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New type of capillary for use as ion beam collimator and air-vacuum interface

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ABSTRACT

Glass capillaries offer a unique way to combine small diameter ion beam collimation with an air-vacuum interface for ambient pressure ion beam applications. Usually they have an opening diameter of a few microns, limiting the air inflow sufficiently to maintain stable conditions on the vacuum side. As the glass capillaries generally are quite thin and fragile, handling of the capillary in the experiment becomes difficult. They also introduce an X-ray background produced by the capillary wall material, which has to be shielded or subtracted from the data for Particle Induced X-ray Emission (PIXE) applications. To overcome both drawbacks, a new type of conical glass capillary has been developed. It has a higher wall thickness eliminating the low energy X-ray background produced by common capillaries and leading to a more robust lens. The results obtained in first tests show, that this new capillary is suitable for ion beam collimation and encourage further work on the capillary production process to provide thick wall capillaries with an outlet diameter in the single digit micro- or even nanometre range.

1. Introduction

Glass capillaries have various uses in scientific instruments. They are used i.e. to deposit small amounts of liquids [1], for chemical analysis using capillary electrophoresis (CE) [2] and for focussing of X-ray beams [3,4]. Recently a new application, ion beam collimation with glass capillaries, has been implemented at various ion beam analysis (IBA) laboratories [5–11] and some publications even reported a gain in fluence after the capillary exit [12,13]. Compared to the usually used electromagnetic lenses a capillary offers a cost effective alternative for micrometre diameter ion beam generation. They require little space, no energy, are easy to install and very good for targeting. Additionally, beam stiffness does not limit the range of usable ion masses or energies and for ambient pressure ion beams a capillary can even be used to replace two components: the focusing optics and the air-vacuum interface.

Investigations into the capillary transmission characteristics show that slightly tapered capillaries with tilt angles of few mill radians improve the ion beam transmission [12] and that, if a conical profile is used, the beam focusing ratio can be enhanced by a factor of two compared to conventional tapered capillaries with a convex inner wall [9]. A possible explanation is Rutherford scattering at the inner glass wall of the capillary occurring at small angles. While even the most efficient profiles only achieve beam focusing ratios close to unity [13], the capillary optics are very attractive for low current techniques like STIM, single cell irradiation and MeV SIMS. PIXE is also possible, but due to the low beam currents relatively long measurement times have to be expected.

The low current obstacle can be removed if there are electromagnetic lenses present in the beam line. They can heighten the current density hitting the capillary entrance and in return the capillary can enhance the focussing ability of the lenses. This way a beam line limited to i.e. 40 μm beam resolution can achieve 1 μm beam diameter or less.

Regarding the ability of capillaries to act also as air-vacuum interface, previous publications report that a long micrometre diameter capillary limits the gas intake sufficiently so the vacuum in the beam line remains stable [5].

In this publication, a new type of conical glass capillary is presented for use as ion beam collimator and air-vacuum interface. The prototype's unique characteristic is a greater thickness of the glass walls compared to conventional tapered capillaries and a different profile of the inner and outer capillary walls. Its usability as well as its special advantages are demonstrated in energy spectra as well as STIM and PIXE measurements.

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2. Experimental

The new type of capillary used for ion beam collimation is produced by drawing of a borosilicate glass tube at high temperature in a heating furnace. The manufacturing process has been implemented at the National University of Córdoba (UNC), and is usually used to produce X-ray capillary optics [14]. The process of drawing has been carefully developed in order to produce capillaries with high quality conical inner profiles combined with thick walls. The resulting capillary is very robust even with outlet diameters in the micrometre range. A picture of one of the prototypes and its tip can be seen in Fig. 1.

Three different capillary types have been tested, one short and cylindrical (KV2) with an outer diameter of 1700 μm at the tip and two long, conical ones with outer diameters 750 μm (KV5) and 710 μm (KV6). To check if the capillaries can be used as ion beam optics they were brought to the Laboratory of Ion Beam Physics at the Eidgenössische Technische Hochschule Zürich (ETHZ). Here a setup for the use of capillaries in ambient Ion Beam Analysis applications has been developed [5]. It is installed at the end of one of the beam lines connected to the 6 MV EN tandem accelerator. The capillary is fixed onto a vacuum flange mounted on a bellow that can be aligned to the beam position and direction by a 2-axis goniometer on an XY stage. The chamber in front of the ambient pressure setup has an inner diameter of 5 cm and is pumped by a turbo molecular pump. Because the capillary opening is fixed in space, imaging is performed by mounting the sample on an XY stage driven by two stick–slip piezo motors. The maximum scanning range of the piezo drives is 7 mm with a closed loop positioning resolution of 50 nm. Adjustments of the sample distance to the capillary tip are done manually employing a micrometer screw.

After fixing the capillaries in an adapter to connect them to the beam line, a test regarding the influence of the capillary opening on the pressure in the beam line was performed. A sufficient vacuum was obtained for all of the three capillaries tested with the pressure remaining in the 10^{-6} mbar range.

