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THE FORMATION AND ANALYSIS OF THIN FILM HIGH TEMPERATURE SUPERCONDUCTORS

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Abstract

Thin films of high temperature superconductors have been fabricated using a variety of physical vapor deposition techniques. Recent results of HTS thin films produced by coevaporation, sputtering and laser deposition will be briefly reviewed. In addition some examples of the utility of high energy ion backscattering for the analysis of film stoichiometry will be given.

I. INTRODUCTION

The recent discovery of oxide superconductors with transition temperatures in excess of 90 K has for the first time offered the possibility of operating a superconductor at liquid nitrogen temperatures. Many of the potential applications for such a superconductor will be in the area of electrical devices and detectors that will require the production of superconducting thin films.

Within a very short time of the discovery of the high temperature oxide superconductor, various research groups around the world reported the successful fabrication of high temperature superconducting (HTS) thin films [1]. The most common method of fabricating HTS thin films is by the simultaneous physical vapor deposition (PVD) of the alloying constituents onto the substrate which are then subsequently annealed at relatively high temperatures (850 -900 C) in flowing oxygen. The purpose of the anneal is to both oxygenate the deposited alloy and to transform the as-deposited, amorphous film into the superconducting phase.

The most widely used techniques for the PVD of the three metallic components include electron beam or resistive deposition, alloy sputtering from a multicomponent target or multiple sources, or laser assisted deposition from the bulk HTS pellets. Recently these PVD techniques have been coupled with various reactive oxygen processes to produce the HTS film directly in the evaporation chamber at relatively low temperatures (400 to 700 C)

In the present paper we will briefly review the HTS thin film PVD techniques; electron beam evaporation, sputtering, and pulsed laser evaporation. In addition, since film stoichiometry is a critical parameter for the production of high quality superconductors, high energy backscattering techniques applied to these materials will also be reviewed.

II. PHYSICAL VAPOR DEPOSITION OF HTS FILMS

A. Electron beam deposition

One of the most common fabrication procedure for producing HTS thin films by PVD is by electron beam or thermal evaporation. Films have been successfully fabricated by the simultaneous evaporation of Y, Ba, and Cu [1-4] as well as by the layered evaporation of these components [5,6] or their oxides [7].

In the most fundamental processes, Y, Ba, and Cu are simultaneously evaporated in reasonably high vacuum (10^{-6} to 10^{-8} torr.). The role of deposition temperature, substrate material, post deposition anneal, and film stoichiometry have all been found to play an important role in the ultimate quality of the HTS film[1-4,8,9]. Most of the reported deposition procedures utilize $SrTiO_3$ <100> single crystal substrates which are not heated during the deposition. The post deposition annealing sequence for Y-Ba-Cu films varies substantially from research group to research group with maximum temperatures ranging between 850 and 900 ^{o}C[1-4]. The primary purpose of this processing

step is to produce fully oxygenated orthorhombic superconducting films with a high degree of c-axis orientation and a minimum of substrate-film interdiffusion. The role of film stoichiometry is clearly important since the $YBa_2Cu_3O_7$ phase is a line compound. Research performed at Stanford showed that film orientation is highly dependent on metal stoichiometry with highly C-axis films being obtained for compositions slightly rich in Ba[8].

While the two step procedure of producing superconducting films, evaporation followed by oxygen annealing, has had moderate success, consistency in the superconducting properties, such as the transition temperature and width, and critical currents have been less than ideal. One major reason for these inconsistencies has been attributed to the highly reactive nature of Ba metal. Films removed from the evaporator are observed to react with the ambient prior to reaching the oxygen furnace. One solution to this problem is in-situ oxygenation. An alternate solution, discovered by Mankiewich et al, is to replace Ba metal with BaF_2 as a deposition source material[10,11]. The advantage of BaF_2 is its stability relative to compounds which might form through reaction with room air [12] and that it is evaporated into the film as molecular BaF_2 [10]. Conversion of a Y-BaF_2-Cu film to an oxide superconductor is achieved by annealing in a flowing $H_2O + O_2$ ambient. The thermodynamics of the process have been recently discussed by Chan et al [13].

One of the primary disadvantages of post deposition oxygenation and annealing is the high processing temperature required. While good films have been produced on $SrTiO_3$ and MgO, processing in the range of 850 to 900°C is unsuitable for semiconductor substrates. An alternative form of producing films is to deposit the metallic components on to a heated substrate in the presence of mildly energetic and reactive oxygen. The temperature conditions assumed in these experiments are that the substrate temperature be at or below the tetragonal-to-orthorhombic transition temperature and also allow sufficient crystallization and oxidation of the film. The successful application of reactive deposition to HTS has been reported by several research groups using both electron and thermal evaporation techniques[14-19].

B. Sputter Deposition

Sputtering is a non thermal thin film PVD process where atoms, (or molecules) are removed from a target by kinetic energy transfer from accelerated ions. The accelerated ions are typically one of the noble gases created in a plasma near the target material. These ions are then accelerated to the biased target. The material ejected from the target then deposits onto the substrate.

Several commercially available methods exist for the generation and acceleration of the plasmas used in sputtering. One of the most commonly used methods for the deposition of HTS thin films is magnetically enhanced diode

sputtering. In this process, the plasma is confined near the target region by strong magnetic fields. The plasma is generated and accelerated with either a DC or RF bias voltage. The RF bias is necessary when insulating targets such as oxides or fluorides are used. This process is schematically illustrated in Figure 1.

Sputter deposition from either single compound targets[20-24] or from multiple elemental targets[25-27] has been equally successful in depositing HTS films. A system that uses multiple targets provides greater flexibility in the choice of deposition parameters (pressure, substrate temperature, rate, film composition, and source-substrate geometry). However, careful attention must be paid to the background gases present in the system when using elemental targets of reactive metals. Small amounts of reactive gases will cause variations to occur in the sputter yields. Load locks for substrate introduction and removal have been employed to minimize these variations[28]. Rotation of the substrates during deposition is required to minimize compositional variations of the films over substrate radii of several centimeters[29]. Use of a single compound target offers, in principle, simplified stoichiometry control. However, in practice, variations in the sticking coefficients and the vapor distributions of the elements require that a new target composition be formulated for a specific film composition and set of deposition parameters[30]. At elevated temperatures for example,

variations in substrate temperature will affect the fraction of volatile species (e.g. copper oxide, barium) in the deposit[31]. Also, if oxygen is introduced during deposition to promote the growth of oxides in the film, careful attention must be paid to its effects on preferential resputtering of cations from the film[31,33].

