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Charge deposition dependence of electron transmission through PET nanocapillaries and a tapered glass microcapillary

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ABSTRACT

Charge deposition dependences of electron transmission through insulating PET nanocapillaries and a tapered glass microcapillary are reported and differences with HCI transmission are noted. Investigations were conducted for electrons with incident energies 500–1000 eV, corresponding to energies per charge similar to those used for HCI studies, incident on (1) an array of PET nanocapillaries (density $\sim 5 \times 10^8/cm^2$) with diameters 100 nm in a foil of thickness 12 μm, and (2) on a tapered glass microcapillary with inlet/outlet diameters of 800/100 μm and a length of ~35 mm. The transmission was measured for incident electrons at small sample tilt angles ranging from 0° to 5° with respect to the beam direction. For most angles, including those near zero degrees, there was an initial quiet period during which essentially no transmission was observed, followed by large rises in the transmission during relatively short periods of charge deposition before equilibrium of the transmission was reached. The resulting equilibrium was stable, blocked or had frequent oscillations depending on the incident energy and the capillary used. Observations for both capillaries show that a negative charge patch is needed to guide incident electrons through the capillaries similar to the manner in which HCIs are guided through capillaries.

1. Introduction

Charge dependence of slow, highly-charged ions (HCI) through nano- and micro-capillaries has been studied extensively but the same has not been reported for incident electrons. For HCIs the results show gradual increases in transmission with the charge deposited into the capillaries followed by the transmission reaching an equilibrium value [1] This charge dependence work grew out of the original work for the transmission of 3 keV Ne^{7+} ions through small diameter (100 nm) insulating polyethylene terephthalate (PET) nanocapillaries [2] in which the deflection and transmission of ions was attributed primarily to the formation of a self-organized charge patch near the capillary entrance. This repulsive charge patch then deflects subsequent incoming ions and prevents interactions with the capillary walls, a phenomenon referred to as *guiding*.

For incident electrons with energies between 200 and 1000 eV, transmission measurements for Al_2O_3 [3] and PET [4,5] nanocapillaries and straight glass microcapillaries [6] showed properties similar to those for slow HCI guiding, but for electrons evidence for inelastic scattering was seen as the tilt angle of the capillary was increased in contrast to HCI transmission for which no inelastic scattering was observed. The transmission of electrons through glass capillaries [7] and PET foils [8] rose with much smaller charge constants, representing faster transmission rates, following significant initial quiet periods, in comparison with HCIs for which the transmission started to increase immediately and rose more slowly before reaching equilibrium with stable or oscillatory behaviors [1]. In a joint experimental and theoretical study for 250 eV electrons incident on Al_2O_3 nanocapillaries [9] it was argued that charge up was not necessary for transmission of electrons to occur. Instead, the transmission was attributed to multiple small-angle scattering events accompanied by significant energy loss and secondary electron emission. An earlier theoretical study [10] of electron transmission through PET nanocapillaries with energies comparable to those used in the present work also predicted a similar interpretation of the results. However, this conclusion based on multiple small-angle scattering does not agree with the findings of our work [8] for electrons incident on capillaries. This discrepancy provided the motivation for this paper and will be examined.

In the present work we investigate the charge deposition dependence of electrons with incident energies 500–1000 eV, corresponding

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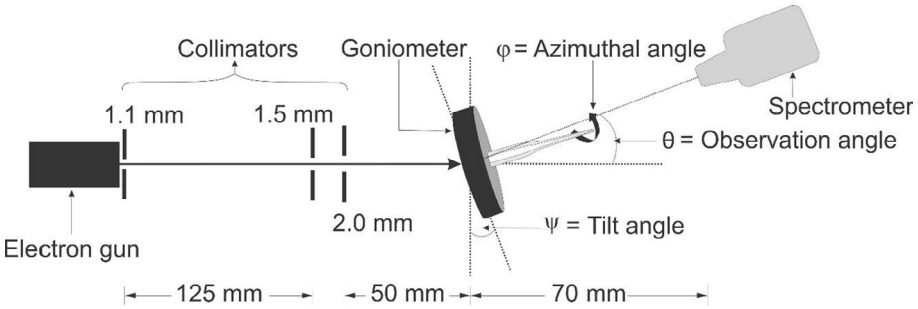

Fig. 1. Schematic of the experimental apparatus used in this work. The diagram shows the setup for the tapered glass microcapillary. For measurements with the PET nanocapillary, the foil array was installed in place of the glass capillary. In the drawing ψ, θ and ϕ are the tilt angle, observation angle and azimuthal angle, respectively. The angles ψ and θ were measured with respect to the incident beam direction. The drawing is not to scale.

to energies per charge similar to those used for HCIs, incident on an array of PET nanocapillaries and on a tapered glass microcapillary. Transmission was studied for electrons incident at small sample tilt angles from 0° to 5° with respect to the beam direction. For all angles studied, an initial quiet period during which essentially no transmitted electrons were emitted was followed by large rises in transmission in relatively short periods of charge deposition into the capillaries. The transmission then reached an equilibrium state that was stable, blocked or had frequent oscillations depending on the incident energy and the capillary used. From these observations, it is concluded that negative charge buildup is important and necessary in initiating the transmission of electrons through the capillaries. A scenario for the buildup of negative charge and the subsequent guiding caused by it is proposed in another paper submitted to these proceedings [11].

