large transport critical current densities (J_c) under the range of intended magnetic field strengths. Since the discovery of as a non-copper oxide Magnesium Diboride (MgB_2) superconducting material with highest critical temperature of 39 K amongst conventional superconductors [1]. Due to its structural simplicity, low anisotropy, lack of intrinsic weak-link effect, low raw material cost and higher J_c values, lots of work has been done in order to achieve higher transport critical current densities for MgB_2 superconducting tapes and wires with different types of sheathing materials till now [2–7]. Some effort has also been made to determine which boron (B) powder will give the best J_c and the best mechanical properties [8–13]. The Mg/B variation has also been studied as factor in controlling the transport properties in MgB_2 superconducting materials [14]. As the materials are far from the optimum values, not standardized, and the sample mass density is usually 50–60% of theoretical value [15,16] MgB_2 superconducting materials still have potential worth investigating.

MgB_2 superconducting wires and tapes are commonly fabricated by powder-in-tube process (PIT). As already well known, there are two different classification approaches depending upon the identifying starting material: a) in-situ, and b) ex-situ. The both approaches have their advantages and disadvantages although the in-situ process has not been standardized yet. The disadvantage of this process is that the density of the MgB_2 core structure after its formation is rather low due to imperfect densification during wire making and conversion of Mg+2B with lower mass density to relatively higher mass density MgB_2. This situation leads to formation of voids during sintering reducing the current carrying cross section of the MgB_2 core. Some extra effort has also been made to overcome this problem but a further mechanical deformation and extra heat treatment did not help this situation [17–19].

The other main problem is to choice the best sheathing material for MgB_2 superconducting wires which leads to higher transport critical current densities at low and high magnetic fields for industrial purposes. To achieve this aim, lots of additions have been tried [8,11,20–24] but to our knowledge of the literature any MgB_2 superconducting material suitable for both low and high magnetic fields strengths has not been reported yet.

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In this present work, we investigate the improvement of transport J_c in low and moderate magnetic field for long in-situ processed MgB_2/Fe superconducting wire by mixture of both amorphous boron (Pavezyum - PVZ Boron 95, 95–97%, particle size: <1 μm) and nano sized amorphous boron (Pavezyum - PVZ nano Boron, >98.5%, particle size: <250 nm) at equal amounts. There are three reasons why we use different-sized boron powders: 1) to compensate the agglomeration problem, 2) to achieve better critical current density values, and 3) to achieve better packing ratio. We prefer simple industrial grade soft iron tube for economical compatibility of the final product in potential industrial applications.

2. Experimental setup

The starting materials in this work were commercially available powders of magnesium (PVZ Atomized Magnesium, 99%, particle sizes ≅ 149–74 μm), amorphous boron (PVZ Boron 95, 95–97%, particle sizes <1 μm), and amorphous nano boron (PVZ Nano Boron, >98.5%, particle sizes <250 nm) from Pavezyum Advanced Chemicals (Turkey). The accurately weighted powders (Mg = 8.00026 g, amorphous boron = 3.53823 g, and amorphous nano boron = 3.53846 g) suitable for Mg+2B stoichiometric ratio are homogeneously mixed by means of rotary ball milling for 3 h at ambient conditions. Through the study, the ball-powder mass ratio was set at 4:1. After mixing the product was collected and filled into an Iron (Fe) tube of 351 mm in length (12 mm OD and 9 mm ID) in argon atmosphere. Both ends of the PIT processed MgB_2/Fe tube was closed by aluminium. Starting mass density of the Mg+2B inside the tube was about 1.5 g/cm^3. Cold drawings through progressively decreasing die diameters were applied with several intermediate heat treatments (600 °C, 1 h) under argon pressure to fabricate the wire samples. Wire samples with the outer diameter of 1.00 mm were cut to about 150 mm long pieces and annealed in a three-zone programmable tube furnace at two different temperatures of 850 and 900 °C for 1 h under 5–10 bar argon atmosphere with 5 °C/min heating/cooling ratios. 20 mm in length of sintered wires were cut off from both ends to avoid any irregularities due to open ends, only middle parts of the wires were used for investigation. Wire samples were named to indicate sintering temperatures as F5E900, the last three digits being sintering temperature in degree Celsius.

Low temperature dc electrical resistivity measurements were carried out on the samples with the four-probe method using a closed-cycled cryostat (Cryo-Industries). Both voltage and current contacts were made with soldering on the wire samples. We measured the temperature (10–50 K) dependence of resistivity of the samples running 100 mA DC current through the sample in the cryostat. Details are given in Ref. [16].