Sputtering, in conjunction with post deposition annealing, has produced high quality films of superconducting material from the rare earth[20-27], bismuth[34,35] and thallium[36-39] based systems. Current densities in excess of 10^6 amps/cm^2 at 77 K [40] have been achieved. As with the other thermal evaporation techniques, reactive, low temperature (650^oC), in situ growth has been demonstrated on a variety of oriented oxide[41,42] and metallic[43] thin film substrates.

These latter films can be of high quality with the c axis perpendicular to the substrate and quite smooth film morphologies. The in situ process offers a viable method for coating large areas with superconducting films if the cation stoichiometry is well controlled.

C. Pulsed Laser Deposition

In this section we will briefly describe the pulsed laser deposition (PLD) technique as it has been applied to the fabrication of high temperature superconducting (HTS) thin films, review some recent results and indicate directions for future research. Laser deposition of thin films has been actively researched over the last 25 years

and has been recently reviewed[44]. Venkatesan, et. al. have recently reviewed their work in PLD of HTS[45].

This physical vapor deposition technique uses laser induced evaporation to generate a vapor plume from a composite target mounted in high vacuum chamber. The plume, which consists of atomic neutrals, ions and metal suboxides is subsequently deposited on a substrate[46]. Depending on the target material, laser energy density, wavelength and chamber pressure the mechanism of material removal may proceed via pyrolytic or photolytic means. In addition, the target-substrate distance and substrate temperature are also important parameters in controlling deposition rates and film quality. Pulsed sources, including Nd:YAG and excimer lasers, have been used to deposit HTSC thin films.[47,48] However, pulsed excimer laser deposition (PLD) at 308, 248 and 193 nm has emerged as the method of choice due to the shorter penetration depths of the UV radiation, availability of high pulse energies and repetition rates, and spatial uniformity of the beam intensity profile.

Many groups have reported deposition of thin films of $YBa_2Cu_3O_{7-\delta}$ [49-53] using PLD. Due to the rapid, nonequilibrium laser induced heating, 10^6 K/s, congruent evaporation can occur, giving rise to stoichiometric film deposition. In addition, this process has been shown to cause a highly directional plume of material, with angular flux distributions of $\cos^{9-11}\theta$[54], to be ejected normal to the target surface. In the simplest mode of operation films

of amorphous metal oxides have been deposited at room temperature and then post-annealed to crystallize the superconducting phase, directly analogous to non-reactive coevaporation or sputtering.

However, an important area of research has been the development of reactive, in-situ pulsed laser deposition to fabricate fully superconducting thin films in a one step process. Improved epitaxy[55], smooth morphology[56,57] as well as ultra-thin[58] $YBa_2Cu_3O_{7-\delta}$ films have been reported by these techniques. Substrate temperatures between 400-800 ^oC have been reported by various groups[55-59] and much work is being done to lower the processing temperature even further by incorporation of activated oxygen during film growth. Methods of activated oxygen production using glow discharge rings[59], microwave discharges[19], ozone generation[18], low energy oxygen ion implantation[15] as well as photodissociation of O_2 and N_2O[55] by the deposition from laser or other photolytic sources are currently being investigated. The broad goal of this work is to lower processing temperatures even further to allow for compatibility with semiconductor processing as well as extend the number of useable substrate materials.

As the majority of PLD studies have been devoted to deposition of $YBa_2Cu_3O_{7-\delta}$ thin films, here we only report some representative results from this area. Thin films of $YBa_2Cu_3O_{7-\delta}$ produced by reactive PLD have reported critical current (J_c) and RF resistance (R_s) values comparable to the

best results derived from other PVD techniques. Jc's as high as 5 x 10^6 A/cm^2 at 77 K and zero applied field[58] have been reported.

An important aspect of the low processing temperatures achieved to date by PLD has resulted in the deposition of HTS thin films on a variety of substrates. Substrate temperatures as low as 400 ^oC have been reported for in-situ deposition on Si at 193 nm[59]. Transport J_C's > 10^3 A/cm^2 at 31 K and zero applied field have been obtained for thin films deposited on Si with a MgO buffer layer[60].

In situ, 248 nm, PLD at 650 ^oC (block temperature) has resulted in the fabrication of very smooth HTS thin films. SEM analysis showed a peak surface roughness of less than 4 nm. A crossectional TEM study of these films confirmed the epitaxial nature of film growth on single crystal substrates[57].

Novel ultra-thin films have also been deposited by reactive PLD at 248 nm with O_2 partial pressures of 0.1 torr. Films as thin as 10 nm were prepared that exhibited Jc's of 1 x 10^5 A/cm^2 at 10 ^oK and zero applied field[58]. High O_2 partial pressures, 0.1-0.3 torr, during deposition between 750 -780 ^oC were also found to be beneficial for producing single crystal thin films by PLD at 193 nm[55].

In summary, it is likely that PLD will emerge as a thin film PVD technique advantageous to deposition of multi-element coatings, of which HTS is a prominent example. However, much work still needs to be done to understand

fundamental issues such as the role of activated oxygen in promoting in-situ film growth at lower temperatures. Additionally, extending the successes of in situ PLD to the $Tl_2Ca_yBa_2Cu_{y+1}O_{7+y-\delta}$,y= 1,2 and $Bi_2CaSr_2Cu_2O_{8-\delta}$ systems[61,62] is clearly a thrust area. The pace of results in PLD for HTS suggest that the technique is well suited for thin film R&D.

III.BACKSCATTERING ANALYSIS IN THIN FILM HTS.

One of the necessary conditions to fabricating good high temperature superconductors is stoichiometry. This is clearly evident when the pseudoternary Y_2O_3-BaO-CuO phase diagram is examined[63]. The oxide phase diagram shows the compound $Y_1Ba_2Cu_3O_x$ (123) to be stoichiometric in its metal components. This indicates that small deviations from 123 in the Y-Ba-Cu stoichiometry will result in the formation of $Y_1Ba_2Cu_3O_x$ and two additional non-superconducting phases.

In addition to the metal stoichiometry, oxygen content has been shown to be critical factor in the formation of the orthorhombic superconducting phase [64] and effects the temperature at which the superconducting transition occurs [65,66].

From the above discussion it is clear that knowledge of the composition of an HTS sample can be a great assistance in the processing and fabrication of this material as well as provide fundamental information for the proper interpretation of results obtained from various physical and transport property experiments.

