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Fabrication of Ultra Fine MgO Particles Added BSCCO 2223 Tapes with Different Precursor Phase Constituent

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Abstract

Ultra fine MgO particles added BSCCO tapes were fabricated by tape casting using Doctor Blade Method and enclosed by silver foil for different starting compositions (that is, 2223 major, 2212 major). In order to obtain optimum microstructure, thermomechanical treatment was done. Microstructure and phase were analyzed by XRD, SEM and DTA. The critical current density was measured under magnetic field at 77K. The tapes fabricated from the precursor powder with BSCCO-2223 phase (>90%) result in a microstructure with a larger grain size and higher transport critical current density value under magnetic field at given thermomechanical treatment conditions.

Keywords: BSCCO 2223, Microstructure, MgO, Flux pinning

I. Introduction

BSCCO 2223 tapes are most prospective conductors for next generation of power application such as transformer (>10MVA), power cable (~3,000A) and motor (>1000hp). There are two obstacles that hinder practical application; i) reproducibility resulted from complexity of the BSCCO-2223 formation and ii) flux creep, resulting in rapid decrease in critical current density (J_c) under magnetic field.

It has been well known that significant magnetic field penetration can occur in the interior of BSCCO superconductors above a critical magnetic field(H_{C1}).

If the magnetic flux lines within the superconductor are not strongly pinned in place, then Lorentz forces can cause the flux lines to migrate, resulting in resistive energy dissipation. The BSCCO superconductors have been found to exhibit significant flux creep in modest magnetic fields, at temperatures well below the critical temperature.

Therefore, the application of these materials in high magnetic fields is seriously limited, unless the flux creep is reduced by enhanced flux pinning. The operating temperature and magnetic field will have to be reduced to less than 30K, and few tesla for the BSCCO system. Recently, nanorods of MgO were grown and incorporated into high T_c superconductors (HTS) to form nanorod-HTS composites[1]. The J_c of the nanorod-HTS composites is enhanced dramatically at high temperatures and magnetic fields as compared with reference samples. In our previous works[2, 4], ultra-fine MgO added Bi-2223/Ag tape made by tape casting method showed increased J_c under magnetic field.

In this study, ultra-fine MgO particles were added to the two different starting precursors (i.e., one consist of BSCCO-2223 phase, the other consists of 2212 and other impurity phase), by way of planetary ball milling followed by ultrasonic treatment to disperse fine MgO particles in the matrix homogeneously.

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BSCCO-2223/MgO/Ag superconducting tape has been fabricated using tape casting followed by repeated thermomechanical treatment. The results were discussed in the viewpoint of microstructure development and magnetic field dependence of J_c with different starting precursors.

II. Experimental procedure

The reagent grade of $Bi(NO_3)_3 \cdot 5H_2O$, $Pb(NO_3)_2$, $Sr(NO_3)_2$, $Ca(NO_3)_2 \cdot 4H_2O$ and $Cu(NO_3)_2 \cdot 3H_2O$ was weighed in the Bi:Pb:Sr:Ca:Cu molar ratio of 1.8:0.4:2:2.2:3 and then dissolved in distilled water. The precursor powder was produced by the spray drying of nitrates solution[5], [6]. Detailed experimental procedure is shown schematically in Fig.1. Partially reacted BSCCO precursor powder (M1) and fully reacted BSCCO precursor powder (M2) were mixed with ultra-fine MgO powders(10nm) respectively. Partially reacted BSCCO precursor powder and fully reacted powder without MgO is donated B1 and B2 respectively. They were mixed with organic vehicles such as solvents(Metyl Isobutyl Keton), binder(Polyvinyl Butyral) and plasticizer(Dibutyl Phthalate) by means of ultrasonic treatment and planetary ball milling. The tape prepared by using a Doctor Blade was enclosed by silver foil (25μm thickness). The organics have been removed at 600°C for 24 hours. The BSCCO/MgO/Ag tape was heat-treated and pressed (2,000 psi) repeatedly. Other detailed sample preparation conditions are summarized in Table 1.

Differential thermal analysis for the tapes was conducted. Cu K_α radiation, using as X-ray diffractometer was used to identify the phase in the samples. The microstructure was observed by SEM and TEM. The transport critical current density was measured by standard DC 4-probe method[7] with a criterion of 1 μV/cm at 77K under magnetic field .

