MATERIALS CHARACTERIZATION USING THE MICROBEAM AT SUNY/ALBANY

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ABSTRACT

The State University of New York at Albany ion scanning microprobe has been used for materials characterization. Focused proton and helium ion beams have been used. Rutherford backscattering spectroscopy (RBS) and particle-induced X-ray emission (PIXE) analysis have been performed on microelectronic circuits with a spatial resolution of approximately 2μ m. Studies on thin films of superconductors will be presented. Several examples of chemical and microstructural analysis will be given.

Key words: Ion scanning microprobe Rutherford backscattering spectroscopy PIXE Thin films of superconductors

I. INTRODUCTION

In recent years there has been rapid growth for the development of equipment for forming a focussed beam (1-2μ m) with high energy ions. There are several installations throughout the world including one at Fudan University, P.R.C.; doing microanalysis with high energy (MeV) ion beams[1-4] and several others under construction stage. Most of these have a provision for scanning a small region, forming an image using secondary electrons, and performing Rutherford backscattering spectroscopy (RBS) and particle induced X-ray emission(PIXE) analysis. The well known advantages of a nuclear microprobe with PIXE and RBS systems are: the sensitivity of heavy particle induced X-ray emission relative to electron induced emission; the depth information available from RBS; light isotope detection with nuclear reactions; and relatively unambiguous interpretation of the data. Nuclear microprobes have been used for biological and mineralogical samples and more recently for solid state physics and material science[5] to obtain chemical and microstructural information from the near surface region of small scale device structures. There have been considerable improvements during the past several years to obtain a small (1-5μ m) size beam spot using magnetic lenses[5-9]. Several sophisticated data collection systems, with several very useful modes for analyzing and displaying data have been developed by Legge et al.[10], Doyle et al.[11] and Fischer[12].

The nuclear microprobe at the State University of New York at Albany (SUNYA) utilizes a quadrupole doublet developed by Martin et al[6]. It has a provision for scanning a region 500× 500μ m^2, forming an image using secondary electrons, and detecting backscattered ions and emitted characteristic X-rays. The primary area of application of this particular instrument has been the chemical and microstructural

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analysis of microelectronic circuits.

Ⅱ. EXPERIMENTAL SETUP

The details of our experimental setup are given by Morris et al.[13-15]. The Dynamitron Accelerator at SUNYA is capable of delivering a wide variety of ions at energies from 0.2－4.2 MeV. During the course of present work only beams of 2 MeV He^+ or H^+ have been used. A quadrupole doublet lens is the final element used to focus the beam on the sample. X－rays are analyzed with a 6mm diam. x 3mm cooled Si(Li) detector which is placed inside the target chamber facing 45^0 to the target plane. The X－ray detector has a resolution of 160 eV at 6－9 keV. For RBS the backscattered ions are detected and energy analyzed by a surface barrier detector placed at 150^0 back angle with a solid angle of 160mse. The RBS detector had a resolution of 18 keV alpha particles.

The microbeam line is permanently setup on one of the several beamlines available at the accelerator facility. A unique feature of this microprobe is that the beam has an auto－aligh system which greatly simplifies setup and control of the microbeam line. Electrostatic scanning of the beam is done by providing scanning voltages to isolated pole pieces of the quadrupole doublet. The secondary electron detector allows us to image a region as large as $500\times 500\mu\ m^2$. As the image is taken inside the magnetic field, it is somewhat skewed. An IBM microcomputer displays both skewed and deskewed images of the surface in order to locate features of interest. The sample is mounted on a motorized x－y stage so that any point wityhin a $3\times 3\ cm^2$ area can be accessed. Energy spectra of the backscattered particles and emitter X－rays are collected and displayed simultaneously. The microcomputer is used to control the multichannel analyzers and to store the RBS and PIXE data. The same computer is used to store the digitized image and to control the x－y stage for the sample holder.

Alignment procedures are simplified by using an autoaligh system[16] which automatically corrects for small amounts of beam drift and keeps the incident beam centered on the slits which define the object size. When system is optimally aligned, one observes no translational sweep of the image when current is varied for the quadrupole doublet. This also gives no observed defocussing at the edges of the scanned image. When properly aligned, we observe a measured beam size of between $1-2\mu$ m with a beam current of 0.2－0.5 nA. Higher beam currents and increased spectral intensities can be obtained at the expense of a larger beam size. A 1000 mesh copper grid is used to check the alighyment, image quality, and magnification factor. The beam current is measured as the beam passes through an opening in the grid into a Faraday cup, and then a stationary beam is placed on a bar of the copper grid to obtain basckscattering and X－ray spectra for calibration purposes.

