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Manufacture of Bi-cuprate thin films on MgO single crystal substrates by chemical solution deposition

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Abstract. $Bi_2Sr_2CaCu_2O_8$ thin films have been deposited on MgO single crystal substrates by spin-coating a solution based on 2-ethylhexanoate precursors dissolved in xylene. Pyrolysis takes place between 200°C and 450°C and is accompanied by the release of 2-ethylhexanoic acid, CO_2 and H_2O vapour. Highly c-axis oriented $Bi_2Sr_2CaCu_2O_8$ as well as Er- or Ho-doped $Bi_2Sr_2(Ca,Ln)Cu_2O_8$ (Ln = Er, Ho) films were obtained after heat treatment at 840°C in air.

1. Introduction

Coated conductor tapes based on $REBa_2Cu_3O_7$ (RE = rare earth) thin films deposited on cube textured substrates can nowadays be produced in long lengths with remarkably high critical current densities (j_c) in excess of 1 MA/cm^2 at 77 K [1,2], which is more than three orders of magnitude higher than in $YBa_2Cu_3O_7$ wires made by the Powder-in-Tube (PIT) technology [3-5]. This large improvement is due to the decrease of the intergrain j_c when the grain misorientation angle exceeds 10° [6]. Nevertheless, the j_c of coated conductors at 77K under external magnetic fields is not high enough for some applications and cooling to 20K – 30K may be needed. In this temperature range, the $Bi_2Sr_2CaCu_2O_8$ (Bi2212) superconductor can also reach very high j_c values under magnetic fields in the form of powder-in-tube wires or ribbons [7-9], with c-axis texture but no in-plane texture. The intergranular j_c of Bi2212 is also lower when the grain boundary misorientation angle exceeds 10° [10], so that the application of a coated-conductor architecture to Bi2212 tapes could result in large performance improvements. Thin films deposited by vacuum techniques show J_c values in excess of 10 MA/cm^2 at 5K [10,11]. Only a few reports dealing with Bi2212 thin films prepared by solution deposition have been published to date [12-19], in spite of the potential of this technique for producing long textured

coated conductors. The films are deposited on MgO single crystals and their T_c is generally between 70K and 84K [13-15,17-19]. In this contribution, we summarize our studies on the pyrolysis of a precursor solution based on 2-ethylhexanoates and on attempts at doping the films with Ho and Er.

2. Experimental details

The coating solutions were prepared by dissolving 2-ethylhexanoate salts of Bi (Alfa Aesar, 70% solution in xylene), Sr (Alfa Aesar, 40% solution in 2-ethylhexanoic acid), Ca (Alfa Aesar, powder), Ho (home made), Er (home made) and Cu (Aldrich, powder) in xylene (Aldrich). For the preparation of Ho- and Er-2-ethylhexanoates, Ho acetate and Er acetate were dissolved in 2-ethylhexanoic acid and excess solvent was evaporated by heating to 90°C. The cation ratio of the coating solutions was Bi : Sr : Ca : (Ho,Er) : Cu = 2 : 2 : 1-x : x : 2 and the solutions had a metal molar concentration of 0.3M.

MgO (100) single crystals from CrysTec GmbH were used as substrates. Details on substrate pre-treatment and film deposition can be found in a previous publication [19]. 10 layers were deposited by spin coating. Annealing of the films was performed in air at 840°C.

Simultaneous thermogravimetry (TG) and differential thermal analysis (DTA) measurements were performed in a STA 449C from Netzsch with a heating rate of 10K/min in air (gas flow 40 ml/min) using Al_2O_3 crucibles. Fourier transform infra-red (FTIR) spectra were recorded in a Bruker Tensor 27 spectrometer coupled to the exhaust line of the TG/DTA device. X-ray diffraction (XRD) patterns were collected in a MD-10 bench-top X-ray diffractometer (for powder samples) or in a Bruker D8 diffractometer (for thin films), both using CuKα radiation. Scanning electron microscopy (SEM) was performed in a TM3000 Tabletop Microscope from HITACHI equipped with a QUANTAX 70 EDS analysis system. T_c was determined from AC-susceptibility measurements.