Table 1. Summarized sample preparation conditions.

MgO	3 vol%
Sample notaion with Calcination condition	B:without MgO, M:with MgO 1: 800°C/2h, grinding, 840°C/15h, ball milling, 840°C/15h, ball milling (2212 major) 2: 1 + 845°C/50h, ball milling, 845°C/200h , ball milling (2223major)
Thermomechanical condition	Temperature range: 830°C-845°C Holding times at Max. temperature: 30-100 hours No. of Pressing: 1-3 times

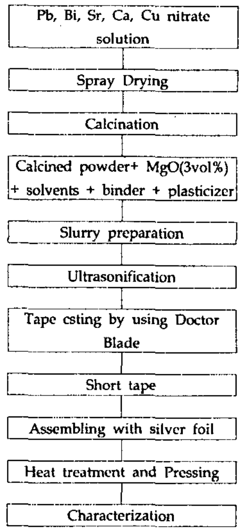

Fig. 1. Flow chart for the experiment

III. Results and Discussion

The XRD patterns showed that the major phase in the precursor powder(M1) was BSCCO-2212; Ca_2PbO_4, $Bi_9Sr_{11}Ca_5O_x$ and CuO were identified as minor phases and major phase in the precursor powder (M2) was BSCCO-2223(>90%) ; BSCCO-2212

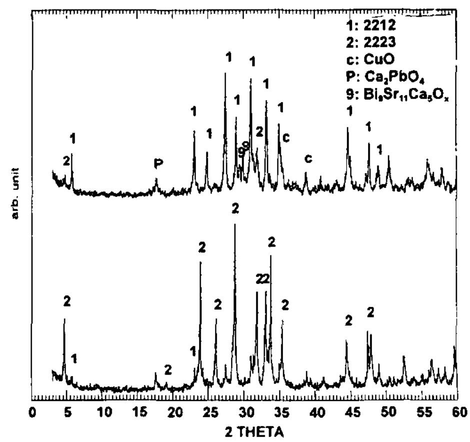

Fig. 2. X-ray diffraction patterns of the starting precursor powder

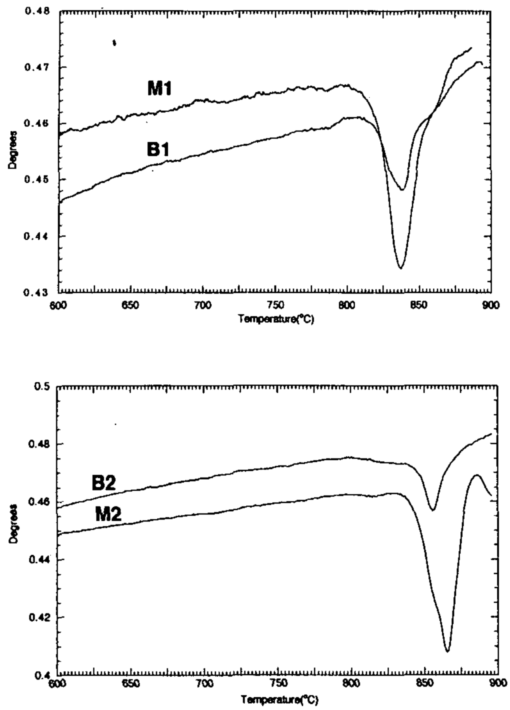

Fig. 3. DTA curve for BSCCO/MgO/Ag tapes.

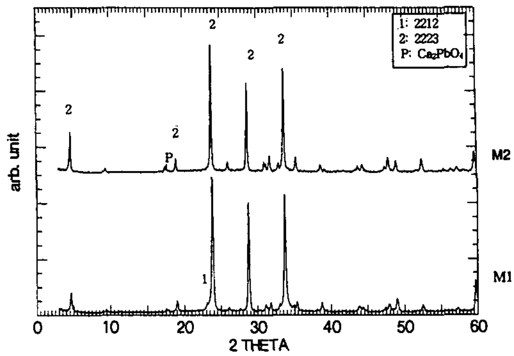

Fig. 4. X-ray diffraction patterns of the oxide core surface for the tape of (a) M1 and (b) M2 heat treated at 840°C for 180 hours and intermediately pressed for two times (840/30/50/100).

and Ca_2PbO_4 were identified as minor phases (Fig.2). The MgO peak was not detected because of relatively small volume.

