Chapter V

TRANSPORT PROPERTIES OF CERAMIC INSULATOR SUPERCONDUCTOR COMPOSITES: A PERCOLATION STUDY

Study of superconducting aggregates, clusters and particles is of great significance from both fundamental and technological standpoint. Small particles are excellent media for studies of percolation and fractal properties, quantum size effects, thermal fluctuations, size effect on superconductivity etc. Because of the granular nature of high T\textsubscript{c} ceramic superconductors with short coherence length and large penetration depth, it is interesting to study the percolation and superconductivity of composites consisting of a superconductor embedded in an insulator medium.

The transport properties of normal metal-insulator percolation systems have been extensively studied over the years. Recently, a few studies on the percolation behavior of the superconductor-normal metal composites based on the electrical transport and magnetisation properties were reported\textsuperscript{(1-5)}. Since the resistivity ratio $\rho_s/\rho_n$ (where $\rho_s$ is the resistivity of the superconductor and $\rho_n$ is resistivity of the normal metal at room temperature) is very small for the superconductor-normal metal composite system, the percolation model which describes the normal state transport behavior of the composite system cannot be strictly applied to such a system. A high T\textsubscript{c} superconductor-insulator composite system is very difficult to obtain without compromising the superconducting properties\textsuperscript{(6)}. High T\textsubscript{c} superconductors require prolonged annealing at the processing temperature ($> 850^\circ$C) for obtaining superconducting phase, and most superconductor-insulator systems do react
chemically with each other under such annealing conditions, reducing the superconducting transition temperature drastically. Recently, we have synthesized a few insulating materials which were found to be chemically non-reacting with YBCO superconductors at their processing temperatures, making percolation studies in superconductor-insulator composite systems possible.

The percolation studies of YBCO superconducting materials with insulators such as SrTiO$_3$, MgO, Al$_2$O$_3$, Cr$_2$O$_3$, etc. have been not successful because of the chemical reaction between the YBCO and the insulators at the processing temperature$^{(7-8)}$. It is also realized that an understanding of the physical properties of insulator-superconductor mixtures will be helpful, for the selection of appropriate substrates for fabrication of HTSC thin and thick films. For fabrication of good quality thin and thick films chemical reaction between HTSC and substrate materials should be minimized. In the present study we have fabricated three superconductor-insulator composites i.e., YBCO-SmBa$_2$SbO$_6$, YBCO-PrBa$_2$SbO$_6$ and YBCO-YBa$_2$SbO$_6$ and the percolation behaviour of these composite system are described in this chapter

5.1 Percolation Model

The percolation model was originally proposed$^{(9)}$ to describe how fluid spreads through porous media, branching polymers form a gel, electron migration occurs in a solid, diseases infect a community and other similar phenomena. Because
of its generality and relative simplicity it has found many applications ranging from the physics of quarks to the extraction of oil from sand stones.

Consider a regular lattice where lattice sites can have two states, either black or white\(^{(9)}\). A cluster is defined as a group of black sites connected by nearest neighbour distances. There is a critical point \( V = V_c \) below which only finite clusters exist, but for \( V > V_c \) a fraction of the black sites belong to an infinite cluster and a percolation is possible. Below the percolation threshold, \( V < V_c \), there is no infinite cluster of black sites. From \( V_c \), the fraction of sites belonging to the infinite cluster grows drastically, it has a non analytic point at \( V_c \). This non analycity is characteristic for the percolation threshold, and is usually described by a power law asymptotically close to \( V_c \).

Consider the case of a metal-insulator composite system. The resistivity of the insulator is very high. If we add metal to insulator, the resistivity of the composite remains more or less same as that of insulator upto a critical volume fraction of metal in the composite. If the volume fraction of metal increases beyond a critical volume, the resistivity reduces drastically to that of pure metal. The critcal volume fraction of the metal required to have a continuous network or to become an infinite cluster is called the percolation threshold value. The transport properties can be described by a set of exponential equation below and above the threshold volumes. The relation are\(^{(9,10)}\)
\[ p = \rho_0 (V_m - V_e)^4 \text{ for } V_m > V_c \quad (1) \]
\[ \rho^1 = \rho_0^1 (V_e - V_m)^u \text{ for } V_c > V_m \quad (2) \]

