SCI. REP. KYOTO. PREF. UNIV. (NAT. SCI. & LIV. SCI.), NO. 40, Ser. A. p. 1~5 (NOV. 1989)

Elemental Mapping with the Uji Proton Microprobe

YOICHI HARUYAMA, ATSUSHI AOKI, MISTUO TOSAKI, MICHIO TOMITA*,

KOJI YOSHIDA* and FUMIO FUKUZAWA*

Abstract

The Uji scanning microprobe system has been improved for elemental mapping. By collimation and focusing with a magnetic quadrupole doublet the proton beam size is reduced to 2×6 μm^2 with currents of $\sim$ 10pA. Elemental mapping was performed by scanning the proton beam in both dimensions across the sample surface and by detecting the secondary electron, X-ray and the scattered charged particle from the surface. Some examples of microprobe measurements are presented.

(Received August 15, 1989)

Introduction

The knowledge on the local concentration and on the spatial variations of the elemental concentrations are necessary in many fields such as biology, medicine, metallurgy, material science and so on. Since the first report on a microbeam with ions of some MeV appeared[1], many other microprobes have been built[2]. Spatial elemental distributions can be measured easily by scanning well focused beam across a sample surface. Especially, scanning microbeam -PIXE (Particle Induced X-ray Emission), -PIGE (Particle Induced Gamma-ray Emission) and -RBS (Rutherford Back Scattering) measurements have been accepted widely as powerful tools for their high sensitivity and spatial resolving power for bulk samples. We have been developing the Uji microprobe system for these four years snd some preliminary results have been published[3-5]. In the present paper, we report on the several improvements during this two years and on the first examples of microprobe measurements for a biological tissue.

Experimental

The experimental arrangement of the Uji scanning microprobe was reported in our previous paper[5] and is not changed fundamentally. We report on the several improvements which have been made during these two years briefly.

A pair of steering magnets for each horizontal and vertical direction were installed upstream of the object aperture to adjust the beam line to the optically aligned magnetic lens system.

In the previous arrangements, according to the Oxford group[6], the beam deflection electrodes were placed upstream of the first quadrupole lens to shorten the distance between the second Q-lens and the target. It was necessary to obtain a high demagnification factor. Two

Laboratory of Applied Physics, Kyoto Prefectural University, Kyoto 606, Japan.

* Department of Nuclear Engineering, Faculty of Engineering, Kyoto University, Kyoto 606, Japan.

dimensional image of RBS scan of a copper grid, however, showed slight distortion with this configuration. Displacement of the deflected beam on the target plane showed linear relation with the applied voltage for each direction. Such distortion was observed only when both vertical and horizontal deflection voltage were applied simultaneously and was caused probably by large displacement of beams from central ray. In the present experiments, small electrodes are inserted for vertical scanning after the second Q-lens to avoid such distortion. This electrodes are designed to deflect 2 MeV proton beam about 1 mm in length with 2kV deflection voltage but not to change the distance between Q-lens and the target. Horizontal scanning was carried out by using the previous electrodes.

