# Characterization of external micro beam produced by glass capillary optics and its application

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***Abstract:*** **A cost effective external microbeam system developed by a tapered shaped glass capillary optic is characterized. We have observed that the ion beam is successfully introduced to the atmospheric environment without any difficulties. The time dependent integrated current shows the satisfactory stability. The high intensity can be obtained few millimeters apart from the exit window of the capillary where Si X-ray produces the focal point. Analyzing different specimens in air, the qualitative result indicates that the system has a merit for elemental analysis.**

***Keywords: Microbeam, Glass capillary, External beam, Qualitative analysis.***

## I. INTRODUCTION

The aspect of developing external microbeam system is to analyze elemental concentration of micron size sample in air. Micro-PIXE analysis is one of the popular examples of this system which is applied on various samples to determine the elemental concentration [1-3]. There are huge attempts have been taken into account to produce external PIXE setup from several years by different research groups [4-6]. The most popular method for producing external microbeam is to use a very thin polymer or metal foil between vacuum and air. In this technique, the high energy ion beam penetrates the exit foil and hits the target in the air. The beam has enough energy and flux intensity for analysis. However, it has a big risk to damage the accelerator when the foil is broken. Several elegant works have been reported for producing microbeam using glass capillary optics for XRF and XRD analysis [7-9]. In those systems, incoming X-ray beams is reduced in size as well as focused by outlet diameter of the capillary.

The facility of ion beam laboratory at Kochi University of Technology (KUT), Japan is extended to produce an external microbeam using glass capillary optics. This optic is an interesting and useful lens for ion beams. The focusing system in this case is completely different contrast to conventional one. Slightly tapered glass capillary optics with a few micrometers of outlet size is placed between vacuum and atmospheric environment. The capillary works as a differential pumping orifice as well as a focusing lens. This is a very simple technique to produce different size of microbeam by changing the capillary only within a few minutes. It can be pointed out that the energy loss of ions during transmission is negligibly small. The capillary is originally designed as an artificial channel for ions and the mechanism of ion deflection at the wall surface is probably by the electric charge effect or guiding effect [10]. So the reason for doing this research is to characterize this cost effective external microbeam system and its applicability.

## II. EXPERIMENTAL SETUP

### *A. Glass capillary:*

The inlet and the outlet diameter of the used glass capillary were 0.8 mm and 10-20 μm, respectively. The length was few cm. The taper angle was kept approximately 5 mrad. The detail fabrication procedures of the glass capillary optics have been reported elsewhere [11]. To fabricate the capillary we used borosilicate glass tube with inner and outer diameter of 0.8 mm and 1.0 mm, respectively. Borosilicate glass is less dense than ordinary glass and makes by silica and boron oxide. This glass consists of 81% m/m $SiO_2$, 13% m/m $B_2O_3$, 4% m/m $Na_2O$, and 2% m/m $Al_2O_3$ [12]. It begins to soften around $821^0$C ($1510^0$F) at room temperature and density is 2.23 g/cm$^3$. The fabrication procedure is very simple and cost effective. It is possible to make various diameter of capillary outlet easily. After fabrication of glass capillary, it was molted into an aluminum pipe. Diameter and length of the aluminum pipe were 1 mm and 5 cm, respectively. Both side of aluminum pipe were pasted with superglue to prevent of air passing.

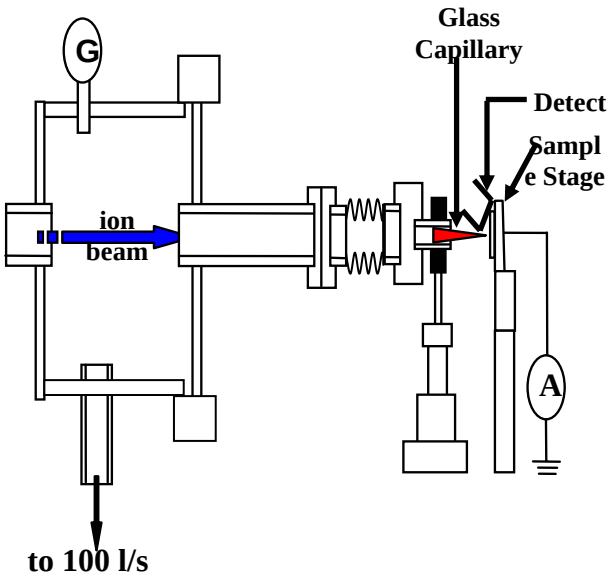


Figure 1: Experimental setup of external microbeam at KUT.

