A MICROBEAM SYSTEM OF HIGH ENERGY IONS AT FUDAN UNIVERSITY

Ren Chigang (任炽刚), Zhou Shijun (周世俊), Che Jianmei(车建美),
Hu Yude (胡毓德), Chen Jianxin (陈建新), Fang Dufei (方渡飞)
and Yang Fujia (杨福家)

(Department of Nuclear Science, Fudan University, Shanghai 200433, China)

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ABSTRACT

Particle induced X- ray emission (PIXE) and Rutherford backscattering spectrometry (RBS) have been used extensively for analytical purpose because of their quantitative accuracy, reliability, simplicity and capability of non- destructive and multielement analysis. When these techniques are combined with a scanning microbeam system and a data acquisition system, three dimensional distribution of elemental composition can be displayed. Samples analyzed so far at Fudan University include a microelectronic circuit and some biological and archaeological samples. The PIXE and RBS spectra and the secondary electron images have been measured.

Keywords: Microbeam system SPM Human hair specimen Examine a crack in steel Elemental maps of copper mirror of Han Dynasty

Ⅰ. INTRODUCTION

Over the past two years, a new microprobe system has been established on a 3MV tandem electrostatic accelerator (9SDH- 2) at the accelerator laboratory of Fudan University[1,2]. High energy ion microbeams are used to determine the composition and distribution of elements in samples by particle induced X- ray emission (PIXE) and Rutherford backscattering spectrometry (RBS). Due to the low bremsstrahlung background when high energy ions interact with a specimen, the sensitivity of PIXE is nearly 3 orders of magnitude higher than that of electron induced X- ray emission[3].

Combining PIXE with RBS, the nuclear microprobe at Fudan can nondestructively analyse such microstructure distributions. Its high resolution, sensitivity, and quickness make the microprobe an appropriate instrument for investigations in a broad range of analytical applications.

The following is the current feature of the microprobe at Fudan: 1) Ion species: proton, helium, and heavy ions; 2) Min. beam spot size: 2μ m; 3) Max. beam current on target: 30 pA/μ m^2; 4) Beam scanning range: 500×500μ m^2; 5) Sample movement range: 2.5×2.5cm^2; 6) Mapping mode: Up to 16 maps of the elemental distribution; 7) Point mode: Automatized measurement on preselected positions.

Ⅱ. DESIGN PARAMETERS AND EXPERIMENTAL PROCEDURES

Fig.1 shows the schematic diagram of the microprobe system, which has been

improved from its original model installed at SUNYA (State University of New York at Albany).

Particle beams from the 2× 3 MV pelletron are guided into the microbeam line after the deflection of 45° by an analysing magnet. The total length of the beam line is shortened to 3.2 meters to fit configuration requirements of the accelerator. The ion beam, focused by the electrostatic quadrupoles and analysing magnet, passes through

