Research Article

Electrical Transport and Lowered Percolation Threshold in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ -Nano- $\text{YBa}_2\text{ZrO}_{5.5}$ Composites

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Received 10 May 2014; Accepted 6 August 2014; Published 25 August 2014

Academic Editor: Dong Qian

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We investigated the chemical reactivity and percolation characteristics of insulating nanocrystalline $\text{YBa}_2\text{ZrO}_{5.5}$ prepared by modified combustion route and the $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor composite system. Structural analysis was done by using X-ray diffraction technique, surface morphology of the samples was studied using scanning electron microscopy, and electrical transport measurements like critical transition temperatures (T_c) and self-field transport critical current (J_c) were done by using standard four-probe technique. It is found that, in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ -nano- $\text{YBa}_2\text{ZrO}_{5.5}$ composite system, the superconductor and insulator materials coexist as separate phases without any noticeable chemical reaction even after sintering at high temperatures. Furthermore, percolation threshold and critical exponent are found to be $V_C = 0.3$, $t = 1.68$, and $u = 2.7$. And the analysis of the current flow in the polycrystalline samples reveals weak link behavior in the majority of grain connections.

1. Introduction

High temperature superconductors with transition temperatures above 77 K in ceramic materials have received tremendous responsiveness because of their scientific and practical potential. The study of superconducting small aggregates, clusters, or particles is very important from both the fundamental and the technological standpoint [1–3]. It will be significant to study the percolation and superconductivity of composites involving superconductor inserted in an insulator medium. Granular nature along with short coherence length [4] and large penetration depth [5] of high temperature superconductors allows us to investigate the percolation behavior, fractal properties, quantum size effects, thermal fluctuations, and size effects on superconductivity. The percolation concept was first employed to describe superconductors by Davidson and Tinkham [6], who analysed resistivity data of composite $\text{Nb}_3\text{Sn}/\text{Cu}$ wires. For the growth of high quality films, the choice of substrate is vital. A high T_c superconductor-insulator system is very difficult to obtain without compromising the superconducting properties. The

chemical nonreactivity of the substrate materials with superconductors indicates their potential as substrates for film deposition. The chemical compatibility of materials with the superconductor at the processing temperature is crucial. Also superconductor-insulator percolation studies are a medium to understand the fundamental mechanism behind high temperature superconductivity.

A percolation model can be regarded as a collection of points or occupied sites distributed in a space; certain pairs of them are randomly linked [7]. Its applications range from transport in amorphous and porous media and composites to the properties of branched polymers, gels, complex ionic conductors, and superconductors [8]. There is a path between two points A and B , if a sequence beginning with A and ending with B can be found, such that successive points in this sequence are linked. This path may allow the flow of charge between two points if we regard the occupied sites as pieces of conductor. The sites may be clustered such that pairs of points belonging to the same cluster are connected but there is no path between points belonging to different clusters. Cluster size (number of points in the cluster) increases

with the number of linkages. Some cluster may become of infinite size at some critical density of occupied sites. Above this critical density, known as percolation threshold (V_s), the infinite cluster spans entire system and the system is in percolating state. For instance, in an ideal mixture of conducting and insulator particles the conductivity is zero outside the percolating region, whereas it takes a finite value inside this region.

It was reported that perovskites could be beneficial for incorporation into YBCO due to their similar crystal structures relative to the host YBCO phase [9]. Thereafter perovskite based additions have gained renewed interest as a class of materials that can be incorporated into REBCO superconductor for dramatic improvements in the pinning properties [10–13]. And a number of perovskites like $\text{Sr}_2\text{HoHfO}_{5.5}$ [14], $\text{GdBa}_2\text{NbO}_6$ [15], $\text{Ba}_2\text{ErZrO}_{5.5}$ [16], $\text{YBa}_2\text{ZrO}_{5.5}$ [17], and so forth were being considered as substrate materials for YBCO film deposition. However substrate selection presents particular challenges for the production of high-quality high-temperature superconducting (HTS) films suitable for applications [18]. In order to confirm their chemical compatibility with YBCO, it is important to study the electrical transport and percolation behavior in these superconductor-insulator composites. Transport properties and percolation behavior of high T_c superconductor-metal composites have been reported extensively [19–24]. The general form of the double perovskites is $\text{A}_2\text{BB}'\text{O}_6$, where for the present work $\text{A} = \text{Ba}$, $\text{B} = \text{Y}$, and $\text{B}' = \text{Zr}$. Paulose et al. studied percolation properties of Ba_2YZrO_6 prepared by conventional solid state route and $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor composite using temperature-resistivity measurement and reported that both superconducting and normal state percolation threshold values of the composite are around 35 vol.% of $\text{YBa}_2\text{Cu}_3\text{O}_7$ in the system [25]. In the present work, the percolation behavior and nonreactivity of nanoparticles of $\text{YBa}_2\text{ZrO}_{5.5}$ (YBZO) ceramic prepared through an autoigniting combustion technique mixed with bulk $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) superconductors are studied. The variations in the microstructure, sintering behavior, sample density, electrical transport such as T_c , and current carrying capacity are also studied.

