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Production of a nm sized slow HCI beam with a guiding effect

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Abstract. We have developed a method to produce a nm sized slow highly-charged ion beam based on a self-organized charge up inside a single tapered-glass capillary. In order to investigate the characteristics of the obtained beams, the transmission of 8 keV Ar^{8+} beam through the capillary of 5-cm long with 800/24 μm inlet/outlet inner diameter was examined. The transmitted beam had the same size as the outlet with the beam-density enhancement of approximately 10. The initial beam was guided through a capillary tilted by as large as $\pm$100 mrad and it still kept the incident charge. A focused ion beam of Ga^+ was employed to manufacture the capillary outlet as small as several hundreds nm in diameter and fabricate a thin glass window at the capillary outlet for biological applications.

1. Introduction

Slow highly charged ions (HCIs) have high ability to modify surfaces and cause efficient sputtering at only the surfaces. For example, a single HCI induces a nanometer dot on graphite [1, 2, 3] and Al_2O_3 surfaces [1]. It was also found that the F-Si bond direction of a Si(001)-F surface can be reconstructed from the 3D momentum distribution of F^+ ions desorbed by slow HCIs, i.e., a stereo-chemical analysis can be done with slow HCIs [4]. Once a microbeam is available, these functions specific to slow HCIs can be used to realize, e.g., micro-patterning of nanodots and element-sensitive micro-imaging. It is noted, however, that a microbeam of slow HCIs was not practically available because the HCI beam is relatively weak and also the emittance is not good. In the present article, we will report that a single tapered-glass capillary can collimate a slow HCI beam with relatively poor emittance down to micrometer or nanometer range, keeping the initial charge state and kinetic energy.

Originally, a similar glass capillary was successfully used to transport a proton or He ion beam in the range of MeV [5, 6]. In this case, however, the beam touches the inner wall and suffers small angle scattering with the wall surface during the transport. On the other hand, the interaction of keV HCI beams with a surface has been well investigated. A thin foil with a multitude of straight holes of $\sim$100 nm in diameter had been used to study above-surface interactions of slow HCIs with insulators [7, 8] and metals [9, 10, 11, 12, 13]. Particularly in the case of metal-plated insulator foils [8, 14], a so-called guiding effect was observed, where

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slow HCIs were deflected along the capillary axis keeping their initial charge states even when the capillary is tilted against the incident HCI beam. Considering the high reactivity of slow HCIs with surface, one can conclude that the single tapered-glass capillary enables the ions to transmit it without touching the inner wall and the ions can be focused due to the taper angle. As well as the thin foil with a multitude of straight holes, the self-organized charge up of the glass capillary inner wall is expected to play the vital role for the guiding effect.

The production of micro- or nano- sized slow HCI beam with a tapered glass capillary has been carried out at RIKEN [15] and CIRIL [16]. Recently, we started a biological application using the glass capillary with a thin glass window at the outlet [17]. Its preliminary results will be shown here.

2. Experimental setup

An ion beam of 8 keV Ar^{8+} was extracted from a 14.5 GHz Caprice ECR (Electron Cyclotron Resonance) ion source at RIKEN, and then transported to an experimental chamber via an analyzing magnet. The ion beam was then collimated by a 2 mmϕ aperture (see Fig. 1(a)), injected in a tapered glass capillary. The divergence of the incoming beam after the aperture was at most $\pm$3.3 mrad.