3. Results and discussion

The TG and DTA traces recorded on the precursor solution dried at 130°C (Fig. 1-left) show that the mass loss takes place in several overlapping steps from 200°C to 450°C. Beyond 450°C and up to 680°C, the sample mass is nearly constant. The mass loss at 500°C (67.3 %) is close to the theoretical value for the decomposition of 2-ethylhexanoates into oxide phases [19].

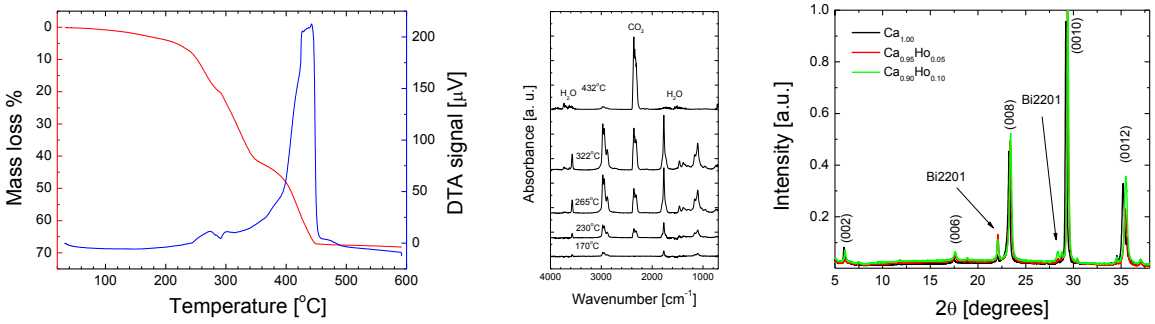

Figure 1: left panel: TG and DTA traces registered on a precursor mixture with $Bi_2Sr_2CaCu_2O_8$ nominal composition after drying at 130°C for 1h. Middle panel: FTIR spectra of the gas evolved during the thermal decomposition of the dried precursor powder. Right panel: XRD patterns of films with $Bi_2Sr_2Ca_{1-x}Ho_xCu_2O_8$ (x = 0.00, 0.05 and 0.10) nominal compositions.

The FTIR spectra of the gas educts (Fig. 1-centre) show that 2-ethylhexanoic acid is released from about 170°C. From 200°C, CO_2 is also detected. The intensity ratio of the CO_2 and 2-ethylhexanoic acid absorption lines increases with temperature. H_2O vapor is also visible from 250°C, suggesting that combustion of 2-ethylhexanoic acid takes place at an increasing rate as the temperature increases.

Although films with a high degree of c-axis orientation were obtained as shown in Fig. 1-right, the T_c of the undoped films is limited to 73 K – 81 K depending on the samples. The T_c of Bi2212 can be

varied by annealing under specific conditions [20-27]. We tried to increase the T_c of the film by post annealing in Ar at 600°C (30 min) and in O_2 at 840°C. According to Triscone et al. [25], these conditions should result in optimum superconducting properties in bulk samples. However, as shown in a previous paper [19], both treatments result in a partial decomposition of the Bi2212 phase and no T_c improvements.

According to Vinu et al. [28], low level Ho doping in Bi2212 can result in an increase of T_c. We therefore tried to dope our films with Ho. Er-doping was also attempted due to the similar ionic size and chemistry of Er and Ho. Fig. 2 shows that the XRD lines of Bi2212 are shifted to higher 2θ angles with doping, as expected from a reduction of the c-axis parameter of the structure [28]. However, both Ho- and Er-doping also resulted in the formation of an increased amount of Bi2201 phase.

