Application of keV and MeV ion microbeams through tapered glass capillaries

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Abstract. We have developed a method to produce micrometer-sized beams of keV energy highly charged ions (HCIs) and MeV energy protons/helium ions with tapered glass capillary optics for the applications of micrometer sized surface modifications and a biological tool, respectively. The transmission experiments of keV HCIs through the glass capillaries show a density enhancement of about 10, beam guiding up to 5°, and the extracted beam keeping the initial charge-state. The combination of MeV ion beams and the capillary with a thin end window at its outlet was used for the irradiation of a part of nucleus of a HeLa cell in culture solution. Escherichia coli cells are irradiated by MeV proton microbeam to determine the minimum dose to stop the single flagellar motor. Scanning irradiation of polymer surface by the beam extracted from the capillary in solution containing acrylic acid was found to provide a deposition layer with large affinity with water.

1. Introduction
Ion beam irradiation has been a common technique in not only physics experiments but also other fields as tools for surface modification, material analysis, investigation of radiation effect in biology, and so on. Ions with keV energy can affect only the surface layer of a sample in vacuum, and ions with MeV energy can be extracted to air or liquid. In the case of the keV energy beam, highly charged ions (HCIs) accelerated by 100 V-10 kV (slow HCIs) are effective for the above purposes. Slow HCI can transfer its large potential energy to only the surface of the sample because it cannot be implanted deeply into the sample. The potential energy is sufficiently large to affect the surface, e.g., that of an Ar^{8+} is about 600 eV. Therefore slow HCIs have high ability to modify surfaces and cause efficient sputtering. Once a micrometer-sized beam (microbeam) is available, these functions specific to slow HCIs can be used to realize, e.g., micro-patterning of modifications and element-sensitive micro-imaging. However, microbeam of slow HCIs is not yet practically available. The use of collimator implies not only the drastic reduction of the beam intensity but also the charge transfer and the energy loss of the ions due to the touching the inner edge of collimating aperture. If the lenses are added, a good emittance of the initial beam is also required. In order to overcome the problems, a different type of optics is needed.

In the case of MeV energy beam, a few MeV protons or He ions are suitable for obtaining the range from 1 μm to 100 μm in liquid, which is almost the same size of a living cell or an organelle in a cell.

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The other advantages of this energy region are that the accelerators are relatively smaller than those for higher energy physics and the radiation level in the experimental area is lower. However, it was difficult to deliver the ions with a few MeV to a living cell in culture solution. Therefore, a delivery method of microbeam to a target in a liquid is required.

For the requirements above, we have developed a method to provide microbeams of keV and MeV ions employing tapered insulator tube whose outlet size is in the order of micrometer. When a beam is transmitted through the tapered insulator tube, the outgoing beam size will be the same as the outlet size of the tube. We selected glass as insulating material. In the present article, we denote the tapered glass tube “single tapered glass capillary” or just “glass capillary”. In the second section, the single tapered glass capillary is briefly explained. In the third and fourth sections, some experiments using slow HCIs and MeV energy ions are described, respectively.

2. Glass capillary

Glass capillaries are sometimes known as glass pipettes for microinjection or glass electrodes for measuring the changing voltage potential within the neurons in biological and medical fields. A number of glass capillary sizes and several kinds of the glass materials are commercially available for these purposes. The first use of the glass capillary in a beam transmission experiment was reported by Narusawa’s group in Kochi University of Technology. They found that a He⁺ microbeam extracted from a glass capillary with the outlet size of 0.3 μmφ kept its initial kinetic energy of 2 MeV [1]. In the case of keV energy ion beams, the transmission through a glass capillary with the outlet size of 24 μmφ was observed for the first time in RIKEN [2] by the present authors. The glass capillaries we use are made of borosilicate, which is typically composed of SiO₂: 80.9 %, Al₂O₃: 2.3 %, B₂O₃: 12.7 %, Na₂O: 4.0 %, K₂O: 0.04 %. Its density of 2.23, which is almost the same as silica glass, is hard enough to prevent the slow HCI beam from penetrating a capillary wall with a thickness of less than 1 μm under the condition of oblique incidence. Soda lime glass capillaries are also used in our experiments. The material consists of SiO₂: 70%, Na₂O: 11%, CaO: 7.0%, and so on. The softening temperatures are 821°C and 700°C for borosilicate and soda lime glass, respectively. The tapered capillary is prepared by heating a straight glass tube, whose dimension is 2 or 3 mmφ in outer diameter, around 1 mmφ in inner diameter and 90 mm in length, and then stretched by pulling both ends with a constant force to have an outlet size of from a few tens μm down to submicron in diameter $D_{\text{out}}$ as shown in figure 1. Any size of $D_{\text{out}}$ larger than 1 μmφ (figure 1(d)) is obtained by a device “microforge” and the capillary taper angle is adjusted by heater temperature and pulling force of another device “puller”.

