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TITLE ION BEAM ANALYSIS AND MODIFICATION OF THIN FILM HIGH TEMPERATURE SUPERCONDUCTORS

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ION BEAM ANALYSIS AND MODIFICATION OF THIN FILM HIGH TEMPERATURE SUPERCONDUCTORS

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1. INTRODUCTION

The application of ion beams to ceramic processing, as the rest of these proceedings has shown, is a very powerful technique. In particular ion beams have been shown to be very effective at producing new and novel material properties through their rather violent interactions with the component atoms of the ceramics. However, in all these cases ions are used to process and produce changes in ceramics, changes which must be monitored by some other techniques. The purpose of the present paper is to show that in addition to modifying materials, ion beams can be used in a more gentle but very powerful way to explore what happens to a ceramic thin film as a function processing. The discussions which will follow will be concerned exclusively with the new and exciting class of ceramic, the high temperature superconductor (HTS). We will discuss the application of various ion beam backscattering techniques, as well as examine the use of ion implantation in the processing of these materials.

2. BACKSCATTERING SPECTROSCOPY

In this section we will review some of the basic physical concepts involved in backscattering spectroscopy. The reader should note that this will only be a brief review and that a more complete treatment may be found in References 1-3. The concepts that will be discussed are the kinematic factor K , scattering cross section σ , and stopping cross section $[S]$. For a discussion on straggling the reader is referred to chapter 1 in reference 1 and chapter 2 in reference 2.

2.1. Kinematic factor K

The perception of mass in a backscattering spectra results from the energy transferred to a target atom by the projectile atom. The kinematic factor K , which is a measure of this, is defined as the ratio of the projectile energy after an elastic collision to that before the collision, and can be derived by applying the principles of conservation of energy and momentum to an elastic collision between two masses M_1 and M_2 . Applying these principles, we have for M_1 scattering from M_2 (4)

$$K \equiv E_1/E_0 = \left[\frac{(M_2^2 - M_1^2 \sin^2 \theta)^{1/2} + M_1 \cos \theta}{M_2 + M_1} \right]^2 \quad (1)$$

From equation 1 we see that the kinematic factor depends only on the projectile and target masses and the scattering angle θ .

In many applications the target will contain more than one type of atom. The ability to resolve backscattering from atoms with similar masses requires that the mass difference, ΔM , produce a large difference in the energy of the backscattered projectile ΔE . In the vicinity of $\theta = 180^\circ$ and for $M_2 \gg M_1$, which are most often the case, it can be shown that (4)

$$\Delta E = E_0(4 - \delta^2)(M_1/M_2^2)\Delta M \quad (2)$$

where $\delta = \pi - \theta$ in units of radians of arc.

Fixing the scattering geometry and ΔM , equation 2 indicates that good mass resolution, large ΔE , can be obtained by either increasing the primary energy E_0 or increasing the mass of the projectile M_1 . However, in applying the later situation some caution is required since a backscattering signal will not be produced from target atoms where M_1 is greater than M_2 .

2.2. Stopping cross section [ϵ]

As a highly energetic projectile travels through a solid it will loss energy primarily through electronic stopping. This energy loss arises from the viscous-like drag that the projectile experiences as it undergoes collisions with the electrons of the target atoms. The amount of energy lost in traversing the target is proportional to the distance traveled and gives the perception of depth in a backscattering spectra.

In Fig. 1 the energy loss components for a projectile scattered from a depth t are presented. For a projectile starting with an energy E_0 , the initial energy loss will occur on the inward path, ΔE_{in} . At a depth t the projectiles energy will have been reduced to E_t . If at this point the projectile suffers a collision with a target atom it will loss energy ΔE_c . The final energy loss occurs on the outward path, ΔE_{out} . The ultimate projectile energy which reaches the detector will be $E_1 = E_0 - (\Delta E_{in} + \Delta E_c + \Delta E_{out}) = KE_t - \Delta E_{out}$. Alternately, if the projectile has a backscattering collision at the surface with out penetrating the solid, the only energy loss will be due to kinematics and the detected projectile energy will be KE_0 .

The total energy difference ΔE between projectiles scattered at the surface and at some depth t is related to the scattering depth through the relationship (4)

$$\begin{aligned}\Delta E &= KE_0 - E_1 = K\Delta E_{in} - \Delta E_{out} \\ &= t[S] = tN[\epsilon]\end{aligned}\quad (3)$$

where $[S]$ is the energy loss factor, $[\epsilon]$ is the stopping power factor, and N is the the atomic density. The energy loss factor can be defined from Fig. 1 and equation 3 as (4):

$$[S] = \left[\frac{K}{\cos\theta_1} \frac{dE}{dx} \Big|_{in} + \frac{1}{\cos\theta_2} \frac{dE}{dx} \Big|_{out} \right] \quad (4)$$

and the stopping cross section factor is defined as (4):

$$[\epsilon] = \left[\frac{K}{\cos\theta_1} \epsilon_{in} + \frac{1}{\cos\theta_2} \epsilon_{out} \right] \quad (5)$$

where ϵ is the stopping cross section and is defined as:

$$\epsilon = \frac{1}{N} \frac{dE}{dx} \quad (6)$$

For situations where the target is composed of more than one element the energy loss is assumed to be equal to the sum of the loss to the constituent elements, weighted by their abundance in the compound. This postulate is known as Bragg's rule (5). Thus, the stopping cross section for a mixture or molecule with the composition $A_m B_n$ is given by

$$\epsilon(A_m B_n) = m\epsilon(A) + n\epsilon(B) \quad (7)$$

where $\epsilon(A)$ and $\epsilon(B)$ are the stopping cross sections for elements A and B.

