COMPOSITIONAL ANALYSIS OF HIGH- TEMPERATURE SUPERCONDUCTOR THIN FILMS BY HIGH ENERGY ELASTIC BACKSCATTERING OF HELIUM IONS

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ABSTRACT

High energy ion backscattering can be used to enhance the sensitivity of oxygen analysis. At He^{++} ion energy of 8.8 MeV, the yield due to oxygen is about 25 times larger than that predicted by Rutherford formula. The elemental stoichiometry of some bulk and thin film superconductor samples was determined. The details of the measuring method are discribed.

Key words: High energy ion backscattering High temperature superconductor

I. INTRODUCTION

It is essential to accurately determine the elemental composition in producing well qualified high-temperature superconductor (HTS) thin films. Low energy Rutherford backscattering spectrometry (RBS) is generally rapid, quantitative and depth sensitive but not applicable to direct oxygen determination in HTS materials because the scattering cross section from oxygen is relatively low to those from heavy elements and oxygen peak rests on a high background. For the analysis of oxygen in HTS material, nuclear reaction $^{16}O(d,p)\,^{17}O$ has to be used[1].

In the case of higher energy ion incident, resonant non-Rutherford backscattering can be utilized to enhance the sensitivity of some light elements. At 8.8 MeV, the scattering yield due to oxygen is large, even comparing with that due to heavier metallic elements[2]. This makes it possible to analyze oxygen as well as other elements simultaneously. The absolute accuracy of determination of oxygen content from such a high energy ion backscattering spectrum depends ultimately on that how accurate the cross section is known.

II. PRINCIPLE AND EXPERIMENTAL

The sensitivity and accuracy of RBS for stoichiometric determination in films depend on both relative magnitudes of scattering cross section of each element and precision of the cross section. Calculated Rutherford cross sections adequately describe He^+ ion scattered from most elements for incident ion energies below about 2 MeV and this fact is the base of the quantitative accuracy of RBS.

Before determining the element composition of HTS by measuring high energy ion backscattering spectrum, it must first be determined that the oxygen cross section for the particular incident beam energy at scattering angle of interest. It is also important to check whether the scattering cross sections at high energy for other elements such as Cu and Y are still predictable by the Rutherford formula.

Some thin – film standards are used to calibrate the ratios of scattering cross sections for several HTS – related elements. Each standard sample consists of two elements which have been evaporated on a pyrolytic graphite substrate with electron beam. The thickness of the film is about 0.1 μ m. For each sample a 2 MeV RBS spectrum is measured and the elemental peaks are integrated to obtain the count ratio A_A/A_B of atoms A and B in the film. The ratio of the elemental concentration N_A/N_B in sample is then determined by calculation of Rutherford scattering cross sections. This procedure sets up a thin – film standard of known elemental stoichiometry. Which is then used to determine scattering cross section ratio σ_A/σ_B in the energy range of interest by integrating elemental peaks in spectra collected.

Fig.1 shows the results of this procedure for the case of He ion scattering from O and Ba in the energy range of 8.2—9.1 MeV. An annular surface barrier dector is used with solid angle about 0.5 sr. The solid line in the figure, 0.018 in value, is the ratio of the Rutherford cross sections at 160°. In energy range from 8.55 to 8.80 MeV, the ratio varied smoothly with the value between 0.460 and 0.453, the increased sensitivity of oxygen at this energy range is then about 25 times greater than that at 2 MeV. So it is appropriate to chose 8.8 MeV as the incident beam energy.

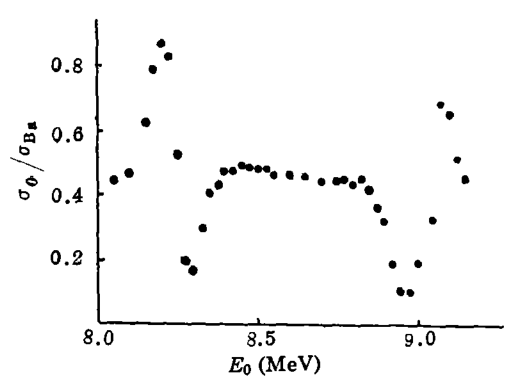

Fig.1 The ratios of scattering cross sections for helium ion from ^{16}O and Ba as a function of incident ion energy

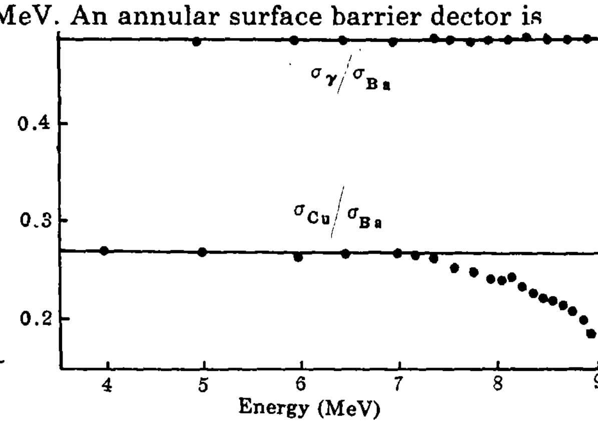

Fig.2 The ratios of scattering cross sections for helium ion from Y and Cu relative to that from Ba as functions of incident ion energy