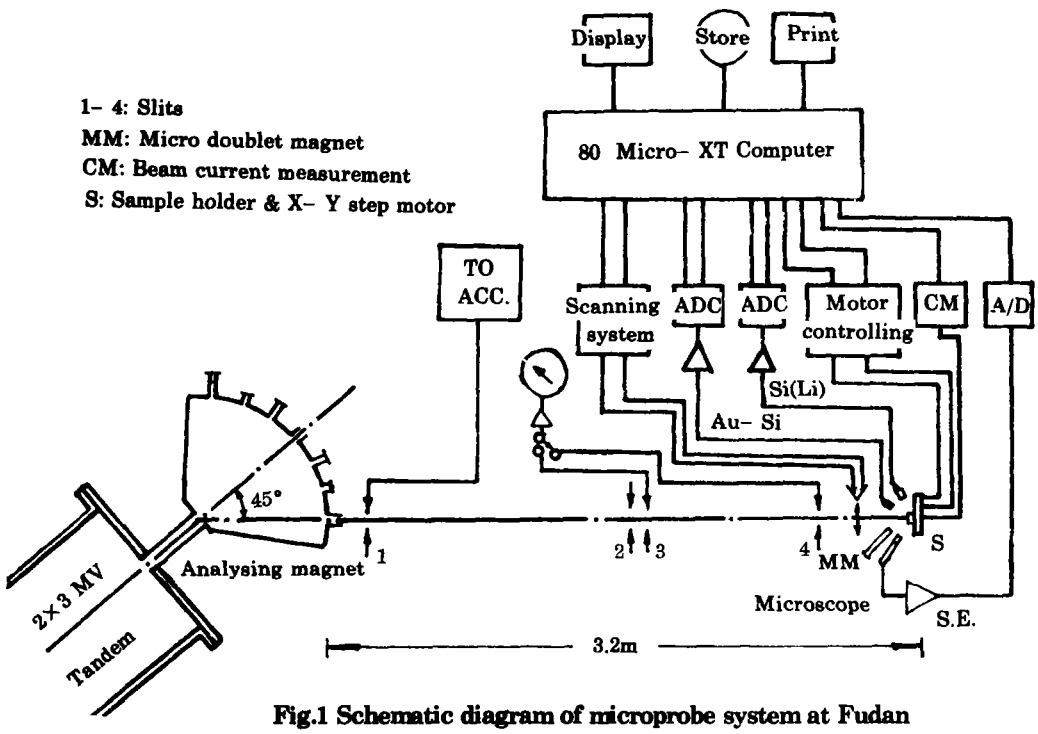


Fig.1 Schematic diagram of microprobe system at Fudan

slit 1 and is focused upon the microslits (slit 3, which is a set of high- precision microslits used as an object slits). A feedback system is equipped between the accelerator and slit 1, thereby improving the energy stability. Slit 2 is used as an aperture to protect the microslits. And slit 4 is used as a divergence slit which can reduce the aberration of the image. Magnetic quadrupole lens doublet suitable for focusing ion beams are offered for sale by the Installation Division of Microscope Associates, Inc., which is now offering a small bore, high field. It is of use for proton and heavy ion microprobes operating in the kilovolt and megavolt region of energy. In order to get a 2 μ m beam focused upon the sample, the microslit is preseted to 10×20 μ m[2][4,5].

The sample holder and X- Y step motor, data read in and spectra acquirement are all controlled by computer. The data collected are stored into a floppy disc directly for later disposal. It can be manipulated to display the spectra extracted in certain area of interest or the distribution maps for preselected elements simultaneously. An updated

program can generate up to 16 elemental maps, which can be shown either 2D or 3D.

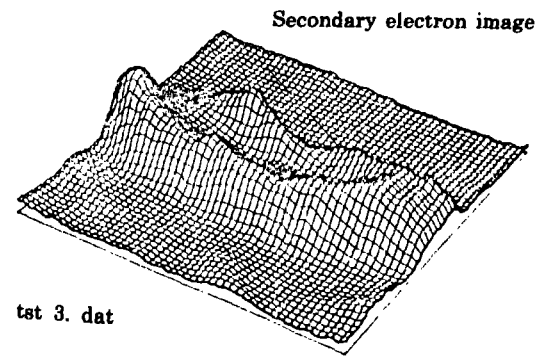
Ⅲ. RESULTS AND DISCUSSION

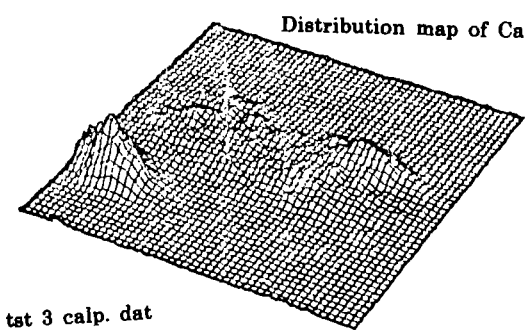
A lot of samples which covered many kinds of subjects have been measured since the set up of the microprobe at Fudan University. Here we introduce some of our work we have done.

