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Direct current magnetron sputtered In_2O_3 films as tunnel barriers

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Insulating films of In_2O_3 were prepared by sputtering indium in the presence of pure oxygen using dc magnetron sputtering. Transmission electron microscopic investigations showed the films to be single phase and polycrystalline. Analysis of the optical transmittance data showed the films to have an optical band gap of 3.71 ± 0.01 eV. Tunnel junctions were made with high T_c superconductors $Bi_2Sr_2Ca_1Cu_2O_y$ and $NdBa_2Cu_3O_{7-\delta}$ using indium oxide as the barrier layer and $Pb_{0.5}In_{0.5}$ as the counter electrode. The conductance spectra displayed prominent structures attributable to energy gap. The reduced gap parameters for $Bi_2Sr_2Ca_1Cu_2O_y$ and $NdBa_2Cu_3O_{7-\delta}$ were found to be 4.0 ± 0.5 and 5.2 ± 0.6, respectively.

INTRODUCTION

Traditionally indium oxide films[1] were studied with a view toward utilizing them as highly conducting and transparent electrodes for various device applications. When doped with tin (usually termed as ITO) the films showed an optical transparency of more than 90% and a resistivity of the order of 10^{-5}–10^{-6} Ω cm. An interesting feature of In_2O_3 films is that depending upon the growth conditions (method of preparation, substrate temperature, residual gas pressure etc) films having totally different properties (insulator, metal, semiconductor) can be prepared, a fact which is of fundamental importance. This is a consequence of the well established fact that each oxygen vacancy in indium oxide acts as a doubly ionized donor. Recently it has been shown[2] that films deposited by ion beam sputtering show amorphous structure; however, they still retain high transparency and conductivity. Further, tunnel junctions of superconducting composite-amorphous indium oxide films[3] on oxidized Al-Mn films showed certain anomalous properties such as differential conductance varying logarithmically with applied bias, at variance with the expected behavior (dependence on the square root of the bias).

In this article we present the results of our studies on deposition and characterization of insulating films of In_2O_3 by reactive dc magnetron sputtering and their possible use as barrier layers in tunnel junctions. For single particle tunneling studies on high T_c superconductors, a variety of barrier layers have been employed. This includes MgO,[4] Al_2O_3,[5] SiO,[6], CdS,[7] Y_2O_3,[8] Cu_2O,[9] Sr_xTiO_y,[10] $BaTiO_3$,[11] Ta_2O_5,[12] *a*-Si,[13] CeO_2,[14] and $BiSr_2CuO_6$[15] films. Good quality tunnel junctions could also be made with native oxides as barriers.[16–18] By good quality we mean where (i) the single step nature of the tunnel current could be established and (ii) the junctions show low leakage. In addition, the Schottky barrier formed between the superconductor and a degenerate semiconductor[19,20] (oxygen deficient SnO_2 or Zn doped GaAs) has also been successfully used to study the tunnel characteristics.

Artificial barriers are often used when the tunnel junction configuration is of the sandwich type, because in the point contact geometry, the tip of the probe usually pierces the sample surface, rendering the barrier layer ineffective. The barriers are generally deposited over the sample surface which is cleaned *in situ* by the usual procedures viz., glow discharge cleaning, ion beam etching, etc. An additional advantage of using artificial barriers is that they may act as passivating layers, retaining the homogeneity of the sample surface, which is essential for the formation of good quality tunnel junctions. Further, if the chosen barrier layer is also a photoconductor (for example Cu_2O), photosensitive experiments[9] such as the modulation of barrier height by a light source can be performed. Recently Zeng *et al.*[21] have studied the characteristics of $YBa_2Cu_3O_{7-y}$/Al/In_2O_3/Si structures. They have used conducting indium oxide films to minimize the interdiffusion between $YBa_2Cu_3O_{7-y}$ and silicon.

