Slip processing of high T_c superconductors

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Abstract. Slip-processing technique has been used to fabricate tapes and alumina-supported films of superconducting $YBa_2Cu_3O_{7-x}$. Good densification and connectivity are revealed by scanning electron microscopy. Both the tape and film show superconductivity well above the liquid nitrogen temperature with a transition range of 3° and 5°K respectively.

Keywords. Superconductivity; slip processing; doctor blade tapes; screen-printed films.

1. Introduction

The discovery of high T_c superconductors (Bednorz and Muller 1986; Wu *et al* 1987) has set off an explosion of research effort in this field. Most of the effort is, however, devoted to understanding the material systems involved and the mechanism of superconductivity. But the promise of technological revolution that the discovery of high T_c superconductivity holds can be realized only if the new materials can be fabricated in the shapes required in their electrical, magnetic and electronic applications. Not enough work has been done in this direction and a number of problems remain in the fabrication of various shapes with good superconducting properties. Doctor blade tapes and films (Togano *et al* 1988; Ishii *et al* 1988), plasma-sprayed tapes (Konaka *et al* 1988) and silver-sheathed tapes (Okada *et al* 1988) have been prepared using Y-Ba-Cu oxide system. Screen-printed films on different ceramic substrates have also been prepared (Bansal and Simons 1988; Sacchi *et al* 1988).

The authors believe that the well-developed ceramic fabrication processes like tape casting, screen printing, injection moulding etc can easily be adopted, with advantage, for the fabrication of superconducting ceramics. All these processes employ dispersion of fine ceramic particles in a suitable organic medium (called slip) as the starting material slip-processing technique has been used in this laboratory to prepare superconducting tapes, films supported by alumina substrate and coating on silver wire. Here we present the results of our initial work on tape and films.

2. Experimental

 $YBa_2Cu_3O_{7-x}$ powder was prepared by the usual method of mixing and grinding Y_2O_3 , BaCO₃ and CuO in the desired ratio, followed by calcination at 950°C. The total heat treatment time at 950°C was 24 h with several intermediate grinding and mixing steps. The single phase Y-Ba-Cu oxide powder obtained was finely ground in steel medium and used for preparing the slip. Ethyl alcohol with some trichloroethylene was used as the solvent, polyvenyl butyrol as the binder and corn oil as the dispersent. The powder was dispersed in the solvent along with the binder and the dispersent by prolonged mixing on a magnetic stirrer before plasticizer (polyethylene glycol/octyl pthalate) was added followed by further mixing. Viscosity was adjusted by adjusting

1110 R K Sinha and R K Sinha

the amount of different organic constituents. No plasticizer was used for screen printing. Tapes (approximate thickness $300 \,\mu$ m) were cast by doctor blading whereas screen printing on alumina substrate was done with a screen of 200 mesh. Silver wire was coated by passing it through the slip. Small pieces (0.5 cm × 1.5 cm) were cut from the tape and sintered at 950°C with prior holding at 500°C to drive out the organic additives. Oxygen flow was started as the temperature reached 900°C and maintained during cooling. The samples were characterized for structure by X-ray diffraction (Philips model 1710 with CuK_a radiation). Microstructure of the as-sintered surfaces was examined with a scanning electron microscope (JEOL JSM 840 A). The standard four-point technique was used to test the superconducting behaviour.

3. Results and discussion

Figure 1 shows a rolled-up tape and a screen-printed film on alumina substrate. The Xray diffraction pattern of the sintered tape (figure 2a) shows a single phase $YBa_2Cu_3O_{7-x}$. The diffraction pattern for the screen-printed film (figure 2b), on the other hand, shows additional peaks for phases like $Y_2Cu_2O_5$ and Y_2BaCuO_5 (not shown in the figure). The presence of $Y_2Cu_2O_5$ and Y_2BaCuO_5 in alumina supported films of $YBa_2Cu_3O_{7-x}$ was reported by others (Budhani *et al* 1987; Sacchi *et al* 1988). This may be due to interaction of the film with the alumina substrate. Alumina has a limited solubility in $YBa_2Cu_3O_{7-x}$ but tends to decompose it (Yan *et al* 1988).

Micrographs of sintered tape and film show good connectivity in a fairly dense structure. Both the samples show regions of thin plate-like crystals closely stacked together. Small isolated pores are present in the microstructure of the tape whereas in the film, pores between the platelets retain their angularity.



Figure 1. Slip-processed products—a roll of tape and screen-printed film on alumina substrate.



Figure 2. X-ray diffraction pattern of (a) tape and (b) screen-printed film.

Figure 3 shows the temperature dependence of resistivity of the sintered tape and screen-printed samples. The tape sample shows an onset temperature of 94 K with zero resistivity being attained at 91 K. The screen-printed film has an onset temperature of 91 K with zero resistivity at 86 K. The lower transition temperature of the screen-printed film may be ascribed to the presence of Y_2BaCuO_5 and $Y_2Cu_2O_5$ phases as impurity. Other investigators have reported superconductivity in alumina-supported screen-printed films of $YBa_2Cu_3O_{7-x}$ with zero resistivity temperatures below the liquid nitrogen temperature. The improved superconducting behaviour of our films



Figure 3. Microstructure of (a) tape and (b) screen-printed film.

may be due to lower sintering temperatures being used. Also different are the organic additives used in this work.

4. Conclusions

Slip-processing using organic medium is a flexible method of fabricating ceramic materials. With proper choice of solvents, binders, dispersant and plasticizers this



Figure 4. Temperature dependence of resistance (a) tape and (b) screen-printed film.

technique may be adopted to fabricated tapes, films and coated wires. A suitable heattreatment schedule is important in obtaining final, sintered product with superconducting properties. It may be possible to extend the technique to injection moulding process to fabricate more intricate structures.

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