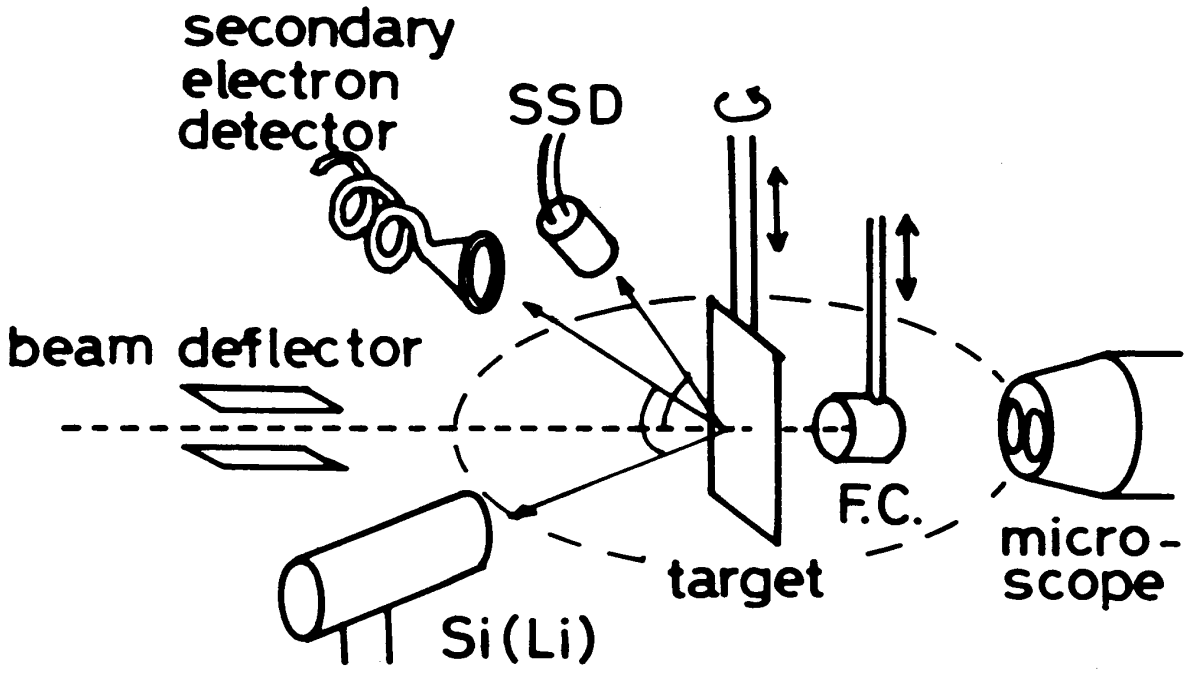


Fig. 1 A schematic diagram showing the layout of the target chamber.

We adopted three types of detectors which can detect secondly electrons (CERATRON), X-rays (Si (Li)) and charged particles (surface barrier detector) to utilize the Uji scanning microprobe for application use. In figure 1 is shown the layout of the target chamber schematically. These detectors are positioned at 135 degree backward from the beam axis. Except for the Si (Li) detector they are mounted in the target chamber. We measured the counting efficiency of these detectors in the present geometrical condition. The output signal of the secondary electron detec tor obtained from a copper grid was about a hundred times greater than those of other two detectors.

According to this improvement of the detector system, the data taking system was improved, too. Firstly, three parameters data taking with multichannel scaling mode became feasible. In the present system, three input signals are processed simultaneously. Secondly, scanning program was revised to shorten the time required for adjustment of a magnetic excitation. To adjust a magnetic excitation of the quadrupole doublet, we measured copper grid images for each direction changing the excitation currents step by step in the past experiments. By using the revised program we can measure both direction images with a single sweep of the beam. Due to employment of the secondary electron detector and the revised scanning program, a beam alignment and a microadjustment of the excitation of two magnetic quadrupoles can be carried out in about half day. In the previous system we need almost two days for this operation.

Results

The primary 2 MeV proton beams are generated in a 4 MV Van de Graaff accelerator at Kyoto Uuiversity. The beam diameter is reduced by collimation and focusing with the magnetic quadrupole doublets. Details of the focusing procedure is described in our previous paper[5]. In figures 2a and 2b are shown the vertical and horizontal spectrum obtained by secondary electron detector for a 250 lines per inch copper grid with the object aperture of 50 μm in diameter. Figure 2b shows that the effective width of the grid in horizontal direction is somewhat longer than the actual width due to the present geometrical condition. It is clearly shown that the secondary electron emission efficiency is enhanced at the grid edge. The beam width for each direction was calculated, however, assuming a Gaussian beam profile and rectangular cross section for the grid by least squares fitting. Solid lines denote the computer fits. Deconvolution calculations show the beam spot dimension of 2 μm and 6 μm for vertical and horizontal plane, respectively.