1. Human hair cross section

Since the establishment of microprobe in 1970[6], many hair specimen have been measured in 2 dimensions[7]. We have also studied a human hair cross section specimen with the microprobe. It is interesting to see 3 dimensional distributions of elements in human hair specimen.

In Fig.2 *a*), *b*) and *c*) are shown respectively the secondary electron image, calcium and sulphur mapping of the hair cross section. From the figures, it can be clearly seen that the image of hair cross section looks just like a crater. There are less elements distributed inside the hair. Also it seems that sulphur spreads uniformly over the whole section of hair and almost all the calcium concentrated at the surface of the hair.





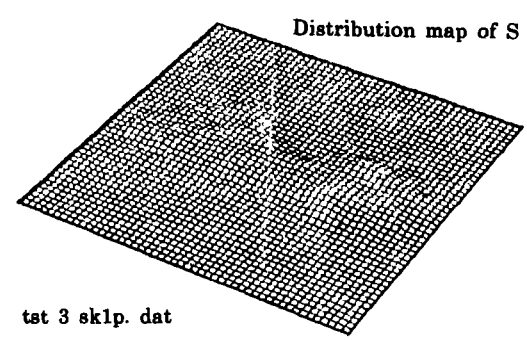


Fig.2 (*a*) Secondary electron image (*b*) Calcium and (*c*) Sulphur distribution of a human hair cross section

Scanned area is $100\times100\mu\ m^2$

2. Creep crack in steel

The microprobe was also used to examine a crack in steel to find out possible causes of the crack formation. Three dimensional Si, Fe and S maps and a secondary electron image of the crack are given in Fig.3 *a*, *b*, *c* and *d*, respectively. The cracks were produced by force. The steel had been pulled under 600kg for 600 h. So we called

the small crack as a Creep Crack. From the secondary electron image of Fig.3, we can see a crack obviously. And we can also see sulphur is mainly distributed inside the crack. The iron profile looks just like the secondary electron image. High level of silicon is also seen at the crack. So it can be sure that sulphur may be one of the reasons to produce a crack in steel.