The sample is moved to the desired region with the help of a motorized x－y stage, utilizing the scanned secondary electron image as a guide. The secondary electron image of desired region is updated enery 2－3 seconds and displayed. The final image (average of eight cycles) of a desired region can be stored and displayed. RBS and PIXE spectra are then obtained from the particular features of interest using a stationary focussed beam. With a beam current of 0.1－0.5 nA, satisfactory spectra can

be obtained within counting time of 5 – 10 minutes.

It has been shown in earlier work[13] that the X – ray yield for K – lines from elements Z = 22 – 30 is much greater for protons than for He. For *L* lines, the difference is not as great, but the *L* lines are generally less intense. In most cases we use He ions for the convenience of obtaining both RBS and PIXE information simultaneously. The analysis of RBS is done using the computer program RUMP developed at Cornell University by Doolittel[17].

Ⅲ. RESULTS AND DISCUSSION

A. Microelectronic circuits

RBS and PIXE provides a very useful information for characterization of thin layers. Fig.1 and 2 show two different areas of Mo conductor runs on a Si integrated circuit. Fig.3 is the backscattering spectrum from one of the conductros, which clearly show the Mo layer and the underlying SiO_2. the simulation program is used to determine that the Mo thickness is 500nm. The value of 1.3μ m for the oxide layer is less certain due to straggling effects. The PIXE spectrum contained only Si K and Mo L lines, confirming that these were the major elements present.

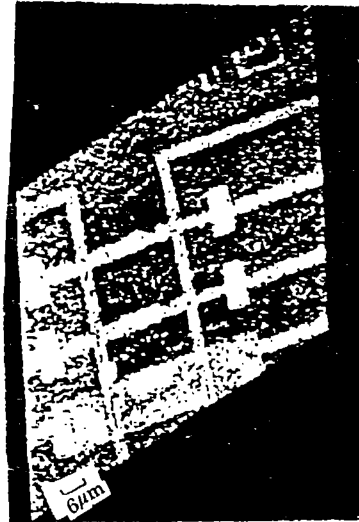

Fig.1 Secondary electron image of an integrated circuit obtained as a 2 MeV beam of He ions is scanned over the surface.
Beam current = 0.2 nA,
scan time 5 sec

Analysis of a nearby region from which the Mo has been etched away (point B in Fig.2) shows the expected oxide layer, determined to be 1.5μ m thick, and the underlying Si substrate (Fig.4). A small peak near channel 750 comes from Ti, which was deposited on the wafer before the Mo. It was incompletely removed along with the Mo during the subsequent etching, perhaps it was partially converted to an oxide. The PIXE spectrum contained only a Si K peak, the Ti being present is too small a quantity to be detected.

Fig.5 shows a secondary electron image of a region of a microelectronic test circuit with Al conductors. A darkened, and possibly contaminated, region was measured. Initially, the most distinguishing feature of the backscattering spectrum in Fig.6 is the lack of well defined "edges" for Si or Al. A large number of possible chemical compositions and layer thicknesses could be fed into the RBS simulation program with equally satisfactory results. By using information in the PIXE spectrum, Fig.7, the composition was restricted to include F, Na, Al, and Si plus lower Z elements such as 0. The presence of a small amount of Cl could fit the data between channels 600 and 660, even though no Cl X – ray peak was observed. This example illustrates the complimentary nature of the RBS and PIXE data, to assist in obtaining better analyses.