Fig.2 showes the results of measurements of the scattering cross section ratios for Cu/Ba and Y/Ba over the energy range of 2—9 MeV. The measuring uncertainty of data is about 3%. The solid lines indicate the calculated Rutherford cross section ratio. The scattering cross section for Ba is expected to be essentially Rutherford even at 9 MeV because of the high atomic number of Ba. The cross section for Cu has an upper limit energy of Rutherford scattering at 7.2 MeV while the cross section for Y keeps Rutherford even at up to 9 MeV

III. RESULTS AND DISCUSSION

This work was carried out at the Accelerator Laboratory of the Institute of Modern Physics, Fudan University. The 8.8 MeV He^{++} ion beam from a 3 MV tandem (9SDH-2, NEC) is applied to perform the backscattering experiment. Fig.3 shows the backscattering ion spectrum of a typical "thick" Y-Ba-Cu-O superconductor sample. The positions of Ba, Y, Cu and O peaks are indicated in the spectrum. It is evident that the peak of oxygen is greatly enhanced because of the increase of scattering cross section at high energy. From the data of Fig.1 and Fig.2 and the stopping power factor of Y, Ba, Cu, O in sample[3], an overall stoichiometry of $Y_{1.1}Ba_{2.0}Cu_{3.0}O_{6.7}$ is obtained. This concentration ratio is very close to the "standard" value of $Y_{1.0}Ba_{2.0}Cu_{3.0}O_{7.0}$. In fact, the *Tc* of this sample is 85 K.

Fig.4 is a scattering ion spectrum of an Y-Ba-Cu-O thin film. The film was deposited on $SrTiO_3$ substrate by magnetron sputtering with thickness of 0.8 μm.

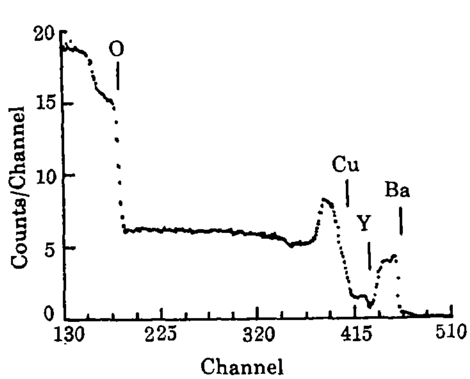

Fig.3 8.8 MeV He^{++} elastic backscattering spectrum from a bulk Y-Ba-Cu-O HTS sample

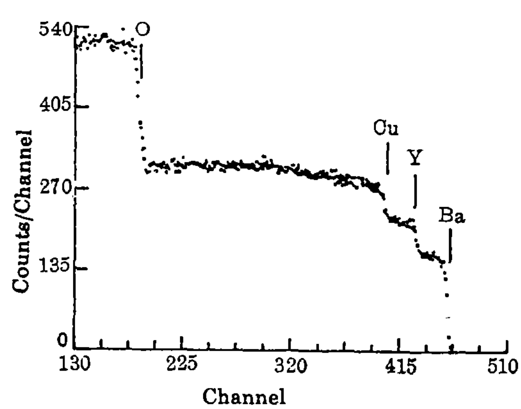

Fig.4 8.8 MeV He^{++} elastic backscattering spectrum from a thin film of Y-Ba-Cu-O on a $SrTiO_3$ substrate

The scattering peaks of Ba, Y, Cu and O are well separated but the contributions of Y and Sr on the interface are overlapped for these two elements have their atomic weight very close to each other. The step on the left of oxygen peak is the contribution of oxygen in substrate. We then determined that the concentration ratio of that sample is $Y_{1.0}Ba_{1.6}Cu_{6.6}O_{5.4}$, which is far deviated from the "standard" value. The experiment showed that it kept non-superconduct even been cooled down to 30 K. On the contrary, many well-qualified superconductor thin film samples have stoichiometry near $Y_1Ba_2Cu_3O_7$. Table 1 is the data we obtained from two of such samples.

Table 1

Elemental composition of two HTS thin films

Sample number	Composition	Tc(K)
564	$Y_{0.94}Ba_{2.0}Cu_{3.0}O_{7.15(\pm 0.28)}$	85
566	$Y_{1.05}Ba_{1.74}Cu_{3.0}O_{7.3(\pm 0.28)}$;	82

Ⅳ. CONCLUSIONS

We have measured the backscattering cross-section ratios of oxygen, copper, and yttrium to barium,respectively. A technique for compositional analysis of high-temperature superconductor material with enhanced sensitivity of oxygen is presented. The technique is proved to be rapid, nondestructive and particularly applicable to the development and analysis of HTS thin film process in view of the critical role of oxygen stoichiometry in HTS materials.

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