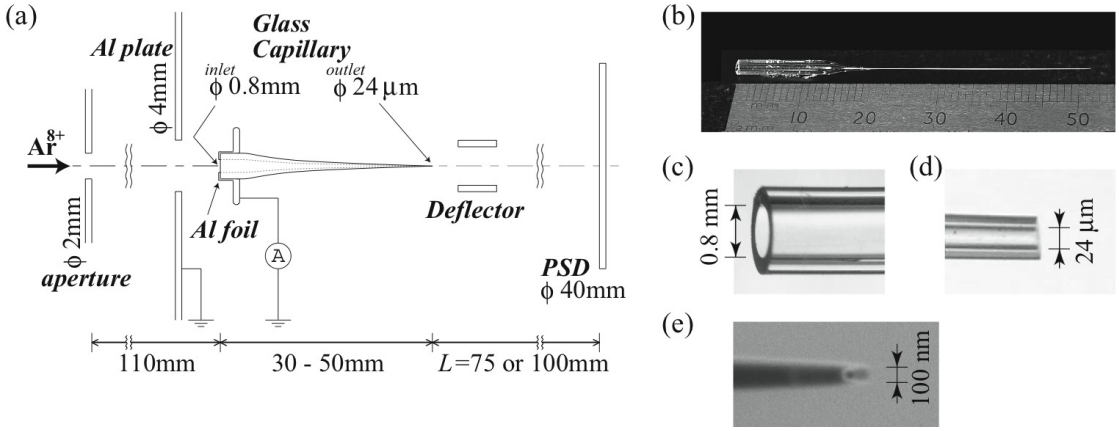

Figure 1. (a) a schematic view of the experimental setup, (b) a tapered glass capillary, (c) and (d) are the inlet and the outlet of a glass capillary, respectively, and (e) shows a microscope image of the outlet whose inner diameter is in the order of 100 nm.

The ions transmitted through the capillary were detected by a Position Sensitive Detector (PSD) with the sensitive area of ϕ 40 mm via a deflector which was for the charge-state analysis of the transmitted ions. A typical length of the capillary was 50 mm (Fig. 1(b)). The outer and inner diameters of the capillary at the inlet were 2 mm and 0.8 mm, respectively (Fig. 1(c)), and the inner diameter of the outlet is 24 μm (Fig. 1(d)). To avoid macroscopic charge-up of the entrance surface of the capillary it was covered by an Al foil with a 0.8 mm hole. The foil was used to monitor the incoming ion current of 0.1 - 10 pA. The secondary electron yield for several keV Ar^{8+} ion from the Al foil is expected to be $\sim$7 [18], the real current should be reduced by 0.47 (= 7/(7+8)) times. The ion current injected in the 0.8 mm hole of the capillary was evaluated from the beam diameter on the Al foil and the hole diameter. The incident currents hereafter correspond to the corrected current above. The PSD consisted of a stack of MCPs and a wedge-and-strip type anode and its detection efficiency was $\sim$50 %.

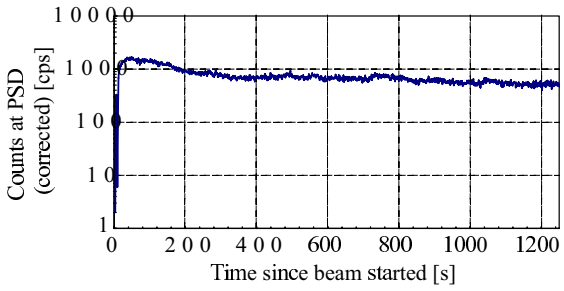

Figure 2. The time dependence of the number of transmitted ions: The counts at PSD were corrected to be divided by the detection efficiency of 50 %. It took several tens of seconds to saturate the transmission rate.

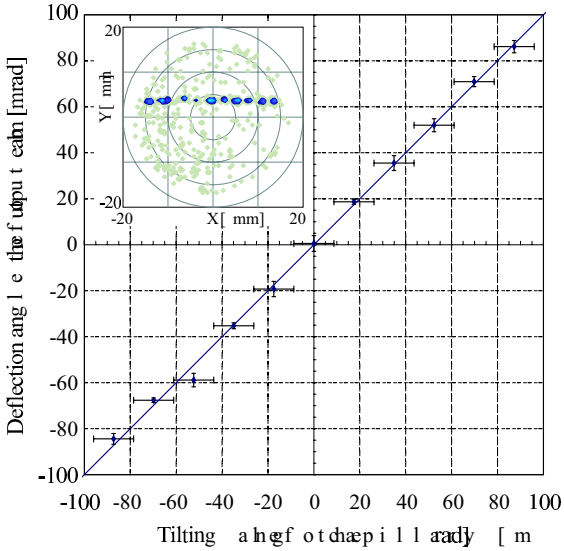

Figure 3. Tilting angle dependence of the peak position for the incident current of ∼0.01 pA: The horizontal axis is the tilting angle. The vertical axis corresponds to the reconstructed deflection angle of the beam from the peak positions at the PSD. The horizontal and vertical error bars show the accuracy of the tilting dial reading and the spot size (FWHM), respectively.

The glass capillary was made of borosilicate with the density of 2.23 g/cm^3, which is typically composed of 80.9 % of SiO_2, 12.7 % of B_2O_3, 2.3 % of Al_2O_3 and others. The tapered capillary was prepared by heating a straight glass tube, and then stretched by pulling both ends with a constant force. It was automatically done by a 'puller' (NARISHIGE CO.,LTD. *PE-21*). The softening temperature of borosilicate is 821°C, which is lower than 1530°C for silica glass. Heating of borosilicate is easier than that of silica glass. The taper angle can be controlled by tuning the temperature and the force. As shown in Fig. 1(e), the inner diameter of the outlet of about 100 nm can be fabricated by this method.