B. Studies on hin films of superconductors

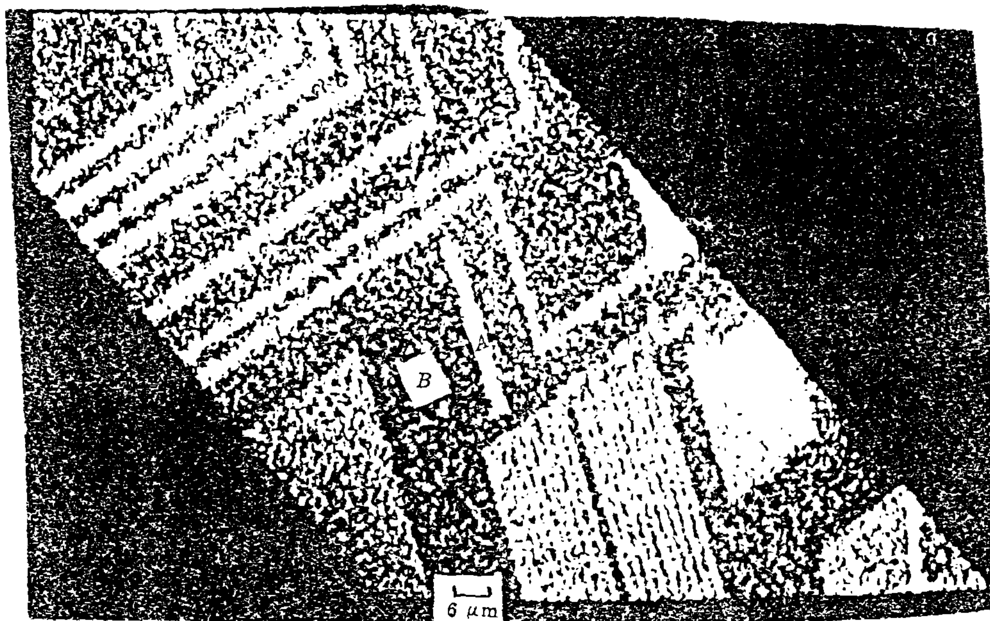

Fig.2 Secondary electron image of an integrated circuit obtained as a 2 MeV beam of He⁻ ions is scanned over the surface. Beam current = 0.2 nA, scan time 5 sec

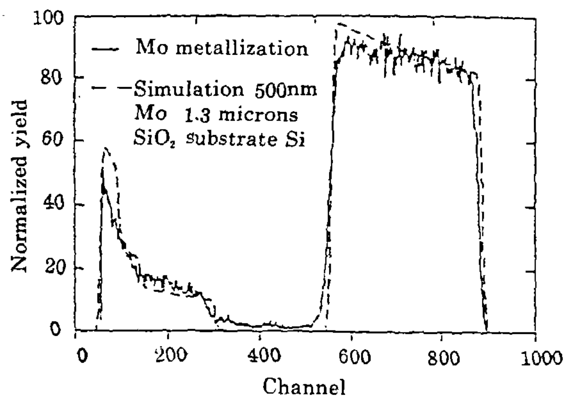

Fig.3 RBS data obtained on a metallized conductor from point A in Fig.2

Beam current = 0.2nA,
Collect time = 5 min

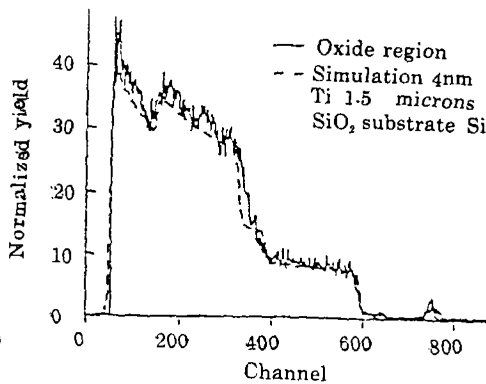

Fig.4 RBS data obtained from point B in Fig.2, Beam current = 0.2 nA

Collection time = 5 min

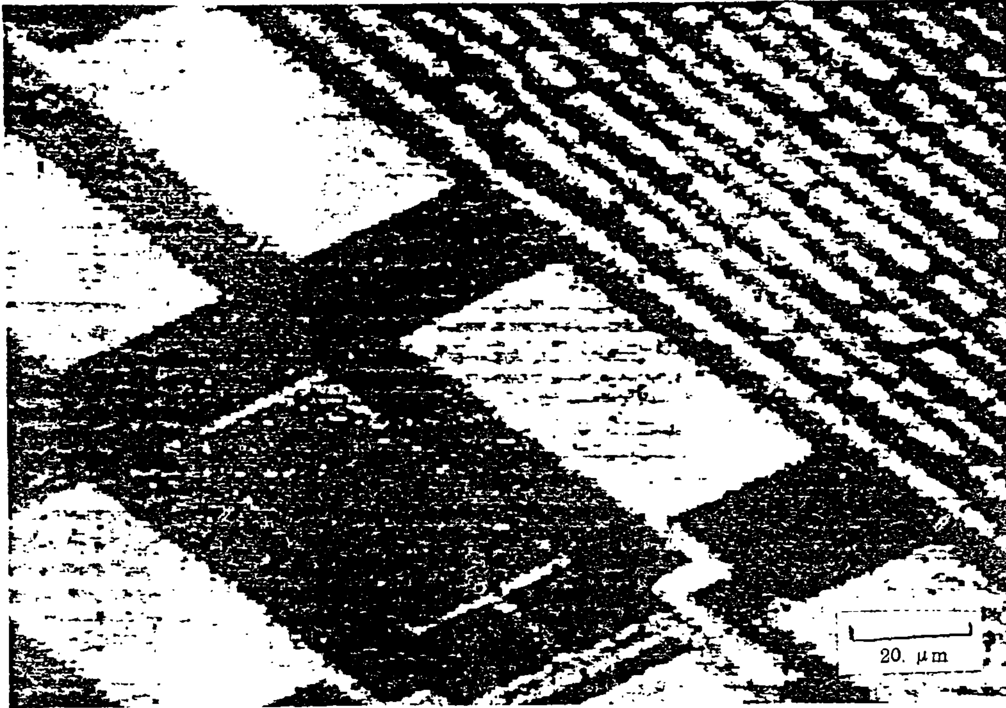

Fig.5 Secondary electron image of a region of a microelectronic test circuit with Al conductors. Beam current = 0.2 nA, scan time 5 sec