A schematic representation of the backscattering process from an idealized free standing compound film is presented in Fig. 2. Similar energy losses will occur in this situation as those depicted in Fig. 1 with the added complexity of kinematic losses from two elements. The energy loss components that arise from a projectile traversing a thickness t and then being backscattered out for the A component are:

$$\Delta E_{in} = \frac{t}{\cos\theta_1} \frac{dE}{dx} \Big|_{in}^{A_m B_n} \quad (8)$$

$$E_t = E_o - \Delta E_{in} \quad (9)$$

$$\Delta E_c(A) = E_t - K_A E_t \quad (10)$$

$$\Delta E_{out}(A) = \frac{t}{\cos \theta_2} \frac{dE}{dx} \bigg|_{A_m B_n}^{out, A} \quad (11)$$

Similar expressions also exist for the P component of our compound target.

The relationship between energy width, $K_A E_o - E_1(A)$, and the film thickness for the two component target is

$$\Delta E_A = K_A E_o - E_1(A) = t N^{AB} [\epsilon(A_m B_n)]_A \quad (12)$$

where N^{AB} is the number of molecules $A_m B_n$ per unit volume and $[\epsilon(A_m B_n)]_A$ is the stopping cross section factor for a projectile scattered from element A while traversing the medium $A_m B_n$ and has the form

$$[\epsilon(A_m B_n)]_A = \left[\frac{K_A}{\cos \theta_1} \epsilon(A_m B_n)_{in} + \frac{1}{\cos \theta_2} \epsilon(A_m B_n)_{out, A} \right] \quad (13)$$

A similar expression to equation 12 can also be written for the energy width of the B portion of the spectra using the cross section factor for scattering from element B, which has the same form as equation 13. The energy width of a compound can also be expressed in terms of a compound energy loss factor, $[S(A_m B_n)]_A$, which can be constructed from equations 8 through 12.

2.3. Scattering cross section σ

In all of the discussion given above we assumed that collisions would occur between the projectile and a target atom which would result in a backscattering event. The likelihood of such an occurrence leads to the concept of the scattering cross section and the capability of performing quantitative composition analysis.

For the experimental conditions in which a uniform beam of projectiles impinges at normal incidence on a uniform target of thickness t, the total number A of backscattered and detected particles is given by

$$A = \sigma N t Q \Omega \quad (14)$$

where σ is the scattering cross section, N is the atomic density which makes Nt the number of target atoms per unit area, Q is the number of incident particles, and Ω is the detector solid angle.

Equation 14 shows that when Ω , Q , and σ are known the number of atoms per unit area, Nt , can be calculated. Both Ω and Q can be experimentally measured, and if the repulsion between the projectile and target atom is coulombic, the scattering is Rutherford and σ is easily calculated (4). At higher energies the probability of the projectile interacting with the nucleus of the target atom increases resulting in an interaction which is not strictly coulombic. In such situations large deviations from Rutherford scattering can occur and experimentally determined values of σ are required in equation 14.

In the hypothetical situation of the free standing alloy film with composition A_mB_n , depicted in FIG. 2, the total number of counts from element A is the area A_A . Assuming that only small changes in the projectile energy occur on the inward and outward paths (surface energy approximation), the area A_A can be described by (4)

$$A_A = \Omega Q \sigma_A(E_0) mN(A_mB_n) t / \cos\theta_1 \quad (15)$$

A similar expression can also be written for A_B using the atomic density of B atoms $nN(A_mB_n)$ and the surface energy scattering cross section for B atoms $\sigma_B(E_0)$. The ratio of atomic densities is then given by

$$m/n = [A_A/\sigma_A(E_0)]/[A_B/\sigma_B(E_0)] \quad (16)$$

In some situation it may not be possible to resolve the full peak of a particular element in a backscattering spectra. In this case equation 16 which utilizes a ratio of peak areas can not be used for composition analysis. However, composition analysis may be possible by comparing the ratios of the surface counts of the backscattering yield. In an analogous expression to equation 15, the backscattering yield at the surface for element A in Fig. 2 is given by

$$H_{A,0} = \Omega Q \sigma_A(E_0) mN(A_mB_n) [L_{A,0} / \cos\theta_1] \quad (17)$$

where $L_{A,0}$ is the thickness of a slab of the target at the surface and is defined by the energy width E of a channel in the detecting system. Projectiles scattered from within $L_{A,0}$ will have a depth scale at the surface given by

$$E = T_{A,0} N^{AB} [\epsilon(A_mB_n)]_A \quad (18)$$

where N^{AB} and $[\epsilon(A_mB_n)]_A$ have the same meaning as in equation 12. A similar set of equations can also be written for $H_{B,0}$ using $L_{B,0}$, $\sigma_B(E_0)$, and $nN(A_mB_n)$. Combining these equations, the ratio of atomic densities can be written as

$$m/n = \frac{H_{A,0} \sigma_B(E_0) [\epsilon(A_mB_n)]_A}{H_{B,0} \sigma_A(E_0) [\epsilon(A_mB_n)]_B} \quad (19)$$

3. 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. The oxide phase diagram presented in Fig. 3 was determined at 950 C (6) and 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. If the stoichiometry is known the fraction of the 123 superconducting phase expected under equilibrium processing conditions can be calculated from the phase diagram by applying the lever rule (7).

In addition to the metal stoichiometry, oxygen content has been shown to be critical factor in the formation of the orthorhombic superconducting phase (8) and effects the temperature at which the superconducting transition occurs (9,10).

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.

3.1. Rutherford backscattering spectroscopy

We have already established in section 2 that backscattering spectroscopy is quite capable of supplying depth sensitive composition information in the near surface region, making it an attractive candidate for examining HTS thin films. However, traditional Rutherford backscattering spectroscopy (RBS) is much more sensitive to elements with a high atomic number. This results directly from the Rutherford scattering cross section, σ_R , which is proportional to $(Z_1Z_2/E)^2$, where Z_1 is the atomic number of the projectile, Z_2 is the atomic number of the target atom, and E is the energy of the projectile. Examining equation 15 in light of this we see that the Rutherford backscattered signal will be much greater for high Z elements such as Ba, Y, and Cu relative to a low Z element such as 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 (11), Fig. 4, thus limiting the maximum energy at which RBS analysis can be performed.