Artificial barriers have played an important role in the case of tunnel junctions made from conventional superconductors.[22] They were highly successful, especially when the base metal did not form a suitable native oxide for tunneling to take place. For instance, niobium based junctions with an Al_2O_3 barrier proved to be invaluable in Josephson junction technology.

In the present study tunnel junctions of two high temperature superconductors namely $Bi_2Sr_2Ca_1Cu_2O_y$ (a well studied system) and $NdBa_2Cu_3O_{7-\delta}$ (a not so well studied system) were prepared with sputtered In_2O_3 as the barrier layer and thin film $Pb_{0.5}In_{0.5}$ as the counter electrode. The junction characteristics were studied at 4.2 K. The results show that indium oxide acts as a reasonably good artificial barrier. A detailed discussion of the tunnel data appears elsewhere.[23,24]

EXPERIMENT

In_2O_3 films were made in a LEYBOLD HERAEUS UNIVEX 450 thin film unit. Indium was reactively sputtered in oxygen atmosphere, using dc magnetron sputtering. Glass/quartz plates were used as substrates. The substrates were held at room temperature during film deposition. The system was initially evacuated to a pressure of better than 10^{-4} Pa and then back-filled with pure oxygen to the desired pressure. Prior to deposition the chamber was flushed several times with oxygen. Sputtering

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was done at an oxygen pressure of 2×10^{-1} Pa and at a power level of 90 W (sputtering was done at constant pressure mode) which led to a power density of $\sim$2.5 W cm^{-2} The film thickness and rate of deposition were monitored by a calibrated INFICON quartz crystal thickness monitor. A typical deposition rate was 1–2 Å s^{-1}. Film thickness was also checked from the interference pattern of the transmittance data.

Room temperature resistivities of the samples were measured using a co-planar configuration with thick vapor deposited copper films as electrodes. Current was measured using a Keithley model 617 electrometer. The films had resistivities more than 10^8 Ω cm. Optical absorption measurements were made using a HITACHI model U-3400 spectrophotometer. Transmittance spectra were recorded in the wavelength range 300–800 nm with unpolarized light at room temperature. Substrate absorption, if any, was corrected by introducing a bare substrate in the reference beam. The absorption coefficient was computed from the transmittance spectra.

Film structure was analyzed using transmission electron microscopy. The adhesion of In_2O_3 films to glass was extremely good. So films for this study were deposited on freshly cleaved KCl substrates. This enabled the films to be easily floated off in water for subsequent studies. The transmission electron microscope (TEM) investigations were carried out using a PHILIPS model EM312 scanning transmission electron microscope. The accelerating potential was in the range 100–125 keV. The beam currents were a few tenths of a microampere. It should be mentioned here that the structure of vapor deposited films did not depend on the type of substrate used and films deposited on different substrates[1] (KCl, NaCl, amorphous carbon, boric oxide coated glass, etc.) have all shown similar structural characteristics. So we believe that the structure of films sputtered on KCl substrates will not be different from those deposited on high T_c superconductors.

Tunneling studies were made on well characterized sintered pellets of $Bi_2Sr_2Ca_1Cu_2O_y$ (BSCCO) and $NdBa_2Cu_3O_{7-\delta}$ (NBCO). The samples were synthesized by the conventional solid state reaction of the constituent oxides. The single phase nature of the samples was confirmed by x-ray diffraction. A four probe resistivity measurement showed BSCCO and NBCO to have zero resistance at 74 and 88 K respectively. Susceptibility measurements made using a commercial Quantum Design SQUID magnetometer showed the samples to have a Meissner fraction of more than 80%.

The details of junction fabrication are described elsewhere.[23,24] A 10-nm-thick In_2O_3 film was sputtered onto one face of the pellet. This was followed by the deposition of a 200-nm-thick $Pb_{0.5}In_{0.5}$ film using a suitable metal mask. This served as the counter electrode. A 200-nm-thick indium film deposited on the other face of the pellet served as the bottom electrode. Prior to the barrier layer deposition the sample surface was glow discharge cleaned for 15 to 30 min in an argon atmosphere so that a fresh surface would be presented for the subsequent barrier layer deposition. The principal aim behind such a cleaning was that it would remove any adsorbed atoms or molecules and the nonsuperconducting layer which is invariably present on the sample surface.

