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Transport properties of sealed MgB_2/Fe/Ni multifilamentary wires heat treated in air

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ABSTRACT

Ni sheathed multifilamentary MgB_2 wires with Fe barrier and Cu stabilizer were prepared by the *in situ* Powder-In-Tube (PIT) method. After rolling, the ends of the wires were sealed by a simple capping technique and the wires were directly heat treated in air, without vacuum or any inert atmosphere. The quality of the wires was assessed by analysing the phase assemblage and measurement of superconducting properties such as R–T, J_C–T and J_C–H. Phase analysis revealed that only traces of MgO was formed in the superconductor core. Typical multifilamentary wires prepared by this method showed a $T_C \approx 38.5$ K and $\Delta T_C \approx 1$ K and J_C of the order of 10^5 A/cm^2 at 6 K (0 T) and 10^4 A/cm^2 at 4.2 K (6 T) respectively. These values are quite comparable with the values obtained for wires heat treated in inert atmosphere.

1. Introduction

MgB_2 is identified as one of the most promising material for future superconducting applications with cost effective operation. Progress of multifilamentary MgB_2 conductor development is so rapid that kilometers long stabilized MgB_2 wires were now industrially manufactured by companies like Columbus Inc., Hyper Tech Research Inc., etc. [1,2]. With a high critical temperature (T_C) of 39 K, MgB_2 offers the advantage of operation at 20–30 K using cryocooler or liquid hydrogen which is not possible for NbTi and Nb_3Sn based low temperature superconductors (LTSs). MgB_2 has the potential to compete and even to excel the performance of NbTi/Nb_3Sn for practical applications if the upper critical field (H_{C2}), the irreversibility feld (H_{irr}) and the in-field critical current density [$J_C(H)$] are improved [3,4]. Carbon doping by various additives like nano carbon, carbon nano tubes, nano metal carbides, carbohydrates, SiC and burned rice husk are found to be very promising for enhancing the field performance of MgB_2 conductors [5–9].

For the fabrication of multifilamentary MgB_2 conductors, Powder-In-Tube (PIT) method is the most popular and industrially preferred process. In PIT the precursor powder is filled in a suitable metal tube. The precursor used may be either a prereacted MgB_2 (*ex situ* method) powder or a mixture of elemental Mg and B powders (*in situ* method). The tubes are then mechanically rolled/extruded/drawn into desired size and shape followed by heat treatment [10–13]. Heat treatment is usually done in vacuum or inert atmosphere to avoid Mg oxidation. Fabrication of MgB_2 wires would become much more easier if the vacuum/inert atmosphere condition could be avoided.

This paper presents a feasibility study of making multifilamentary MgB_2 wires by direct heat treatment in air and their characterization.

2. Materials and methods

2.1. Wire preparation

Stoichiometric quantities of Mg powder (Goodfellow, -325 mesh, 99.8% purity) and amorphous B powder (Merck, -325 mesh, 99% purity) were weighed using an electronic balance (Mettler AE240) and homogeneously mixed. Fe tubes 5 cm in length, 6 mm in outer diameter (OD) and 4 mm in inner diameter (ID) were used for the fabrication of PIT wires. The tubes were filled with homogeneously mixed Mg and B powders (without any additives) and mechanically compacted. Iron studs were used to plug the ends of the tubes. The composite tubes were groove rolled down to wires of diameter 2.2 mm. To prepare multifilamentary wires the groove rolled monofilamentary wires were cut, bundled and packed inside Ni tubes of typical dimensions of 8 mm in OD, 6 mm in ID and 10 cm in length. Cu wires of 0.75 mm in