The DTA traces(Fig. 3) show the temperature of main endothermic peak of B1 (without MgO) was little higher than that of M1 and on the contrary, M2 is higher than that of B2 (without MgO). In case of M1 starting from complex phase assembly, that is a possibility that fine MgO particles react with other minor phase. In case of M2, which consists of mostly BSCCO-2223, assuming no reaction with MgO and BSCCO-2223 matrix, melting point of matrix can be raised by addition of MgO particles that have high melting point.

Fig. 4 shows XRD patterns of fully heat-treated tapes. Most of the peaks correspond to those of the (00*l*) plane of the BSCCO-2223 phase and small amounts of BSCCO-2212 and Ca_2PbO_4 phase.

Fig. 5 (a) and (b) are the typical SEM images of the top fracture surface of the oxide core for the tape at given thermomechanical treatment conditions. A comparison of photographs in Fig.5(a) and (b) indicates that size of BSCCO grains for the M2 tape is larger than that of M1 and homogeneity of M2 matrix is better than that of M1 at given thermomechanical treatment conditions. These results suggest that microstructure control is possible by selecting the starting phase assembly along with fine MgO particles. In case of M1, starting from BSCCO-2212

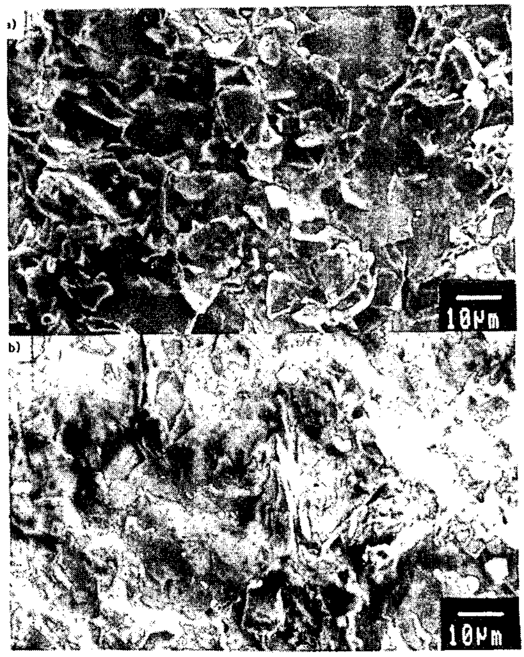

Fig. 5. SEM image of the top fracture surface of the oxide core for the tape of (a) M1 and (b) M2 heat treated at 840oC for 180 hours and intermediately pressed for two times (840/30/50/100).

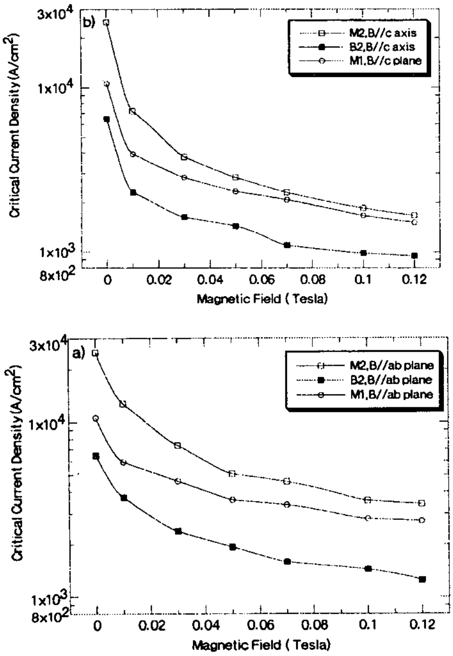

Fig. 6. Magnetic field dependence of the critical curren densities of the BSCCO-2223/MgO/Ag tapes heat treated at 840°C for 180 hours and intermediately pressed for tw times(840/30/50/100)
(a) magnetic field applied parallel to tape surface (B//a-b plane), (b) magnetic field applied perpendicularly to the tape surface (B//c-axis).