For glow discharge cleaning, usually an inert gas plasma is preferred as it does not react with the sample surface. However for high T_c superconductors its use is disputed because it was shown that cleaning the sample surface with argon ion beam results in structural changes viz., breaking of Cu–O bonds and subsequent loss of oxygen. Photoemission studies[25,26] have shown that this leads to the formation of a few nm thick nonsuperconducting layer on the sample surface. To avoid this the use of oxygen plasma[27] was suggested, even though with this it was not possible to remove the nonsuperconducting layer completely. In the present case we have used glow discharge cleaning, where the ion energy is relatively low. Therefore we believe that such a cleaning process should be effective in removing the adsorbates and a few layers of the nonsuperconducting layer without causing any structural changes on the sample surface.

A typical junction area was 1 mm^2. A four probe method was employed to measure the junction characteristics and all the measurements were made at 4.2 K. The data handling was already described.[24] Briefly, the raw data was smoothed, splined, and differentiated. Smoothing was done chiefly because the current-voltage (I-V) data were taken manually at discrete points and when differentiated the random errors were expected to be high. An important aspect of data smoothing is that the magnitude of the conductance or its variation with bias did not change significantly when smoothing parameters were varied.

RESULTS AND DISCUSSION

Figure 1(a) shows a typical selected area diffraction pattern for a In_2O_3 film having a thickness of 70 nm. The corresponding micrograph is shown in Fig. 1(b). The polycrystalline nature is evident from the well defined rings and the formation of the crystallites is clearly seen in the micrograph. The magnification used is 10^5. The crystallites are nearly 200 Å in diameter. The crystallite size is comparable to that of the films deposited at a substrate temperature of 165 °C by electron beam evaporation. The lattice spacings were calculated from the diffraction relation $d_{hkl}=2\lambda L/D$ where λ is the DeBroglie wavelength, which can be calculated from the accelerating potential after applying the relativistic correction. L is the distance between the sample and the photographic film and D is the diameter of the diffraction rings. The calculated d_{hkl} values showed excellent agreement with the ASTM (No. 06-0416) data. Table I gives the calculated lattice spacings together with ASTM data. It is to be mentioned here that electron beam deposited films showed certain additional reflections[1,28] whose significance was not yet understood. However our films showed no such reflections.

The absorption coefficient was calculated from the transmittance data alone. In principle the determination of optical constants would involve both transmittance and reflectance measurements.[29] However, this method is rather complicated because the relevant equations have