Where \( \rho \) and \( \rho^1 \) are the resistivity of the composite, \( \rho_0 \) and \( \rho_0^1 \) are constants. \( V_e \) is the critical volume fraction at which the transport properties change drastically (called threshold value), \( V_m \) is the vol\% of metal in the system, ‘t’ and ‘u’ are critical exponents describing the transport properties of composite system. The value obtained for percolation threshold is around 17\% of metal in the system for a perfect metal-insulator composite without any interaction or reaction between the two. The expected values of critical exponents are \( t \approx 1.7 \) and \( u \approx 0.7 \) approximately. The values obtained by experimental methods vary slightly around this value. An appreciable variation of critical exponents from the above values indicates that the system is not forming a perfect composite.

The normal state transport properties of the ceramic insulator-superconductor composite system can be described by a set of equations considering the superconductor as a metal. The equations are

\[ p = \rho_0 (V_s - V_e)^4 \text{ for } V_s > V_e \quad (3) \]
\[ \rho^1 = \rho_0^1 (V_e - V_s)^u \text{ for } V_s < V_e \quad (4) \]

Where \( p \) and \( \rho^1 \) are the resistivity of the composite, \( \rho_0 \) and \( \rho_0^1 \) are constants, \( V_s \) the vol\% of superconductor YBCO in the system and \( V_e \) the critical volume at which the
transport properties change drastically, called the percolation threshold value, ‘t’ and ‘u’ are critical exponents describing the transport properties of the composite system. In this chapter the percolation studies of three superconductor-insulator systems(YBCO-SmBa$_2$SbO$_6^*$, YBCO-PrBa$_2$SbO$_6$, YBCO-YBa$_2$SbO$_6^*$) based on X-ray diffraction and electrical measurements are presented.

5.2 Percolation study of YBa$_2$Cu$_3$O$_{7.5}$ - SmBa$_2$SbO$_6$ system

Pure YBCO has been prepared from high purity Y$_2$O$_3$, BaCO$_3$ and CuO by the solid state reaction method. YBCO - SmBa$_2$SbO$_6$ composites were prepared by mixing different volume percentage of the compound (from 0 to 100%) SmBa$_2$SbO$_6$. It was then pressed in the form of pellets and sintered at temperatures in the range of 950°C to 1020°C for 15h depending on the SmBa$_2$SbO$_6$ volume fraction. The samples were then cooled slowly at the rate of 2°C/min. to room temperature in air. Pure SmBa$_2$SbO$_6$ was sintered at 1500°C for 5h. Table 5.1 shows the different volume ratios of composites prepared and the sintering temperature for zero vol% to 100 vol% of SmBa$_2$SbO$_6$.

Table 5.1

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<th>Vol% of YBCO</th>
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5. 2. 1 X-ray diffraction studies on YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> - SmBa<sub>2</sub>SbO<sub>6</sub> composite.

The reactivity and the phase analysis of the composites were carried out by X-ray powder diffraction techniques. Fig 5.1 shows the X-ray powder diffraction pattern of different volume percentage of composition. In the XRD patterns of the composites having upto 70 vol% of SmBa<sub>2</sub>SbO<sub>6</sub>, two distinct sets of peaks, one corresponding to YBCO having an orthorhombic structure and the other corresponding to SmBa<sub>2</sub>SbO<sub>6</sub> are clearly visible. There was no evidence of any additional peak other than those of YBCO and SmBa<sub>2</sub>SbO<sub>6</sub> in the XRD patterns, indicating that there is no detectable reaction (within the precision of XRD) between the two components even after severe heat treatment upto 1020°C.

5. 2. 2 Temperature-resistivity studies on YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> - SmBa<sub>2</sub>SbO<sub>6</sub> composite