2. Materials and Methods

Conventional technique of solid state route was used for the preparation of YBCO superconductor, by which high purity Y_2O_3 , BaCO_3 , and CuO were thoroughly mixed in the stoichiometric ratio of $\text{Y}:\text{Ba}:\text{Cu} = 1:2:3$. The mixture was then calcined at 930°C for 72 hours with two intermediate wet grindings. High quality nanoparticles of $\text{YBa}_2\text{ZrO}_{5.5}$ (YBZO) were synthesized through modified autoigniting combustion technique as reported by Jose et al. [26]. In a typical synthesis, aqueous solution containing ions of Y, Ba, and Zr was prepared by dissolving high purity Y_2O_3 in dilute HNO_3 , $\text{Ba}(\text{NO}_3)_2$, and ZrOCl_2 (99%) in double distilled water in a glass beaker. Citric acid (99%) was then added to the solution containing Zr ions which serves as the complexing agent. Oxidant/fuel ratio of the system was adjusted by adding nitric acid and ammonium hydroxide

FIGURE 1: TEM micrograph of $\text{YBa}_2\text{ZrO}_{5.5}$ nanoparticles.

and the ratio was kept at unity. The solution containing the precursor mixture at a pH of ~ 7.0 was heated using a hot plate at $\sim 250^\circ\text{C}$ in a ventilated fume hood. The combustion product was subsequently characterized as single phase nanocrystals of $\text{YBa}_2\text{ZrO}_{5.5}$. The YBCO and nano-YBZO were thoroughly mixed in different ratios and made into rectangular pellets of dimension $12 \times 4 \times 1$ mm. These pellets were sintered at temperature ranges from 975°C to 1500°C for 12 hours with a heating rate of $3^\circ\text{C}/\text{minute}$ depending on the volume percentage of YBHO in the composite material. The samples were then cooled to 550°C and kept 24 hours for oxygenation and then cooled to room temperature.

The structural characterization of YBCO-nanocrystalline YBZO composites was done by powder X-ray diffraction (XRD) technique using a Bruker D-8 X-ray diffractometer with Nickel filtered $\text{Cu K}\alpha$ radiation. The surface morphology of the sintered samples was studied using scanning electron microscopy (SEM) (JEOL JSM 6390 LV). The qualitative and quantitative elemental composition of the materials in the compounds was studied using energy dispersive X-ray spectroscopy (EDS) JEOL model JED-2300. The critical transition temperatures (T_c) of the samples were measured using standard four-probe technique. For the samples with higher vol.% of insulator, a two-probe method was used to measure their resistivity. For this experiment the entire data collection was run using a Lab VIEW 7.1 program on a PC. This was connected to Keithley source meter 2440 and nanovoltmeter 2182A along with lakeshore temperature controller equipped with PT-111 platinum sensor using a GPIB connection. The electrical contacts on the surface of the pellets were done by adhesive silver paste after making a narrow scratch on the sample surface. The self-field transport critical current (J_c) measurements were done at liquid nitrogen temperature using the standard $1\mu\text{V}/\text{cm}$ criterion.

3. Results and Discussion

The powder morphology of the as-prepared $\text{YBa}_2\text{ZrO}_{5.5}$ nanopowder was done by using transmission electron microscopy (TEM) and is shown in Figure 1. The result suggested that the self-aligned nanoparticles of YBZO are of cuboidal shape with sharp grain boundaries and with size less

FIGURE 2: XRD pattern of different vol.% of nano-YBZO mixed YBCO superconductor ((a) = 0%, (b) = 10%, (c) = 30%, (d) = 50%, (e) = 70%, and (f) = 100%).

than 20 nm. The selected area electron diffraction (SAED) pattern, shown inset to Figure 1, reveals that the $\text{YBa}_2\text{ZrO}_{5.5}$ nanoparticles were crystallized, with bright polycrystalline diffraction rings. SAED patterns were composed of a number of bright spots arranged in concentric rings. The electrons were reflected and diffracted from crystallographic planes of the unit cells of the sample to produce bright spots. The rings were diffuse and hollow, showing that the products were composed of nanocrystals with different orientations. This is indicative of the polycrystalline nature of the crystallites, but the spotty nature of the SAED pattern could be due to the fact that finer crystallites with related orientations were agglomerated together, resulting in a limited set of orientations.