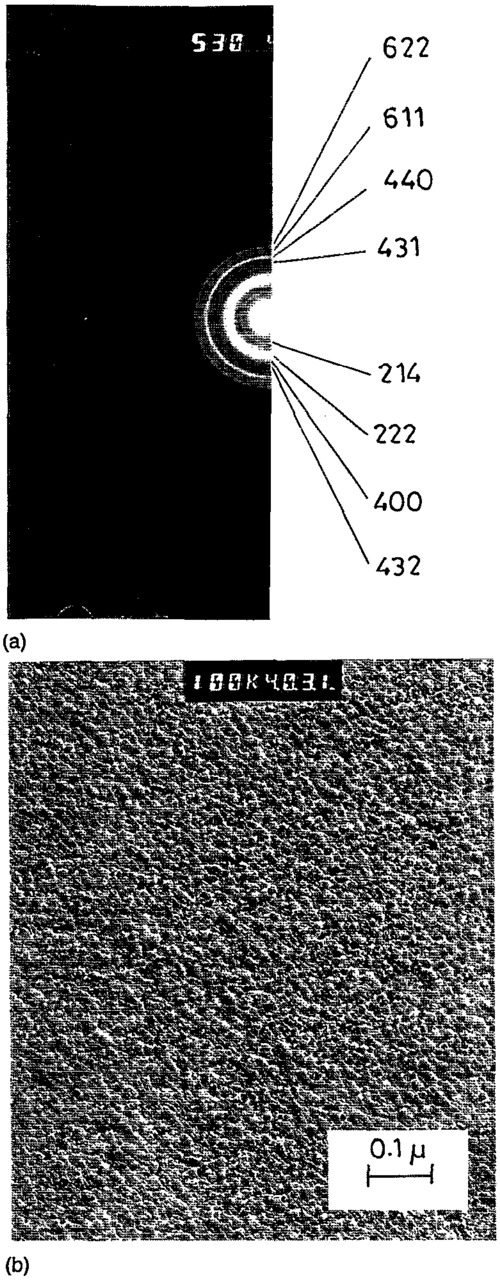

FIG. 1. (a) Selected area diffraction pattern for an as deposited indium oxide film and (b) the corresponding micrograph.

multiple solutions, which depend critically on film thickness. Alternate methods, valid under specific conditions, involve the analysis of either the transmittance[30] or the reflectance[31] data alone, and the evaluation of the absorption coefficient from either.

Neumann *et al.*[32] have suitably modified the general expressions for the optical absorption to calculate the absorption index and hence the absorption coefficient from transmittance data alone for the case of a thin absorbing film on a thick nonabsorbing substrate. Above the band edge, the absorption coefficient increases steeply reaching a value of about 10^5 cm^{-1} for In_2O_3. Further, the substrate thickness is very large compared to the film thickness. This

TABLE I. Lattice spacings calculated from the electron diffraction pattern for a typical indium oxide film. Values within the brackets give intensity ratios.

No.	d_{hkl}	d (Å) (measured)	d (Å) ASTM Std.	Remark
1	214	4.17	4.130 (14)	···
2	222	2.94	2.921 (100)	···
3	400	2.53	2.529 (30)	···
4	432	2.18	2.157 (6)	···
5	431	1.99	1.984 (10)	···
6	440	1.79	1.788 (35)	···
7	611	1.62	1.641 (6)	···
8	622	1.49	1.525 (25)	···
9	721	1.35	1.377 (4)	Faint line
10	820	1.22	1.227 (2)	Faint line
11	840	1.13	1.1312 (4)	Faint line
12	932	1.04	1.0436 (2)	Faint line

situation may be considered as a thin absorbing film on a thick nonabsorbing substrate. Taking the refractive index of In_2O_3 to be 1.96,[1] the relevant equations for this case were solved using the Newton–Raphson technique. The absorption coefficient was computed such that the difference between the computed and the experimental transmittance values is less than a preset minimum.

The energy gap was evaluated from the absorption coefficient data using the relation for direct allowed transition. Figure 2 shows a plot of transmittance as a function of energy. The films have a transmittance of 85% in the visible region. In order to evaluate the band gap the absorption data were plotted using the relation $[\alpha h\nu \propto (h\nu - E_g)^{1/2}$, where E_g is the band gap] for direct allowed transitions. The inset to Fig. 2 shows such a plot where $(\alpha h\nu)^2$ was plotted against energy. A least squares method was used for the extrapolation and to find the intercept on the abscissa, viz., the band gap. The band gap was found to be 3.71 ± 0.01 eV, which agrees well with the reported values.[1]