The electrical resistivity measurements were carried out by a four-probe method for the superconducting samples using a Keithley nanovoltmeter (model 181) and a current source (model 220) as described in Chapter 2. Resistivity measurements of the composite in the higher resistance range were found by a Keithley electrometer (model 602) at room temperature. Fig. 5.2 shows the temperature resistance curves for YBCO - SmBa<sub>2</sub>SbO<sub>6</sub> composite for different volume ratios of composites. For clarity and readability \( \rho / \rho_r \), where \( \rho \) is the resistivity of the composite at different temperatures and \( \rho_r \) the room temperature resistivity, is
Fig. 5.1  X-ray diffraction pattern for different vol% of SmBa$_2$SbO$_6$ in the YBa$_2$Cu$_3$O$_{7.5}$ - SmBa$_2$SbO$_6$ composites. (a) 0%  (b) 10%  (c) 30%  
(d) 50%  (e) 70% and (f) 100%.
Fig. 5.2  Variation of normalized resistivity $\rho/\rho_r$ with temperature for different vol% of SmBa$_2$SbO$_6$ in the YBa$_2$Cu$_3$O$_{7.8}$-SmBa$_2$SbO$_6$ composites. (a) 0% (b) 10% (c) 20% (d) 30% (e) 50% (f) 70% (g) 80%
plotted against temperatures. From the figure it is clear that for up to 70 vol% of SmBa$_2$SbO$_6$ in the composites, the resistivity shows a metallic behaviour, where as vol% $\geq$ 80 of SmBa$_2$SbO$_6$, it shows a semiconducting behaviour. The resistivity measurements indicate that the YBCO-SmBa$_2$SbO$_6$ composite shows superconducting transition (onset) up to 80 vol% of SmBa$_2$SbO$_6$ even though for 80 vol% of SmBa$_2$SbO$_6$ in the composite, no zero resistivity transition was obtained up to 77 K. It can be due to the absence of a superconducting network through the mixture of the composite because of the low vol% of YBCO. Thus the superconducting percolation threshold for the composite lies around 20 vol% of YBCO.

Fig 5.3 explains the electrical transport in the normal state. The resistivity ($\rho$) and the temperature coefficient of the resistivity $\left(\alpha = \frac{1}{\rho} \times \frac{d\rho}{dT}\right)$ at $T = 300$ K are shown in fig. 5.3. The normal state resistivity of the composite sample is dominated by the presence of YBCO, with a significant drop of $\rho$ occurring near 20 vol% of YBCO. The behaviour of $\rho$ correlates with that of $\alpha$. The value of $\alpha$ increases sharply around 20 vol% of YBCO. Thus the superconducting percolation threshold and normal state percolation threshold values of SmBa$_2$SbO$_6$ - YBCO composite lie in the same range. The normal state transport properties of a superconductor - insulator composite can be expressed by a set of relation (3) and (4). The values of $\rho_0$, $\rho_0^{1/4}$ t and u are found from the log-log plot of $\rho$ vs ($V_s$-$V_c$) and $\rho^1$ vs ($V_c$-$V_s$). Fig 5.4 (a-b) shows the log-log plot of $\rho$ vs ($V_s$-$V_c$) and $\rho^1$ vs ($V_c$-$V_s$). The value of $V_c$
Fig. 5.3 (a) Variation of normal state resistivity ($\rho$) and (b) Temperature coefficient of resistivity ($\alpha = \frac{1}{\rho} \frac{d\rho}{dT}$) at room temperature for different vol\% ($V_s$) of YBa$_2$Cu$_3$O$_{7-\delta}$.
Fig. 5.4.a Log-Log plot of resistivity $\rho$ vs $V_s - V_c$ where $V_s$ is the vol% of YBa$_2$Cu$_3$O$_{7-\delta}$ in the composite and $V_c$ is the percolation threshold value.
Fig. 5.4.b  Log-log plot of resistivity $\rho'$ vs $V_c - V_s$ where $V_s$ is the vol% of YBa$_2$Cu$_3$O$_{7.8}$ in the composite and $V_c$ is the percolation threshold value.
was chosen such that the two graphs yielded a straight line. The value of \( \rho_0, \rho_0', t \) and \( u \) are calculated from the figure

\[ V_e = 0.22, \quad \rho_0 = 3.8 \ \Omega \ \text{cm}, \quad \rho_0' = 6.7 \times 10^{11} \ \Omega \ \text{cm}, \quad t = 1.66, \quad u = 0.66 \]

The values of the exponent obtained agree with the predicted values for ideal metal insulator composite.