The major negative side-effects associated with performing backscattering experiments in the low energy regime where the O cross section is still Rutherford are the limitations imposed on film thickness and composition analysis. As indicated by equations 3 and 12, the backscattering peak width ΔE is related to the film

thickness t . From equation 2, we also observe that mass resolution decreases with decreasing projectile energy. Together these points suggest that decreasing projectile energy and increasing film thickness will ultimately lead to signal overlap between the high Z components in our superconducting film and making depth dependent composition analysis difficult. This point is demonstrated in Fig. 5 with a RBS spectra that was simulated with the program RUMP (11). The simulation was carried out assuming 2 MeV He projectiles and a 4000 Å thick free standing $\text{YBa}_2\text{Cu}_3\text{O}_7$ film. Also shown are the component RBS signals from Ba, Y, Cu, and O, which make up the total spectra. While this data ignores the additional spectra complexities which will be present when a substrate is included in the simulation, it clearly demonstrates how complicated and convoluted the RBS data can be under the given conditions.

Two solutions to the problem of Ba, Y, and Cu peak overlap exist and are presented in the RUMP backscattering simulations of Fig. 6. A possible but not very practical solution would be to only work at energies where O is Rutherford and limit the thickness of the film to avoid peak overlap. The low energy spectra, which was simulated assuming 2 MeV He, suggests a maximum $\text{YBa}_2\text{Cu}_3\text{O}_7$ thickness of 1200 Å in this case. An alternate approach would be to take advantage of the improved mass resolution (equation 2) and the lower stopping cross section that occur at higher He energies. The high energy portion of Fig. 6 shows that complete peak separation in a 4000 Å film is possible at 4 MeV. This latter data suggests that a complete composition analysis may be possible by acquiring backscattering spectra from the same film at two different energies; A high energy spectrum to determine the Ba, Y, and Cu composition profiles and a lower energy (≤ 2.2 MeV) spectrum to determine the oxygen depth profile.

While the two step depth profiling solution presented above is possible in theory, the low yield of backscattered O at energies ≤ 2.2 MeV and the incorporation of backscattering signals from the substrate make accurate O analysis difficult to carry out in practice. In Fig. 7 a RUMP simulated RBS spectra is presented for 2 MeV He on a 4000 Å thick $\text{YBa}_2\text{Cu}_3\text{O}_7$ film on a SrTiO_3 substrate. Also shown are the individual backscattering signals from Ba, Y, Cu, and O in the surface film and signals from Sr, Ti, and O in the substrate. Examining Fig. 7 we see that the small O signal from the superconductor sits on top of a large background which is made up of the backscattering signals from the substrate components Sr, and Ti. It should be pointed out that the simulated spectra is highly idealized and does not contain any of the background noise which is present in reality. The combination of the low O yield (counting statistics) and the ability to properly subtract the background from the O signal greatly limits the accuracy of O composition analysis.

3.3. Non-Rutherford backscattering

3.3.1 Elastic resonance of 3.045 MeV He with O^{16}

Solutions to the limitation imposed on performing O composition analysis in the Rutherford scattering regime have been offered by several research groups (13-16). One suggested solution is to take advantage of the enhanced, non-Rutherford, elastic resonant O^{16} cross section which occurs for incident 3.045 MeV He. This resonance cross section was first described by Cameron (17) and has recently been applied to oxide superconductors by Blanpain et al (16). The enhancement in the measured cross section relative to the Rutherford cross section at 3.045 MeV can be observed in Fig. 4 (11), and has been estimated to produce an O yield which is 30 times greater than expected for Rutherford scattering (18). Backscattering data presented in Fig. 8 clearly shows the effect of this enhancement on the O backscattering yield from a 3050 Å thick Ta_2O_3 film with 3.097 MeV He relative to 2.030 MeV He (16). However, since the resonant scattering from O^{16} only occurs over a narrow energy window of 11 keV (16), concentration depth profiles require that several backscattering spectra be taken at slightly increasing energies which probe the O content at greater depths in the sample. An example of such a depth probe is given in Fig. 9 for a Zirconia sample (from Ref 16).

The results obtained by Blanpain et al (16) from a 3.086 MeV He backscattering experiment on a Y-Ba-Cu-O/Zirconia film/substrate combination are presented in Fig. 10. Oxygen concentration and depth resolution under these operating conditions are quit good. Concentration accuracy is proposed to be less than 5%. The O depth resolution, ignoring straggling effects, will be primarily dependent on the resonance energy width, 11 keV. Assuming an energy loss of $dE/dx = 36$ eV/Å in $YBa_2Cu_3O_7$ at 3.1 MeV suggest a depth resolution of approximately 300 Å at normal incidence. However, accurate O depth profiling requires the use of a calibration sample, which is ran in concurrence with runs of the unknown (16). Also, as the high energy portion of Fig. 10 shows, additional spectra at higher He energies will be necessary to achieve complete separation of the Y, Ba, and Cu peaks.

3.3.2. Elastic resonance of 2.5 MeV protons with O^{16}

While the use of the non-Rutherford elastic resonant O^{16} cross section which occurs for incident 3.045 MeV He has clear advantages, it does require the acquisition of multiple spectra at different energies to obtain the O composition as a function of depth. In a recent article by Rauhala et al (13) a alternate backscattering technique has been described which also provides composition analysis in $YBa_2Cu_3O_7$ material and does not require multiple spectra to determine the oxygen composition profile. The approach taken by these researchers was to combine the elastic non-Rutherford scattering of protons and the Rutherford scattering of He to examine the composition of O and the

higher Z elements respectively. This technique utilizes an enhanced O scattering cross section which is relatively flat over a wide range of proton energies. The C cross section occurs for proton projectiles of energy ≤ 2.5 MeV, and is enhanced by a factor of 5 relative to the Rutherford cross section (19).