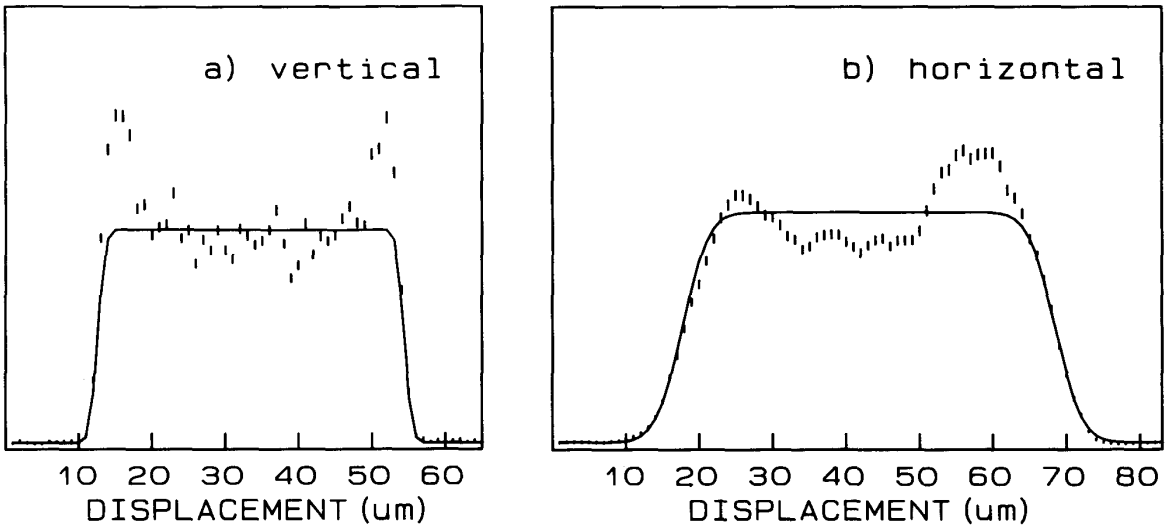


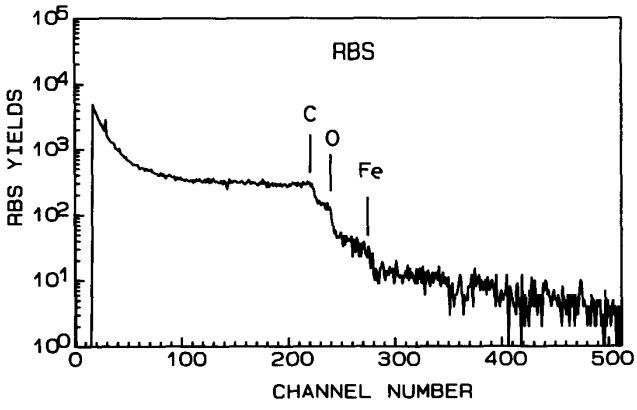
Fig. 2 Secondary electron spectrum for (a) vertical and (b) horizontal scanning. Solid lines denote the computer fit to the measured spectrum. (see text)

We used this microprobe for elemental distribution analysis of biological tissue at the first time. Tooth of *Liolophura Japonica* (LISCHKE) was chosen for a sample tissue because it is well known that it has magnetite in its tooth. A microscopic photo of teeth is shown in figure 3. The tooth sample was extracted and mounted on a thin polycarbonate film. The sample was bombarded with a 2 MeV proton beam. In figures 4a and 4b are shown the RBS and PIXE spectrum of this sample. From energy calibration the highest proton energy scattered with carbon, oxygen and iron atoms are marked in the RBS spectrum. As the sample is too thick, it is difficult to evaluate the absolute quantities of these elements from RBS spectrum. A rough estimation tells that the quantities of carbon and oxygen atom are nearly same and that the main components of the sample are the such organic elements. In the PIXE spectrum, only iron Kα and Kβ lines are clearly observed. From these spectrum, it is concluded that heavy element except for iron was not included in this sample. The results of the two dimensional elemental mapping are shown in figures 5a and 5b. Changing the beam deflection voltage, scanning area was adjusted to cover the sample tissue. Scanning area of these spectrum was 1×1 mm^2. It took nearly three hours to obtain these spectrum. Figure 5a shows the three dimensional image of the sample measured by RBS and gives the all-over shape of the sample.

In figure 5 b is shown the iron elemental distribution measured by PIXE. By comparing these figures, it was found that root part of the tooth is composed by the organic elements and the magnetite is concentrated at the top of the tooth.