Critical current of the wires after sintering was measured in liquid helium with the four probe method. These transport measurements were carried out at ILHMFLT (International Laboratory of High Magnetic Fields and Low Temperatures) in Wroclaw using a Bitter 14 T magnet on 20 mm long wire samples. The magnetic field was applied perpendicular to the wire axis. The 1 μVcm^{-1} criterion was used.

The magneto-resistivity of samples was measured at 10–35 K using Quantum Design physical properties measurement system (PPMS) with a magnetic field sweep rate of 0.050 T/s and the amplitude of up to 14 T at ILHMFLT in Wroclaw.

Phase formation of the samples are characterized by X-ray diffraction (XRD) investigation by means of a Rigaku Multiflex + XRD diffractometer with a monochromatic beam (wavelength of 1.5418 Å) derived from a CuKα target in the range of 2θ = 10-90° at a scan speed of 3°/min and a step increment of 0.02° at room temperature. A scanning electron microscope (SEM, JEOL 6390-LV) with the accelerating voltage of 20 kV was used to investigate the surface morphology of the monofilamentary MgB_2/Fe wires.

3. Experimental results

Fig. 1 shows the transport measurements of the MgB_2/Fe monofilamentary F5E850 and F5E900 wire samples measured in PPMS. The T_c^{offset} is 36.5 K (ΔK = 1.5 K) and 37.1 K (ΔK = 1.0 K) for F5E850 and F5E900 wire samples, respectively. The F5E850 sample has a lower T_c and a wider transition width in the transport measurement. The Δρ value of the F5E850 sample is higher than the value of the F5E900 sample. This may be an evidence of relationship between sintering temperature and superconducting properties as well as boron depletion in to the iron sheath causing Mg rich core with better connectivity.

The effect of the applied magnetic field, B, on the resistivity has been examined in the temperature range between 10 K and 35 K. Fig. 2 shows a set of ρ(B, T) curves for several applied field strengths for F5E850 and F5E900 wire samples. As can be seen from Fig. 2 the irreversibility fields (B_{irr}) of both samples are almost equal at 35 K and B_{irr} of the wire F5E850 gradually becomes superior as the temperature decreases. This behaviour may be attributed to better pinning ability of the wire F5E850 due to higher granularity since the wire F5E900 are more crystallized.

Fig. 3 shows the engineering critical current (I_{ce}) measurement of the F5E850 and the F5E900 monofilamentary wires under high magnetic fields ranging from B = 3 T–9 T. The magnetic field was applied perpendicular to the wire axis in the Bitter magnet. It was found that a high I_{ce} (4.2 K) > 150 A (J_{ce} >1.91 × 10^4 A/cm^2) at B = 3 T was obtained for F5E850 wire, sintered at 850 °C. Critical current measurement was also applied on the F5E900 sample and measurement results can be seen as inset in Fig. 3. The critical current values of both samples at B = 3 T are above 150 A but the I_{ce} values of the F5E850 wire are higher than the F5E900 wire at magnetic fields higher than 3 T.

Fig. 4(a) shows the transport engineering critical current density results of the wire samples of F5E850, F6-850 and F7-850 measured at T = 4.2 K for different magnetic field strengths up to 9 T. As given in Fig. 3, the J_{ce}-B performance of the F5E850 wire is significantly better than that of the wire F5E900. This situation indicates that the flux pinning ability of F5E850 is improved for higher magnetic field

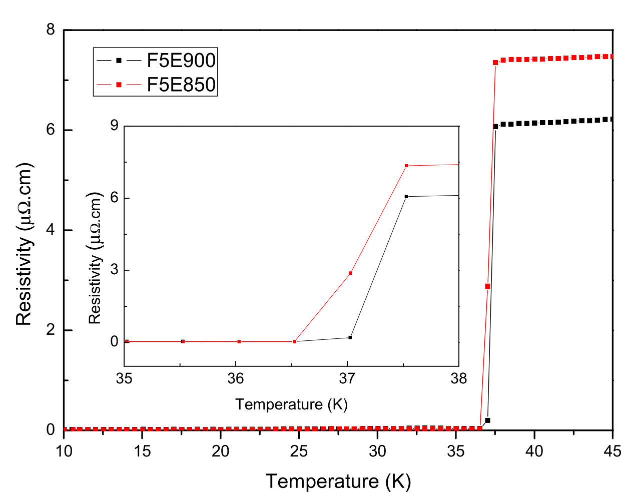

Fig. 1. Transport measurement of the F5E850 and F5E900 in-situ monofilamentary MgB_2/Fe wire samples.