Ion beam analysis has been successfully applyed to this problem using a variety of techniques. The most prominent of these techniques is Rutherford backscattering spectroscopy (RBS)[67]. However, tradition RBS is much more sensitive to elements with a high atomic number[68]. indicating that the Rutherford backscattered signal will be much greater for elements such as Ba, Y, and Cu relative to elements such O. Oxygen RBS analysis is further complicated by the experimental observation that He projectiles on oxygen start to show deviations from Rutherford scattering near 2.2 MeV [69], which limits the maximum energy, and ultimate film thickness at which RBS analysis can be performed on HTS films.

Several ion beam solutions to this problem have been presented in the literature which take advantage of a variety of non-Rutherford backscattering techniques. These techniques all enhance the oxygen backscattering signal[70-74].

Of the non-Rutherford techniques applied to HTS thin films, one of the easiest to apply utilizes high energy backscattering and the elastic resonance of 8.8 MeV He with O^{16} [72,73]. This procedure allows the experimentalist to obtain both good mass separation and the depth dependent composition information from all the components in the film from a single spectrum. The utility to oxygen analysis by working at this energy can be realized by examining the cross section data of Martin et al [73], Fig. 2. The

increased sensitivity to O^{16} at 8.8 MeV is about 25 times greater than that for Rutherford scattering.

To show the mass separation advantage of working with 8.8 MeV He, Fig. 3 contrasts spectra taken of an unannealed film on graphite substrates 8.8 and 3.05.MeV He. Both spectra were take at the same scattering geometry.

In Fig. 4, 8.8 MeV He backscattering data is presented from Y-Ba-Cu films which were prepared by electron beam coevaporation in a vacuum of approximately $5x10^{-8}$ torr. Films were simultaneously deposited onto both $SrTiO_3$ and graphite substrates. A Y capping layer, approximately 150 Å thick, was deposited prior to removing the samples from vacuum. This was done in an attempt to minimize atmospheric contamination during the step of taking the films out of vacuum and placing them in the O_2 annealing furnace.

The spectra in Fig. 4 are from films in their as-deposited state and allows us to examine the influence of the $SrTiO_3$ and C substrate signals on the Ba, Y, Cu, and O peaks. Clearly evident in this data is the the good mass separation between Ba, Y, and Cu as well as the enhanced O yield. This data indicates that the Ba, Y, Cu, and O distributions for both substrates are identical, including the small O peak which has formed in the thin Y capping layer. The step which appears at the low energy end of the Y peak and produces the background for the the Cu peak is backscattering from Sr in the $SrTiO_3$ substrate. The large step occurring in the vicinity of channel 275 corresponds to

backscattering from O in the $SrTiO_3$ substrate. The graphite substrate offers a clear advantage in peak separation, and allows rapid composition analysis of the as-deposited film by simply measuring the number of counts in the Ba, Y, Cu, and O peaks

To produce a superconductor out of the coevaporated Y-Ba-Cu films, an O_2 annealing sequence of 650 ^{o}C for 1hr, 750 ^{o}C for 1hr, 850 ^{o}C 1hr, 650 ^{o}C 1hr, followed by a slow cool to room temperature. Only films deposited on $SrTiO_3$ were annealed. High energy backscattering spectra from films on $SrTiO_3$ before and after O_2 annealing are presented in Fig. 4.

A comparison of the data presented in Fig. 5 clearly shows the effects of the O_2 annealing procedure. These data show that the heights of the Ba, Y, and Cu peaks have all undergone a reduction while the height of the O in the film has grown larger than the Ba height. In addition, the Ba, Y, Cu, and O film peaks have all become wider and the Sr and O substrates signals have undergone a corresponding shift to lower energy positions. Qualitatively, these results are consistent with the production of a thicker film which has a lower Ba, Y, and Cu atomic density and a higher O atomic density after annealing, relative to the as-deposited film.

To successfully analyze the thickness and composition of an HTS thin film with 8.8 MeV He backscattering requires the Y, Ba, Cu, and O cross section information obtained by

Martin et al [73] as well as stopping data Zigler et al [75]. Analysis of the annealed film gives a thickness of 8980 Å and a composition of $Y_{1.11}Ba_{2.00}Cu_{2.91}O_{7.04}$. Similar analysis on the unannealed film, using data from both the C and $SrTiO_3$ spectra (Fig. 4), gives us a film thickness of 7400 Å and a stoichiometry of $Y_{0.97}Ba_{2.00}Cu_{2.91}O_{0.21}$. The above calculations assumed atomic densities of 4.17 and $7.69x10^{22}$ atoms/cm^2 for the unannealed and annealed films respectively.

A careful comparison of the metal stoichiometries before and after annealing indicates that the number of Ba and Cu atoms has remained constant while the number of Y atoms has increased by 14 %. This increase in the calculated Y stoichiometry results directly from a greater than expected increase in the height of the Y backscattered peak. Since the Ba and Cu data suggest that mass was conserved during the annealing procedure, the increased Y yield indicates that backscattering from near surface Sr has occurred. This may be the result of Sr diffusion into the surface film and/or the formation of holes in the Y-Ba-Cu layer during annealing. If we assuming that the increase is the result of Sr contamination, the estimated Sr concentration in the film would be approximately 1.1 atomic %.

The transport quality of the annealed film, as determined by four point probe measurements, is presented in Fig. 6. The film possess a superconducting transition which

is 8 K wide, reaching zero resistance at 79 K. The normal state resistance shows mild metallic behavior but does not drop very quickly with decreasing temperature as would be expected from a high quality superconducting film. The moderate transport quality of this film is consistent with the backscattering data which has shown the metal stoichiometry to be less than perfect and suggests possible Sr contamination. Deviations from a 1:2:3 atomic ratio for Y:Ba:Cu are expected to result in the formation of other, non-superconducting, phases. As in other ceramics, second phases in the $YBa_2Cu_3O_7$ superconductor will most likely accumulate at grain boundaries, producing Josephson junctions which tend to spread the superconducting transition out [8,76]. The addition of Sr has similar detrimental effects on HTS materials, studies have clearly demonstrated that T_c decreases with increasing Sr content [77,78].

5. SUMMARY

Several physical vapor deposition techniques and their application in the production of thin film high temperature superconductors have been discussed. While the method of deposition differs for electron beam evaporation, pulsed laser deposition, and sputtering, each technique offers specific coating advantages and all are capable of producing high quality superconducting films. The superconducting quality of a film produced by any of these techniques is

highly dependent on composition. In this area, ion beam analysis has been very successful in providing depth sensitive information. In particular, 8.8 MeV He backscattering supplies information from both the metal and oxygen components in a single spectra.