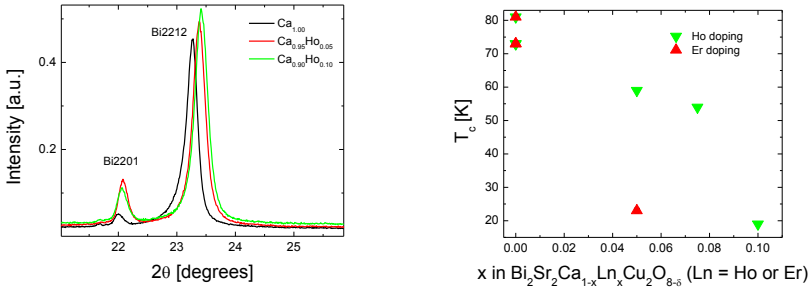

Figure 2: Left panel: detail of the XRD patterns of films with $Bi_2Sr_2Ca_{1-x}Ho_xCu_2O_8$ (x = 0.05, 0.10) nominal compositions. Right panel: T_c values measured on films with various $Bi_2Sr_2Ca_{1-x}Ln_xCu_2O_8$ (Ln = Ho, Er) nominal compositions.

The T_c of the Bi2212 phase was not increased upon doping with either Ho or Er. In contrast, as shown in Figure 2, T_c decreases with doping and this occurs apparently much faster for Er doping compared to Ho doping. The microstructure of the surface of a few selected films is shown in Figure 3. The undoped film is covered mostly with platelet-like grains that have grown parallel to the substrate plane, as reflected by the strong c-axis texture observed in the XRD patterns. Ho doping does not seem to induce significant changes in the surface microstructure. In contrast, the Er doped sample consists of much smaller grains, with evidence for many platelets with ab-axis oriented growth as suggested by the appearance of plate edges in the right-hand picture of Fig. 3.

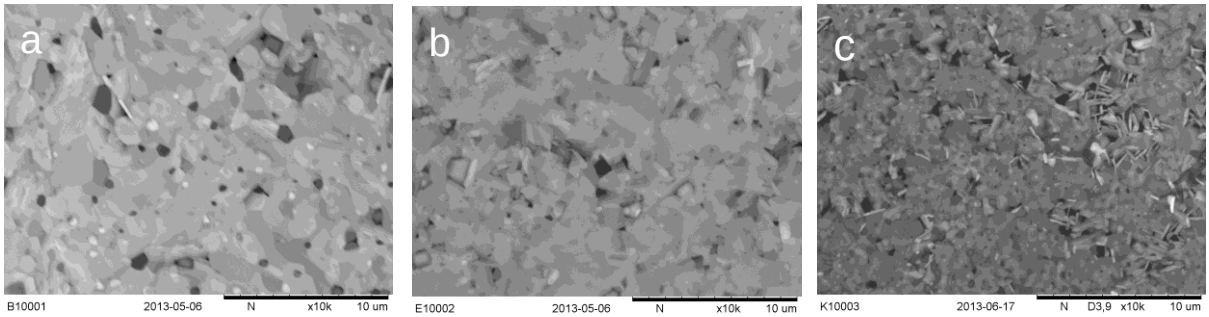

Figure 3: SEM pictures (secondary electron mode) of the surface of films with different stoichiometries. a: $Bi_2Sr_2Ca_1Cu_2O_{8+\delta}$, b: $Bi_2Sr_2Ca_{0.95}Ho_{0.05}Cu_2O_{8+\delta}$ and c: $Bi_2Sr_2Ca_{0.95}Er_{0.05}Cu_2O_{8+\delta}$.

4. Conclusions

$Bi_2Sr_2Ca_{1-x}Ho_xCu_2O_8$ ($0.00 \leq x \leq 0.10$) films with good c-axis orientation were grown on MgO single crystal substrates by means of chemical solution deposition. The T_c of the films was however not larger than 81 K in the best un-doped sample and decreases upon Ho doping. Attempts at doping with Er instead for Ho resulted in a degradation of the microstructure and a sharper decrease of T_c.

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