

Peculiarities of swift proton transmission through micro- and nanocapillary structures

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Tasks and Objects



- Introduction and motivation
- Experimental setup designed for studying the transmission of swift ions through capillary systems
- Micro- and nanocapillary systems
- Transmission of swift protons through tapered micro- and nanocapillaries





Transport of charge particle beams through dielectric capillaries

The principle of new optics discussed in this report is based on interaction of glancing beams of charged particles with a charged insulating surface of capillary walls. To a certain degree, it resembles us the motion of charged particles in **channeling** regimes along the lowindex directions of crystal lattices, or quanta motion in the **waveguide** regime.





Potential implementations of the phenomenon

Analysis of the effect of surface charge on the character of motion of the ion beam forming this charge on an insulator is of interest both for new ion optics and for analysis of the interaction of particles with insulators at small angles of beam incidence with respect to the surface.

The phenomenon of glancing motion of ion beams along a charged dielectric surface can be used to develop systems of transformation, control, and transport of charged particle beams; in particular, to obtain micro- and nanosized beams, which are interesting in local elemental and structural analysis, nanolithography, X-Ray radiography and applications in biology and medicine.





Potential implementations of the phenomenon

- In comparison with the existing tools for formation of micrometer- and nanometer-sized beams, this method is undoubtedly simpler and less expensive.
- At the same time, is satisfies all the requirements of submicron Rutherford backscattering spectrometry or analysis with application of induced characteristic X rays (PIXE). For example, this technique enables in-air PIXE measurements of various samples that are not compatible with the vacuum environment (wet solids, liquids and gases). Slightly tapered glass capillary optics can be applied as a differential pumping orifice as well as a focusing lens.



Fabrication of nanocapillaries in polymers

As a unprecedented result on the low energy ion transmission through such capillaries I illustrate an experiment of N. Stolterfoht *et al.*, NIM B203 (2003) 246.

They measured the transmission of 3 keV Ne⁷⁺ ions through capillaries of 100 nm diameter and 10 µm length produced by etching ion tracks in a polyethylene terephthalate polymer foil. The foils were tilted up to $\pm 25^{\circ}$. The majority of Ne⁷⁺ ions were found to survive the transmission in their initial charge. This capillary guiding of the Ne⁷⁺ ion provides evidence that the inner walls of the capillaries become charged and electron capture from the surface is suppressed in a self-organizing process.



Time dependence of the transmitted Ne⁷⁺ intensity showing the charging and discharging phenomena of capillaries in PET



A beam of 1.3 nA Ne⁷⁺ ions is directed onto the PET foil tilted at 10°.

The transmitted Ne⁷⁺ intensity measured at 10° increases exponentially with a time constant of 2.5 min. After 10 min the beam is turned off. Short beam pulses probe the decrease of the transmission with a time constant of 40 min.



Experimental Setup





Schematic of the experimental setup: (MC) matching circuit, (VFC) voltage-frequency converter, (VD) voltage divider, (EA) electrometric amplifier, (FVC) frequency-voltage converter, (RM) programmable rate meter, (FA) forming amplifier, (CSP) chargesensitive preamplifier, (SBD) silicon surface barrier detector, and (PC) personal computer.



Experimental Setup





Analytical equipment to study processes of charged particle interaction with capillary systems

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This setup, which enters the implantation complex, formed on the basis of an ESU-2 electrostatic ion accelerator, consists of four units: (1) a system of ion beams formation, (2) a scattering chamber, (3) a measuring chamber, and (4) a system for detecting scattered ions.

The parameters of the setup are as follows:

- ✓ the error in determining angles in measurement of angular distributions is not larger than 3.3×10⁻³ deg.
- ✓ the error in the capillary orientation with respect to the beam axis is not larger than 2.5×10⁻² deg.
- ✓ the angular divergence of the initial beam \pm 3.0×10⁻² deg.
- the total measured energy resolution of the recording system does not exceed 16 keV
- a mobile silicon surface barrier detector is positioned at a distance of 90 cm from a capillary holder





- ✓ glass (borosilicate) capillaries with a diameter of 0.5 mm and length of 178 mm;
- ✓ glass tapered capillaries with an inlet diameter of 0.5 and 3.5 mm and outlet diameter of 0.1 mm and a taper angle of 0.5, 1.7 and 2.2 deg;
- arrays of uniform-sized nanopores with a diameter of 30-70 nm in anodic alumina.







The individual capillary systems used for the transportation of proton beams





Shape and angular distribution of a 200 keV proton beam transmitted through the capillary with a taper angle of 1.7 deg





It should be mentioned that a practically uniform distribution of ion beam density with sharp edges is registered in this case. Moreover, a spot size amounts to 3.8 mm that corresponds to a beam divergence of ± 0.13 deg just as an initial beam divergence was ± 0.015 deg.

