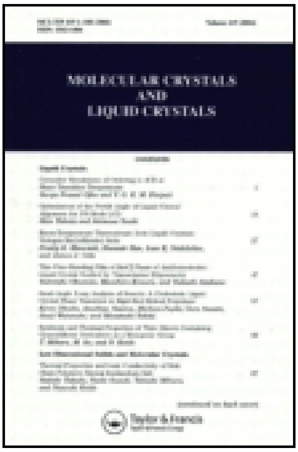

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Tomoko Goto [a]
[a] Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya, Japan
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EFFECTS OF SPINNING CONDITION ON CRITICAL CURRENT DENSITY OF HIGH-Tc OXIDE SUPERCONDUCTING FILAMENTS PRODUCED BY SUSPENSION SPINNING METHOD

TOMOKO GOTO
Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya, Japan

Abstract We have studied the preparation of a high-Tc oxide superconducting long filament using a suspension spinning method: The oxide powder was suspended in a polymer solution and the viscous suspension was extruded as a filament into a precipitating medium and coiled on a winding drum. The obtained filament was heated to remove volatile component and to generate the superconducting phase. The Jc was dependent on the suspension medium and the oxide powder contents of the spinning dope,as well as the heating condition. The highest Jc values at 77 K and 0 T, 1285 A/cm^2, 1940 A/cm^2 and 1045 A/cm^2 were attained for the Y-Ba-Cu-O, (Bi,Pb)-Sr-Ca-Cu-O and Tl-Ba-Ca-Cu-O filaments respectively.

INTRODUCTION

The discovery of a high-Tc oxide superconductor with Tc exceeding the liquid-nitrogen temperature promised wide application in future technology. However, such oxide superconductors are usually brittle and difficult to be fabricated into tapes or wires, which are useful in large scale engineering applications such as superconducting magnets, power transmission lines, etc.. Its applications are also hindered by the weak-link problem in the sintered materials, which is typified by very low transport critical current densities (Jc).

We have studied the preparation of the high-Tc oxide superconducting long filament using a suspension spinning method[1-4]: The oxide powder was suspended in a polymer solution and the viscous suspension was extruded as a filament into a precipitating medium and coiled on a winding drum. The obtained filament was heated to remove volatile component and to generate the superconducting phase. It is important in this technique how to enhance the densification of the oxide in the filament and to form the fewer and cleaner grain boundaries. The dispersion behaviour, alignment and densification of the oxide were dependent on the spinning condition, hence the microstructure and superconducting properties of the filament heated was affected by the spinning conditions. The present paper deals with the effects of spinning condition on Jc of high Tc $Y_1Ba_2Cu_3Ox$, $(Bi,Pb)_2Sr_2Ca_2Cu_3Ox$ and $Tl_2Ba_2Ca_2Cu_3Ox$ superconducting filaments produced by suspension spinning method.

EXPERIMENTAL

A fine mixed powder with nominal composition of $Y_1Ba_2Cu_3Ox$ was prepared by coprecipitating the carbonates of Y, Ba, and Cu. The powder was calcinated at 1123 K for 7.2 ks and the particle size of the heated powder was up to 200 nm. The powder was suspended in PVA or PAN solution. The suspension dope was extruded as a filament into a precipitating medium of methyl alcohol and coiled on a winding drum. The as-drawn filament was sintered at 1253 K for 300 s in oxygen gas flow, followed by furnace cooling.

Appropriate amount of Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$ and CuO powders was mixed, calcined at 1073 K for 15 h and pressed into pellet and then sintered at 1123 K for 120 h in air to form the high-Tc phase of the Bi system. The resultant pellet was milled into a fine powder and the filament was made by the suspension spinning method. The pyrolyzed filament was densified by pressing and sintering.[4]

$Tl_2Ba_2Ca_2Cu_3Ox$ samples was prepared from a mixture of Ba-Cu-O, Tl_2O_3 and CaO. The filament was made by the same method for the Bi system oxide.

The resistivity of the filament heated was measured by a standard four-probe method and the transport Jc measurement was performed at 77 K, 0 field with criterion of 1 μV/cm.

RESULTS AND DISCUSSION

$Y_1Ba_2Cu_3Ox$ Filament

The suspension spinning of a $Y_1Ba_2Cu_3Ox$ superconductor under various conditions was initially examined in an aqueous PVA solution and a nonaqueous PVA solution to prepare a long filament with high Jc.[3] The filament with diameter ranged from 15 μm to 300 μm was prepared and the electrical resistivity of the filaments sintered was measured. A rapid resistivity drop was observed around 90 K and zero resistivity was 85 K. The filament with higher Jc was obtained through the nonaqueous solution. The Jc was dependent on the fineness of the starting oxide powder, solvent , precipitating medium, degree of polymerization of PVA, the kind of dispersant used, and the oxide powder contents of the spinning dope, as well as the heating condition.[3]

The effects of PVA suspension medium on structure and Jc of the Y-Ba-Cu-O filament sintered were explored with PVA of various degrees of polymerization (DP) in DMSO, HMTA and the mixed solution of DMSO and HMTA (1:1). Figure 1 shows the effect of DP of PVA on Jc of the filament spun through various PVA solutions. The Jc of the filament