The X-ray diffraction (XRD) patterns of composites for 0–100 vol.% of nano-YBZO in the YBCO system are shown in Figure 2. XRD patterns of composites show that all the peaks could be indexed for orthorhombic $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ and cubic $\text{YBa}_2\text{ZrO}_{5.5}$ and there is no extra peak detectable. This implies that $\text{YBa}_2\text{ZrO}_{5.5}$ and $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ remain as two different separate phases in the composite even after severe heat treatment up to 1020°C . For YBCO-YBZO composites with 80 vol.% YBZO, the sintering temperature was above the peritectic temperature of YBCO ($\sim 1030^\circ\text{C}$) and therefore the formation of a 211 phase with YBCO is expected, but in the present study the XRD patterns did not show the presence of a 211 phase in the system. Thus, the composites synthesized are suitable for the percolation studies.

The surface morphology of the composite samples was illustrated by scanning electron microscopy (SEM). Figure 3 shows the SEM images of different vol.% of nano-YBZO added YBCO composites. These indicate that the surface of the samples presents a crystalline character, which is typical of a polycrystalline ceramic material. The $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ grains form groups which have appearance of clusters and there is no detectable interface interaction between the YBZO and YBCO grains. However, there are different grain sizes

and a random orientation of grain boundaries, whereby the presence of different intergrain conductivities is expected.

The effect of $\text{YBa}_2\text{ZrO}_{5.5}$ on the superconducting transition temperature T_c of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ was studied by four probe measurements for temperatures ranging from 77 to 300 K. Variation of normalized resistivity with temperature of the YBCO superconductor mixed with different vol.% of insulating nano-YBZO samples is shown in Figure 4. It is observed that the composite sample with less than 50 vol.% of YBCO showed a metallic behavior and gave zero resistivity superconducting transition temperature above liquid nitrogen temperature. However the YBCO-nano-YBZO composite with 80 vol.% of YBZO showed a conducting behavior; there is no superconducting transition up to 77 K. The resistivity of composites is dominated by YBZO for volume less than 50% of YBCO with a significant drop of ρ occurring near these values of volume. The absence of a superconducting network through the composite sample or a low vol.% of YBCO may be the reason for this behavior. These results show that when YBCO is ~ 30 vol.% or above there are interconnected networks of superconducting grains for the super current to pass through the composite material, but for lower vol.% of YBCO, the continuous network of superconducting grains breaks away and the resistance becomes nearly equal to that of pure insulator. Hence the value of percolation threshold for the YBCO-nano-YBZO composite sample lies between 30 and 40 vol.% of YBCO in the composite.

The resistivity (ρ) and temperature coefficient of resistivity, $\alpha = (1/\rho)(d\rho/dT)$ at room temperature, are shown in Figure 5 as a function of vol.% of YBCO (V_s) in the composite. In the normal state, YBCO behaves as a metallic conductor and shows resistivity $\sim 10 \mu\Omega\text{m}$. However the resistivity of composites is dominated by $\text{YBa}_2\text{ZrO}_{5.5}$ for lower vol.% of YBCO with a significant drop of ρ occurring near 30–50 vol.%. The behavior of ρ correlates with that of α , which increases sharply towards that of YBCO starting from

FIGURE 3: SEM images of different vol.% of nano-YBZO mixed YBCO superconductor ((a) = 0%, (b) = 10, (c) = 30%, (d) = 50%, (e) = 70%, and (f) = 100%).

$V_S \sim 30$ vol.%. So if we assumed that the electrical transport behavior in YBCO-nano-YBZO composites is percolative, the percolation threshold V_C is between 0.3 and 0.4. Thus, the superconducting percolation threshold and normal state percolation threshold values of YBCO-nano-YBZO composites lie in the same range. The electrical properties of the superconductor-insulator system can be described in [27, 28]

$$\begin{aligned} \rho &= \rho_o (V_S - V_C)^{-t} \quad \text{for } V_S > V_C, \\ \rho' &= \rho'_o (V_C - V_S)^u \quad \text{for } V_C > V_S, \end{aligned} \quad (1)$$

where ρ_o and ρ'_o are constants, V_C is the critical volume fraction of superconductor at which ρ changes dramatically and is called the percolation threshold, V_S is the vol.% of superconducting material in the composite, and t and u are

the critical exponent describing the transport properties of the composite system. The values ρ_o , ρ'_o , t , and u are found from the log-log plot of ρ versus $(V_S - V_C)$ and ρ' versus $(V_C - V_S)$ with $V_C = 0.3$. The value of V_C is adjusted so that the log-log plots of ρ and ρ' give a straight line.