Barbour et al (14) have also taken advantage of this cross section and backscattering data from their work on bulk $\text{YBa}_2\text{Cu}_3\text{O}_7$ is present in Fig. 11. The inset in Fig. 11 shows the behavior of $\text{O}^{16}+\text{p}$ cross section as a function of energy. Examining both the cross section and energy loss factor for HTS material suggest that an O analysis range of 4 μm and a depth resolution of 2000 $\text{\AA}$ is possible for 2.5 MeV incident protons (14). While this technique is relatively simple to apply to oxygen analysis, the loss of depth and mass resolution that is observed in the high energy portion of the spectrum clearly shows that accurate composition analysis is not possible for Ba, Y, and Cu. As proposed by Rauhala et al, additional experiments must also be performed with He projectiles to obtain the composition information for the higher Z elements.

3.3.3. High energy backscattering and the elastic resonance of 8.8 MeV He with O^{16} . Up to this point we have primarily concerned our selves with backscattering experiments at relatively low energies where complete composition analysis of a $\text{YBa}_2\text{Cu}_3\text{O}_7$ film required collecting multiple data, changes in projectile and/or incident energy. As noted previously (Fig. 6), going to higher energies offers the advantage of producing greater mass separation between the high Z components. However, it is well know that the likelihood for scattering to deviate from a Rutherford nature increases with increasing projectile energy. Deviations are expected when the projectile velocity is large enough to allow it to penetrate deep into the the orbitals of the atomic electrons and interact with the nucleus of the target atom. Mayer and Feldman have shown that the onset energy for deviation from Rutherford scattering to be roughly proportional to $(Z_1 Z_2 / A^{1/3})$ (20), where A is the mass number of the target atom and Z_1 and Z_2 are atomic numbers of the projectile and target atom respectively. Thus, if any sense is to be made of the information obtained from higher energy backscattering experiments, it will first be necessary to know the behavior of the scattering cross sections for all elements involved.

In the best of all possible situations, one would ideally like to obtain both good mass separation and the depth dependent composition information from all the components in the film from a single spectrum. Recently, Barbour et al showed that this could be achieved by performing backscattering experiments with 8.7 MeV He particles (14). The utility to oxygen analysis by working at this energy can be realized by examining the cross section data of Hunt et al (21), Fig. 12. The data in

Fig. 12 shows the high energy behavior of the O^{16} scattering cross section as a function of incident He (α -particle) energy for a variety of scattering geometries. This data indicates that as the geometry becomes progressively more backscattering (increasing $\theta_{C.M.}$), a very broad plateau appears in the cross section which is centered near 8.6 MeV.

A typical backscattering spectrum, taken from the work of Martin et al (15), for 8.8 MeV He incident on a 7700 Å thick film of $YBa_2Cu_3O_{7-x}$ sitting on a $SrTiO_3$ substrate is presented in Fig. 13. Indicated in the spectrum are the surface energies for Ba, Y, Cu, and O. The unlabeled step edges which appear at 2.80, 5.96, and 7.00 MeV correspond respectively to sub-surface O, Ti, and Sr in the substrate. Clearly evident in this data is the good mass separation between Ba, Y, and Cu as well as the large O yield from the superconducting film. However as noted above, the absolute accuracy of determining the film composition from such a spectrum depends on how well the scattering cross sections are known.

To determine how the scattering cross sections behave as a function of He energy, Martin and co-workers used a thin film standard technique to measure the ratio cross section for several HTS elements. The production of the standards consisted of electron beam coevaporating a Y-Ba-Cu film onto SiO_2 which was capped with a thin layer of Ti, and evaporating Ba in the presence of O_2 onto a Ti coated graphite substrate and then capping with another layer of Ti. The substrates were chosen in each case because of their low kinematic factors. The Ti layers were employed to minimize environmental contamination and aid in the adhesion to the graphite. For each standard the film thickness was chosen to ensure elemental peak separation in a 2 MeV RBS spectrum. The RBS data for these two standards at 2 MeV is presented in Fig. 14 (22). Performing peak integration on this data and applying equation 16, the composition of Y and Cu relative to Ba and the composition of O to Ba can be determined to within a few percent. Once the ratio of m/n is determined, values for σ_A/σ_B at various energies can be measured by integrating the elemental peaks at the energy in question and again applying equation 16.

The results of this procedure for He scattering from Ba and O in the energy range 8.2 and 9.1 with a scattering angle of 166° is presented in Fig. 15 (from Ref 15). The dashed line in the figure represents the ratio of the cross sections assuming Rutherford scattering. The increased sensitivity to O^{16} at 8.8 MeV is about 25 times greater than that for Rutherford scattering. The statistical uncertainty in measuring this data was determined to be less than 2 %.

In Fig. 16 measured cross section ratios for Cu/Ba and Y/Ba are presented over the energy range 2 to 9 MeV (15). Again the dashed line represents the Rutherford cross section ratios for a scattering angle of 166° . Assuming the scattering cross section for Ba to remain Rutherford up to 9 MeV (large Z) (14,15), this data

indicates that the Cu and Y cross section start to deviate from a Rutherford nature near 6.5 and 8.0 MeV respectively.

4. APPLICATIONS OF HIGH ENERGY BACKSCATTERING TO THIN FILM PROCESSING AND ION IMPLANTATION OF HTS.

To successfully analyze the thickness and composition of an HTS thin film with 8.8 MeV He backscattering requires the cross section information presented in Figs. 15 and 16 as well as stopping data. Zigler et al (24) have developed a reliable, consistent parameterization of stopping data for essentially all elements. In Fig. 17, stopping data calculated by the program TRIM (24) is presented for the case of He projectiles on a Y-Ba-Cu-O target in the energy range 10 keV to 10 MeV. This data was calculated assuming a target atomic composition of 7 % Y, 15 % Ba, 23 % Cu and 54 % O, and a mass density of 6.54 g/cm³.

4.1. Thin Film Processing

In Fig. 18, 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 5×10^{-8} torr (22). Films were simultaneously deposited onto both SrTiO₃ 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 O₂ annealing furnace.