*B. Beam line:*

A 1.7 MV tandem accelerator at the Kochi University of Technology, Japan was used to carry out the present development. Douplasmatron ion source was fixed up. The microbeam setup was installed on the $-15^0$ line of the accelerator. The glass capillary mounted on a 4-axis goniometer. The x, y positions and the tilt angle of the capillary with respect to the ion beam line was controlled by the goniometer. A 100 l/s turbo-molecular pump was used as the differential pump. The pressure of the chamber was kept approximately $10^{-4}$ Pa before capillary experiment. The ion beam was collimated to 2mm x 2mm by a 4-jaw slit. The measured ion beams current before and after the capillary was 10 nA and 100 pA, respectively. This indicates that an enhancement of the beam flux intensity is by ~2 orders. The experimental setup is shown in figure 1. The capillary was only 1 mm or less apart from the sample surface. Detector to sample distance was tried to keep as close as possible but it was limited to approximately 2 cm due to the detector housing diameter. Gate valve can isolate the differential pump from the air environment when there is no capillary. Therefore, it is possible to make different size of external microbeam just only replace the capillary with desire diameter within a few minutes. Generally 2-3 MeV proton beam is used for PIXE analysis but in the present study 4MeV $He^{++}$ (Helium) beam was used because of some regulations in our laboratory.

*C. Acquisition system:*

The sample stage places on a three axis stage. Though there is no obstacle or any other equipment in front of capillary, enough space is available to place and analyze any size of sample (i.e. micro to macro size). Therefore, the sample was kept as close as possible to the capillary exit window for analysis. The collected charge was measured by current integrator from the sample holder which was served as a Faraday cup. The characteristic X-rays are detected by a RÖNTEC XFlash 2001 detector capable to detect low and medium high-energy X-rays. The detector is positioned at ~$135^o$ to the beam line. Detector is a silicon drift type, having a resolution of 139 eV with 1 μs shaping time at 5.9 KeV. The silicon thickness and the active area of the detector are 0.3 mm and 10 $mm^2$, respectively. A beryllium window of 8 μm thick coated by polymer is equipped with the detector. The collimator in the detector is made by zirconium with an aperture of 3.4 mm. The detector was connected to a high resolution pulse processing unit with a single cable. Detected signal was amplified by an ORTEC 572 amplifier which was coupled to the pulse processing unit. The output of the ORTEC 572 amplifier was then connected to a computer via an analog to digital converter (ADC) and a multi-channel analyzer (MCA). The data acquisition was configured into two ways: a front-end computer with MCAWIN software system for real-time data-acquisition, and a back-end system for data collection from the detector via processing unit, pre-amplifier and multi-channel analyzer (MCA).

## III. CHARACTERIZATION

*A. Beam Profile*

In present study, the beam profile was examined using a capillary of 10 μm outlet diameter. The Cu target was placed 1 mm apart from exit window of the capillary to measure the actual beam profile. The measured x intensity profile was obtained by fitting the experimental data with a function resulting from the convolution of Gaussian. The FWHM of the Gaussian best fit for x is approximately 10 μm which represents the width of the beam shown in figure 2. The beam divergence is roughly estimated about 7 mrad.

Figure 3 shows the ion ranges passing from capillary outlet and travels through in air as a function of distance. The range gradually decreases with respect to distance, indicates that the ions are losing their energy in air environment. SRIM simulation result suggests that the 4MeV $He^{++}$ ion can travel in air is about 25.21 mm. Therefore, our observed result shows good agreement with simulation one. Suggest that the capillary is capable to pull out the beam even though the beam is micron size.