### 5.3 Percolation study of YBa\(_2\)Cu\(_3\)O\(_{7.5}\) - PrBa\(_2\)SbO\(_6\) system.

Pure YBCO has been prepared from high purity Y\(_2\)O\(_3\), BaCO\(_3\) and CuO by solid state reaction method. The compound PrBa\(_2\)SbO\(_6\) has been prepared from constituent oxides by the method described in chapter 3. The phase purity of the compound was verified with X-ray powder diffraction method using Ni filtered CuK\(\alpha\) radiation. YBCO-PrBa\(_2\)SbO\(_6\) composites were prepared by mixing different volume percentages of the compound (from zero to 100\%) of PrBa\(_2\)SbO\(_6\). It was then pressed in the form of pellets and sintered at temperatures in the range of 950-1030\(^\circ\)C for about 10h depending on the PrBa\(_2\)SbO\(_6\) volume fraction. The samples were then cooled slowly at the rate of 2\(^\circ\)C/min. to room temperature in air. The details of various composition and the corresponding sintering temperatures are provided in Table 5.2. The sample prepared as above were used to study their normal state and superconducting properties.
Table 5.2

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5.3.1 XRD studies of $YBa_2Cu_{3}O_{7-\delta}$-PrBa$_2$SbO$_6$ system

The reactivity and phase compatibility of the composite YBCO -PrBa$_2$SbO$_6$ system were studied by XRD. Fig. 5.5 shows the XRD pattern for different volume ratios of composites. In the XRD patterns of the composites having up to 70 vol% of PrBa$_2$SbO$_6$, two distinct sets of peaks, one corresponding to YBCO having an orthorhombic structure and the other corresponding to PrBa$_2$SbO$_6$ are seen. There was no evidence of any additional peak other than those of YBCO and PrBa$_2$SbO$_6$ in the XRD pattern, indicating that there is no detectable reaction between the two components in the composite.

5.3.2 Temperature Resistivity studies on $YBa_2Cu_{3}O_{7-\delta}$-PrBa$_2$SbO$_6$ composites

The variation of resistivity with temperature for different volume ratio of YBCO-PrBa$_2$SbO$_6$ composites are shown in Fig. 5.6. For simplicity and clarity, the normalized resistivity ($\rho/\rho_r$) where $\rho$ is the resistivity of the composite at different temperature and $\rho_r$ is the room temperature resistivity, is plotted in the figure. The
Fig. 5.5  X-ray diffraction pattern for different vol% of PrBa$_2$SbO$_6$ in the YBa$_2$Cu$_3$O$_{7.5}$ - PrBa$_2$SbO$_6$ composites. (a) 0%  (b) 10%  (c) 30%  (d) 50%  (e) 70% and (f) 100%. 
Fig. 5.6 Variation of normalized resistivity $\rho/\rho_0$ with temperature for different vol\% of PrBa$_2$SbO$_6$ in the YBa$_2$Cu$_3$O$_{7.8}$ - PrBa$_2$SbO$_6$ composites. (a) 0\% (b) 10\% (c) 20\% (d) 30\% (e) 50\% (f) 70\%
temperature-resistance curves show a metallic behaviour upto 70 vol% of PrBa$_2$SbO$_6$ in the system with a superconducting transition around 92 K. As the vol% of PrBa$_2$SbO$_6$ increases, the transition temperature decreases slightly. The composite with 70 vol% of PrBa$_2$SbO$_6$ is superconducting above liquid nitrogen temperature, but for higher vol%, the resistivity increases to a higher value and there is no superconducting transition till 77 K. The system YBCO-PrBa$_2$SbO$_6$ shows a semiconducting behaviour, if the insulator percentage increases beyond 80 vol%. This may be due to the absence of a superconducting network through the bulk of the composite for the conduction of supercurrents. The superconducting percolation threshold value of YBCO-PrBa$_2$SbO$_6$ composites lies around 20 vol% of YBCO. That is if the vol% of YBCO is more than 20, the composite system shows superconductivity and if the volume% of YBCO is less than 20%, the system YBCO-PrBa$_2$SbO$_6$ does not show any superconducting transition by resistivity measurements.

