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EDX analysis and microstructural properties of the $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 superconducting composites

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We have fabricated and characterized $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 a realized high T_c superconductor - ceramic insulator composite system in which particles of the superconductor and ceramic insulator could coexist with well defined separated phases left intact by stringent processing conditions. All the composites exhibit superconductivity at 92K. EDX analysis show that element% and atomic% of the composites are in a good agreement with those of their respective components. SEM studies show homogenous surface morphology and particle size distribution. There is no detectable interface interaction between component grains and Ba_2HoSbO_6 grains are distinguishably distributed in the $YBa_2Cu_3O_{7-\delta}$ matrix.

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1. INTRODUCTION

High T_c superconductor (HTS)–insulator composites offer the attractive feature of improved chemical and physical stability for the application viability of these materials. Experimentally, fabrication of HTS-insulator composites is a difficult task due to chemical interaction between the component materials at the high processing temperatures which affects the superconducting properties drastically. In particular, $YBa_2Cu_3O_{7-\delta}$, which are essentially line compounds in the phase diagram, off-stoichiometric mixtures inevitably lead to multiphase samples with poor superconductivity or without superconductivity at all[1,2]. We have successfully fabricated a realized HTS-ceramic insulator composite

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system $YBa_2Cu_3O_{7-\delta}$ -Ba_2HoSbO_6 in which particles of the superconductor and ceramic insulator could coexist with well defined separated phases left intact by stringent processing conditions[3].

In the present work we have studied structural and microstructural characteristics of the $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composite system by X-ray diffraction (XRD), energy–dispersive x-ray (EDX) analysis and scanning electron microscopy (SEM). Superconducting properties of the composites were investigated by a. c. magnetization measurements. This article reports the fabrication processing, structural, microstructural characteristics and superconducting properties of the $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composites.

2. EXPERIMENTAL DETAILS

$YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 powder materials were prepared by solid state reaction process[3]. Quantitative elemental analysis of $Ba_2HoHfO_{5.5}$ and $YBa_2Cu_3O_{7-\delta}$ was carried out by EDX technique. EDX spectra of the samples were recorded using X-ray OXFORD model PENTAFET detector with Be-window and 128 eV resolution. The accelerating voltage used was 20KV, the beam current 200pA and the counting 100s. We have synthesized $YBa_2Cu_3O_{7-\delta}$. Ba_2HoSbO_6 composites with 0 to 50 wt% of Ba_2HoSbO_6 component in the respective composites. The component materials were mixed in desired wt% ratios and the mixture was pelletised as circular discs at a pressure of 2ton/cm^2. These discs were heat treated at 950 °C for 24h in flowing oxygen and cooled down slowly at a rate of 2°C/min to room temperature for proper oxygenation.

Surface morphology and microstructure of sintered Ba_2HoSbO_6 and $YBa_2Cu_3O_{7-\delta}$ materials and $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composites were studied by scanning electron microscopy, using both secondary and back-scattered electrons. SEM micrographs were recorded by a Leico-Cambridge model stereoscan 440 electron microscope.

Chemical stability of $YBa_2Cu_3O_{7-\delta}$ with Ba_2HoSbO_6 was examined by x-ray diffractometry of $YBa_2Cu_3O_{7-\delta}$ - Ba_2HoSbO_6 composites. Powder x-ray diffraction spectra of the component materials and composite samples were recorded by a Siemens D-5000 x-ray diffractometer using nickel filtered Cu-K_α radiation (λ = 1.5406 Å).

Superconducting properties of the $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composites and the effect of Ba_2HoSbO_6 addition on the superconductivity of $YBa_2Cu_3O_{7-\delta}$ superconductors was investigated by measuring a.c. magnetization of $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composites in the temperature range 5 to 300K at a frequency of 31Hz and with an ac field amplitude of 3 Oe, using a Quantum Design (MPMS-5S) SQUID magnetometer.

3. RESULTS AND DISCUSSION

Quantitative elemental analysis of the single phase $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 component materials and $YBa_2Cu_3O_{7-\delta}$ - Ba_2HoSbO_6 composites was carried out by EDX analysis. The results of EDX analysis are presented in Tables 1 - 3. As seen from these Tables , there is no evidence of impurity traces in the component samples. The element% and atomic% of the composites are in a good agreement with those of their respective components.