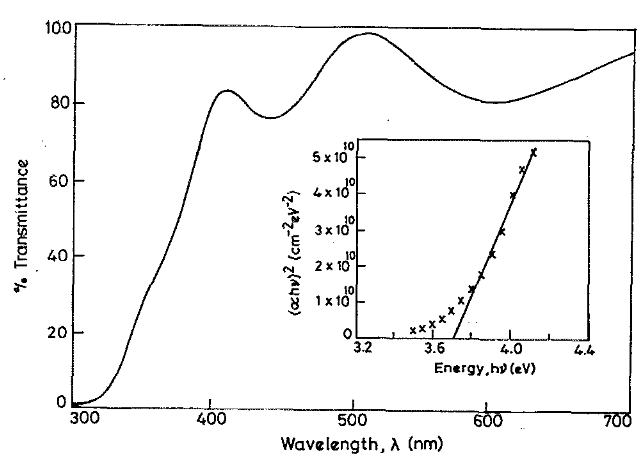

FIG. 2. Variation of transmittance as a function of wavelength for a typical indium oxide film. The inset shows a plot of $(\alpha h\nu)^2$ against energy. The continuous line shows the least squares fit.

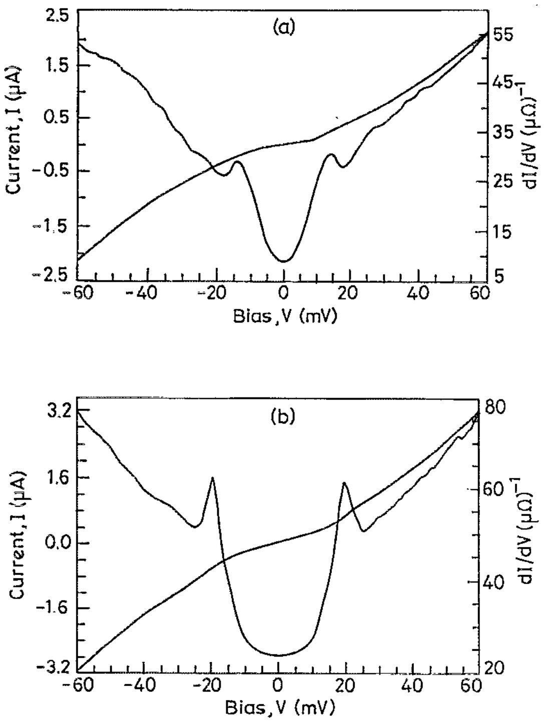

FIG. 3. (a) The *I-V* characteristics together with the conductance data for a typical BSCCO junction and (b) the corresponding plot for the NBCO junction.

The results of the tunneling studies are shown in Fig. 3. In Fig. 3(a) the *I-V* characteristics, together with the numerically evaluated first derivative, are plotted for the BSCCO junction. Figure 3(b) shows the corresponding plots for the NBCO junction. In the figures the voltages are referred to with respect to the counter electrode. As seen from the figures the conductance spectra display prominent overshoots, clearly suggestive of a superconducting gap. The structures appear at about ± 20 meV for NBCO and ± 13 meV for BSCCO. The characteristics are almost symmetric with respect to the bias, implying a large and symmetric barrier. In addition the trace shows finite conductance at zero bias. What we presented here is the best of our results, although curves of this type with some variation in shape and peak position could be obtained with reasonable reproducibility. Tunneling[33,34] and photoemission [35] studies on BSCCO single crystals showed the energy gap in BSCCO to be anisotropic with $2\Delta_{ab}\sim 46$ meV and $2\Delta_c \sim 26$ meV. We have obtained 13 meV which agrees well with the gap value measured along the *c* axis direction. For NBCO, as there are no reported tunnel measurements, we compare our value with that measured using far infrared reflectance (FIR) spectra. The FIR spectrum of NBCO reported by Crawford *et al.*[36] shows appreciable drop at about 500 cm^{-1}. If it is taken as an indication of the opening of a gap then it gives 25 meV for the gap, which is reasonably close to our value of 20 meV. Taking the T_c for BSCCO and NBCO to be 74 and 88 K, respectively, we obtain the reduced gap $2\Delta/K_BT_c$ to be 4.0 ± 0.5 and 5.2 ± 0.6 in the two cases.