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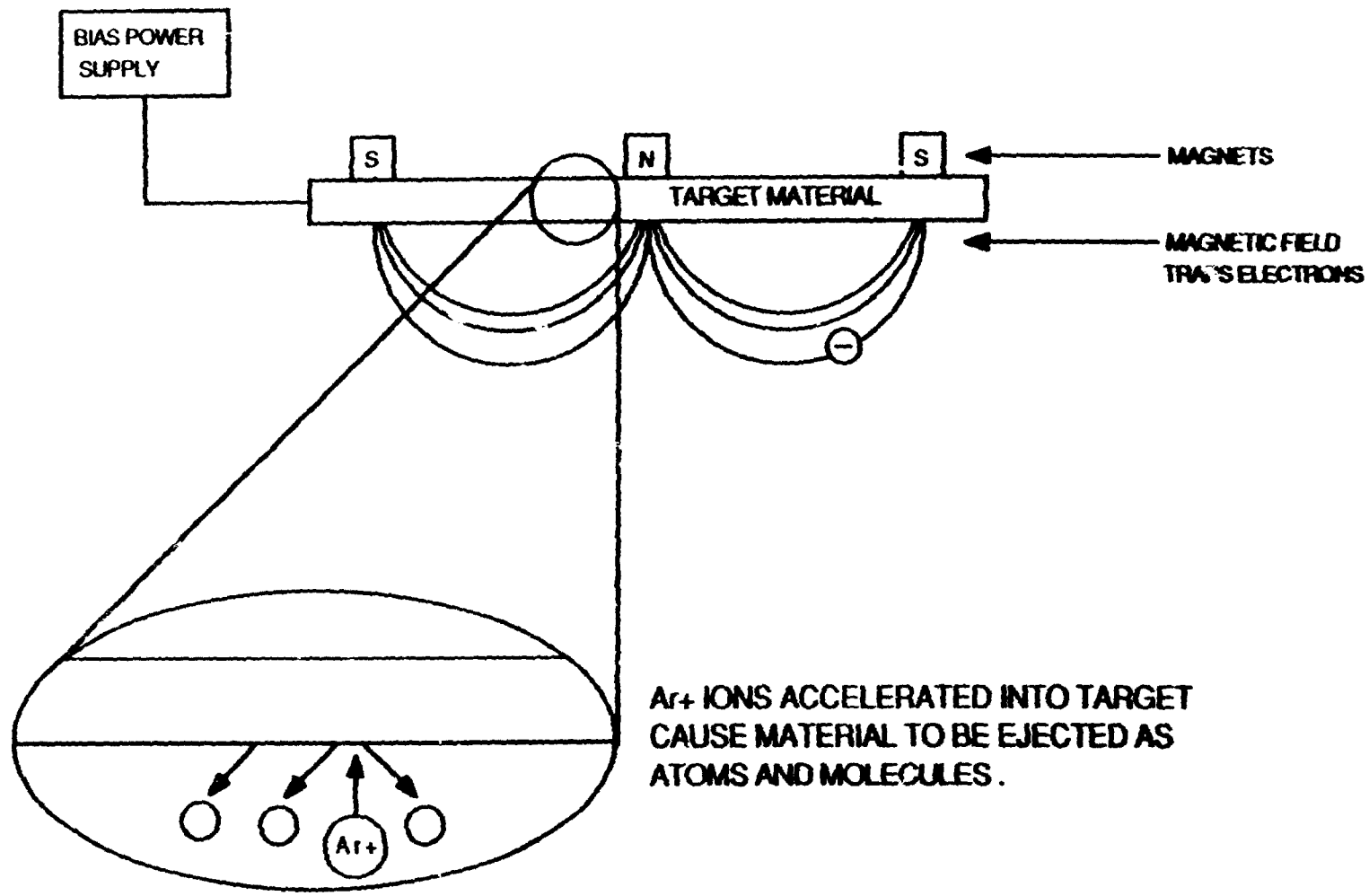
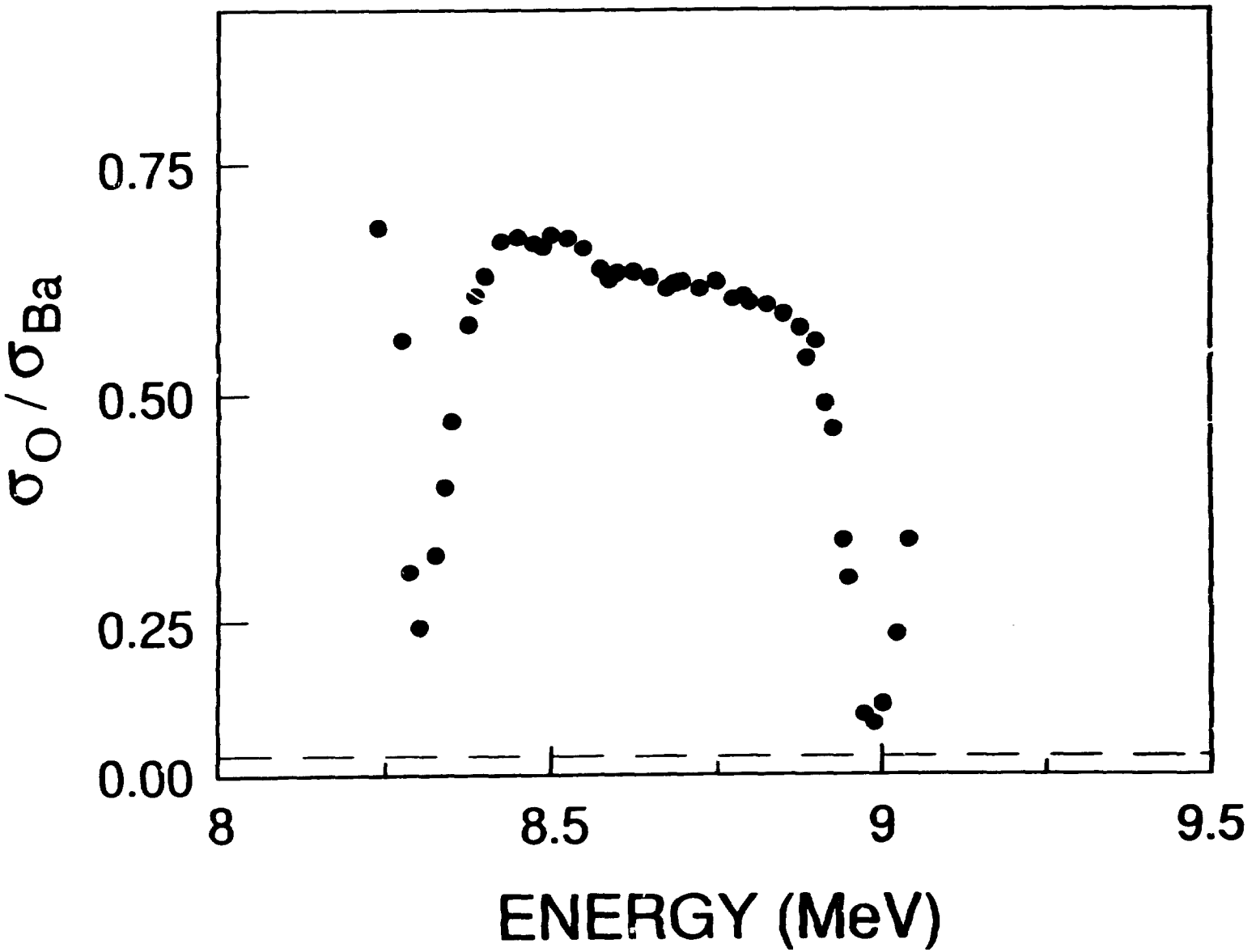