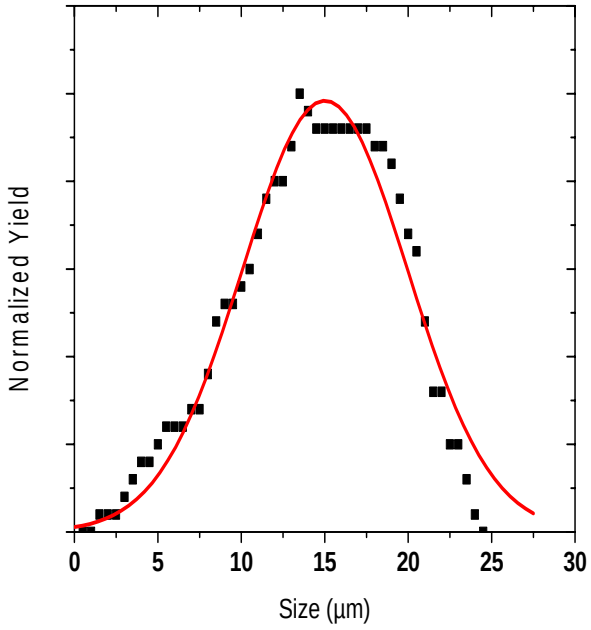


Figure 2: Estimation of beam size produced by 10µm capillary at energy of 4MeV $He^{++}$.

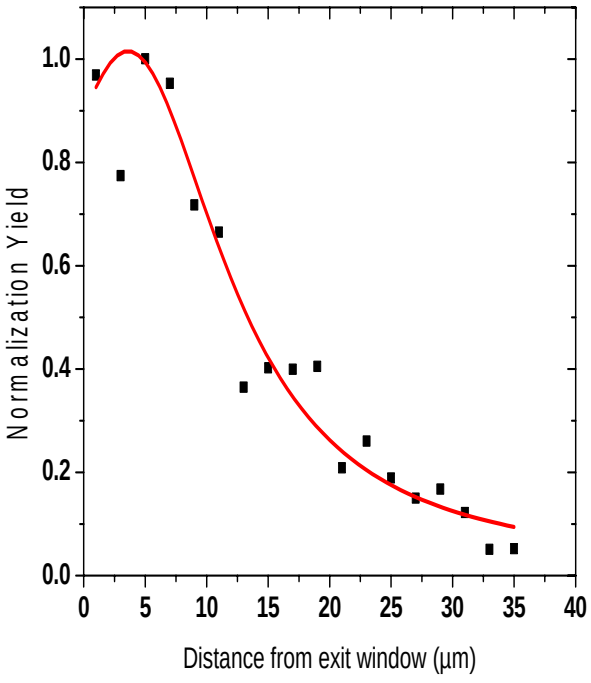


Figure 3: Stopping range of 4MeV $He^{++}$ ion beam in air.

*B. Stability of the outlet current*

The stability of beam current is one of the most important factors for external ion beam analysis. Due to constant pressure, beams can propagate smoothly without any interference in vacuum. However, in atmosphere beam cannot propagate easily and face some difficulty. Therefore, low energy ions especially for micro beams will be scattered, loss the energy in air and interrupt the propagation of ion beams. Tokihiro Ikeda et al have reported the stability of transmitted ion as a function of time with an 8 KeV $Ar^{8+}$ for a fixed distance [13]. We investigated the stability of ion beams with various distance as a function of time from exit of the capillary outlet.

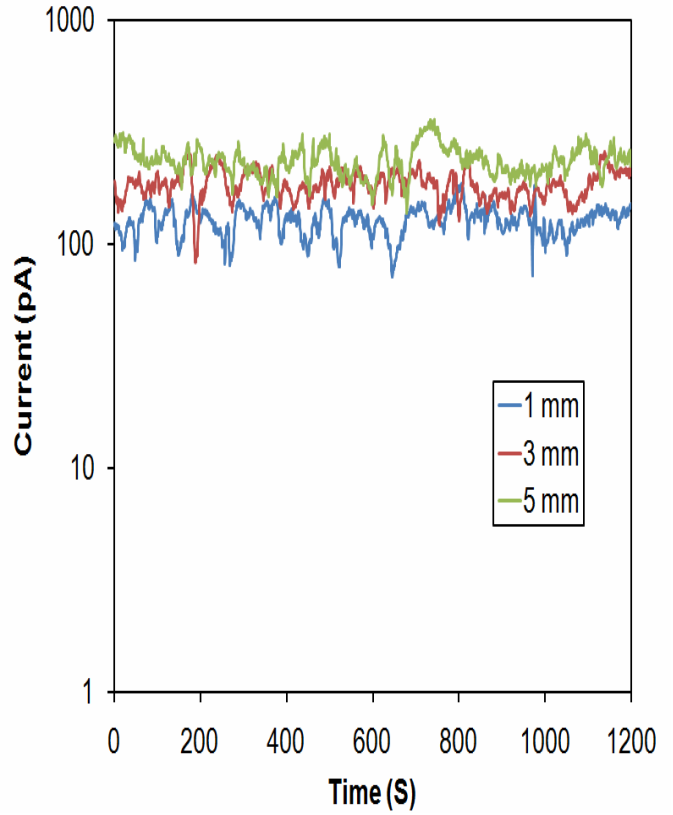


Figure 4: Integrated current with respect of time at different distances from the exit window of the capillary.