3. Transmission characteristics

Figure 2 shows the transmitted ion intensity as a function of time for the glass capillary injected with an 8 keV Ar^{8+} ion beam of 0.2 pA (∼$1.5\times10^5$$Ar^{8+}$ions/s) with a stability of ∼10 %. It is seen that the transmission current grew slowly with a time constant of several tens of seconds, and then got more or less stable for more than 1200 s. The peak counts in Fig. 2 was about 1600 cps. The focusing factor defined by the ratio of N_t/S_o to N_i/S_i is estimated to be ∼10, where N_t is the number of transmitted ions, N_i the number of injected ions into the capillary, and S_o and S_i are the geometrical outlet and inlet cross sections of the capillary, respectively. This factor depends on the taper angle, the outlet size and the monitored current at the Al foil in the range from 0.1 pA to 10 pA.

The upper left inset of Fig. 3 shows the position of the transmitted beam on the PSD (L=100 mm) when the capillary was tilted stepwise relative to the axis of the incident current of ∼0.01 pA (0.1 pA at Al foil). The relation between the tilting angle of the capillary and the deflected angle of the beam is shown in Fig. 3, which proves that the beam was well guided in the direction

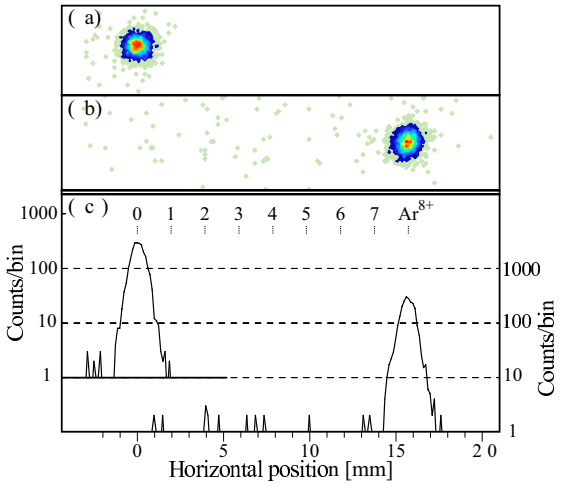

Figure 4. (a) and (b) show the spots at the PSD without and with the deflection bias, respectively. (c) The projected beam profiles corresponding to (a) and (b).

of the capillary tilted by as large as 100 mrad. It is 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 trapezoid.

4. Quality of extracted beams

The charge-state distribution of the transmitted HCIs for the incident current of 0.3 pA was measured by biasing the deflector downstream of the capillary without tilting. Figure 4(a) shows the circular spot at the PSD (L=75 mm) without the deflector bias. While, Fig. 4(b) shows the spot with the bias of 280 V, which was still circular and shifted to the position corresponding to the initial charge. The peaks in Fig. 4(c) show the projected beam profiles corresponding to Figs. 4(a) and (b). In the region corresponding to charge-changed components, there were no peaks but some background events, i.e., the transmitted ions were guided along the capillary without changing the incident charge state. The angular divergence of the transmitted beam was evaluated from the spot sizes seen in Fig. 4(c), and found to be $\sim \pm 5$ mrad.

5. Nanometer sized beams

Figure 5(a) shows the extracted beam profile on the PSD (L=75 mm) when the capillary outlet was ϕ 900 nm as shown in Fig. 5(d). The intensity of the initial Ar^{8+} beam with the energy of 64 keV was 10 pA at the Al foil. The spot had the widths (FWHM) of 0.30 mm and 0.40 mm in vertical (Fig. 5(b)) and horizontal (Fig. 5(c)) directions, respectively. The corresponding divergences were ± 2.0 mrad and ± 2.7 mrad, respectively.