The spectra in Fig. 18 are from films in their as-deposited state and allows us to examine the influence of the SrTiO₃ and C substrate signals on the Ba, Y, Cu, and O peaks. 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 Cu peak is backscattering from Sr in the SrTiO₃ substrate. The large step occurring in the vicinity of channel 275 corresponds to backscattering from O in the SrTiO₃ 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 O₂ annealing sequence of 650 °C for 1hr, 750 °C for 1hr, 850 °C 1hr, 650 °C 1hr, followed by a slow cool to room temperature. Only films deposited on SrTiO₃ were annealed. The graphite substrate samples which are very effective for composition analysis of the as-deposited film are not very practical for the actual formation of the superconductor. Graphite is readily converted into CO₂ under the annealing sequence described above. High energy backscattering spectra from films on SrTiO₃ before and after O₂ annealing are presented in Fig. 19.

A comparison of the two spectra in Fig. 19 clearly shows the effects of the O₂ annealing procedure. Examining the data we see 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 substrate 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 determine the composition of the annealed film and the as-deposited film on SrTiO₃, a peak height analysis, similar to that suggested by equation 19 will be require. However, instead of using the surface height analysis we will use an average height analysis with the cross sections evaluated at an energy corresponding to the average energy of the incoming projectile, $\bar{E}_{in} = [E_o + E_t]/2$ (see Fig. 2 and equations 8-11). Correspondingly, an average stopping cross section factor will also be used, and for element A has the form (4)

$$[\bar{\epsilon}(A_m B_n)]_A = \left[\frac{K_A}{\cos\theta_1} \epsilon(\bar{E}_{in}) + \frac{1}{\cos\theta_2} \epsilon(\bar{E}_{out}) \right] \quad (20)$$

where the average energy of the out going particle, after a collision with element A is expresses as

$$\bar{E}_{out} = [K_A E_t + E_1(A)]/2 \quad (21)$$

The average stopping cross section will also be used in equation 12 to determine the film thickness.

While not explicitly stated, values of ϵ in equation 20 are for the compound target and should be calculated according to equation 7. For the superconducting compound YBa₂Cu₃O₇, ϵ on an atomic fraction basis will be equal to

$$\epsilon(YBa_2Cu_3O_7) = 1/13\epsilon(Y) + 2/13\epsilon(Ba) + 3/13\epsilon(Cu) + 7/13\epsilon(O) \quad (22)$$

where the component stopping cross section have been taken at the energies of interest, $\bar{E}_{in}$ and $\bar{E}_{out}$. The result of this calculation over a wide range of energies is presented in Fig. 17.

Iterative calculations of the film thickness, composition, E_{in} , E_{out} , and the average stopping cross section factor indicate that the annealed film is 8980 Å thick and has a composition of Y_{1.11}Ba_{2.00}Cu_{2.91}O_{7.04}, normalized to Ba = 2. Similar analysis on the unannealed

film, using data from both the C and SrTiO_3 spectra (Fig. 18), gives us a film thickness of 7400 Å and a stoichiometry of $\text{Y}_{0.97}\text{Ba}_{2.00}\text{Cu}_{2.91}\text{O}_{0.21}$, normalized to Ba = 2. The above calculations assumed atomic densities of 4.17 and 7.69×10^{22} atoms/cm² for the unannealed and annealed films respectively. The stopping cross section factors (equation 20) calculated for the unannealed material are 82.8, 82.4, and 82.0×10^{-15} eV cm² for the Ba, Y, and Cu peaks respectively, and in the annealed material, 53.2, 52.9, 52.8, and 59.9×10^{-15} eV cm² for Ba, Y, Cu, and O respectively. This indicates that for a scattering geometry of $\theta_1 = 0^\circ$ and $\theta_2 = 15^\circ$, and a detector resolution of 15 keV, our depth resolution for 8.8 MeV He is approximately 440 Å in the unannealed material and 370 Å in the annealed material.

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 assume that the increase is the result of Sr contamination, the estimated Sr concentration in the film would be approximately 1.1 atomic %.

To clearly show the advantage of working with 8.8 MeV He, Fig. 20 contrasts the same high energy data presented in Fig. 19 to RBS spectra taken on the same as-deposited and O₂ annealed samples. The RBS spectra were taken at the same scattering geometry employed for the 8.8 MeV data. The 2 MeV He RBS spectra also include data from O backscattering.

The transport quality of the annealed film, as determined by four point probe measurements, is presented in Fig. 21 (22). The film possesses 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 has suggested Sr contamination. As indicated by the phase diagram in Fig. 3, deviations from a 1:2:1 atomic ratio for Y:Ba:Cu results in the formation of other, non-superconducting, phases. As in other ceramics, second phases in the $\text{YBa}_2\text{Cu}_3\text{O}_7$ superconductor will most likely accumulate at grain boundaries, producing Josephson junctions which tend to spread the superconducting transition out (24, 25). The addition of Sr has similar detrimental effects on HTS

materials, studies have clearly demonstrated that T_C decreases with increasing Sr content (26,27).

4.1. Formation of $YBa_2Cu_3O_7$ superconducting films by ion implantation

One possible method of producing superconducting material that will be compatible with the fine structures required in potential an electronic devices is ion implantation. Early work by Koch et al (28) demonstrated that the radiation damage which results from ion implantation into $YBa_2Cu_3O_x$ thin films renders the new class of oxide superconductors normal at moderate doses (29,30). Koch and co-workers used implantation and masking techniques to destroy the superconductivity in specific portions of a superconducting $YBa_2Cu_3O_x$ film to define the structure of a superconducting quantum interference device.

An alternate technique, which has proven to be quite effective in producing devices in semiconducting materials (31), is to use ion implantation to introduce additional elements into a base material and alter the electrical properties of the implanted region. Recently Nastasi and co-workers (32) examined the effectiveness of implanting Y ions into a non-superconducting Ba-Cu-F base material to produce the superconducting $YBa_2Cu_3O_7$ phase.