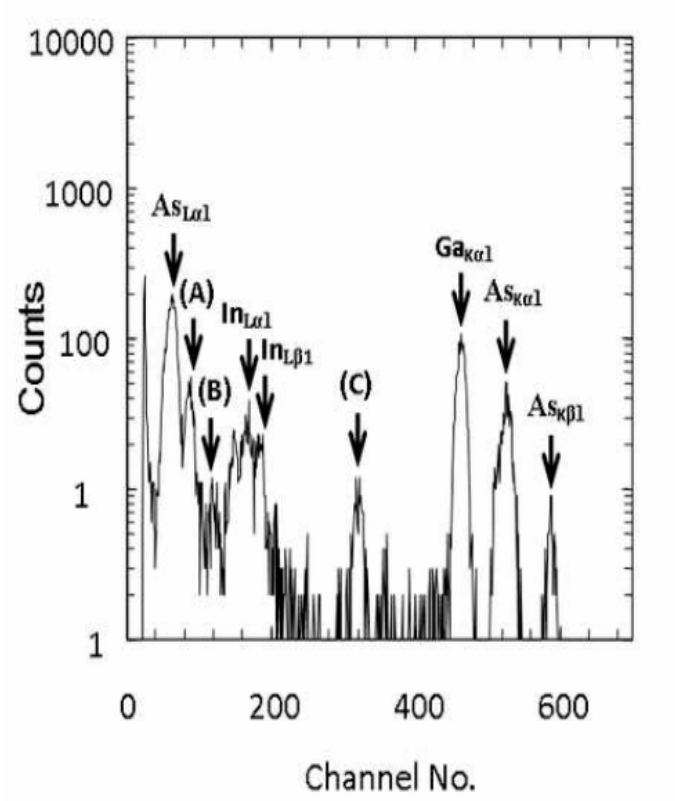


Figure 5: PIXE spectra of GaInNAs sample obtained with a 4 MeV $He^{++}$ in-air. The ion beam dose is 1.4 μC.

Figure 4 shows the transmitted ion intensity as a function of time for tapered glass capillary with a 4 MeV $He^{++}$ ion beam. We measured the current at three different distances from the capillary outlet with same energy. External microbeam produces very low current about several pA. Therefore, it is required to analyze the sample for longer time to provide certain beam dose. So, it is quite important to examine that whether external beam current is stable or not for a long period. We considered 1200 S but more than that is also possible. It is seen from this figure that the current is almost stable with respect to time. Careful monitoring of the beam parameters can increase the stability of the current throughout the measurement. The intensity of beam current is increased with respect to distances from capillary outlet which is seen in this figure. We suspect that the capillary works as a focusing lens and focal point is little away from exit window. It can be explained that the highly charged ions hit the inner wall of the capillary and produces some Si X-rays. These X-rays propagate through the capillary and make a focal point together with $He^{++}$ ions at a certain distance from capillary outlet. As a result we found high intensity at focal point. It can be obtained higher beam dose if the sample is placed at the focal point.

## IV. Result and Discussion

The elemental composition and concentration are very important for analysis of a material. We analyzed $Ga_{0.7}In_{0.3}As_{0.99}$ crystal sample to examine the feasibility of external micro- PIXE system. For doing this crystal sample was mounted on a ~12 μm thick carbon foil and was irradiated by 4 MeV $He^{++}$ ion beam. Figure 5 shows the resultant spectrum. The designated elements of crystal are shown in the figure. The system was calibrated at 20 eV per channel, prominently shown $As_{L\alpha 1}$ (1.28 KeV) at channel no 64.

