JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS Vol. 10, No. 11, November 2008, p. 2981 - 2984

PIT method for obtaining of MgB_2 wires with SiC powder added

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The most popular process of MgB_2 wire fabrication is the so-called in situ powder-in-tube (PIT) process, which is characterized by filing metallic tubes with Mg and B mixed powder, drawing and rolling the material into wires and, finally, by applying a heat treatment. X-ray diffraction analyses were carried out for MgB_2 cores and SiC-added wires. XRD patterns reveal that nearly single-phase MgB_2 was obtained. Also, in this paper are presented the obtained magnetization curves.

(Received September 1, 2008; accepted October 30, 2008)

Keywords: MgB_2 wires, SiC, PIT method, Magnetization curves, XRD

1. Introduction

The discovery of the magnesium diboride superconductor, MgB_2 with a transition temperature near 40 K has promoted a new interest in the area of fundamental and applied research on superconducting materials, has caused a renaissance of interest in intermetallic superconductivity [1].

This, combined with the discovery of superconductivity in YPd_2B_2C and the RNi_2B_2C series several years ago [2, 3], seems to indicate that the old idea of looking for high intermetallic T_c values in compounds rich in light elements is still a valid guiding principle.

MgB_2 is considered a promising candidate to replace low temperature superconductors (LTS) or to fill in niches of applications requiring intermediate characteristics between LTS and high temperature superconductors (HTS).

2. Preparation of MgB_2 wires by the PIT method

A very convenient way of producing MgB_2 wires of industrial lengths is the so-called PIT method, which is currently studied in a large number of laboratories. The PIT method has the advantage of low costs of material and deformation techniques, which are relatively simple. Particularly, drawing or rolling in wires with very fine filaments represents a difficult problem. MgB_2 is a relatively tough and hard phase, and thus cannot be plastically deformed. The only way to obtain a filamentary configuration is to start with powders that are packed in metallic tubes. There are two methods for obtaining. The first is ex situ method in which the MgB_2 powder, available commercially or prepared is packed in a metallic tube. The other is in situ method in which the powders composed by Mg and B are packed in a metallic tube and then, the MgB_2 phase forms inside the tube by heat treatment.

The tapes manufactured by ex situ method exhibited higher values of critical current density (J_c), than those by in situ method. However, critical current density for the tapes manufactured by ex situ method must be improved for practical applications. The high values of Jc for tapes manufactured by ex situ method were achieved using materials for sheath with high mechanical forces.

MgB_2 wire bars formed by ex situ process [2, 3] are manufactured by filling of metallic tubes with MgB_2 powder already reacted, in Ar atmosphere. Then, the tubes are drawing, rolling and finally, an annealing heat treatment is applied. The processing steps are presented in Fig. 1.

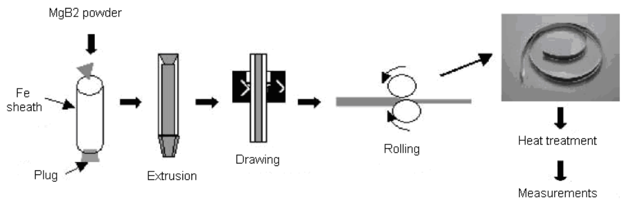

Fig. 1. PIT method for manufacturing of MgB_2/Fe mono-filamentary tape.

The transition temperature of MgB_2 phase in filaments is strongly affected of stresses induced during the deformation process. In spite that, the wire bars produced by using the pre-reacted powder of MgB_2 carry a high critical current after deformation, even before of any heat treatment [4, 5]. Connectivity between grains is reached after a final heat treatment, between 850 and 950ºC, and after that the tapes are presenting a single superconducting transition.