For the following measurements each capillary was prepositioned using a Laser beam running through the beam line. Angles have to be aligned carefully towards the beam direction in order to allow the beam to transmit through the small opening. Afterwards the unfocussed 2 MeV proton beam was sent through the capillary and detected by a Hamamatsu S3590-09 large area PIN diode (10×10 mm detection area) placed behind the opening. While monitoring the intensity detected by the diode, the capillaries' position and inclination was further optimized.

After intensity optimization energy spectra of the ion beam after passing through the capillary were taken with the diode. The same detector was used for Scanning Transmission Ion Microscopy (STIM) measurements of a gold mesh (mesh dimensions: 50 μm thick bars with 200 μm space between each bar). Furthermore spectra for in-air PIXE were taken using an Amptek X-123 SDD X-ray spectrometer with a detection angle of 45° to the sample surface.

3. Results

Energy spectra of a 2 MeV proton ion beam after passing through the three tested capillaries (KV2, KV5 and KV6) taken with Hamamatsu diode and are shown in Fig. 2. The Spectra are normalized to the intensity of the high energy peak for comparison and all show two characteristic differences compared to the ion beam before passing through the capillary: a slight increase of FWHM and a low energy tail. Both effects can be explained by scattering of the ion beam at the capillary tip. The differences in peak broadening between the capillaries originate from the different ratio of scattered to directly transmitted ions depending on capillary position and shape. Additionally a smaller exit diameter leads to fewer directly transmitted ions and a higher influence of scattered ions. The capillary with the highest FWHM and most pronounced low energy tail is KV6 which hints to a smaller exit diameter for this capillary but could also be explained by bad positioning. The energy distributions of the capillary collimated beams show the potential of the capillaries for in-air Ion Beam Analysis as mostly non scattered ions are transmitted which can be used for STIM or PIXE.

Imaging was also tested with: A small gold grid was raster-scanned between the tip of capillary KV5 and a Si-PIN diode detector using the piezo positioners. In Fig. 3 the resulting intensity profile is displayed. A beam diameter after passing through the capillary of (25 ± 2) μm has been calculated, were the error results from low statistics of the obtained data and beam fluctuations during the measurement. Further decrease of the outlet diameter is possible with newly produced capillaries.

Lastly the PIXE capabilities of the capillaries have been studied. Spectra obtained from an iron target using capillary KV6 with and without steel shielding between the detector and the capillary tip are compared to a spectrum taken with a thin wall capillary from ETHZ (Fig. 4). The low energy background present in the spectrum of the ETHZ capillary, produced by excitations in glass near the capillary outlet, disappears completely for thick walled capillaries. The outer diameter of the capillary KV6 tip is 710 μm which leads to a wall thickness of at least 300 μm around the ion transmitting channel, assuming that the channel's diameter is similar to KV5's. This is well above the sum of the range of 2 MeV protons in glass (44 μm for 2 MeV protons in Borosilicate glass according to SRIM [15]) and the approximately 5 μm attenuation length [16] for Si K-line X-rays in SiO_2. This explains the missing characteristic Si X-ray background, which is the major background source in PIXE measurements with the ETHZ capillaries. A few new background events, not visible in the ETHZ capillary's spectrum, appear in the higher energy range. They can be attributed to characteristic X-ray emission from arsenic and rubidium. The addition of a steel shield between the detector and the capillary eliminates these peaks (Fig. 4).

This proves that the arsenic peaks originate from impurities in the capillary material as exactly these peaks disappear after adding a steel shielding between detector and capillary. The attenuation

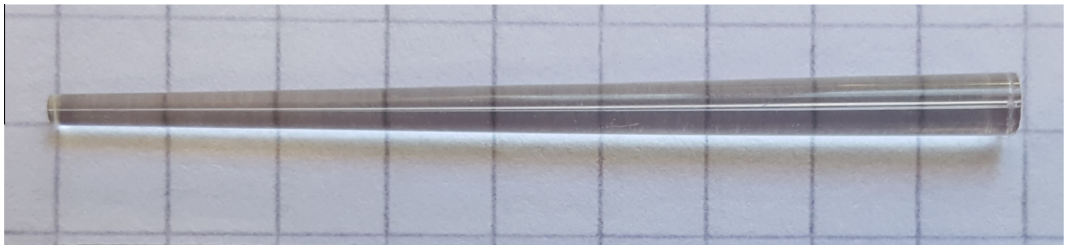

Fig. 1. One of the capillaries recently produced in the UNC. Notable are the thick wall and the different inner and outer profiles. For reference, the squares on the paper on the back are 5 mm × 5 mm.

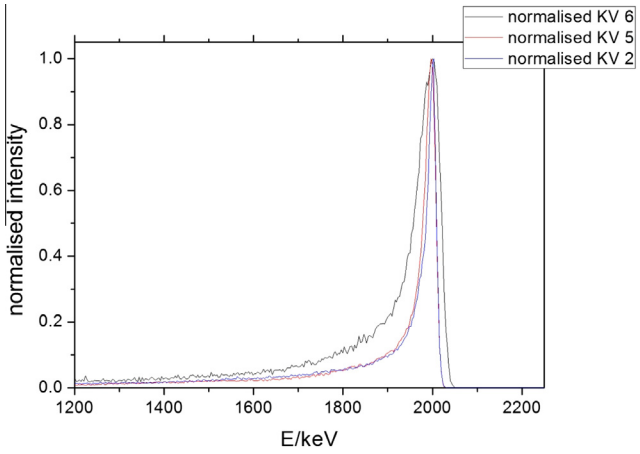

Fig. 2. Normalized energy spectrum of a 2 MeV proton beam measured in air after passing through the thick-wall capillaries.