The experimental procedure followed by these researchers was to form a base material for Y implantation by the electron beam coevaporation of BaF_2 and Cu. A 4000 Å thick alloy layer with an approximate composition of $Ba_2Cu_3F_4$, was deposited in a vacuum of $\sim 5 \times 10^{-8}$ torr on to room temperature substrates of jewelers quality $SrTiO_3$ and single crystal Al_2O_3 . A 200 Å layer of BaF_2 was used as a capping layer to minimize contamination and material loss from sputtering during ion implantation.

During the Y ion implantation process the coevaporated BaF_2 -Cu films were held at 80 K in a vacuum of 5×10^{-7} torr. Two different energy Y implantations were employed in an attempt to produce a more uniform Y distribution. The films were first implanted with 400 keV Y^{1+} to a dose of 6×10^{16} atoms/cm², followed by 100 keV Y^1 to a dose of 2×10^{16} atoms/cm². TRIM Monte Carlo simulations (23), which did not consider sputtering, estimated the range and straggling of the implanted Y ion to be 1947 Å and 776 Å respectively at 400 keV, and 646 Å and 217 Å at 100 keV.

Figure 22 shows 8.8 MeV He backscattering data from as-deposited and Y implanted films on Al_2O_3 . Analysis of the as-deposited spectra, using the cross section data presented in Fig. 16 and equation 16, gives a total integrated Ba and Cu composition of Ba_3Cu_4 . Backscattering studies at 2 MeV have determined that BaF_2 is evaporated stoichiometrically (22) indicating that the composition of the coevaporated film is $Ba_2Cu_4F_4$.

A comparison of the two spectra in Fig. 22 shows that ion implantation produces a decrease in the width of both the Cu and Ba peaks as well as a decrease in the surface

density of Ba. An analysis using the total counts in the Ba and Cu peaks yields a post-implantation composition of $\text{Ba}_{1.8}\text{Cu}_3$, indicating that Ba was preferentially sputtered during the implantation of Y. A further analysis using equation 19 and the surface heights of the Ba, Y, and Cu peaks gives a surface composition of $\text{Y}_{0.9}\text{Ba}_{1.6}\text{Cu}_3$ in the as-implanted film. The implanted Y distribution was peaked at the film surface with a fwhm of approximately 1600 Å, and a low energy tail which extends to a depth of approximately 2900 Å.

Both the as-deposited and Y implanted samples were annealed using a wet oxygen procedure similar to that proposed by Mankiewich et al (33,34). The samples were annealed for 1 hr at 750 C in dry oxygen, 1 hr at 850 C in wet oxygen, 1 hr at 650 C in dry oxygen, 1 hr at 550 C in dry oxygen, and a slow cool to 50 C, over approximately 3 hr, in dry oxygen.

In Fig. 23, 8.8 MeV He backscattering data from the as-deposited, Y implanted, and Y implanted plus oxygen annealed samples, on SrTiO_3 substrates, are presented. Evident in the low energy portion of this data is the evolution of the F and O content as a function of processing. Both F and O are observed in the spectra from the as-deposited and Y implanted films. Following oxygen annealing a large increase in the oxygen signal is observed along with the nearly complete attenuation of the F signal.

Examining the high energy portion of Fig. 23, we see that annealing has increased the width of the Ba, Y, and Cu peaks. In addition, annealing has left the surface height of both the Ba and Y peak very close to their as-implanted heights, while the Cu surface height has been reduced. However, a close examination of the Y peak region reveals that the total Y yield has increased after annealing relative to before annealing. This data indicates that surface or near-surface scattering of Sr is occurring. A SEM examination of this sample did detect some surface porosity, which leads us to conclude that Sr substrate backscattering is contributing to the enhanced Y yield. Additional experiments will be necessary to determine if Sr has also become incorporated into the surface layer. A surface height analysis of the Ba, Cu, and O peaks after oxygen annealing and the Y as-implanted peak gives a surface composition of $\text{Y}_1\text{Ba}_{1.7}\text{Cu}_{2.5}\text{O}_{6.4}$.

Electron diffraction data, presented in Fig. 24, indicates a minimum of two phases were formed during coevaporation; only BaF_2 could be positively identified. Additional diffuse rings from the carbon support film were also observed. Diffraction data from the implanted region, close to the surface (Fig. 24b, region 1), shows that low temperature implantation of Y results in a near complete amorphization of the surface. The diffraction pattern from a deeper region (region 2) is identical to that presented in Fig. 3a. The diffraction pattern from the Y implanted and annealed film (Fig. 24c) indicates that substantial grain

growth occurred during the oxygen annealing process. The indexed diffraction pattern suggests that a minimum of two phases are present, $\text{YBa}_2\text{Cu}_3\text{O}_7$ and BaCuO_2 .

Resistance versus temperature data is presented in Fig. 25 for both the as-deposited and Y implanted $\text{Ba}_2\text{Cu}_3\text{F}_4$ films after oxygen annealing. As anticipated, oxygen annealing the as-deposited film results in the formation of an insulating material which possess a room temperature (RT) resistance of 1.0×10^5 ohms which increases to 6.4×10^5 ohms by 245 K. The Y implanted film shows a moderately sharp superconducting transition at approximately 85 K which is followed by broader transition starting at approximately 76 K. At higher temperatures the resistive quality of the film is semiconductor in nature.

The resistance data shows for the first time that ion implantation alloying is capable of producing material which possess a relatively high superconducting transition temperature. The semiconducting normal state behavior and low temperature tail in the superconducting transition indicate that multiple phases, both superconducting and non-superconducting, have formed in the Y implanted region. This interpretation is consistent with the backscattering data of Fig. 23 which showed the annealed film to be off the ideal 123 stoichiometry and contaminated with Sr, and the electron diffraction data of Fig. 24c which showed the presence of both superconducting and non-superconducting phases.