![Figure 1](image-url)
Both devices are commercially provided by NARISHIGE, Inc. Figure 1(e) is a photo of an outlet modified by a focused ion beam device.

3. Microbeams of slow HCI with glass capillaries and Teflon tube optics

Transmission of slow HCI beams through a single tapered glass capillary can be explained as follows: The incident ions entering the capillary hit the inner wall and consequently cause it to become charged, although the ions stop at the hitting point. When the accumulated charge becomes large enough to prevent the following incident ions from touching the inner wall, the ions travel more or less parallel to the wall, and then some of the ions can exit from the outlet. Although some entering ions do not transmit, this has the following advantages. (1) Extracted beam keeps its initial charge state and kinetic energy. (2) The taper can enhance the density of the output beam. (3) The size of the output beam is the same as that of the outlet diameter. Considering these points, single tapered glass capillaries are one of the feasible techniques to produce a microbeam of slow HCIs. This technique is based on a self-organized charge up process of the inner glass wall mentioned above, which was experimentally demonstrated by Stolterfoht et al. [3] using insulator capillaries with a 3 keV Ne\(^{7+}\) beam. In the experiments, polyethylene terephthalate (PET) foils with multi-capillaries of 100 - 200 nm\(\phi\) in diameter and 10 \(\mu\)m in length were employed. In order to explain the phenomenon, some model calculations have been carried out [4,5].

3.1. Transmission properties for slow HCIs

Using the guiding process, the first microbeam by a single tapered glass capillary was obtained with 8 keV Ar\(^{8+}\) as mentioned in the previous section [2]. Figure 2 shows the number of the transmitted ions as a function of time using an injected beam of 0.2 pA (~1.5x10\(^5\) Ar\(^{8+}\) ions/s) with a stability of ~10\%, where the vacuum level of the experimental chamber was ~10\(^{-5}\) Pa. The capillary with \(D_{out} = 24\; \mu m\phi\) was used in this experiment. The number of the transmitted ions grew slowly with a time constant of several tens of seconds (charge-up time), and then became more or less stable for more than 1200 s. However, no transmission was obtained in the first a few seconds as shown in inset. There was a dip around \(t = 9\; s\), which has been sometimes observed in experiments. In order to understand the structure, theoretical works have been carried out using the measured shapes of capillaries [6].

![Figure 2](image)