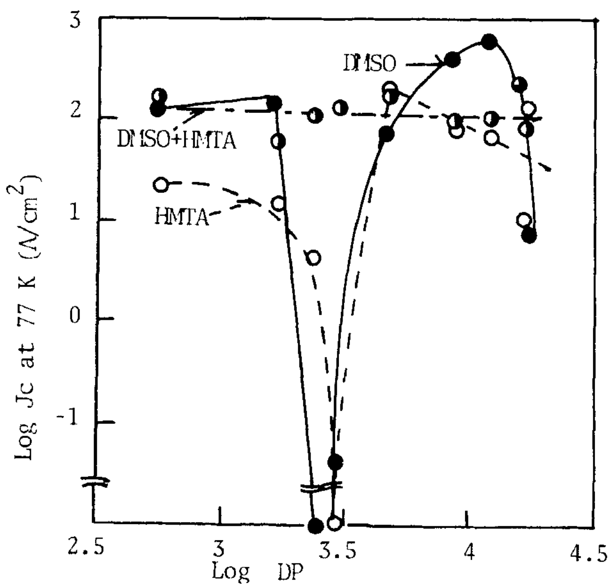

FIGURE 1 Relation between Jc of the Y-Ba-Cu-O filament and DP of PVA in various solutions.

produced by pure solution of DMSO and HMTA was not detected at DP of 3000 and 2450, respectively due to the effect of entanglement value of molecular weight. The filament spun through the mixed solution had a high Jc more than 100 A/cm^2 at wide range of DP. The crystal grains of the oxide in the filament sintered grew finer with increasing the DP of PVA. The Jc of the filaments was dependent on the oxide powder content. The maxium Jc of the filament spun in DMSO, HMTA and the mixed solution was observed at the oxide content of 95, 93 and 96 wt%, respectively. The maximum Jc spun by using PVA-DMSO solution was 680 A/cm² and the oxide powder was drawn up the blade type grains with a grain size of 3 μm×5μm×1μm.

The spinning of Y-Ba-Cu-O was also examined in various PAN solutions and the oxide powder contents of spinning dope. The Jc of the filament spun in various PAN solutions was measured and the Jc at 77 K and resistivity (ρ) at 100 K of the filament spun from the spinning dope containing the powder oxide of 92 wt % were listed in Table 1. The highest Jc of 1285 A/cm² at 77 K and 0 T was attained for the filament spun through PAN-DMF suspension. The high Jc is considered due to a coarse texture allowed of large superconducting crystal growth of 12 μm×3 μm.

TABLE I Jc at 77K and ρ at 100 K of the Y-Ba-Cu-O filament spun through various PAN solutions.

PAN solution	Jc (A/cm²)	ρ (mΩ · cm)
N-N′ dimethyl sulfoxide	109	0.38
N-N′ dimethyl acetamide	448	0.31
N-N′ dimethyl formamide	1285	0.15
ethylene carbonate	268	0.38
malononitrile	208	0.21

$(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ Filament

The suspension spinning of the single high-Tc phase Bi-Pb-Sr-Ca-Cu oxide was made by using the mixed PVA solution of DMSO and HMTA or PAN-DMF solution.[4] The as-drawn filament was cold pressed and heated at 773 K for 3.6 ks to remove volatile components. The filament was then cold pressed and was sintered at 1113 K for 54 ks under the oxygen pressure of 1/13 atom. The densification was repeated to obtain higher Jc. The resistivity of the filaments showed a sharp drop at about 115 K and became zero at 90 and 104 K for the filament from PAN and PVA respectively as shown in Fig.2. A high Jc value of 1940 A/cm² at 77 K was attained for the filament from PVA, whereas the filament from PAN had a low Jc of 55 A/cm². These filaments consisted of a single high Tc phase and the grains were approximately oriented with the c-axis perpendicular to the longitudinal direction of the filament. The alignment of the grain of the filament from PAN medium was poor.

$Tl_2Ba_2Ca_2Cu_3O_x$ Filament

Tl-Ba-Ca-Cu-O superconducting filaments were successfully prepared using a technique in which suspension spun and pyrolyzed

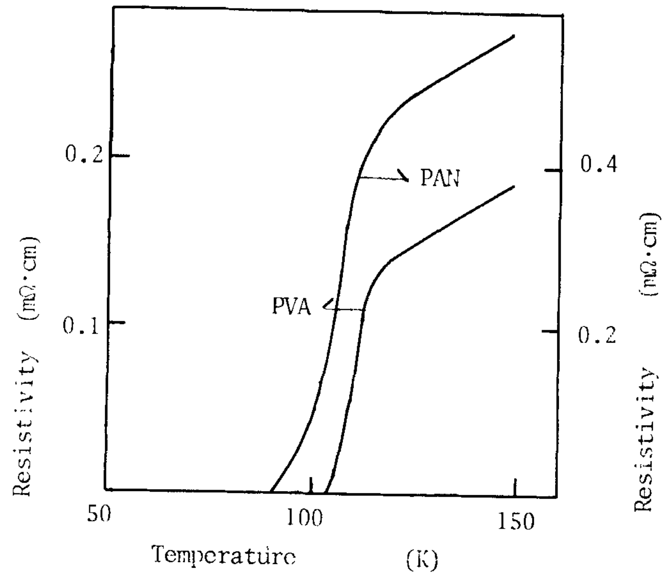

FIGURE 2 The temperature dependence of the electrical resistivity for the (Bi,Pb)-Sr-Ca-Cu-O filaments spun from PVA and PAN suspension media.

filaments were densified by pressing and sintering. The Tl-Ba-Ca-Cu oxide filament was spun from the mixed PVA solution of DMSO and HMTA. The as-drawn filament was heated at 773 K for 1800 s and was cold pressed and then was sintered at 1183 K for 600 s. A zero resistance state was achieved at 98 K and the maimum Jc at 77 K, 0 T was 1045 A/cm^2. The filament consisted of a single low Tc phase of 98 K and perfered orientation of the crystals was not detected.

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