5. SUMMARY

The preceding text has demonstrated that ion beams are quite capable of supplying a variety of depth sensitive information from thin film high temperature superconductors as well being useful in the production of these new materials. A variety of backscattering approaches have been discussed including classical Rutherford backscattering with He ions, and non-Rutherford backscattering techniques at both low and high energies, employing proton and He projectiles. The utility of using 8.8 MeV He backscattering was clearly demonstrated in the fabrication of thin film superconductors by both conventional evaporation techniques and non-conventional ion implantation techniques. Finally, the application of ion implantation alloying to the production of high temperature superconducting material was discussed.

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References

- 1) Mayer J.W. and E. Rimini, Ion Beam Handbook for Material Analysis (Academic Press, New York, 1977).
- 2) Chu W-K., J.W. Mayer, and M-A. Nicolet, Backscattering Spectrometry (Academic Press, New York, 1978).
- 3) Feldman L.C. and J.W. Mayer, Fundamentals of Surface and Thin Film Analysis (North-Holland, New York, 1986)
- 4) Ref. 4, chapt. 2.
- 5) Bragg W.H. and R. Kleeman, Phil. Mag. 10 (1950) S318.
- 6) Frase K.G., L.G. Liniger, and D.R. Clarke, J. Am. Ceram. Soc. 70 (1987) C204.
- 7) Lupis C.H.P. Chemical Thermodynamics of Materials (North-Holland, New York, 1983) chapt. 10.
- 8) Jorgensen J.D., M.A. Beno, D.G. Hinks, L. Soderholm, K.J. Volin, R.L. Hitterman, J.D. Grace, L.K. Schuller, C.U. Segre, K. Zhang, and M.S. Kleetisch, Phys. Rev. B36 (1987) 3608.
- 9) Cava R.J., B. Batlogg, C.H. Chen, E.A. Rietman, S.M. Zahurak, and D. Werder, Phys. Rev. B36 (1987) 5719.
- 10) Shi D. and D.W. Capone II, Appl. Phys. Lett. 53 (1988) 159.
- 11) Leavitt J.A., P. Stoss, D.B. Cooper, J.L. Seerveld, L.C. McIntyre Jr., R.E. Davis, S. Gutierrez, and T.M. Reith, Nucl. Inst. and Meth. B15 (1986) 296.
- 12) Doolittle L.R., Nucl. Inst. and Meth. B9 (1985) 344.
- 13) Rauhala E., J. Keinonen, and R. Jarvinen, Appl. Phys. Lett. 52 (1988) 1520.
- 14) Barbour J.C., B.L. Doyle, and S.M. Myers, Phys. Rev. B, in press.
- 15) Martin J.A., M. Nastasi, J.R. Tesmer, and C.J. Maggiore, Appl. Phys. Lett. 52 (1988) 2177.
- 16) Branpain B., P. Revez, L.E. Doolittle, K.H. Purser, and J.W. Mayer, Nucl. Inst. and Meth. B, in press.
- 17) Cameron J.R., Phys. Rev. 90 (1954) 849.

- 18) Mezey G., J. Gyulai, T. Nagy, E. Kotai, and A. Manuba, in Ion Beam Surface Layer Analysis, eds. O. Meyer, G. Linker, and Kappeler (Plenum Press, New York, 1976) p. 303.
- 19) Luomajarvi M., E. Rauhala, and M. Hautala, Nucl. Inst. and Meth. B9 (1985) 255.
- 20) Ref. 3, chapt 2.
- 21) Hunt W.E., M.K. Mehta, and R.H. Davis, Phys. Rev. 160 (1967) 782.
- 22) Nastasi M., J.A. Martin, J.R. Tesmer, M.G. Hollander, and C.J. Maggiore, unpublished data.
- 23) Zeigler J.F., J.P. Biersack, U. Littmark, The Stopping and Range of Ions in Solids (Permagon Press, New York, 1985)
- 24) Naito M., R.H. Hammond, B. Oh, M.R. Hahn, J.W.P. Hsu, P. Rosenthal, A.F. Marshall, M.R. Beasley, T.H. Geballe, and A. Kapitulnik, J. Mater. Res. 2 (1987) 713.
- 25) White A.E., K.T. Short, D.C. Jacobson, J.M. Poate, R.C. Dynes, P.M. Mankiewich, W.J. Skocpol, R.E. Howard, M. Anzlowar, K.W. Baldwin, A.F.J. Levi, J.R. Kwo, T. Hsieh, and M. Hong, in High Temperature Superconductors, eds., M.B. Brodsky, R.C. Dynes, K. Kitazawa, and H.L. Tuller (Materials Research Society, Pittsburgh, 1988) p. 531.
- 26) Veal B.W., W.K. Kwok, A. Umezawa, G.W. Crabtree, J.D. Jorgensen, J.W. Downey, L.J. Nowicki, A.W. Mitchell, A.P. Paulokas, and C.H. Sowers, Appl. Phys. Lett. 51 (1987) 279.
- 27) Ono A., T. Tanaka, H. Nozaki, and Y. Ishizawa, Jpn. J. Appl. Phys. 26 (1987) L1687.
- 28) Koch R.H., C.P. Umbach, G.J. Clark, P. Chaudhari, and R.B. Laibowitz, Appl. Phys. Lett. 51, 200 (1987)
- 29) Clark G.J., A.D. Marwick, R. Koch, and R.B. Laibowitz, Appl. Phys. Lett. 51, 119 (1987)
- (30) Clark G.J., F.K. LeGoues, A.D. Marwick, R.B. Laibowitz, and R. Koch, Appl. Phys. Lett. 55, 1462 (1987)
- 31) Mayer J.W., L. Eriksson, and J.A. Davis, Ion Implantation in Semiconductors (Academic Press, New York, 1970)
- 32) Nastasi M., J.R. Tesmer, H.G. Hollander, J.F. Smith, C.J. Maggiore, Appl. Phys. Lett. 52 (1988) 1729.