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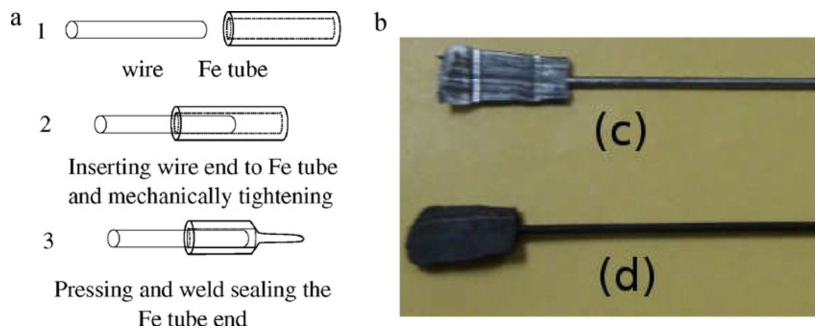

Fig. 1. (a) Schematic diagram of the end sealing by capping technique. (b) Photograph of the end sealed wires by capping technique before(c) and after(d) heat treatment.

diameter were bundled inside the Ni tube along with the monowires. The composite tubes were then groove rolled to the desired dimensions typically, 1.67 mm in OD and about 2 m in length.

2.2. End sealing & heat treatment

The rolled multifilamentary wires were cut into pieces of 10 cm length and these pieces were used for the end-sealing and air-heat treatement experiments. The end-sealing was done by a capping technique before heat treatment. For this, the ends of the conductors were inserted into suitable iron tubes of short length (1.5 cm) and mechanically fixed with the conductor using a hydraulic press and then the ends of the iron tubes were welded using DC arc without any flux by keeping the sample cooled using a wet cloth. Fig. 1(a) shows a schematic illustration of the capping technique.

Fig. 1(b) shows the photograph of a typical sample end-sealed by the capping technique before (c) and after (d) heat treatment. Sealing by direct welding/fusing of the ends of the wire was also tried initially, but with limited success only. This is because during welding, Mg + 2B core got oxidised at the ends and hence the multifilamentary wires could not be sealed properly. After sealing the ends by capping, the wires were heat treated at 650 °C for 2 h in a muffle furnace without vacuum or any inert atmosphere. Monofilamentary MgB_2/Fe wires of similar dimensions were also end-sealed by the capping technique and heat treated together with the multifilamentary conductors for phase analysis. We have heat treated a number of wire samples with this type of sealing for various studies. These include multifilamentary wires of different length, diameter and configurations and the failure rate in all these cases was almost zero. All the samples showed equally good phase purity and superconducting properties as those heat treated in argon atmosphere. The only drawback we observed by heat treating in air is the slight surface oxidation of the outer sheaths. This doesn't matter since the surface oxidation is minimal for sheath materials such as Ni, monel, inconel, etc. at temperatures in the range of 600–700 °C for short duration.

2.3. Characterizations

Phase analysis of the samples were performed using X-ray diffractometer (Philips X'pert Pro) with Cu Kα radiation employing a high sensitive detector called X'Celerator and a monochromator at the diffracted beam side. Phase identification of the samples was performed using X'Pert Highscore Software in support with ICDD-PDF-2 database. Microstructural investigation of the samples was done using an optical microscope. The resistance versus temperature (R–T) measurements were carried out by a closed cycle cryocooler integrated in the cryostat by DC four probe resistive method. The in-field transport J_C from 4 T to 8 T at 4.2 K was measured using a 16 T magnet-cryostat system (Oxford). R–T, $J_C - T$, $J_C - H$ measurements were done on 3 samples of the same batch and variations in these measurements were within 5%. The graphs are plotted using the mean values of the data.

3. Results and discussions

3.1. Phase and microstructural analysis

A typical XRD pattern taken on the powdered MgB_2 core of the monofilamentary wire heat treated in air is shown in Fig. 2(a). The pattern in general is found to be as good as or better than the samples processed in inert atmosphere [14,15]. The pattern shows sharp MgB_2 peaks with only traces of residual Mg, Fe and MgO. The presence of Fe is from the sheath during the mechanical recovery of core. The presence of MgO observed is due to the air entrapped inside the tube during the filling of the powder, before sealing the ends. Fig. 2(b) shows the cross section of a typical 4 filament wire of 1.67 mm in diameter heat treated in air. Optical microscopy measurements, taken with the help of a scale built into the eyepiece, were used for the exact estimation of core and sheath cross sectional areas. For a typical multifilamentary conductor the cross sectional area is around 8% for the MgB_2 core, ∼8% for Cu, ∼27% for Fe and ∼57% for the outer Ni sheath.