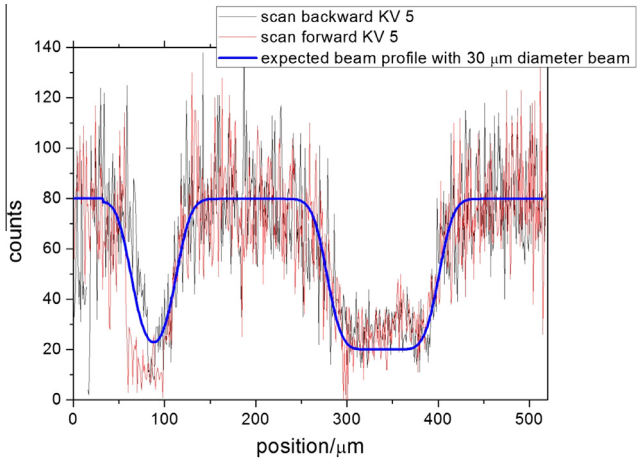

Fig. 3. STIM raster scan of a gold mesh. Red in one direction and black on the way back. The ion beam diameter after passing through the capillary is (30 ± 2) μm. In blue the expected scan profile for a beam with 30 μm FWHM. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

length for the characteristic As K-lines is about 300 μm for As K-lines at 10.5 keV according to [16]. A slightly thicker capillary, i.e. KV2, should suffice to suppress this background without shielding in front of the detector.

The overall low background is very useful for PIXE of light elements, making it possible to work without additional shielding.

4. Conclusion

In conclusion the capillaries produced at the UNC have shown great potential for in-air and low current Ion Beam Analysis. They have unique characteristics compared to regular, thin capillaries: Due to their thick glass walls even at the capillary tip, they are easier to handle and very sturdy. This should enable their usage over a long time, because accidental damaging of the capillaries is almost impossible as is deformation of the tip over long storage times. One minor drawback is that targeting on the sample with such capillaries is slightly less precise than with thin wall capillaries due to the thick glass at the tip, which limits it to sub-mm precision. For PIXE applications they have additional benefits as they show significantly less low energy X-ray background from the capillary. Imaging capability was also demonstrated.

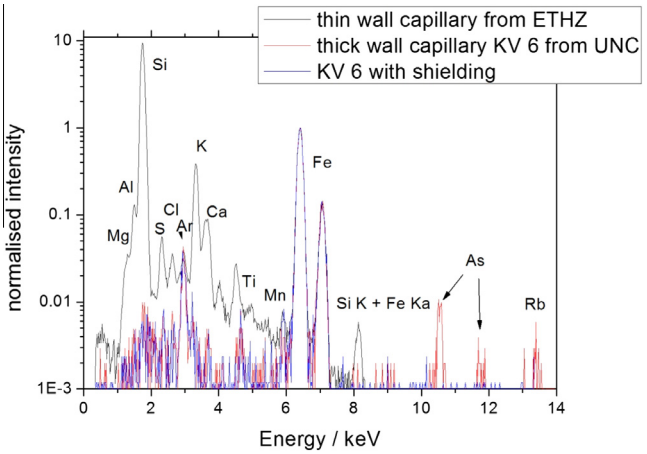

Fig. 4. Comparison of PIXE spectra normalized to the Fe-Kα peak from an iron sample using a common thin wall capillary from ETHZ (black), a UNC thick wall capillary (red) and a UNC thick wall capillary with shielding in front of the detector (green). Spectrum acquisition durations were about 30 min for the kV 6 spectra and 2 min for the ETHZ capillary. Clearly visible: the suppression of the capillary wall material induced characteristic X-rays when using the thick walled capillary and the deletion of the thick wall capillary lead background after introducing a shielding. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Compared to the capillary optics used for X-ray beam focusing [3,4] for micro X-ray fluorescence (μXRF) setups, the beam diameter achieved is similar for the tested thick wall capillary and significantly smaller for the ETHZ capillary [5]. The gain was not measured for the thick wall capillaries but earlier experiments have shown it to be around 1 which is much less then achieved by poly- and monocapillary X-ray optics. On the other hand it has been shown in [5] that the divergence of the ion beam compares favourably to the divergence of X-ray beams after passage through capillary optics.

The future objective will be to produce capillaries with outlet diameters in the single digit and sub-micron range, comparable to thin walled capillaries produced in other labs like i.e. at the ETHZ. This will provide a valuable addition to the air-vacuum interface tool set. Also by usage of impurity free glass like borosilicate the already low background from As K-lines can be eliminated. Overall these thick wall capillaries might offer a way to produce nano ion beams with a cheap and sturdy lens.

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