With conventional machining under an optical microscope, it is impossible to manufacture the outlet of the capillary with a diameter smaller than several hundreds nm. Therefore a focused ion beam (FIB: Hitachi *FB-2100*) of 40-keV Ga^+ was employed to cut the glass capillary with smaller diameter. Figure 6(a) shows a thus manufactured outlet of a glass capillary with an inner diameter of 500 nm. The scanning parameters of the FIB, e.g., scanning directions, interlace rate, scanned region, etc., were tuned to prevent the Ga^+ beam from being bent due to the charge up around the capillary outlet. After the tuning, various shapes can be realized. (Fig. 6(b))

6. Application

The outlet of the glass capillary is of the order of 1 μm, which is smaller than the diameters of some living cells. The microbeam or nanobeam extracted from the glass capillary could then be aimed at a small part of a cell. As an application, a thin glass window (a glass lid) was put at the capillary outlet to keep the vacuum inside the capillary connected with the beam line even

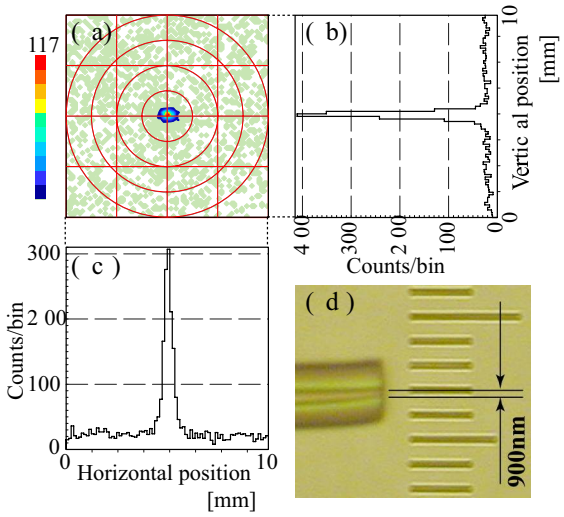

Figure 5. (a) The beam profile at the PSD: The beam of Ar^{8+} of 64 keV extracted through the capillary outlet of ϕ 900 nm, (b) and (c) are the projections to the vertical and horizontal axes, respectively, and (d) shows a photograph of the capillary outlet.

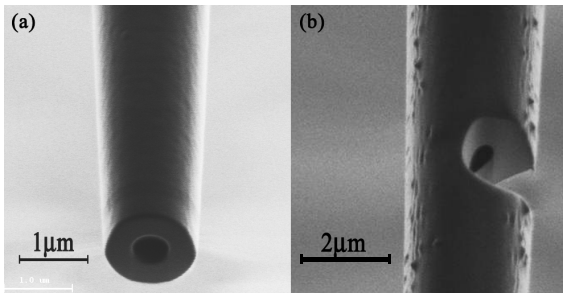

Figure 6. (a) a manufactured outlet of a glass capillary by FIB, (b) various shapes can be realized.

if the capillary outlet is in the liquid sample where cells can live. The thickness of the lid can be controlled using FIB. When the thickness is selected to be slightly smaller than the range of a initial beam ($\geq$MeV), the ions must be stopped just after they penetrate the lid. The beam energy in MeV region can be also kept during the transmission [5]. It means that the deposited energies of the stopping ions can be concentrated at the small region. For calibration a liquid scintillator of about 0.2 cm^3 was used instead of a living cell. Then the scintillation emitted from a volume within at most 10 μm was observed with the lid of less than 1 μm-thick and a beam of α particles of 4 MeV. This application has the advantage of selective irradiation to a small well defined region not only in transversal but also longitudinal direction.

7. Discussion

The results for slow HCIs reported here can be explained as follows: The saturation time shown in Fig. 2 suggests that the incident ions entering the capillary hit and charge up the inner wall. 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 the guiding is established. The charge-state distribution (Fig. 4) indicates that the transmitted ions never touched the inner wall of the capillary.

Conventionally, magnetic and/or electrostatic lenses have been employed to produce microbeams. Using such lenses, submicron ion beam of a few tens keV region was reported [19], where the lenses with small aberration and an ion source with good emittance were employed. Our method presented here is not very sensitive to the initial beam with broad energy-spread and divergence, and it was realized just by a tiny tapered glass tube of $\sim$50 mm in length. In irradiation experiments, the respective beam position can be determined easily.

8. Summary

We have observed stable transmission of slow HCIs through a single tapered glass capillary with a focusing factor of approximately 10 for an outlet diameter of 24 μm. The transmitted beam obtained by such a simple setup was guided to as far as $\pm$100 mrad, and had a circular spot and a divergence of $\pm$5 mrad without charge transfer inside the capillary. A nm sized slow HCI beam was produced. Using an FIB, a capillary outlet smaller than several hundreds nm can be realized and an outlet covered by a lid can be also manufactured.

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