3.2. Measurement of superconducting properties

Multiwire samples of 5 cm in length, cut from the middle of the air-heat treated wires were used for measurement of superconducting properties. The wire has an outside diameter of 1.67 mm and has 4 MgB_2 filaments. Fig. 3(a) shows the resistance versus temperature plot of a multifilamentary wire, heat treated at 650 °C in air. Fig. 3(b) shows the enlarged view of the plot near the superconducting transition region. The wire exhibits a sharp superconducting transition ($T_{C-\mathrm{onset}}$) at around 38.5 K with transition width $\Delta T_C < 1$ K. This gives a clear indication of the high phase purity, good crystallinity and thermal stability of the sample.

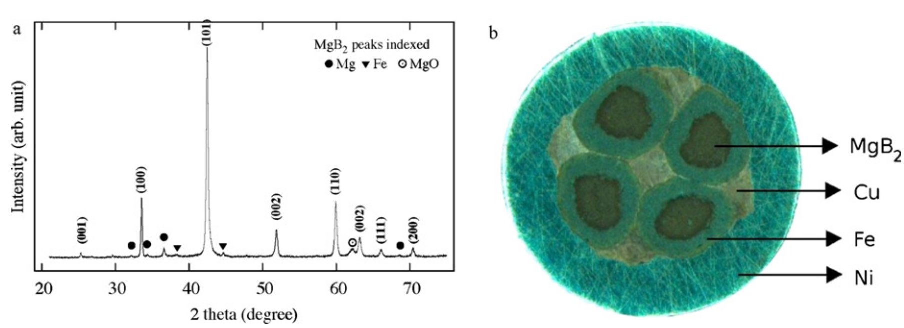

Fig. 2. (a) XRD pattern of the MgB_2 sample recovered from the wire heat treated in air after end sealing by capping technique. (b) Cross section of a typical multifilamentary wire (OD ∼ 1.67 mm).

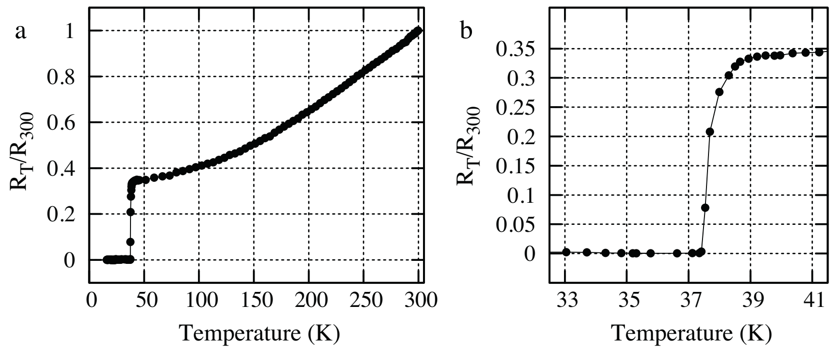

Fig. 3. (a) *R*–*T* plot of a typical multifilamentary wire heat treated in air. (b) Enlarged portion of *R*–*T* plot near the superconducting transition region.