The maximum intensity in figure 2 was about 1600 cps (counts per second). The corresponding transmission efficiency was about 1\%. The density enhancement factor defined by the ratio of \(N_{out}/S_{out}\) to \(N_{in}/S_{in}\) is estimated to be ~10, where \(N_{out}\) is the number of transmitted ions, \(N_{in}\) the number of injected ions into the capillary, and \(S_{out}\) and \(S_{in}\) are the geometrical outlet and inlet cross sections of the capillary, respectively. The factor more than 1 means that the optics makes the beam density higher (focusing effect). This factor depends on the taper angle, the outlet size and the injected current. The angular divergence of the transmitted beam was estimated to be ~5 mrad. The beam transmission was
examined when the capillary was tilted stepwise by 1° from −5° to +5° (= 87 mrad) relative to the axis of the injected beam of ~0.01 pA keeping the capillary inlet position. The extracted beams were obtained for all the tilting angles by using a position sensitive detector (PSD) downstream of the capillary. This proves that the beam was well guided in the direction of the capillary tilted by as large as 100 mrad (guiding effect). It was noted that the deflection angle is an order of magnitude larger than the half opening angle (~8 mrad) of the tapered capillary when the cross section of the capillary along its axis is assumed to be a trapezoid. And the divergence of the injected beam was a few mrad. The charge-state distribution of the transmitted HCIs through the capillary without tilting was also measured by a combination of the PSD and a deflector between the capillary outlet and the PSD. There was no indication of charge-changed ions on the PSD but some background events, i.e., the ions were transmitted along the capillary without changing the incident charge state. To realize a sub-micron beam, extraction from the outlet of \( D_{\text{out}} = 900 \text{ nm} \) was obtained for an \( \text{Ar}^{8+} \) beam accelerated by 8 kV (corresponding to the energy of 64 keV). The vertical and horizontal divergences of the transmitted beam were 2.0 mrad and 2.7 mrad, respectively \[7\]. There have been some reports for slow HCI transmission experiments with \( \text{Ar}^{8+} \) of 80 keV and \( \text{Xe}^{23+} \) of 230 keV \[8\], \( \text{I}^{q+} \) (\( q = 10 - 50 \)) of 3 keV/q \[9\], \( \text{Ar}^{9+} \) of 4.5 keV \[10\], \( \text{Ar}^{8+} \) of 8-60 keV \[11\], and so on. Not only the stable transmission reported above but also unstable behaviour has been sometimes observed especially with higher incident currents. Figure 3 shows transmitted ion intensity through a 4 \( \mu \text{m} \) outlet for 64 keV \( \text{Ar}^{8+} \) as a function of time. The incident current was kept around 20 pA. The plot is an extracted view of 5 minutes (\( t = 600 - 900 \text{ s} \)) from a 25-minute measurement. Several spikes were observed with approximately constant transmission of 4,000 cps, whose corresponding density enhancement factor is ~10. This fluctuating behaviour is possibly due to the higher incident current comparing with that of figure 2. Although the transmission is supported by a self-organized charge up, discharge process must contribute to the charge distribution. Considering the almost constant input charge onto the surface, the spikes in transmission are expected to be due to a dynamically resistive change of the glass.

In order to investigate the resistive switching of bulky glass under irradiation of slow HCI, a guiding experiment using an \( \text{Ar}^{8+} \) of 104 keV beam through a gap (0.1 mm) of a paired glass plates was performed and compared with a hysteretic resistive switching model \[12\]. We observed a slowly and regularly oscillating transmission current through the gap during an injection of a steady beam. The oscillation frequency was almost proportional to the injection current intensities. This phenomenon can be attributed to the charge–discharge iteration cycle with dynamic resistive change of the glass plate, which includes switching like insulator-metal transition according to the electric filed induced by the deposit charge from the input beam. The model calculation was applied to bulky insulator sample for the first time and well reproduces the experimental observation.
3.2. Beam guiding with flexible Teflon tubes

Based on the guiding effect of the glass capillary, a guiding experiment by larger angles for Ar$^{8+}$ of 8 keV beam through flexible Teflon tubes is in progress [13]. Figure 4(a) shows a schematic drawing of the sample setup. Teflon tubes with outer diameter of 2 mm and inner diameter of 1 mm were used. The sample tube was placed inside a groove having a fixed curvature machined in the holder. The definition of the bending angle $\phi$ is given in figure 4(a). The tube holder had three grooves of different curvatures and one for a straight tube. The tested bending angles were $\phi = 9.6^\circ$, $17.5^\circ$, $26.7^\circ$, and $0^\circ$, which correspond to the radii of curvature $R = 270$, $150$, $100$ mm, and straight tube, respectively. The holder was made of aluminium and the grooves were filled with an electrically conductive paste while inserting the Teflon tube. Both ends of each tube, i.e. the faces of inlet and outlet but not inside, were also coated with the conductive paste. The transmitted ion current, $I_t$, was measured with an aluminium plate set just after the outlet of the tube, and its time dependence was recorded every second by a PC data acquisition system. The beam current on the holder, $I_h$, was also monitored during the measurement. The total current, $I_{tot}$, namely the beam current passing through the 2 mm diameter aperture of the shield, was determined as the sum of on-holder and transmitted currents, i.e. $I_{tot} = I_h + I_t$. Figure 4(b) presents the ratio of transmitted current to total current. The region from $0^\circ$ to $7^\circ$ in horizontal axis corresponds to the transmission ratio for the straight tube (in dashed-line region). Open squares denote the measurement with tilt angle (from $-7^\circ$ to $+5^\circ$). The bars denote the statistical error of one standard deviation. Assuming a certain value for the beam fraction injected into the tube, one can convert the ratio $I_t/I_{tot}$ into a transmission efficiency. If 32% of the total current, which is estimated from a Gaussian beam profile with FWHM=2 mm, is injected, $I_t/I_{tot} = 0.28$ at $0^\circ$ (open square) corresponds to a transmission efficiency of 87.5%. The transmission efficiency became zero when the tube was tilted more than $7^\circ$ from the centre. The ratio $I_t/I_{tot}$ for the curved tubes is also summarized in figure 4(b). When the primary beam intensity was of the order of a few nA, the maximum transmitted current was high, but unstable. In these three cases, the lower the incident current, the more stable the ion transmission. This suggests that there are appropriate intensities for a good balance of charge and discharge to guide the ion beams stably. The results demonstrate that these curved tubes can guide 8 keV Ar$^{8+}$ ions with transmission efficiencies of several tens of per cent. The beam-guidable bending angles of the curved tubes turned out to be indeed much larger than the limit of guiding with a straight tube of the same size. This may open a way to develop a new scheme of flexible ion beam deflector systems like fibre optics guides.