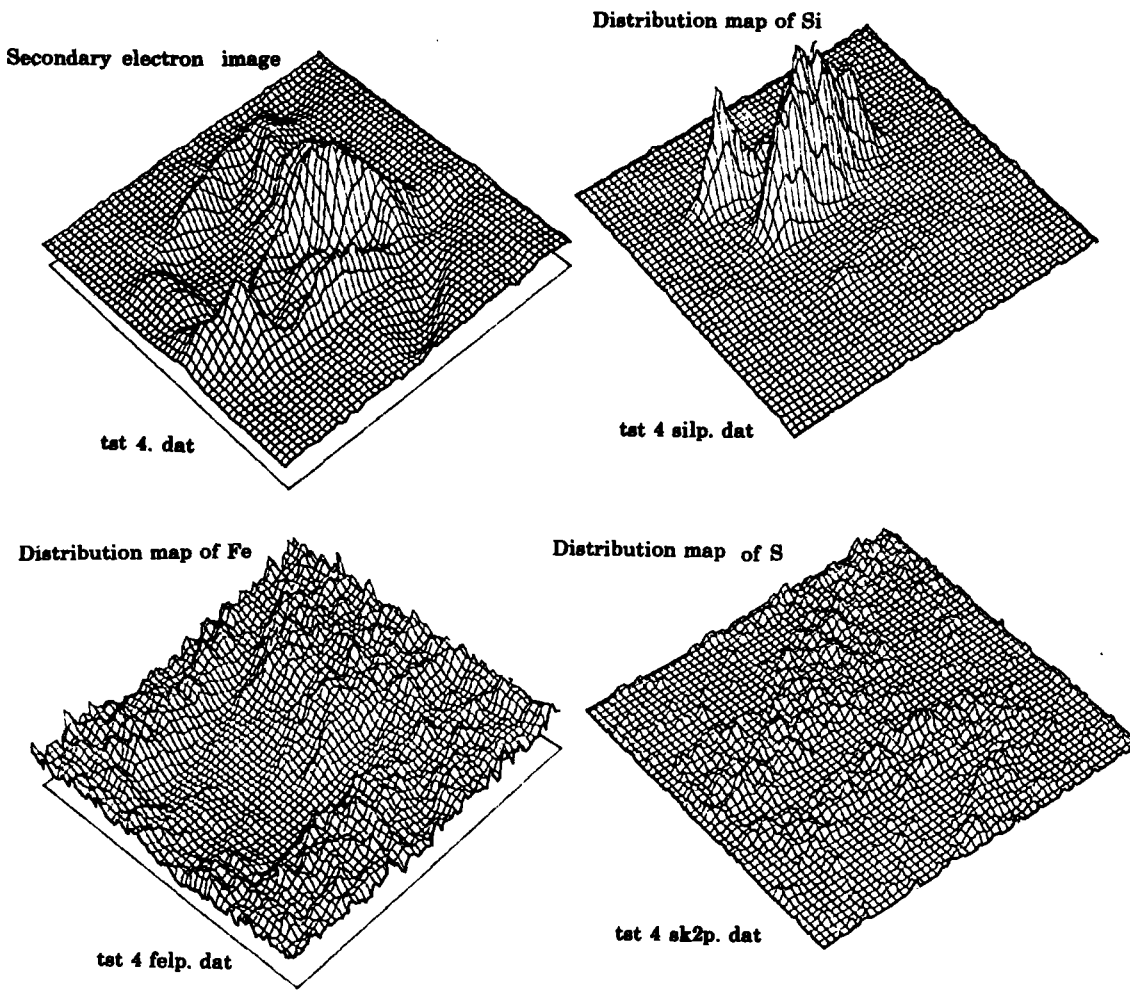


Fig.3 Secondary electron image and elemental maps of silicon, iron and suophur of creep crack in steel

Scanned area is $500\times500\mu$ m^2

3. Ancient copper mirror

Another important application of the microprobe is in archaeology. The copper mirror we have measured was made in Han Dynasty in about 200A.C. It has two sides. The frontside was highly polished to be used as a mirror. On the backside, some beautiful decorative pattern was moulded. We cut a small piece off a broken mirror and polished the cross section to analyse its microstructure. Fig.4 shows 2D elemental maps created by scanning the proton beam over a $500\times500\mu$ m^2 area near the front

edge.

From the elemental maps of Fig.4 we can clearly see that the element tin was adhered to the mirror's surface, the element copper was uniformly distributed while