The selection of metallic sheath was reduced at those elements or alloys which do not react or react very slightly with MgB_2. As materials for sheath for manufacturing of MgB_2 plated wires were tested Ni, Cu, Ag and Fe. For mechanical consolidation of the sheath were used composed materials as Cu-Ni [6], Nb-Ta/Cu/stainless steel [7], stainless steel [6, 8], Ag/ stainless steel [7] and Fe/ stainless steel [9]. Until now, Fe was identified to be the most indicated material of sheath for wires manufacturing, presenting a weak reaction with MgB_2 during the final heat treatment. In tapes with iron sheath, only a thin reaction layer < than 1 µm was observed at Fe/MgB_2 interface after 30 minutes, at 950ºC [10]. Because, the optimized parameters for high J_c carry on towards lower temperatures and smaller times, this reaction layer is expected to be decreased. The MgB_2 wires obtained by PIT method achieve high critical current densities or even higher than 10^5 A/cm^2 in self field, at 4.2 K [11].

The addition of Y_2O_3 and SiC nanopowders has been successful in raising both J_c and the irreversibility field, as reported by Wang et al. [12] and Dou et al. [13], respectively. The final heat treatment also leads to a densification of the filaments [14], which is crucial for improving the J_c values.

3. Experiments

For our experiments, we used simple or added (SiC) MgB_2 powder and for sheath, Fe.

The addition of nanometre-size SiC powder increases B_{c2}, and hence the J_c, in high magnetic fields [15]. This J_c improvement could be attributed to the substitution of carbon from SiC for boron in MgB_2. Commercially powder of MgB_2 (Alfa-Aesar, average size of particles is of 4.0 µm) was packed in Fe tubes, of two types of Fe, the last being of Armco type with lengths of 7-10 cm. This process of packing was achieved in air. The inner and outer diameter of tubes was 4 mm, respectively 6 mm. these tubes were cold drawing in wires with sizes presented in table 1, where de and di represent the outer diameter and respectively inner diameter of the sheath before rolling, and l represents the side of the square section achieved after rolling. All wires were cold processed to final dimension without cracks. Figure 2 presents the longitudinal section of a superconducting wire of MgB_2/Fe.

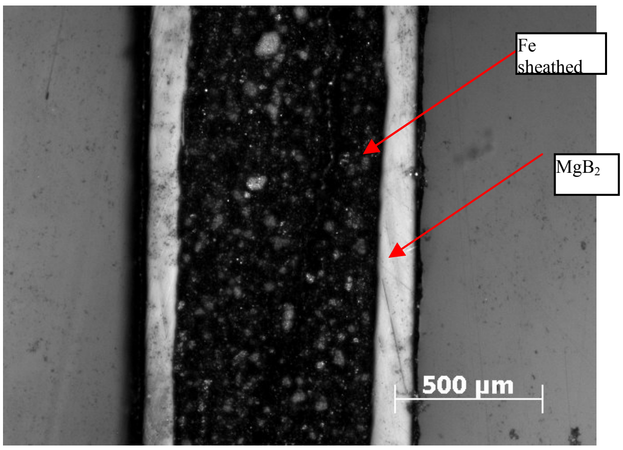

Fig. 2. Longitudinal section for a superconducting wire of MgB_2 /Fe.

Heat treatment of the obtained wires was achieved in argon atmosphere, and the treatment parameters, also are indicated in Table 1. It must be mentioned that in Table 1 are presented the average values of 5 determinations for each type of sample.

Table 1. Conditions for obtaining of MgB_2/Fe wires.

Sample no.	Sheath material	Additive, SiC[%]	T [ºC]	Profile shape	de [mm]	di [mm]	l [mm]
1	Fe	0	750	square	5	-	0.92
2	Fe	5	750	square	5	-	0.95
3	Armco Fe	5	750	square	7.5	3.1	0.95
4	Armco Fe	5	800	round	7.5	3.1	Φ 2

4. Results and discussion

For identification of the crystalline chemical compounds by X-ray diffraction was used the diffractmeter by type D8 Advance-Bruker-Analytical X-ray systems. The X-ray patterns for the analyzed samples were compared with data registered in corresponding ASTM cards.

Comparisons among the analyzed samples with Mo tube after these were polished, so that can be seen maximal surface of the core, are presented in Fig. 3.