![Figure 4](image-url)
4. Microbeams of MeV ions with glass capillaries

4.1. Glass capillary with thin end-window

Several research groups have been intensively working on the preparation of microbeams for biological applications. In a conventional scheme, a well-focused energetic ion beam is extracted in air via a vacuum isolation window, then injected into a biological cell in culture solution. Another straightforward scheme involves passing energetic ions through a micrometre-sized aperture with or without a thin window at the end. Drawbacks of these schemes are (1) a relatively large cylindrical volume is damaged along the beam trajectory in addition to the targeted point, (2) serious energy and angular stragglings are induced during passage through the vacuum isolation window and air, which deteriorates the beam quality, and as a result determines the lower limit of the beam size at the target, and (3) real-time control/monitoring of the bombarding point is not easy even when a micrometre beam is prepared. In order to overcome such technical but serious problems, we have developed a scheme using a tapered glass capillary. The transport mechanism for MeV ions is governed by small angle scattering with the inner wall, which is different from that for slow HCIs. The point for the break-through for cell irradiation is that the capillary has a thin window at its outlet [14]. This scheme can realize pinpoint energy deposition and three-dimensional selection of the bombarding point by observing the outlet through a microscope with a precision of a micron or better in an arbitrary position of a living cell or in any liquid object. As the material of the end window, borosilicate glass [14,15] or plastic is available.

4.2. Irradiation to human cells

Figure 5. The target of the irradiation was a nucleus of HeLa cell in culture solution, whose nucleus was labelled with green fluorescent protein (histone H2B-GFP). The left and right panels are fluorescent images before and after the irradiation combined with the phase contrast images, respectively.

Figure 6. Plastic scintillator end-window with diameter and thickness are 5 μm and ~4 μm, respectively. When an ion passes through the plastic scintillator end-window, the window yields scintillation photons due to the energy loss at the window.

Figure 5 shows the demonstration of a cell irradiation using a glass capillary with a borosilicate end window. The target was a HeLa cell in culture solution, whose nucleus was labelled with green fluorescent protein (histone H2B-GFP). The left panel of figure 5 is a fluorescent image before irradiation combined with the phase contrast image of the same sample. A proton beam of 1 MeV was generated by Pelletron Accelerator at RIKEN, then extracted with a current of ~10 pA from the end window whose diameter and thickness were 5 μm and 8 μm, respectively. After 20 s irradiation, only the irradiated area was bleached, as shown in the right panel. The total dose of this irradiation was relatively high so that the bleaching of GFP was demonstrated at a 3-dimensionally confined volume. However, in some cases, irradiation by only one ion is sufficient to create a change or damage inside the target cell. Therefore, single ion irradiation is required for accurate dose control. To reduce the
beam intensity, we have already installed a beam chopping system which generates short pulsed beams of the order of hundreds ns. The number of ions in a pulse can be adjusted to be about 1. The number of ions in a pulse follows a Poisson distribution, i.e., some pulses are empty or have more than one ion. If the ion energy is larger than ~10 MeV, a detector behind the target cell would be available for counting the ions. However, in the case of our a few MeV ions, plastic scintillator end-window has been introduced to confirm the single ion irradiation before stopping at the target [16]. Figure 6 is a photo of the window whose diameter and thickness are 5 μm and ~4 μm, respectively. The end-window diameters from about 1 to 100 μm are available. The thickness is similar to the diameter. When a few MeV ion passes through the plastic scintillator end-window just before the target, the end-window yields scintillation photons due to the energy loss at least 100 keV for several μm thickness (for protons). The intensity of the scintillation will be monitored by a photo multiplier. The method for the dose control is in progress.