Fig 5.7 shows the variation of normal-state resistivity at room temperature as a function of vol% of insulating phase PrBa$_2$SbO$_6$. The normal state resistivity showed a sharp deviation in its value for about 75 vol% of Pr Ba$_2$ Sb O$_6$. The temperature coefficient of resistivity $\left(\alpha = \frac{1}{\rho} \times \frac{d\rho}{dT}\right)$ at room temperature is also plotted in Fig. 5.7. Both the normal state resistivity and temperature coefficient of resistivity showed a deviation at around 75 vol% of PrBa$_2$SbO$_6$ (or 25 vol% of YBCO). Thus the percolation threshold value for normal state resistivity lies around 25 vol% of YBCO below which the composite shows a higher resistance which is close to the
Fig. 5.7  
(a) Variation of normal state resistivity ($\rho$) and (b) Temperature coefficient of resistivity $\left(\alpha = \frac{1}{\rho} \frac{d\rho}{dT}\right)$ at room temperature for different vol% ($V_s$) of YBa$_2$Cu$_3$O$_{7-\delta}$. 

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Fig. 5.8  (a) Log-Log plot of resistivity $\rho$ vs $V_s - V_c$
5.8. (b) Log-log plot of resistivity $\rho$ vs $V_c - V_s$
resistivity of the insulator. The actual value of percolation threshold volume can be calculated from the percolation model equations governing the transport properties of the composite system (refer equations 3 and 4). The percolation threshold value $V_e$ calculated from the log-log plot of equations 3 and 4 for the YBCO-PrBa$_2$SbO$_6$ composite is 24 vol% of YBCO. The critical exponents calculated from Fig 5.8 (a-b). $t=1.56$, $u=0.57$, and the value of constants are, $\rho_0=2.6$ mΩcm, $\rho_0'=4.3 \times 10^{11}$ mΩcm.

The values of the critical exponents ‘$t$’ and ‘$u$’ matches with that for an ideal metal-insulator composite system

5.4 Percolation studies in a rapidly quenched superconducting YBa$_2$Cu$_3$O$_{7.5}$ - YBa$_2$SbO$_6$ (YBCO - YBSO) composite system.

It has been discussed earlier in chapter III Section 3.3.1 that superconductivity in YBCO-YBSO composite system is possible by rapidly quenching the composite directly from the sintering temperature unlike the widely reported slow-cooling process necessary to obtain superconductivity in YBCO, this unusual observations demands detailed investigation because of its tremendous technological implication in the processing of superconductor thin-film technology. In this section we discuss the percolation studies of rapidly quenched YBCO-YBSO composites.

The phase pure YBCO was prepared from high purity (99.9%) $Y_2O_3$, $BaCO_3$ and $CuO$ by solid state reaction. Single-phase YBSO was synthesized from constituent oxides $Y_2O_3$, $BaCO_3$ and $Sb_2O_3$. YBCO and YBSO, thus prepared, were mixed thoroughly in different vol% of YBSO and were pressed at a pressure of 5 ton
cm² in the form of discs having a diameter 10 mm and thickness 1.5 mm. These discs were sintered between 950 and 1020°C, depending on the vol% of YBSO in the composite for 15 h. in air and then quenched in air directly from the sintering temperature to room temperature. The details of various compositions and the corresponding sintering temperature are given in table 5.3. Quenching experiments were always performed on samples having identical size and shape.

**Table 5.3**

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<th>Vol% of PrBa₂SbO₆</th>
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The X-ray diffraction patterns of YBCO - YBSO composites for 0 to 100 vol% YBSO in the system quenched directly from the sintering temperature to room temperature are given in Fig 5.9. For the composites having 0 vol% YBSO (pure YBCO) a tetragonal structure was observed, where as all composites with YBSO greater than 2 vol% an orthorhombic structure was observed. In the XRD pattern two distinct sets of peaks, one corresponding to YBSO and the other to YBCO were clearly visible. There was no evidence of any additional peak other than those of YBCO and YBSO, indicating that there was no reaction between the two components, even under severe heat treatment for a prolonged period.
Fig. 5.9 XRD patterns for different vol% of YBSO in YBCO - YBSO composites. (A) 0\% (B) 10\% (C) 20\% (D) 30\% (E) 40\% (F) 60\% (G) 100\%
The resistivities of the composites with different vol% of YBSO were measured in the temperature range 77 - 300 K, and in Fig 5.10 the temperature dependence of normalised resistivities of the samples with different vol% of YBSO is given. Composite upto 80 vol% of YBSO showed metallic behaviour and a zero resistivity superconducting transition was obtained upto 60 vol%. For higher vol% (>80 vol%) of YBSO the composites showed semiconducting behaviour with no superconducting transition. This can be due to the absence of a continuous network of superconducting YBCO in the matrix, because of the low vol% of YBCO in the insulating medium.

