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V. 4. Recent Application of Tohoku Microbeam System

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Introduction

High energy ion microbeam is a powerful analytical tool by combining various ion beam analysis techniques such as PIXE, RBS, STIM and SEM¹-³) and is also attractive as a direct lithographic technique. A microbeam system was constructed at the Tohoku University Dynamitron laboratory for biological applications in a sub-micron resolution. The first purpose is to develop a 3D micron CT, where microbeam is used as a monoenergetic point X-ray source. The second is to develop microbeam analysis system for biological samples. The microbeam line was installed in July 2002 and optimization of the system has been performed. A measured beam spot size of 0.4×0.4 μm² at a beam current of 20 pA is currently being obtained. This paper describes recent applications of Tohoku microbeam system.

Microbeam and analysis system

The microbeam system was designed to achieve sub-micron beam sizes and was developed in collaboration with a domestic company (Tokin Machinery Corporation)⁴). The system is connected to the 4.5 MV Dynamitron accelerator at Tohoku University. To achieve sub-micron beam seizes, a high resolution energy analysis system is installed upstream of the microbeam line. The microbeam line is composed of a quadrupole doublet, three slits systems of microslits (MS), divergence-defining slits (DS) and baffle slits (BS). These components are mounted on a heavy rigid support with vibration isolation. The microslits (MS) define the object size and are composed of two wedge-shaped slits. The divergence-defining slits (DS) define beam divergence into the
quadrupole doublet and placed 3 m downstream of the MS. As for the quadrupole magnet, the bore radius is 5 mm and dimensions of the yoke are 60 mm long×220 mm outside diameter. An iron piece of the magnet with poles and yoke was cut as one body using a computer numerically controlled machine to reduce mechanical irregularities causing sextupole contamination\(^4\). The demagnification factors are 9.2 and 35.4. The focused microbeam is scanned across a target by using an electrostatic beam scanner.

The target chamber is a rectangular box and applicable to either in-vacuum or in-air analysis without changing the main body. For multimodal analysis, two X-ray detectors and three charged particle detectors can be mounted simultaneously\(^5-7\). For 3D micron CT, a CCD, a rotating sample stage and a target to produce X-rays are also mounted on this chamber\(^8-10\).

**Recent application of the microbeam system**

The microbeam analysis system at Tohoku University is routinely applied to various fields. Typical applications are presented.

* Aerosol study

Elemental analysis of atmospheric aerosol is useful for source identification and for study of its formation mechanism. These studies were carried out by analyzing bulk samples thereby averaging over many single particles. Therefore, analysis of single aerosol particles is superior to bulk analysis. Our system can quantify single aerosol particles containing from hydrogen to heavy metals and reveal chemical composition of these particles\(^8\). The microbeam system was applied to the analysis of yellow sand dust particles. These particles were impacted on a thin polycarbonate film developed for this purpose. Yellow sand dust particles from the Asian continent sometimes cause turbid conditions in Japan especially in spring. Typical elemental images of yellow sand dust particles are shown in Fig. 10.

The elemental distribution of Al is similar the one of Si and Oxygen, originating mainly from soil dust of Alumino-Silicate particles. Distribution of hydrogen is clearly seen and equal to the one of Ca. These particles are sometimes deformed by mixing with anthropogenic aerosols. Then the system will be useful to study these deformation processes.
Cell analysis

An in-air simultaneous PIXE, RBS and a consecutive STIM analysis is a powerful tool for analysis of single cell and tissue section in biological and biomedical research. We have developed an in-air micro-PIXE system in collaboration with the Japan Atomic Energy Agency (JAEC), Takasaki\textsuperscript{11,12} and has applied to various kind of cells\textsuperscript{13-15}. Our in-air analysis system is also focused on this purpose\textsuperscript{7}. While in-air analysis reduces the damage during beam irradiation\textsuperscript{16}, morphological change has occasionally occurred by beam irradiation, and cell thickness or density is reduced to ~60 \% of the initial value\textsuperscript{7}. In this case, density of the cell should be monitored simultaneously with analysis and correction of X-ray self absorption should be done according to irradiation dose. On/off-axis STIM enables to perform simultaneous density imaging in PIXE and RBS analysis. Figure 12 shows elemental maps of cells and density map measured by the new in-air system. Distributions of P, S, Cl and K elements are clearly seen and correspond to the density. Changes in cell density during the beam irradiation can be monitored and will be effective in cell analysis.

Proton beam writing

Proton beam writing was realized by scanning the microbeam with a corresponding scanning pattern translated from a bitmap file. The spacing between pattern to pattern was made by moving the beam at the fastest speed of the beam scanner. Details of the system will be written elsewhere. Typical pattern written on a Mylar film is shown in Fig. 13. While this pattern was written before removing the field contamination from the annular detector, shape of Japan Island are clearly seen. We are planning to use this technique for an efficient analysis by combining STIM image.

3D-micron CT

3D micron CT is composed of a point X-ray source, a rotating sample stage and a high-speed X-ray CCD camera. The details of the 3D micron CT and applications are written precisely in refs 9, 10, 11. Microbeam is used as a monoenergetic $\mu$ X-ray source by bombarding pure metals. 2D transmission data are obtained by rotating the sample encapsulated in a micron tube. 3D images were reconstructed from the obtained transmission data. 3D images of living ant’s head were obtained by this system with the spatial resolution of 6 $\mu$m. Typical CT image of ant’s head is shown in Fig. 14. Since
our micron CT uses monoenergetic low energy X-rays, contrast of the image for small insects is superior to that of other CT system. Furthermore, our system can easily change X-ray energy to get better contrast depending on the elements contained in the insects. This system can measure small insects of ~2 mm in-vivo and will be useful in the biological field.

**Conclusion**

A microbeam system was constructed at the Tohoku University Dynamitron laboratory and optimization of the system has been carried out. Minimum beam spot size of the microbeam was saturated at $1.5 \times 1.5 \, \mu m^2$ by parasitic field contamination. This parasitic field contamination was ascribed to the tungsten carbide slits and the annular Si surface barrier detector in the case of RBS analysis. By replacing these components, the parasitic field contaminations of the system were greatly reduced, which was confirmed by the grid shadow method, and the performance of the microbeam system was improved. Minimum beam spot size of $0.4 \times 0.4 \, \mu m^2$ at a beam current of several ten pA is being obtained which is the optimum expected from the accelerator performance. The results obtained by the grid-shadow method show that a beam spot size less than $0.4 \, \mu m$ can be obtained in the low current range.

For an easy tuning and an operation of the microbeam system, the human-machine interface of the control system has been developed based on the user participatory design concept. The operational log of a skilled operator has been utilized to realize the HMI, which is user-friendly and has higher usability both for novice and experienced users. The new HMI has been favorably accepted by the users and is utilized in routine operations.

While improving the microbeam system, simultaneous in-air/vacuum PIXE, RBS, SEM and STIM analysis and 3D micron CT are developed and are now being applied to biology and other fields.

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Figure 1.  Elemental maps of yellow sand dust particles.

Figure 2.  Elemental maps and density distribution of cells.

Figure 3.  Optical image of pattern written by microbeams.

Figure 4.  Cross sectional view of ant’s head.