Ar+ IONS ACCELERATED INTO TARGET CAUSE MATERIAL TO BE EJECTED AS ATOMS AND MOLECULES .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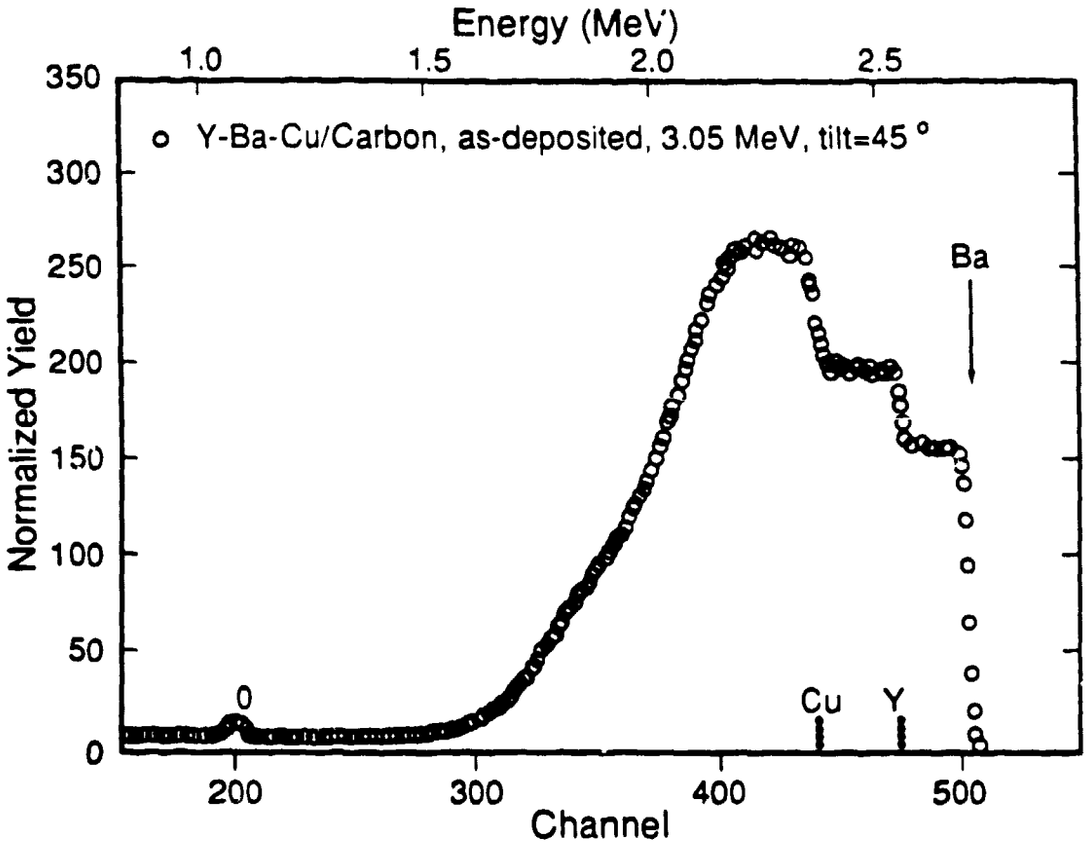
0.50
0.25
0.00
σ_O / σ_{Ba}
8
8.5
9
9.5
ENERGY (MeV)