Log-log plot of ρ versus $(V_S - V_C)$, for $V_S > V_C$, is shown in Figure 6 which gave the exponents $t = 1.68$ and $\rho_o = 5.08 \mu\Omega$ m. And Figure 7 shows the log-log plot of ρ' in function of $(V_C - V_S)$ for $V_C > V_S$ system which gave the values $u = 2.7$ and $\rho'_o = 58.31 \times 10^4 \mu\Omega$ m. Least square fits were performed to determine the slope of the plots which gave the values of exponents t and u . However for an idealized metal-insulator system $t = 1.7$ and $u = 0.7$ [29]. A great number of experiments on conducting-insulator systems show percolation thresholds in agreement with percolation

FIGURE 4: Temperature-normalised resistivity graph for different vol.% of nano-YBZO mixed YBCO superconductor ((a) = 0%, (b) = 20%, (c) = 50%, (d) = 70%, and (e) = 80%).

theory [19–21]. However, there are few others which reported variation from the universal values [27, 30].

The values of critical current densities (J_c) measured for different samples at zero applied magnetic field are shown in Table 1. The self-field J_c is found to be decreasing on the addition of nanocrystalline YBZO in YBCO. A judicious explanation is the existence of larger defect density, since J_c is more sensitive to the defect density. The analysis of the current flow in the composite samples reveals the weak link behavior in the majority of grain connections. But this does not necessarily mean that weak (Josephson) grain coupling is absent in these samples. However, the number of stronger grain connections must be significantly above the percolation threshold [28], which is defined as the minimum fraction for a continuous current path.

$\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor will start to melt and decompose when it is sintered at higher temperature.

FIGURE 5: Variation of (a) normal state resistivity ρ and (b) temperature coefficient of resistivity $\alpha = (l/\rho)(d\rho/dT)$ at room temperature for different vol.% of YBCO.

FIGURE 6: Log-log plot of ρ versus $(V_S - V_C)$.

FIGURE 7: Log-log plot of ρ versus $(V_C - V_S)$.

TABLE I: Sintering temperature, density, and electric transport data of different YBCO-nano-YBZO composites.

Vol.% of YBCO	Vol.% of YBZO	Sintering temp. (°C)	Density (gm cm ⁻³)	T_c (K)	Self-field J_c ($\times 10^4$ A/cm ²)
100	0	975	5.35	92	1.47
90	10	983	5.57	92	0.88
80	20	990	5.66	90.5	0.26
70	30	1000	5.75	89	0.09
50	50	1015	5.84	82	—
30	70	1120	5.88	—	—
20	80	1230	6.03	—	—
10	90	1370	6.15	—	—
0	100	1500	6.27	—	—

The values of V_C , t , and u obtained for different systems of composites vary due to many factors. The critical exponents, t and u , are a measure of the order of interaction between normal metal and insulator. The value for ideal percolation threshold is around 17 vol.% of metal in the system for a perfect metal-insulator composite without any interaction or reaction between the two. The normal state and superconducting percolation threshold values of YBCO-nano-YBZO (0.3 vol.% of YBCO) composites are in closer ranges as expected for an idealized percolation system and are lower than those observed in conventionally prepared YBZO-YBCO (0.35 vol.% of YBCO) composite [25]. This may be due to the better chemical nonreactivity between YBCO and nano-YBZO. Furthermore, there is a negligible conductivity below percolation threshold while it is expected to be zero in ideal percolative systems. The nonreactivity of YBCO with nano-YBZO even at high processing temperature also points to the fact that nano-YBZO is a suitable substrate for YBCO superconductor and thus to develop devices based on high temperature superconductor films and junctions.

4. Conclusion

In this paper we have studied the chemical reactivity and percolation behavior using electric transport measurements in polycrystalline $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor-nano- $\text{YBa}_2\text{ZrO}_{5.5}$ composite. From structural analysis it is observed that the mixture of materials $\text{YBa}_2\text{ZrO}_{5.5}$ insulator with $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor is a system where the particles of superconductor and insulator materials are found to coexist in a composite with two well-defined separate phases. Resistivity measurements of the composites synthesized in this work show an apparent percolative behavior with percolation threshold $V_C = 0.3$ and critical exponents values $t = 1.68$ and $u = 2.7$. Compared to the conventionally prepared YBZO, nanocrystalline YBZO mixed superconductor-insulator system showed a better percolation behavior. Thus, as discussed earlier, YBZO is chemically stable with the YBCO superconductor and, at the same time, it did not have any deteriorating effect on the superconducting property characterized by the transition temperature.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

Sukumariam Vidya acknowledges CSIR for financial assistance.

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