An integral of an area under the curve in this figure shows that the fraction of transmitted protons is equal to 80%, i.e. the number of transmitted ions relative to those entering into the capillary. Therefore, taking into account that the outlet diameter of the capillary is 100 μ m and the initial beam diameter amounts to 500 μ m, the focusing factor reaches up to 20.





Count rate of particles transmitted through the capillary versus proton current at the input of the capillary

10/11/10





The results presented in this figure demonstrates a strong nonlinear behavior of the current at the output of the capillary on intensity of the input beam up to an input current of 5x10⁻¹³ A. It is well accepted that such protons are guided electrostatically due to the charging up of the inner wall of capillaries made of insulating material.





Time distributions of protons transmitted through a tapered capillary (a) and cylindrical capillary (b)





In spite of the practically equal currents at the input of these capillaries, time evolutions of beam currents at the output of the capillaries are strongly different in shapes and frequencies of the beam intensity oscillations. Current pulse frequencies of ions transmitted through the tapered capillary exceed essentially those for the cylindrical capillary. On the contrary, more shorter pulse durations are typical for the tapered capillary.

The mentioned above experimental results confirm our recent assumption on a dominant role of charging up a face part of the capillary in the transformation of continuous ion beams into oscillating ones. This effect is not observed if an input hole of capillaries exceeds the beam diameter.







The transparency of this capillary with a taper angle of 0.5 deg is 300 times less than this parameter for the capillary with a taper angle of 1.7 deg

Shape and angular distribution of 200 keV protons transmitted through a capillary with a taper angle of 0.5 deg





Angular distribution of 320 keV protons transmitted through a capillary with a taper angle of 2.2 deg

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Energy spectrum of transmitted protons with an initial energy of 320 keV with and without capillary. The taper angle is 1.7 deg. **Curve 1** is for the initial beam and **curve 2** is for the transmitted beam

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The most of ions transmit the capillary without energy loss, however, the low energy tail certainly exists (**curve 2**). It means that those transmitted ions moving with higher transverse energies and suffering the small angle scattering lose their energy interacting with an inner surface of the capillary. It should be noted that these particles cause only a very modest widening of the initial energy distribution (less than 5 to 6%).





Diameters of entrance holes on the face side of the sample. The thickness of anodic alumina wafers was 42 μ m. Diameter of entrance holes was in a range of 60-70 nm and the density of holes was 1.2x10¹⁰ cm⁻²





SEM cross-section images of a cleaved sample





Angular distribution of protons with an energy of 190 keV transmitted through the AI_2O_3 nanostructure sample oriented along the beam axis





Angular distribution of protons with energy of 290 keV transmitted through the AI_2O_3 nanostructure sample oriented along the beam axis





Angular distribution of 290 keV protons transmitted through the AI_2O_3 sample at a misorientation angle of 0.07 deg

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It should be noted that the FWHM of central peaks in the both angular distributions is considerably narrower than the width of the initial beam.

The presented in the last three figures proton beam intensities were measured behind the sample with a thickness of 42 µm that considerably (more than one order of magnitude) exceeds ranges of protons in this material.

This is an evidence of an anomalous motion of protons like the hyperchanneling of charged particles along the low-index directions of crystal lattices. The transmission coefficient in this system achieved a few percent if the face part of the sample was covered with a thin Au layer.

Micro – and nanocapillary systems



The Kumakhov's

microcapillary system with a diameter of 30 nm of individual glass capillaries

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(A) the glass capillary optics, (B) the X-ray detector, (C) the sea water droplet





PIXE spectrum of sea water





External PIXE spectrum of a gallbladder tissue





The experimental setup for micro-beam production using a tapered glass capillary (J. Hasegawa *et al.*, NIM B 266 (2008) 2125)



Proton-induced X-ray radiography



A schematic of quasi-monochromatic X-ray imaging using the glasscapillary-based micro-beam generator

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then insert it again

Application of capillary ion optics



An image of a miniature bulb filament taken by Cu K X-rays. Capillary tip: $D 25 \mu m$, exposure time: 1 h, magnification: 10x

(J. Hasegawa et al., NIM B 266 (2008) 2125)





- We have confirmed that a few hundred keV proton beams are successfully focused by the tapered capillary optics.
- The areal density of the transmitted beam is enhanced by approximately 20 times.
- Charging up a face part of the capillary causes the transformation of continues ion beams to oscillating ones.
- ✓ The most of protons (94–95%) in the energy range of 150 to 320 keV transmit the capillary without energy loss.
- Changing a taper angle from 0.5 deg to 1.7 deg evidences increase of the transmission coefficient more than by 300 times keeping the initial energy spectrum of ions.
- Compared with the conventional micro-beam facilities, the usage of tapered capillaries is certainly simple and low-cost, thus providing an interesting technique of submicron RBS or PIXE elemental analyses. Moreover, if the ion species are extended to heavier elements, the present method provides highly local versatile maskless ion implantation technique.









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