Energy (MeV)
1.0
1.5
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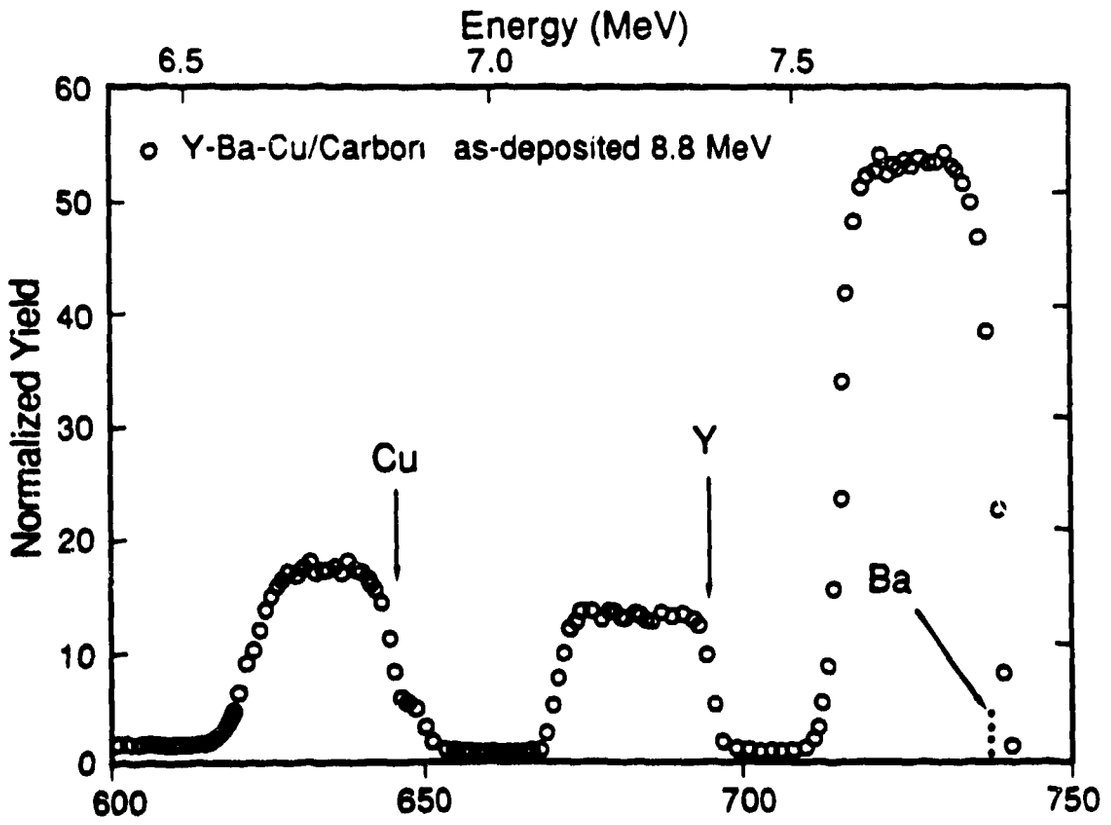
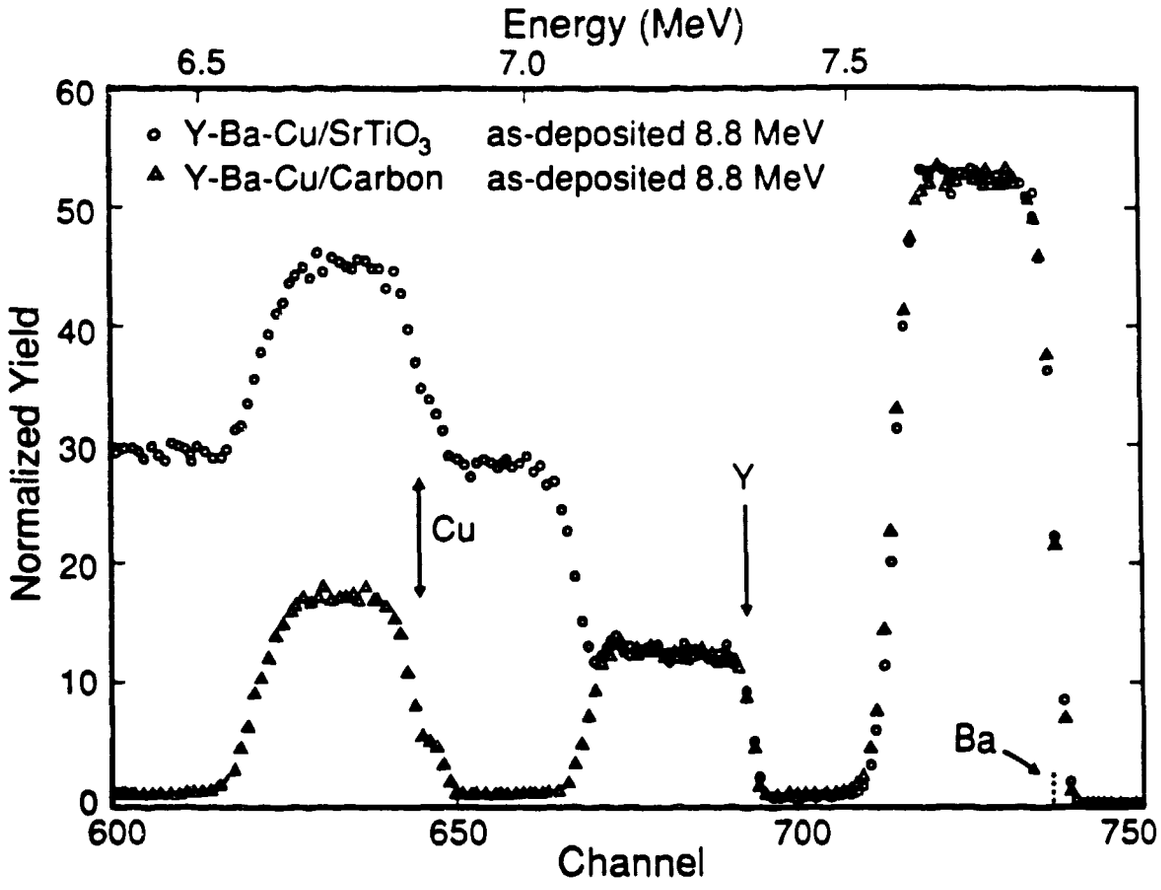