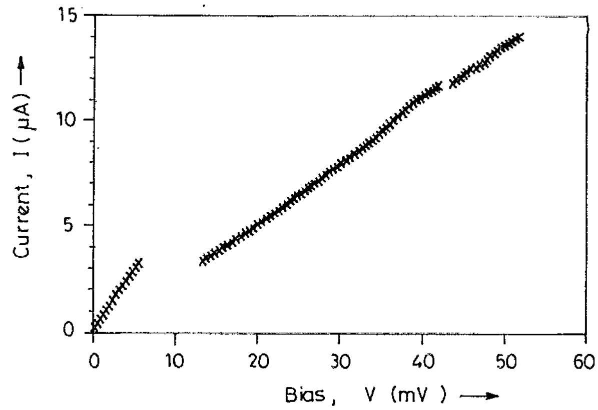

FIG. 4. The *I-V* characteristics of a typical BSCCO junction with a 30-Å-thick barrier layer.

To verify that indium oxide layer indeed acts as the barrier, junctions were fabricated with much thinner indium oxide layer. The idea is to find out whether the product of the junction resistance and its area shows the expected exponential dependence on barrier thickness.[37] Figure 4 shows the typical *I-V* characteristics of a BSCCO-In_2O_3-$Pb_{0.5}In_{0.5}$ junction with a 30-Å-thick indium oxide layer. The *I-V* characteristics of such junctions show Josephson-like behavior, completely different from those with a thicker barrier (100 Å). For a fixed junction area, at low biases the current increased by nearly 2 orders as the junction thickness was reduced from 100 to 30 Å. As seen from the figure, even at low biases there is a finite voltage drop indicating the presence of shots and the "leaky" nature of the junctions. An interesting feature that may be noticed is the voltage jump seen at about 13 mV [In principle as $T\rightarrow 0$ a switch-over should occur to $(\Delta_1+\Delta_2)/e$ where Δ_1 and Δ_2 are the energy gaps of a Pb-In alloy and BSCCO, respectively]. If we take this to be the Δ of BSCCO then it shows an excellent agreement with the Δ value obtained from our single particle tunneling characteristics. In addition the characteristics show a discontinuity at about 40 mV whose origin is not clear at present.

For a tunnel junction with a simple rectangular potential barrier, the product of junction resistance and its area at low biases can be written as [38]

$$RA=3.17\times 10^{-11}(t/\phi^{1/2})\exp(t/\beta),$$

where R is the junction resistance, A is the junction area, t is the barrier thickness, ϕ is the barrier height, and β is the effective tunneling length. The ratio of the RA's for the junctions with two different barrier thicknesses can be written as

$$(RA)_{100}/(RA)_{30}=\exp(t_{100}-t_{30})/\beta,$$

where we have taken into account only the dominant exponential term. The subscripts denote the sample thicknesses. From the above relation β can be calculated. (In principle measurements should be made on junctions with a wide range of barrier thicknesses and β should be computed from the slope of the plot of junction thickness against *RA*). The *RA* value for each junction was determined from the low bias (ohmic) *I-V* characteristics measured at 4.2 K. Then β was calculated using the above equation. The calculated value of β was ~18 Å, which is rather large when compared to the value (1–2 Å) for a perfect barrier.[39] Similar high values have been observed in Nb/Pb junctions [40] with zirconium fluoride barrier and was attributed to the presence of intermediate states in the barrier.

In the present case the high value of β may be due to the nonuniformity of the barrier and/or due to the interface layer that would be formed between the barrier and the high T_c material. If the deposited indium oxide does not form a uniform film when the thickness is low, then the barrier may consist of two components, viz., small islands of sample surface in parallel with the deposited barrier layer. This will lead to alternate paths for current in addition to tunneling. Some evidence for the distortion of the single step tunnel current could also be seen in our single particle tunneling results where particle conservation was not strictly satisfied (Fig. 3).