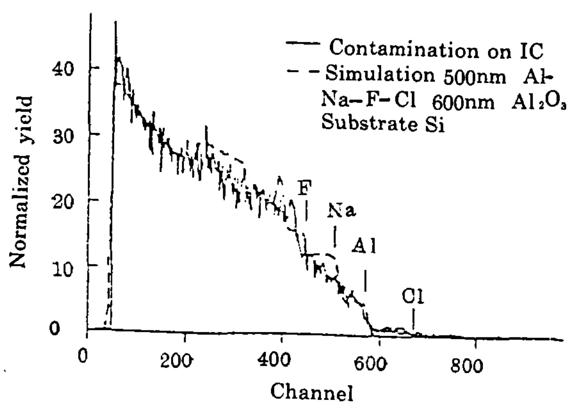

Fig.6 RBS data from a contaminated region on a Si integrated circuit

Beam current = 0.2nA
Collect time = 5 min

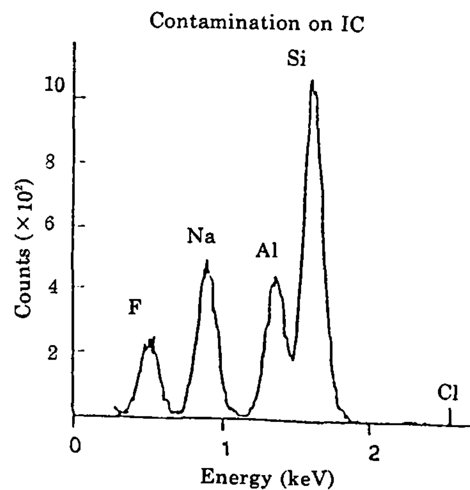

Fig.7 PIXE spectrum obtained from the same area as Fig.6

Beam current = 0.2nA
Collect time = 5 min

Thin films of superconductors (Y BaCuO) have ben studied using the microbeam system. Figure 8 shows the RBS spectrum of a 800nm thick Y BaCuO superconducting film. The PIXE spectrum of above film is shown in Fig.9. Studies on maps of Y, Ba and Cu show features of $20-30\mu$ m areas where Y was enhanced, the Ba concentration was less and areas where Ba was enhanced, the Y concentration was less.

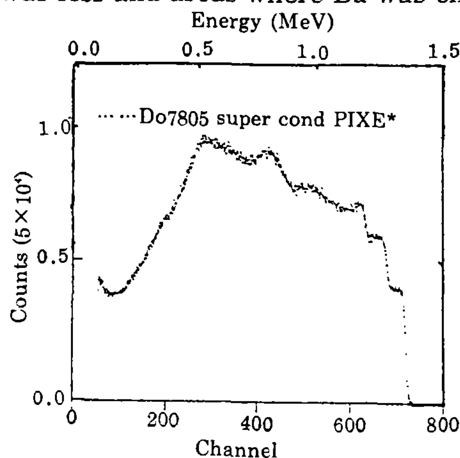

Fig.8 RBS spectrum of 800000 nm thick YBaCuO superconducting film

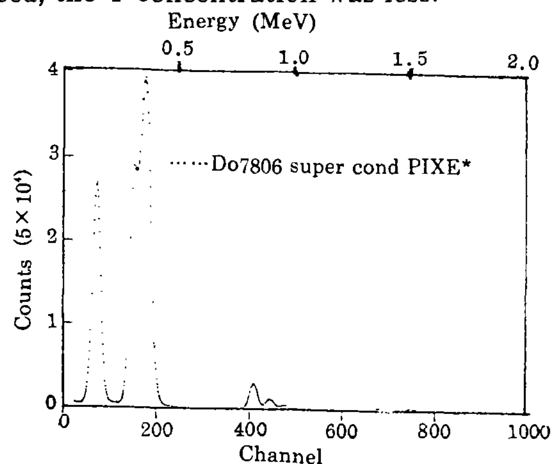

Fig.9 PIXE spectrum of 800000 nm thick YBaCuO superconducting film

IV. CONCLUSION

The RBS and PIXE analysis using MeV microbeam is very useful in the analysis of composition and microstructural features in microelectronic circuits and superconducting thin films. Recently we have started a program of structural studies of biological cells using the above microbeam system.

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