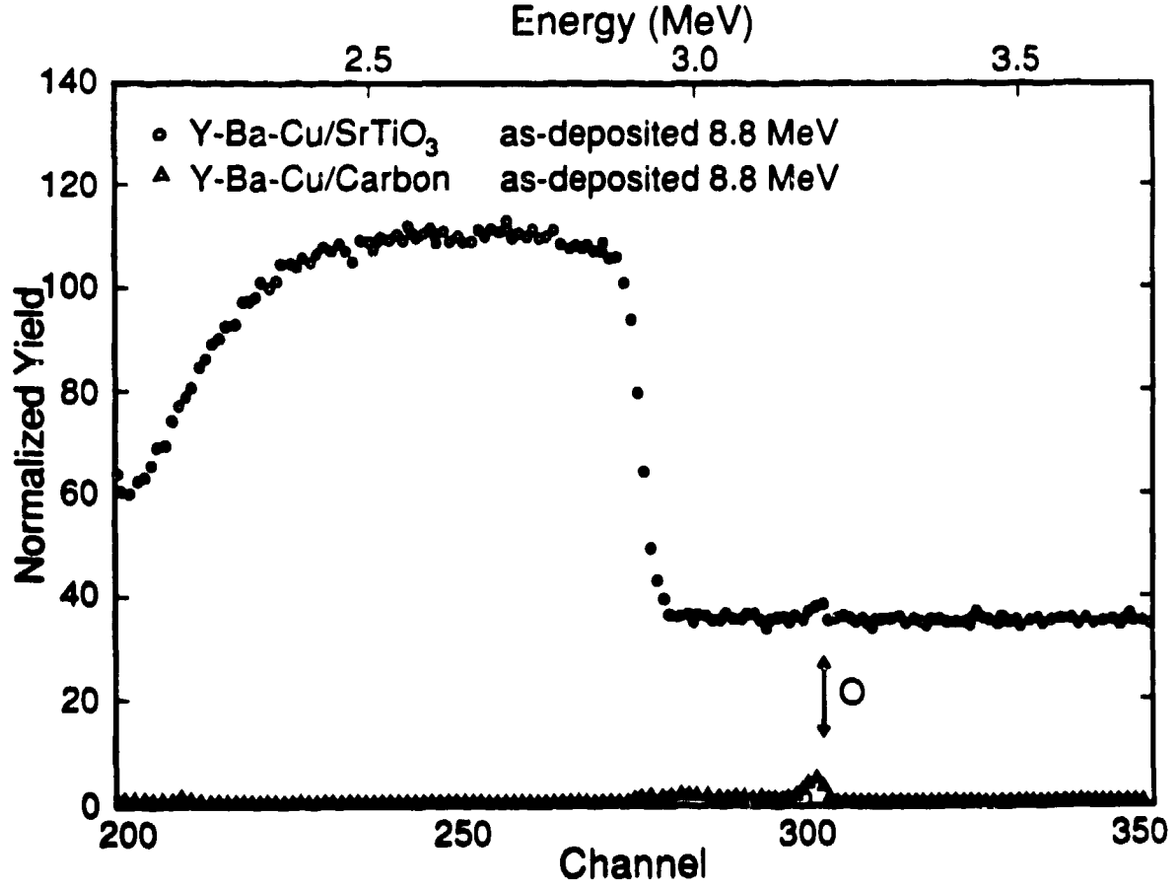
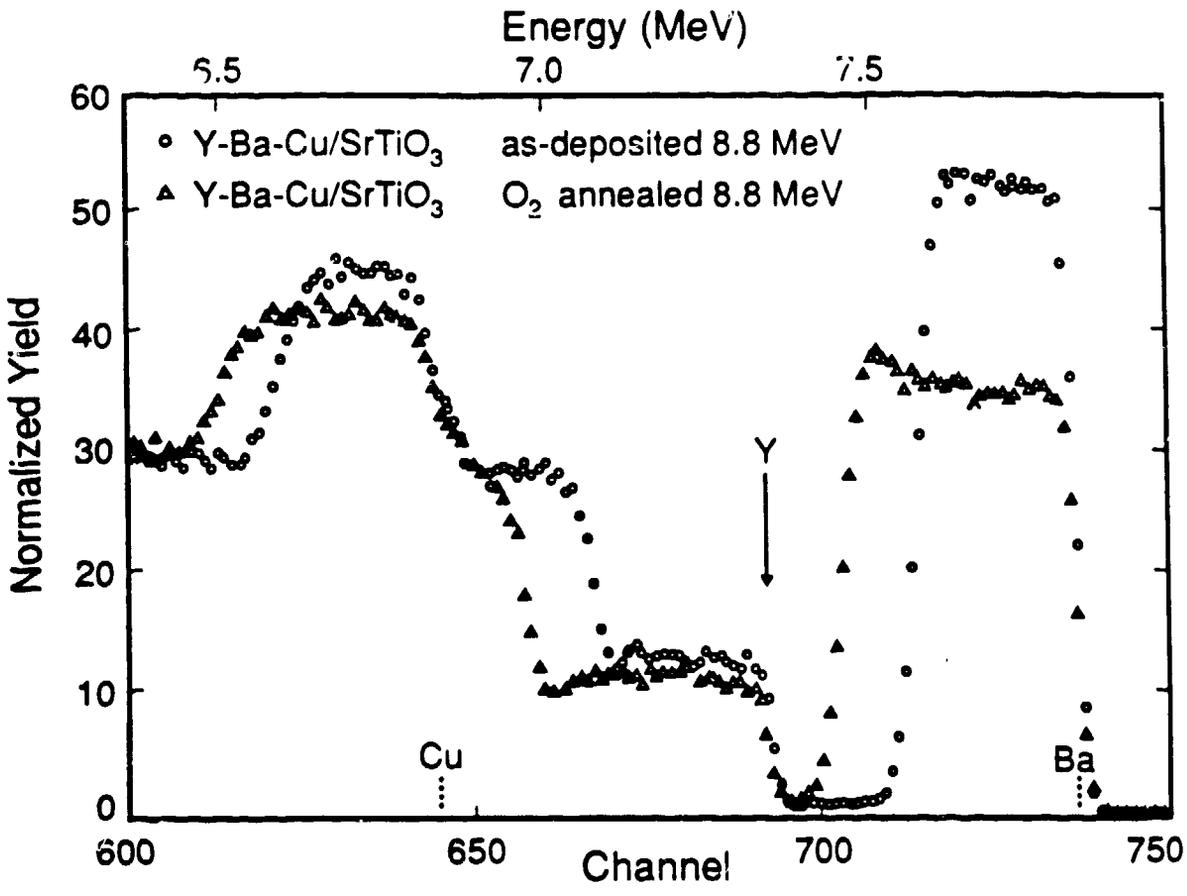
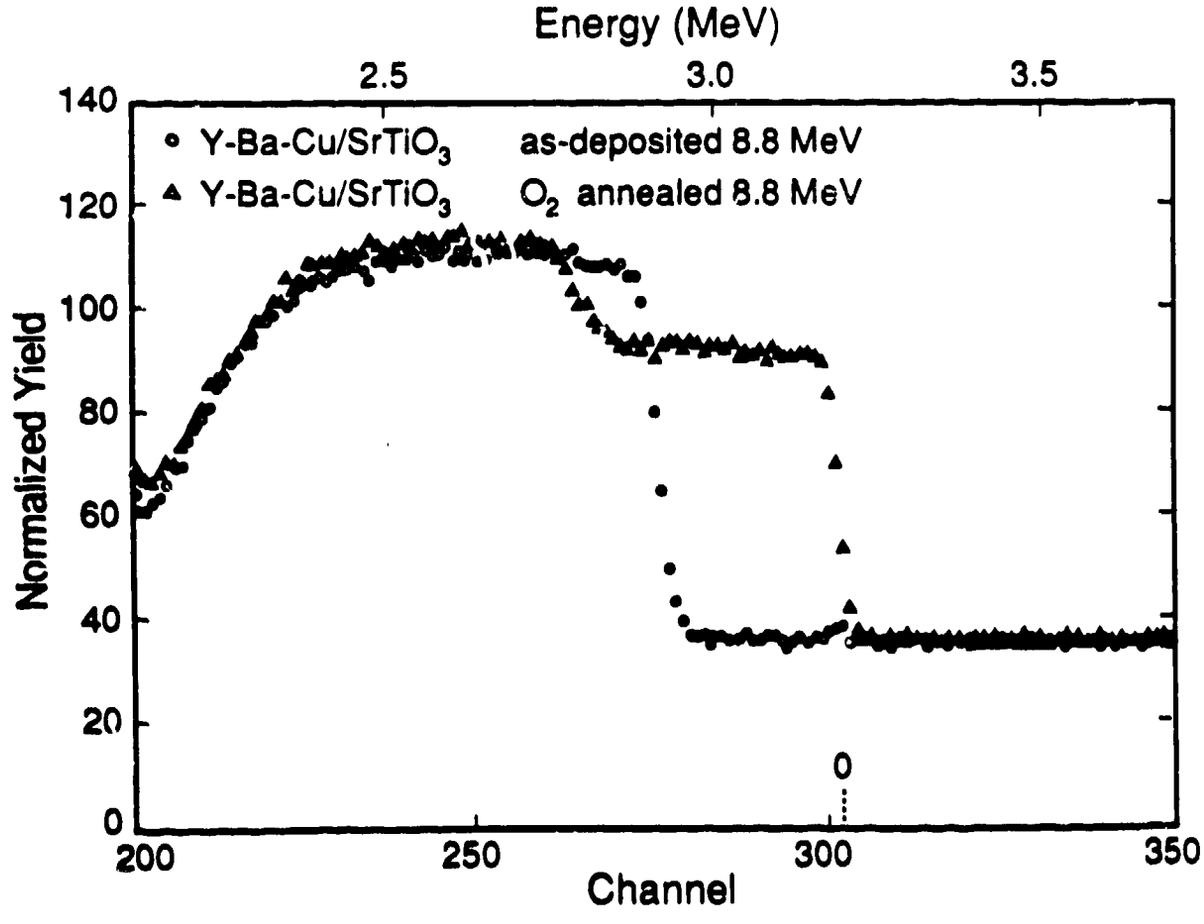
Normalized Yield
Ba
O
Cu
Y
Channel