Element	Element %	Atomic %
Ba	41.08	13.58
Y	12.24	6.25
Cu	24.63	17.59
O	22.06	62.58
Total	100.00	100.00

Table 1
Quantitative elemental analysis data of $YBa_2Cu_3O_{7-\delta}$

Element	Element %	Atomic %
Ba	40.35	16.19
Ho	20.14	7.05
Sb	21.26	10.09
O	18.26	65.90
Total	100.00	100.00

Table 2
Quantitative elemental analysis data of Ba_2HoSbO_6

Element	Element %	Atomic %
Ba	41.60	15.43
Y	7.09	4.06
Cu	18.71	15.00
O	19.05	60.65
Ho	7.41	2.29
Sb	6.13	2.57
Total	100.00	100.00

Table 3
Quantitative elemental analysis data of a representative $YBa_2Cu_3O_{7-\delta}$ - (20wt%) Ba_2HoSbO_6 composites

X-ray diffraction spectrum of $YBa_2Cu_3O_{7-\delta}$, a representative $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composite with 50 wt% component and Ba_2HoSbO_6, are shown in Figure1. The XRD spectra of the composites contain XRD peaks corresponding to either $YBa_2Cu_3O_{7-\delta}$ or Ba_2HoSbO_6 and there are no extra peaks due to any impurity phase. It establishes chemical stability of the two component materials in the composites.

For the structural compatibility it is worth discussing the lattice matching of $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 materials, at this point. $YBa_2Cu_3O_{7-\delta}$ has a orthorhombic crystal structure with lattice parameters a=3.8214 Å, b=3.8877 Å and c=11.68 Å. Ba_2HoSbO_6 has a $A_2BB'O_6$ type ordered complex cubic perovskite structure, with lattice constant a=8.3712 Å. Based on the doubling of primitive ABO_3 simple cubic perovskite cell, (½ a=4.1856Å of Ba_2HoSbO_6), the component materials have a lattice mismatch ~ 10%, which indicates reasonably good lattice matching.

SEM micrographs of the $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 materials are shown in Figure 2. Surfaces of the samples present a crystallinity, with that is typical of a polycrystalline ceramic materials with homogeneous surface morphology and particle size distribution. The average particle size of the $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 grains were estimated to be 10-20 μm and 1-2 μm, respectively. Back-scattered SEM has been used to study the interface interaction between $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 grains. In back-scattered electron scanning electron microscopy high energy incident electrons undergo Rutherford scattering from the surface atoms and reemerge from the surface.

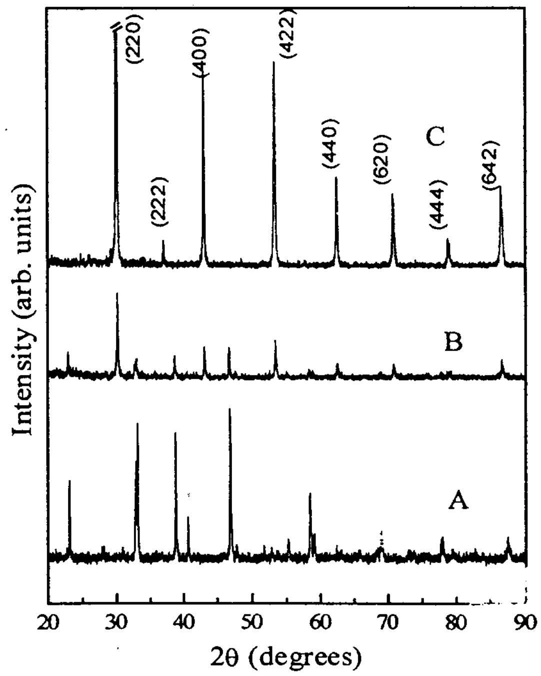
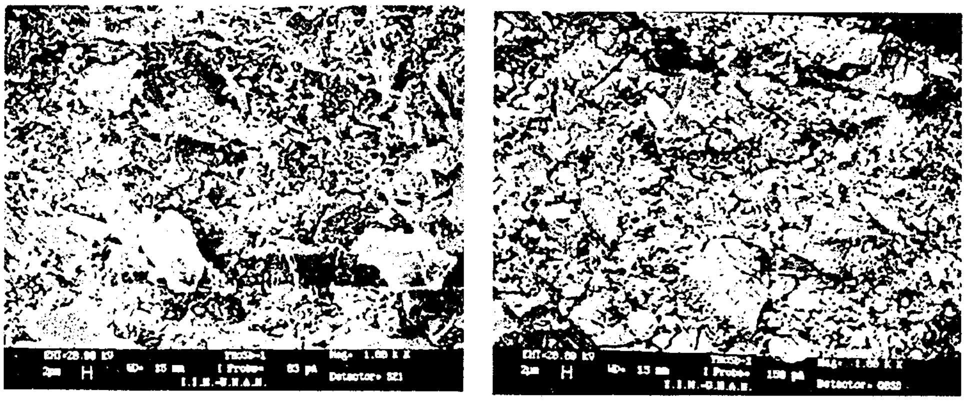