4.3. Irradiation to Escherichia coli cells

In contrast, some targets need a higher dose to change. A microbeam extracted from the glass capillary with end-window was applied to a single cell of *Escherichia coli* in liquid media to evaluate the effect of irradiation on cellular physiology [17]. As *E. coli* cells can move in the liquid media by rotating a bundle of their peritrichous flagella, the rotational behaviour of the single flagellar motor of the cell tethered to the substrate was observed during irradiation and the growth profiles of the cells attached to the substrate were monitored after irradiation. Then the irradiation dose required to stop the flagellar motor was evaluated. A proton microbeam of 2 MeV (before end window) with an intensity of 7.2 x 10⁴ particles /s was extracted from the plastic end window whose diameter and thickness were 11 μm and less than 10 μm, respectively. Figure 7 is a bottom view of the specimen. The area targeted by the microbeam is indicated with a circle. Actively rotating tethered cells, marked with arrows, exist both inside (the cell #1) and outside (the cell #2) the target area. The cell #1 stopped rotating, while the cell #2 continued to rotate, indicating that the beam was thin enough to irradiate the specific area.

**Figure 7.** A bottom view of the specimen. The area targeted by the microbeam is indicated with a circle. Actively rotating tethered cells, marked with arrows, exist both inside (the cell #1) and outside (the cell #2) the target area. The cell #1 stopped rotating, while the cell #2 continued to rotate, indicating that the beam was thin enough to irradiate the specific area.
cellular processes in the future works, e.g., damage to cell membrane, effects on membrane potential and respiratory chain enzymes.

4.4. Surface modification in liquid

The following application is not for biology but for surface modification. The range of the 3 MeV protons in water after passing through a Kapton end window with a thickness of 7 μm was calculated to be 140 μm using the SRIM software package. The surfaces of polyethylene (PE, −[CH₂CH₂]ₙ−) and polytetrafluoroethylene (PTFE, −[CF₂CF₂]ₙ−) sheets were selected as the samples, and aqueous acrylic acid (AAc, CH₂CHCO₂H) in concentrations between 0 and 10 wt.% was used as solution. Acrylic acid readily combines with itself or other monomers by reacting at its double bond site to form hydrophilic polymers. A scanning irradiation was performed with the distance of about 100 μm between the capillary outlet and the sample surface. After the irradiation, deposition on only the scanned area due to grafting and polymerization of AAc monomers was found to have a large affinity with water [18]. The deposited layer was stable and showed good adhesion to the base polymer. This method can provide techniques of coating or surface modification with fine pattern.

Other activities employing the tapered single glass capillaries with MeV ions were reported for PIXE [19-20] and NRA [21], as well as fundamental studies of the beam transport mechanism [22-25]. This glass made optics has been applied to not only ion beams but also electron [26-28], positron [29,30], and muon beams [31,32].

5. Summary

A method to produce microbeams of keV energy HCIs with single tapered glass capillaries based on a self-organized charge-up process has been developed. The transmission in this energy showed focusing effect up to the enhancement of 10, the guiding effect, and preservation of its initial charge state. Employing a flexibly curved Teflon tube, a beam guiding by large angles up to 26.7° was obtained. Microbeams of a few MeV protons/He ions provided by glass capillaries with end-windows have been used for the irradiation of HeLa cells and Escherichia coli cells. A method for dose estimation is in progress using plastic scintillator end-window combined with a pulsed beam system. A scanning surface modification experiment by the irradiation of proton beam was performed. The deposition on only the scanned area due to grafting and polymerization of AAc monomers was found to have a large affinity with water.

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