Regarding the interface between indium oxide and high T_c superconductors, no detailed investigations have been reported so far. However, a lot of work has been done on metal-high T_c superconductor interfaces. Secondary ion mass spectrometry[41] and photoemission investigations[42–47] on metal-high T_c superconductor interfaces showed that except for gold and silver,[41,42] most of the other metals (Al, Cu, Fe, In, Pd, Pb, Sn, Ti, W, Zn)[42–45] take up oxygen from the superconductor surface and become oxidized. This depletion of oxygen from the sample surface results in the destruction of superconductivity and the formation of a semiconducting layer on the superconductor surface. This has also been borne out by contact resistance studies between various metals[43,46–48] and high T_c superconductors and also between metal alloys (Ag-Pd, In-Cd, In-Pb, In-Sn, etc.)[49,50] and high T_c superconductors. These studies have shown that only gold and silver exhibit very low contact resistances ($\sim 10^{-9}$ Ω cm) as they do not easily form oxides and hence do not react with the sample surface. Studies on interfaces between high T_c superconductors and different insulators (MgO, $SrTiO_3$, YSZ, sapphire, TiO_2, ZrO_2, Cr_2O_3, Al_2O_3, CaF_2, MgF_2, etc)[51,52] have also been reported. The mechanism of formation of interface layers in these junctions is rather complicated and is significant only at high temperatures.

Studies on indium-high T_c superconductor interfaces[50] indicated the formation of an In_2O_3 layer at the interface which was also responsible for the high contact resistance ($\sim 10^{-6}$ Ω cm) observed. This has also been confirmed by the studies of *I-V* characteristics of In-high T_c superconductor contacts,[53] which showed nonohmic behavior. In the present case the deposited layer is an oxide. Therefore the modification of the superconductor surface viz., the formation of a nonsuperconducting region at the interface is avoided. Recently conducting In_2O_3 and ITO films were used as buffer layers to grow oriented $YBa_2Cu_3O_{7-y}$ films on silicon[21] and Al_2O_3[54] substrates. It was found that the use of these buffers improved the structural perfection of the $YB_2Cu_3O_{7-y}$ films. This shows that even at high temperatures, indium oxide-high T_c superconductor interface will not affect the film properties. In the present study indium oxide was deposited onto a high T_c superconductor surface which was kept at room temperature. So we believe that the indium oxide-high T_c interface will not have a noticeable effect on the junction characteristics. Further if there are any oxygen deficient regions on the sample surface, the indium oxide layer might saturate the region, preventing further degradation of the surface (the exact nature of the interface can be determined only by studies like x-ray photoelectron spectroscopy).

These observations grossly show that although indium oxide does not form an ideal barrier it does have control over the junction characteristics. One final point should be mentioned. In our studies although the measurements are made at 4.2 K, which is below the T_c of the counter electrode $Pb_{0.5}In_{0.5}$ (6.52 K),[55] features representing tunneling between two different superconductors were not seen. Similar observations have been made by a number of workers.[56,57] In some cases the single step nature of the tunnel current was established by the presence of either the counter electrode gap structures at low biases or its phonon spectra. In the present case the noise level due to the highly resistive nature of the junctions rendered such observations difficult. The absence of the "sum" and "difference" features could to some extent be due to the distortion of tunnel currents by other inelastic processes in the gap. The main reason may be that the width of the peaks are larger than the T_c of $Pb_{0.5}In_{0.5}$. This will certainly smear out the extra structures. In addition, the measurements are made with a counter electrode of finite area especially over sintered pellets. So the finer features will be smeared out and only average properties will be obtained.

In conclusion we have deposited insulating films of In_2O_3 using dc magnetron sputtering from a pure indium target. The as deposited films were polycrystalline in nature and showed an optical band gap of 3.71 ± 0.01 eV in accordance with the reported values. Tunnel junctions made with BSCCO and NBCO samples with In_2O_3 as the barrier layer and $Pb_{0.5}In_{0.5}$ as the counter electrode showed features characteristic of an energy gap. The investigations show that indium oxide acts as a reasonable tunnel barrier. The reduced gap parameter for BSCCO and NBCO was found to be 4.0 ± 0.5 and 5.2 ± 0.6, respectively.

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