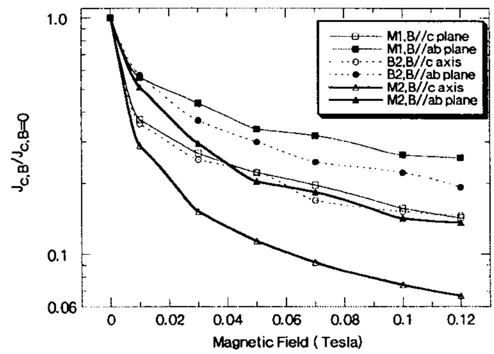

Fig. 7. Magnetic field dependence of the normalized critical current densities of the BSCCO-2223/MgO/Ag tapes heat treated at 840°C for 180 hours and intermediately pressed for two times (840/30/50/100).

phase and other impurity phases which supplies liquid phase, there are some enhancement in properties such as healing of microcracks, grain growth and grain connectivity caused by lager amount of liquid phase. However, it is difficult to control the microstructure because there is a possibility of the MgO particles react with alkaline earth cupurates which exist during heat treatment. In case of M2, starting from BSCCO-2223 phase, driving force for grain growth is less than for M1 because most reactions have been occurred during preparing precursor powder. Processing temperature also becomes little higher and that gives rise to grow alkaline earth cupurates grains that cause deterioration of superconducting properties. However, it can be overcome by using reactive precursor powder such as spray dried granules in this study.

Fig. 6 (a) and (b) show the magnetic field dependence of transport critical current density for M1, M2 and B2 samples at 77K, with applied field parallel to the a-b plane and the c-axis. Fig. 7 is a normalization of Fig. 6 (a) and (b), and obviously observed enhancement of flux pinning strength with ultra-fine MgO addition. In Fig.6 (a) and (b), J_c values of M2 always exhibit higher value than those of M1 and B2. These can be well explained as the improvement of weak links at BSCCO-2223 grain boundaries and enhancement of flux pinning strength resulting from the ultra-fine MgO addition, which is consisted with the microstructure analysis shown in Fig. 8. Many dislocation lines in TEM micrographs were observed along MgO particles and formed networks around MgO particles. It suggests that improvement in flux pining could be due to defects such as dislocations produced by finely dispersed MgO particles, which can provide pinning site.

IV. Conclusions

The tapes fabricated from the precursor powder with BSCCO-2223 phase (>90%) result in a microstructure with a larger grain size and higher transport critical current density value under magnetic field at given thermomechanical treatment conditions. Ultrafine MgO particles were finely dispersed in BSCCO-2223 matrix. The transport current density was less sensitive to the magnetic field for MgO added BSCCO tapes due to the introduction of pinning sites such as dislocations produced by finely dispersed

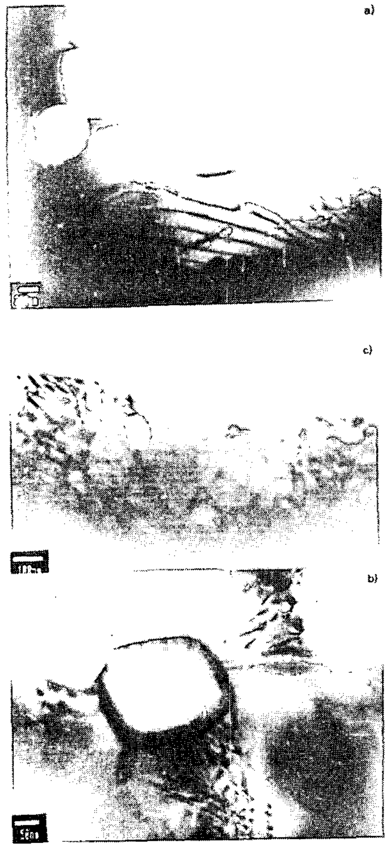

Fig. 8. TEM micrographs of the BSCCO-2223/MgO/Ag tapes heat treated at 840°C for 180 hours and intermediately pressed for two times(M1, 840/30/50/100)
(a) shows dislocation lines around MgO particles, (b) shows dislocations around MgO particle, (c) shows dislocation networks around MgO particles

MgO particles in BSCCO-2223 matrix.

Acknowledgments

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