However, it can be seen that it has three extra peaks indicated as (A), (B) and (C) those are not related to the crystal. The X-ray energy of peak (A) clearly indicates that it comes from Si. This is probably from the glass capillary itself. Three possible reasons can be classified for this peak as shown in figure 6. (i) The $He^{++}$ ions with 4 MeV energy have the stopping range in borosilicate glass is about 14 μm calculated by SRIM software. The wall thickness near the outlet of used capillary is less than 10 μm. Therefore, Si X-rays emitted inside the capillary have some possibilities to penetrate the wall and come towards the detector. (ii) X-rays generated inside the capillary come out through the outlet and reflect at the sample surface. (iii) Scattered ions can hit the outside of the capillary and comes towards the detector. The X-ray energy of peak (B) corresponds to Ar because the presence of Ar gas in the air. On the other hand, peak (C) clearly indicates as the energy peak of Fe. Though the exact reason of Fe is not cleared but we suppose that this is again due to scattered ions or reflected X-rays. This unique technique has a great advantage to produce external microbeams easily with a minimum cost even though it has some demerits.

Application of this external PIXE setup has been investigated and qualitative results are presented, which demonstrate the applicability of this method in different areas. We analyze seabed sludge as an environmental sample. The system can provide the facility to measure the wet or liquid sample directly without any sample preparation. Trace elements are tabulated in table. It can be seen from table that this system detects less elements for both dried and wet sludge than vacuum. It can be explain in terms of low beam dose and atmospheric interference for in air measurement. It is also seen that few elements is absence in wet sludge in contrast to dried sludge. We measured the wet sludge directly without any sample preparation. Most of elements of sludge are diluted and dissolved in water. Therefore, when beam hits the wet sludge, some flux of ion beam is absorb by the sample.

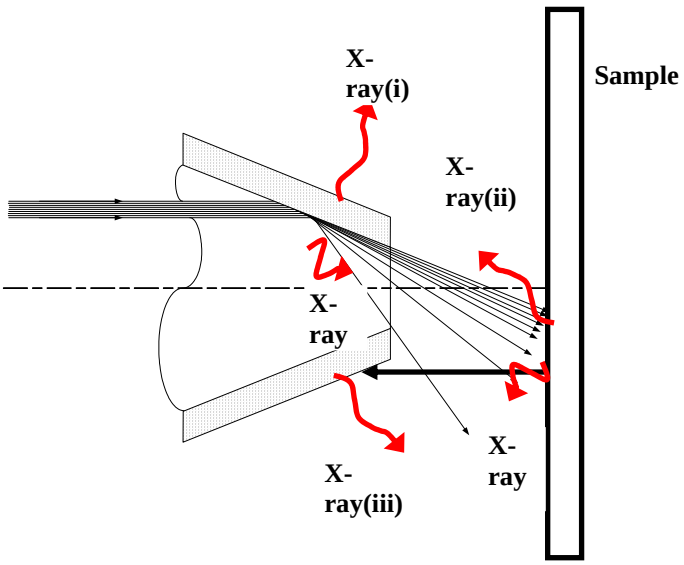


Figure 6: Possibility of Si X-ray generation due to glass capillary itself.

TABLE: LISTED THE ELEMENTS DETECTED BY THE EXTERNAL SYSTEM IN AIR.

| Elements | In Air | | | In Vacuum |
|---|---|---|---|---|
| | Dried Sludge | Wet Sludge | Shell fish | Dried Sludge |
| Si | √ | √ | √ | √ |
| S | | | √ | √ |
| Cl | √ | √ | | √ |
| K | √ | √ | √ | √ |
| Ca | √ | √ | √ | √ |
| Ti | √ | | | √ |
| Cr | | | | √ |
| Mn | √ | | | √ |
| Fe | √ | √ | | √ |
| Ni | | | | √ |
| Cu | | | | √ |
| Zn | √ | | | √ |

Beam intensity becomes weaker inside the sample. As a result, beam cannot bombard the heavy elements atom. The technique is also used to analyze the elements of biological sample such as shellfish without any sample preparation. The elements from Na to Ca have been detected by the system. Though shellfish contains many others heavy elements. However, it is required the sample preparation as well as higher beam dose to detect other elements from the shellfish.

## V. CONCLUSION

A unique external microbeam system is characterized using a glass capillary optics. The in-air PIXE spectra can be easily obtained from virtually any type of samples such as solids, liquids and gages. The beam profile and stability show a justification of perfect in-air microbeam. Results suggest that the present facility is certainly useful for PIXE analysis of various samples that are not compatible with the vacuum environment.

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