Fig. 3 Microscopic photo of teeth of *Liolophura Japonica* (x100) .

The measurement of iron distribution in a tooth of *Liolophura Japonica* (LISCHKE) with the Uji scanning proton microprobe demonstrates the usefulness of such an instrument in combination with RBS and PIXE analysis as a tool for the analysis of biological sample. The authors with to express their thanks to Prof. Y. Maeda at Research Reactor Institute of Kyoto University for his provision of the sample.



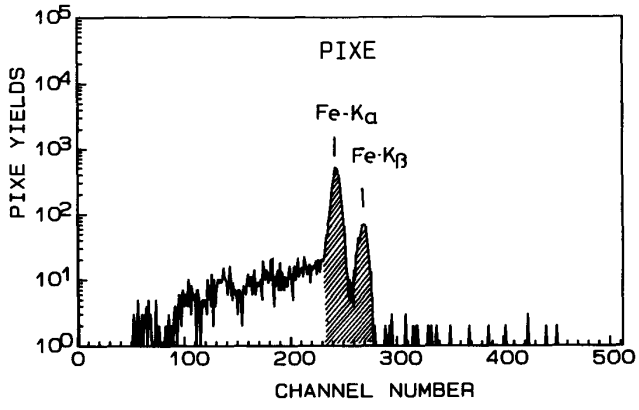


Fig. 4

a) RBS spectrum of the sample tooth.

b) PIXE spectrum of the sample tooth. The hatched area denotes the iron Kα and Kβ lines.

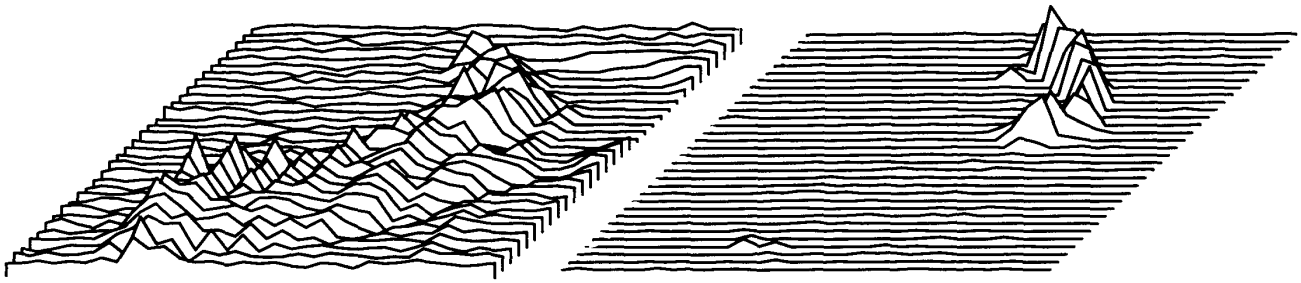


Fig. 5

a) Three dimensional image of the tooth obtained by RBS measurement without energy gate.

b) Mapping of the spatial distribution of the iron concentration in the tooth sample obtained by PIXE.

Reference

1) J. A. Cookson, A. T. G. Ferguson and F. D. Pilling, J. Radioanal. Chem. **12**, 39 (1972)
2) "Proceedings of the Second International Confererence on Chemical Analysis", Nucl. Instr. and Meth. **197** (1982)
3) Y. Haruyama, A. Aoki, K. Yoshida and F. Fukuzawa, Sci. Rep. Kyoto Pref. Univ. (Nat. Sci. & Liv. Sci.), **36**, 9 (1985)
4) Y. Haruyama, A. Aoki, K. Yoshida and F. Fukuzawa, Sci. Rep. Kyoto Pref. Univ. (Nat. Sci. & Liv. Sci.), **37**, 13 (1986)
5) Y. Haruyama, A. Aoki, K. Yoshida and F. Fukuzawa, Sci. Rep. Kyoto Pref. Univ. (Nat. Sci. & Liv. Sci.), **38**, 29 (1987)
6) G. E. Grime, J. Takacs and F. Watt, Nucl. Instr. and Meth. **B3**, 589 (1984)