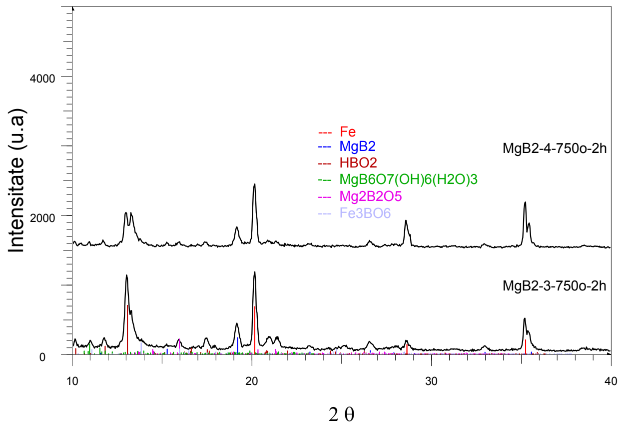

Fig. 3. X-ray diffraction pattern of 1 and 2 samples (Mo tube).

The ratio between the peaks intensity for Fe phase and peaks intensity for MgB_2 phase is different from one sample to other, in terms of the sheath walls thickness, Fig. 4.

XRD patterns, both for un-doped samples, and for those doped with SiC (Figs. 4 and 5), indicate the presence of two main phases, Fe and MgB_2, also minor impurities of MgB_4 and MgO. The diffraction lines appear at angles larger than in the case of Mo tube, because theirs position is direct proportional with the wave length of the tube (for a X-ray tube with copper anode, λ = 1.5406 Å, for Mo anode

λ = 0.70930 Å). When the reaction temperature is relative low, there is un-reacted Mg in samples; when the reaction temperature is relatively high, MgB_4 phase appears, [16]. The forming of MgB_2 from Mg and B powders is exothermic. The sintering temperature of 750ºC is too low to improve the crystallinity. The lattice parameters determined for MgB_2, are: a = 3.0827 Å and c= 3.5245 Å.

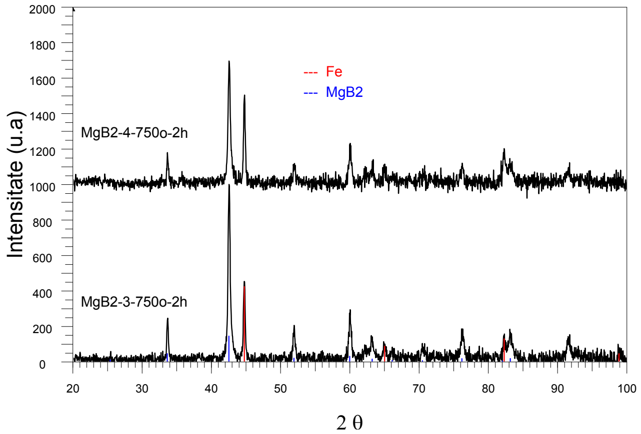

Fig. 4. X-ray diffraction pattern, comparative sheath-core, for 1 and 2 samples .

Fig. 5 presents the temperature dependency of electrical resistivity in zero magnetic field of MgB_2 material. The transition temperatures for initial and ending points are 39 K and respectively 38 K, indicating thus that the superconductivity is indeed realized in this system, [16].

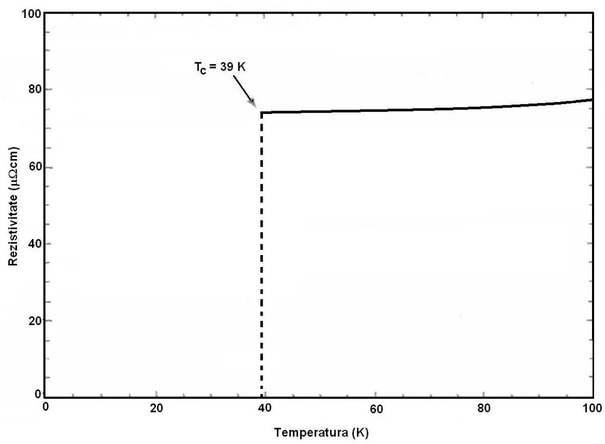

Fig. 5. The temperature dependency of electrical resistivity in zero magnetic field of MgB_2 material.