33) Mankiewich P.M., J.H. Scofield, W.J. Skocpol, R.E. Howard, A.H. Dayem, and E. Good, Appl. Phys. Lett. 51, 1753, (1987)

34) Mankiewich P.M., R.E. Howard, W.J. Skocpol, A.H. Dayem, A. Ourmazd, M.G. Young, and E. Good, in High Temperature Superconductors, eds., M.B. Brodsky, R.C. Dynes, K. Kitazawa, and H.L. Tuller (Materials Research Society, Pittsburgh, 1988) p. 119.

Figure Captions

Figure 1. The energy loss components for a projectile scattered from a depth t in a single element target. See equations 3 through 6.

Figure 2. A schematic representation of the backscattering process from an idealized free standing compound film with composition A_mB_n and thickness t .

Figure 3. The pseudoternary Y_2O_3 -BaO-CuO phase diagram determined at 950 C shows the compound $Y_1Ba_2Cu_3O_x$ (123) to be stoichiometric in its metal composition. From reference 6.

Figure 4. Measured backscattering cross section for He on O^{16} in the energy interval 1.8 to 48 MeV. The O cross section starts to deviate from Rutherford scattering near 2.2 MeV. From reference 11.

Figure 5. RBS spectra simulated with the program RUMP (11) for 2 MeV He projectiles on a 4000 Å thick free standing $YBa_2Cu_3O_7$ film. Also shown are the component RBS signals from Ba, Y, Cu, and O.

Figure 6. RUMP backscattering simulations of two possible solutions to the problem of Ba, Y, and Cu peak overlap. The low energy spectra, which was simulated assuming 2 MeV He, suggests a maximum $YBa_2Cu_3O_7$ thickness of 1200 Å at this energy. The high energy portion shows that complete peak separation in a 4000 Å film is possible at 4 MeV.

Figure 7. A RUMP simulated RBS spectra 2 MeV He on a 4000 Å thick $YBa_2Cu_3O_7$ film on a $SrTiO_3$ substrate. Also shown are the individual backscattering signals from Ba, Y, Cu and O in the surface film and signals from Sr, Ti, and O in the substrate.

Figure 8. Backscattering spectra from a 1050 Å thick Ta_2O_5 film on Ta. The data contrasts the effect of an enhanced O cross section, which exists for 1.097 MeV He, compared to the Rutherford O cross section for 2.030 MeV He. From reference 16.

Figure 9. Multiple He backscattering spectra for He projectiles on Zirconia at increasing energy. From reference 16.

Figure 10. Backscattering data obtained from a 1.086 MeV He on a Y-Ba-Cu-O/Zirconia film-substrate combination. From reference 16.

Figure 11. Backscattering data from 2.5 MeV protons on bulk $\text{YBa}_2\text{Cu}_3\text{O}_7$. The inset shows the behavior of $\text{O}^{16}+\text{p}$ cross section as a function of energy. From reference 14.

Figure 12. High energy O^{16} scattering cross section as a function of incident He (alpha-particle) energy. The data indicates that as the geometry becomes progressively more backscattering a very broad plateau appears in the cross section which is centered near 8.6 MeV. From reference 21.

Figure 13. A typical backscattering spectrum for 8.8 MeV He incident on a 7700 Å thick film of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sitting on a SrTiO_3 substrate. From reference 15.

Figure 14. RBS data at 2 MeV from two films which were used to measure the relative cross sections of Cu, Y, and O to Ba as a function of He energy.

Figure 15. Ratio of scattering cross section for He ions scattering from O^{16} and Ba as a function of energy. The dashed line shows the ratio of the Rutherford scattering cross sections. From reference 15.

Figure 16. Ratio of scattering cross sections for He ions from Y and Cu relative to Ba as a function of He energy. The dashed line represents the ratio of the Rutherford scattering cross sections. From reference 15.

Figure 17. TRIM (ref. 24) calculated stopping data for He projectiles on a Y-Ba-Cu-O target. This data was calculated assuming a target atomic composition of 7 % Y, 15 % Ba, 23 % Cu and 54 % O, and a mass density of 6.54 g/cm^3 .

Figure 18. 8.8 MeV He backscattering data from Y-Ba-Cu films prepared by vacuum electron beam coevaporation onto SrTiO_3 and C substrate.

Figure 19. 8.8 MeV He backscattering from Y-Ba-Cu films on SrTiO_3 substrates before and after annealing.

Figure 20. 2 MeV He RBS spectra (top) and 8.8 MeV He backscattering spectra taken from the same as-deposited and O_2 annealed samples. Both sets of data were taken at the same scattering geometry. The 2 MeV He RBS spectra also include data from O backscattering.

Figure 21. Four point probe data following an O_2 anneal. The film possess a superconducting transition which is 8 K wide, reaching zero resistance at 79 K.

Figure 22. 8.8 MeV He backscattering data from as-deposited and Y implanted films on Al_2O_3 . These spectra were taken with $\theta_1 = 60^\circ$.

Figure 23. 8.8 MeV He backscattering data from the as-deposited, Y implanted, and Y implanted plus oxygen annealed samples, on SrTiO_3 substrates. These spectra were taken with $\theta_1 = 60^\circ$.

Figure 24. Electron diffraction patterns from the as-deposited (a), Y implanted (b), and Y implanted plus oxygen annealed (c) films.

Figure 25. Resistance data versus temperature data for both the as-deposited and Y implanted $\text{Ba}_2\text{Cu}_3\text{F}_4$ film after oxygen annealing.

ENERGY LOSS

SCATTERING AT DEPTH t :

SCATTERING SURFACE:

$$\Delta E_{in} = \frac{t}{\cos \theta_1} \left. \frac{dE}{dx} \right|_{in}$$

$$\Delta E_s = E_0 - KE_0$$

$$E_t = E_0 - \Delta E_{in}$$

$$\Delta E_c = E_t - KE_t$$

$$\Delta E_{out} = \frac{t}{\cos \theta_2} \left. \frac{dE}{dx} \right|_{out}$$

 

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