2. Experimental procedure

The experimental apparatus has been described in detail previously [5,12], and will be discussed only briefly here. The measurements were made at Western Michigan University and a schematic of the apparatus is shown in Fig. 1. The setup shown is for the tapered glass capillary but for the PET nanocapillaries the only difference was that the foil array was installed in the sample position instead of the glass capillary. Both samples were isolated from ground and were not polarized so that the current on them could be read directly, a measurement that was done with an electrometer having the capability of reading currents less than a picoampere. An electron gun generated beams with energies between 500 and 1000 eV. The beam was initially collimated through a 1.1 mm aperture at the exit of the e-gun followed by additional collimation with two apertures of sizes of 1.5 mm and 2.0 mm separated by 10 mm located ~12.0 cm downstream of the e-gun. The beam divergence for these conditions was calculated to have a FWHM of ~0.53° (~0.0092 rad).

A two-dimensional goniometer located about 50 mm downstream from the 2.0 mm collimator was used to mount and rotate the foil array or the glass capillary. The goniometer had a tilt angle rotation capability of $-20^\circ < \psi < +20^\circ$ around a vertical axis and an azimuthal rotation of $0^\circ < \psi \leq 360^\circ$ about a horizontal axis. When the sample was tilted the goniometer was always started with positive values of ψ and moved towards negative values (the designation of positive and negative angles is arbitrary) in small steps in order to overcome any backslash in the goniometer mechanism. The transmitted electron beam was observed with an electrostatic parallel-plate analyzer placed ~5 cm behind the goniometer. This analyzer had an energy resolution of 3%, an angular resolution ~0.3° and a detection efficiency of ~50% [13]. Transmitted electrons were counted with a channel electron multiplier (CEM). The pressure in the experimental chamber was maintained at $\sim 2 \times 10^{-6}$ Torr.

The PET foil used in this work had capillary diameters of 100 nm, an array density of $\sim 5 \times 10^8/\text{cm}^2$ and a thickness of 12 μm. The aspect ratio (length/diameter) of the capillary is 120. Additionally, a thin gold layer (~20 nm) was evaporated at an angle of 30° from four directions onto both sides of the foil to prevent charge up of the sample from the primary beam and to allow measurement of the incident current on the foil. The sample was prepared at the GSI laboratory in Darmstadt, Germany and was subsequently shipped to the Helmholtz-Zentrum Berlin where the sample was mounted into an aluminum frame enabling it to be installed onto a sample holder.

The tapered glass capillary was made of borosilicate glass (80.9% SiO_2, 12.7% B_2O_3, 2.3% Al_2O_3, 4.0% Na_2O, 0.04% K_2O, and 2.23 $g{\cdot}cm^{-3}$ density) with inlet/outlet diameters of 0.80 mm/0.10 mm and a length of 35 mm. The inlet region of the capillary (inner diameter 0.80 mm) remained constant along a length of ~22 mm with a straight shape, followed by a length of 5 mm, over which the diameter decreased from 0.80 mm to 0.16 mm with a funnel (exponential) shape. Then, the capillary had a conical shape with the diameter decreasing from 0.16 mm to 0.10 mm over a length of 8 mm. (A picture of this capillary can be found in Ref. [12].) The aspect ratio of this capillary is ~70. The capillary was prepared at the RIKEN laboratory in Japan. The entrance surface (facing the incident beam) of the capillary and its holder were coated with conducting silver paint at Western Michigan, to allow excess charges to be carried away thereby preventing charge up of the entrance and allowing measurement of the current incident on it.

Before the charge deposition measurements began, the zero degree angles for both the PET capillary array and the glass capillary were determined from electron transmission measurements. This procedure gave the zero value to about ± 0.2°. The capillary was then left to discharge for at least 24 h prior to making the charge deposition measurements. A similar discharge time was used between each of the measurements for the various angles studied.

To obtain the transmitted intensities, electrons were counted over energy ranges for which significant transmission was observed for each incident energy. These ranges were 470–540 eV for 500 eV, 755–845 for 800 eV, and 800–1060 for 1000 eV, with each range requiring about a minute to complete (see Fig. 8 in Ref. [5] and Fig. 3 in Ref. [12] for typical spectra taken, although these spectra were taken for significantly longer times). The counts were then summed to give the integrated intensity for that point. This procedure was repeated until equilibrium was reached and it was clear that the pattern was no longer changing significantly. To get the total charge deposited corresponding to each measurement over the energy range, the incident current into a single capillary was multiplied by the total time elapsed from the beginning of sample irradiation. The time was determined by the counting time for each energy range scan (about one minute). The current was calculated from the area covered by a single capillary to the total area of the beam on the sample (about 3 mm diameter). Results for each capillary are presented below.

3. Results

As already noted, two distinct samples were used in this work. First an array of PET nanocapillaries was investigated for incident energies of 500 and 800 eV and current densities of ~1.6 and ~4.0 nA/mm^2, respectively. The second sample was the tapered glass capillary for which a current of 60 pA was incident into the capillary entrance