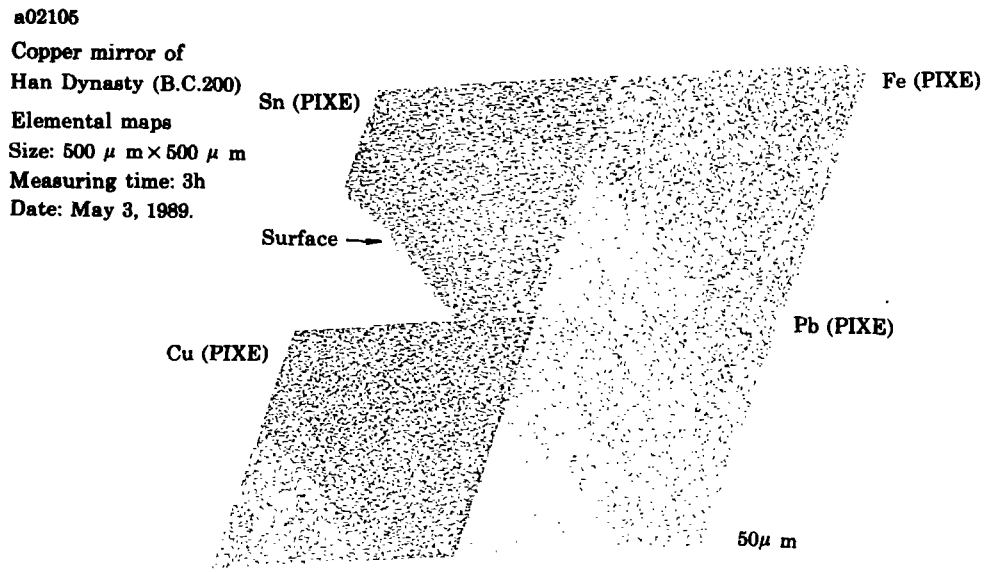


Fig.4 Elemental maps of copper mirror of Han Dynasty

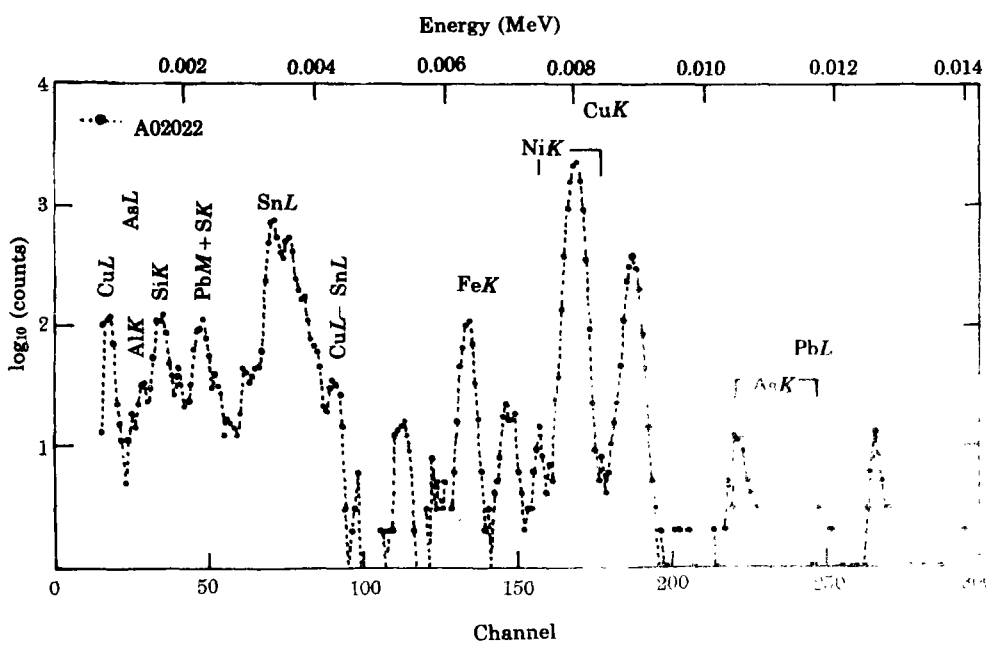


Fig.5 PIXE point spectra of copper mirror's surface

iron was not, and there still exist lead in the mirror body. Fig.5 shows the PIXE point

spectra of a spot at the mirror's surface. We can clearly see that the copper mirror is mainly made up of tin and copper. It contains not only Cu, Sn, Fe and Pb, but also Si, S, Cr, Ni and As. It is a magic thing how ancient people in Han Dynasty could adheretin to a copper mirror's surface, and also how a copper mirror can be preserved perfectly for almost 2000 years. It also shows high level of smelting technology in ancient China.

IV. CONCLUSION

A microprobe with 2MeV proton or helium ions and a minimum beam spot size of 2 μ m was obtained at Fudan University. It is a powerful tool to analyse microstructure of many samples in different fields.

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