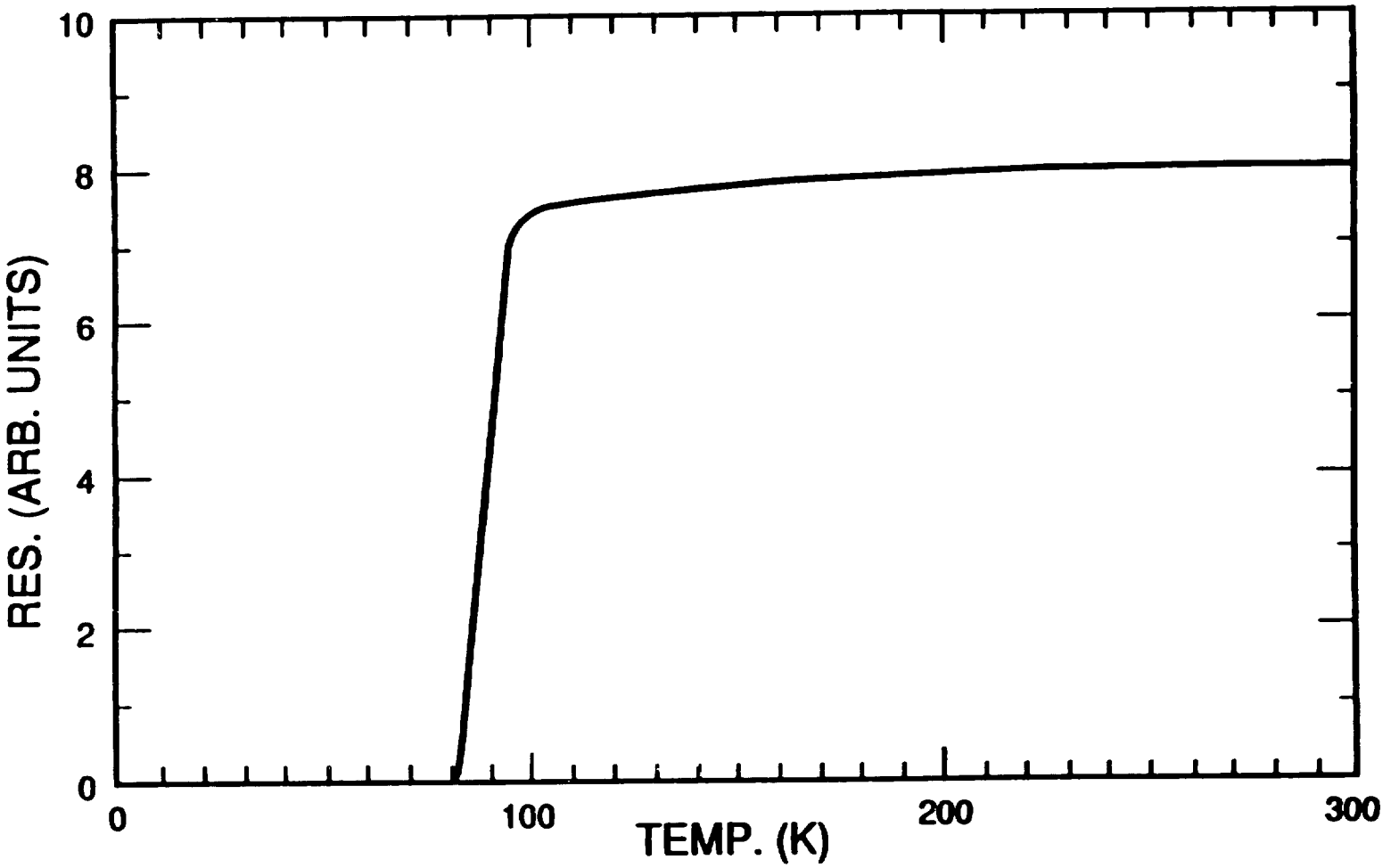

Energy (MeV)
6.5
7.0
7.5
Y-Ba-Cu/Carbon as-deposited 8.8 MeV
Normalized Yield
Cu
Y
Ba


Energy (MeV)
6.5
7.0
7.5
Y-Ba-Cu/SrTiO3 as-deposited 8.8 MeV
Y-Ba-Cu/Carbon as-deposited 8.8 MeV
Normalized Yield
Cu
Y
Ba
Channel
600
650
700
750


Energy (MeV)
2.5
3.0
3.5
Y-Ba-Cu/SrTiO3 as-deposited 8.8 MeV
Y-Ba-Cu/Carbon as-deposited 8.8 MeV
Normalized Yield
O
Channel
200
250
300
350


Energy (MeV)
6.5
7.0
7.5
Y-Ba-Cu/SrTiO3 as-deposited 8.8 MeV
Y-Ba-Cu/SrTiO3 O2 annealed 8.8 MeV
Normalized Yield
Y
Cu
Ba
Channel
600
650
700
750
0
10
20
30
40
50
60


Energy (MeV)
2.5
3.0
3.5
Y-Ba-Cu/SrTiO3 as-deposited 8.8 MeV
Y-Ba-Cu/SrTiO3 O2 annealed 8.8 MeV
Normalized Yield
O
Channel
200
250
300
350
0
20
40
60
80
100
120
140


10
8
6
4
2
0
RES. (ARB. UNITS)
0
100
200
300
TEMP. (K)