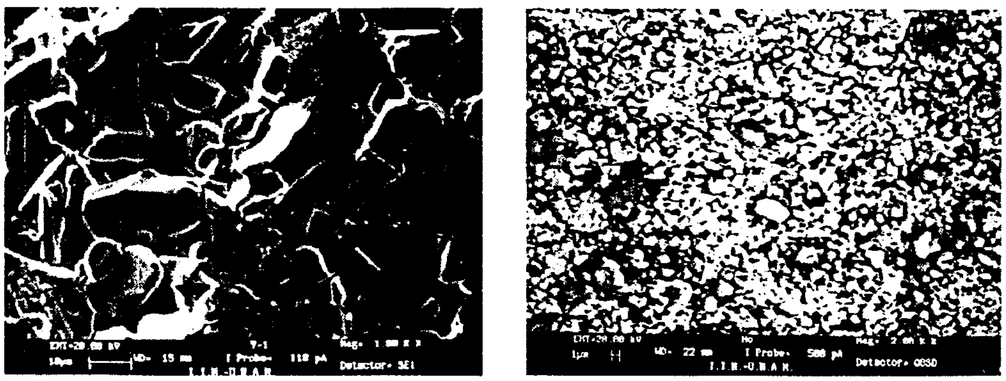
Figure 1. XRD spectra of $YBa_2Cu_3O_{7-\delta}$ (A) 1:1wt% $YBa_2Cu_3O_{7-\delta}$ - $HoBa_2SbO_6$ composite (B) and $HoBa_2SbO_6$ (C).

The resulting image is in some way like the secondary electron image but there are a number of important differences. First, the back-scattered electron come from a greater depth in the sample, and because of the spreading of the electrons in the sample they represent a larger area. Also, since the back-scattered electrons come from deeper in the sample, they contain less information about the surface and more about the bulk material[4]. A representative back-scattered electron micrograph of a $YBa_2Cu_3O_{7-\delta}$-(20wt%)Ba_2HoSbO_6 composite is shown in Figure 3 along with a secondary electron SEM micrograph of the same composite. As seen from these micrographs, there is no detectable interface interaction between $YBa_2Cu_3O_{7-\delta}$ and Ba_2HoSbO_6 grain interfaces. Ba_2HoSbO_6 grains are distinguishably distributed in the $YBa_2Cu_3O_{7-\delta}$ matrix. For the ceramic superconductor-insulator composites, a magnetization measurement is a effective tool in characterizing the superconducting properties because it can directly probe individual superconducting grains[5]. We have measured a.c. magnetization of $YBa_2Cu_3O_{7-\delta}$-Ba_2HoSbO_6 composites in the temperature range 5 to 300K at a frequency of 31Hz and with an ac field of 3 Oe.



(a) (b)

Figure 3. (a) Secondary and (b) electron SEM micrographs of a $YBa_2Cu_3O_{7-\delta}$ - (20wt%) $HoBa_2SbO_6$ composite.



(a) (b)

Figure 2. Secondary electron SEM micrographs of (a) $YBa_2Cu_3O_{7-\delta}$ and (b) $HoBa_2SbO_6$

Figure 4 shows the representative a. c. magnetization versus temperature curves of a 1:1wt% $YBa_2Cu_3O_{7-\delta}$ - Ba_2HoSbO_6 composite and a pure $YBa_2Cu_3O_{7-\delta}$ superconductor. In all the composites, there is sharp superconducting transition at 92K, corresponding to the T_c of the pure $YBa_2Cu_3O_{7-\delta}$ superconductor sample. Ba_2HoSbO_6 addition did not have any deteriorating effect on the superconducting properties of $YBa_2Cu_3O_{7-\delta}$ superconductor.

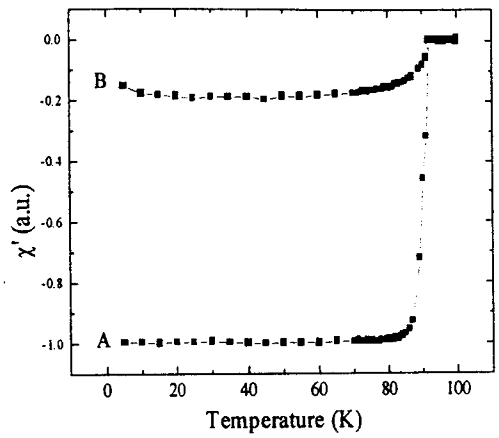


Figure 4. a.c. magnetization versus temperature curves of $YBa_2Cu_3O_{7-\delta}$ (A) and 1:1wt% $YBa_2Cu_3O_{7-\delta}$ - $HoBa_2SbO_6$ composite (B).

4. CONCLUSIONS

In conclusion, we have successfully fabricated $YBa_2Cu_3O_{7-\delta}$ - Ba_2HoSbO_6 a high T_c superconductor - ceramic insulator composite system in which particles of the superconductor and ceramic insulator could coexist with well defined separated phases left intact by stringent processing conditions. All the composites exhibit superconductivity at 92K and the element% and atomic% of the composites are in a good agreement with those of their respective components. Composites present homogenous surface morphology and particle size distribution. Ba_2HoSbO_6 grains are distinguishably distributed in the $YBa_2Cu_3O_{7-\delta}$ matrix and there is no detectable interface interaction between the component grains.

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