Fig. 6 presents the curve of electrical resistivity versus temperature for a superconducting wire sample of third type. The T_c for the analyzed sample was of 38.45 K.

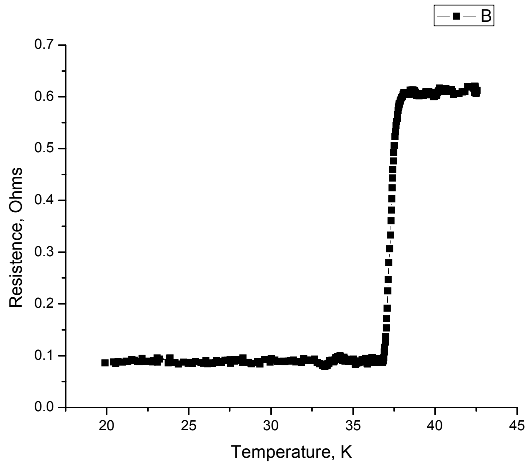

Fig. 6. The curve of electrical resistivity versus temperature for the sample 3.

For characterization of magnetic properties are achieved hysteresis loops at different temperatures using a magnetometer. Didn't notice a significant dependence of temperature for hysteresis loops measured at some temperatures from 45 K to 80 K, in low field region. Thus, the loop obtained at T = 45 K was considered to be a good approximation for magnetic part, in entire range of temperature from 5 K to 35 K. Magnetization loops corrected are presented in figure 7, for (a) H / / c and (b) H / / ab.

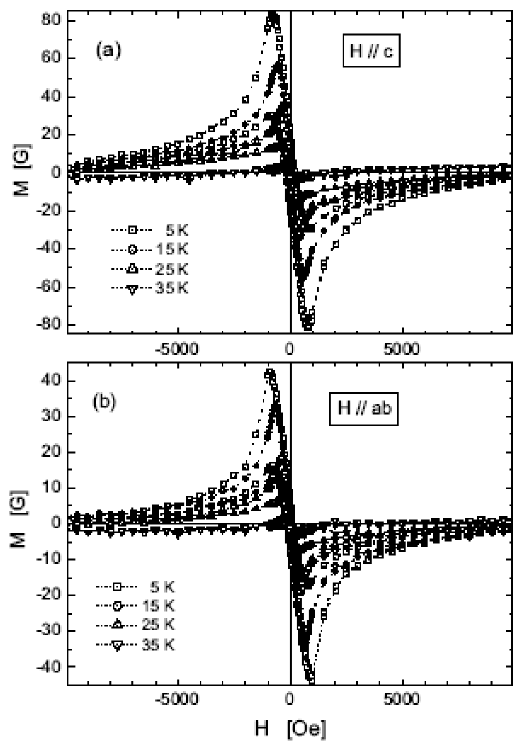

Fig. 7. Hysteresis loops for wires of MgB_2/Fe, measured of T = 45 K, [17]

5. Conclusions

Is presented the most known method used for manufacturing of MgB_2 monofilamentary and multifilamentary wires, the PIT method. In summary wires samples of SiC-doped MgB_2 superconductor were fabricated from PIT method. A common problem for all PIT techniques represents the necessity for a densification of the filaments, during the final reaction (in situ), or during the re-crystallization (ex situ), with dense cores, which achieve improved values of J_c. In the experiments for obtaining of MgB_2 superconducting profiles were prepared more samples of precursory powders mixtures using as additive, SiC.

X-ray diffraction analyses were carried out for MgB_2 cores and SiC-added wires. XRD patterns reveal that nearly single-phase MgB_2 was obtained.

These tapes and wires were cut in small pieces with 3-4 cm in length and was measured the electrical resistivity versus temperature. As transition temperature was identified the 38.45 K value.

Also, in this paper are presented the obtained magnetization curves.

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