The resistivity ($\rho$) and temperature coefficient of resistivity ($\alpha = \frac{1}{\rho} \times \frac{d\rho}{dT}$) at $T = 300$ K. are shown in Fig. 5.11 as a function of $V_s$, vol% YBCO in the composite. From the curve it can be seen that the superconducting percolation threshold is nearly $V_s \approx 20$ vol%. Values of $\rho_0$, $\rho_0^1$, $t$ and $u$ are found from the log-log plot of $\rho$ versus $V_s - V_c$ and $\rho^1$ versus $V_c - V_s$. Fig. 5.12 (a-b) shows the plot of $\rho$ vs $V_s - V_c$ and $\rho^1$ vs $V_c - V_s$. The least-square fits were performed to determine the slopes of the plots which gave the exponents $t$ and $u$ as 1.66 and 0.92 respectively with percolation threshold value $V_c = 0.22$. These values agree well with the theoretically predicted values for an idealized conductor-insulator system.
Fig. 5.10 Variation of normalised resistivity $\rho/\rho_r$ with temperature for different vol% of YBSO in the composite. (A) 0% (B) 10% (C) 20% (D) 30% (E) 40% (F) 60%.
Fig. 5.11  Variation of normal state resistivity ($\rho$) and (b) Temperature coefficient of resistivity ($\alpha = \frac{1}{\rho} \frac{d\rho}{dT}$) at room temperature for different wall% ($W_w$) of $YBa_2Cu_3O_{7-x}$.
Fig. 5.12.a Log-Log plot of resistivity $\rho$ vs $V_s - V_c$ where $V_s$ is the vol% of YBa$_2$Cu$_3$O$_{7-\delta}$ in the composite and $V_c$ is the percolation threshold value.
Fig. 5.12.b  Log-log plot of resistivity $\rho$ vs $V_c - V_s$ where $V_s$ is the vol% of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ in the composite and $V_c$ is the percolation threshold value.
5. 5 Discussion.

The studies on YBCO-SmBa$_2$SbO$_6$ system have shown that there is no reaction between YBCO and SmBa$_2$SbO$_6$ at normal temperature of processing. Even at elevated temperature of 1020°C, YBCO retains its superconducting properties without any reaction with SmBa$_2$SbO$_6$ or any decomposition by itself. The percolation theory has been applied to the composite system to explain its transport properties by considering YBCO as a normal metal and SmBa$_2$SbO$_6$ as an insulator. The model obeys very well as in the case of a metal insulator composite system. The superconducting and normal state percolation threshold value ($V_c = 22$ vol%) to be in the same range which is about 17 vol% of YBCO for metal-superconductor system. The critical exponent ‘t’ which describes the transport properties in the metallic side of the composite system agrees with that expected for an ideal conductor-insulator. It means that the system form a good composite upto the threshold volume, that is bout 78 vol% of SmBa$_2$SbO$_6$. The critical exponent ‘u’ which describes the transport properties in the insulating sides of the composites also agrees with the expected value for an ideal metal-insulator system. It means that beyond the threshold value i.e., for more than 78 vol% SmBa$_2$SbO$_6$, the system reacts and does not form a perfect composite.

In the case of YBCO-PrBa$_2$SbO$_6$ composite, studied secondly, the percolation threshold is bout 24 vol% of YBCO in the system. Also the critical exponent ‘t’ and ‘u’ agrees with the theoretically expected values. Results obtained from the quenching studies of YBCO - YBSO composite shows that the normal state
percolation threshold and critical exponents describing the transport properties of the composite agree very well with that of an ideal conductor-insulator composites. XRD studies on the above composite systems have shown that particularly there is no reaction between YBCO with SmBa$_2$SbO$_6$, PrBa$_2$SbO$_6$ and YBa$_2$SbO$_6$ throughout the entire volume range.

Due to the non-reactivity of YBCO with the above insulating materials, it is expected that good quality films of YBCO can be fabricated on substrates made of these materials and the results are given in Chapter 6.
References