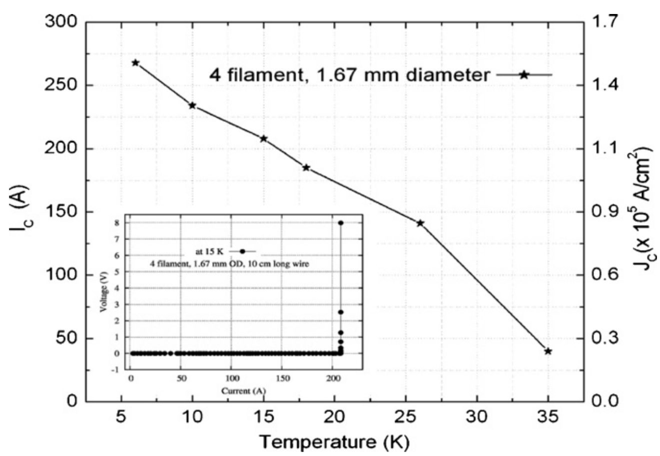

Fig. 4. Variation of I_C and J_C values of a typical multifilamentary MgB_2 wire heat treated in air with temperature. Inset: *I*–*V* plot of the wire at 15 K.

Fig. 4 shows the variation of self-field I_C and J_C of the wire with respect to temperature. The distance between the voltage contacts was 1 cm and critical current was defined at 1 μV criterion. The wire shows a self-field J_C of ~1.5×10^5 A/cm^2 at 6 K and ~2.3×10^4 A/cm^2 at 35 K. The inset of Fig. 4 shows the self-field *I*–*V* characteristics of the wire at 15 K. The *I*–*V* measurement was done with a current ramp rate of 1 A/s. The wire shows a critical current of ~208 A, corresponding to a critical current density of

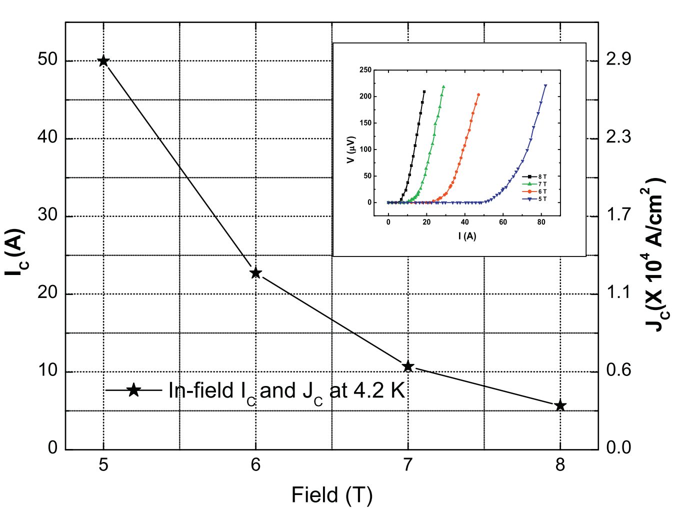

Fig. 5. Variation of I_C and J_C values of a typical multifilamentary MgB_2 wire heat treated in air with magnetic field. Inset: *I*–*V* plot of the wire at 4.2 K at different magnetic fields.

1.18×10^5 A/cm^2 at 15 K. Fig. 5 shows variation of I_C and J_C with applied field. A J_C of 2.86×10^4 A/cm^2 has been measured in the sample at 4.2 K and 5 T. The inset shows *I*–*V* plots of the wire at different applied fields ranging from 4 T to 8 T. The values are quite comparable or better compared to those measured for pristine MgB_2 wires heat treated in inert atmosphere [8,16].

4. Summary

The feasibility of making MgB_2 based multifilamentary wires by direct heat treatment in air has been demonstrated. The study has been conducted on multifilamentary wires with Fe barrier, Cu stabilizer and Ni sheath. The ends of the wires were sealed by a simple capping technique using Fe tubes followed by heat treatment in air. Characterization and evaluation of the samples have shown that they are as good as or better with respect to phase purity and superconducting properties than the samples prepared by the usual method of heat treatment in inert atmosphere. The method can be adopted for preparation of short samples for research and development as well as long conductors for industrial applications. The possibility to avoid vacuum or inert atmosphere for production of MgB_2 superconductors will make the process simple, economical and attractive.

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