

## Vacuum ion emission from solid electrolytes: An alternative source for focused ion beams

Conrad Escher, Sandra Thomann, Cornel Andreoli, and Hans-Werner Fink<sup>a)</sup>  
 Physik Institut der Universität Zürich, Winterthurerstrasse 190, CH-8057 Zürich, Switzerland

Julien Toquand and Dieter W. Pohl  
 Physik Institut der Universität Basel, Klingelbergstrasse 82, CH-4056 Basel, Switzerland

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A bright ion source based on the solid electrolyte  $(\text{AgI})_{0.5}(\text{AgPO}_3)_{0.5}$  has been developed. The solid electrolyte source provides stable currents of  $\text{Ag}^+$  in the microampere regime that make it suitable for focused ion beam applications. Similar conditions are expected for different solid electrolyte materials and their corresponding ions. This opens a broad field of applications in structuring and modifying devices on a nanometer scale using focused ion beams. © 2006 American Institute of Physics. [DOI: 10.1063/1.2264092]

Focused ion beams play a vital role in structuring devices on the nanometer scale.<sup>1</sup> A prerequisite for the generation of such focused ion beams are ion sources with a high degree of confinement and brightness as well as stability in continuous operation. This excludes volume plasma sources due to their lack of confinement as well as gas field ionization sources due to their limited brightness. So far, the only source that has been satisfying these three requirements routinely is the liquid gallium ion source<sup>1</sup> with emission currents in the microampere regime.

The operating principle of the liquid gallium source is based on a tungsten tip coated with liquid gallium. By means of an electric field, a delicate balance of surface tension and electrostatic forces forms a cone of gallium at the apex of the tip. The pointed cone is required to confine the extraction field to a small source region. The loss of ions is compensated by diffusion from a liquid metal reservoir, which enables continuous operation.  $\text{Ga}^+$  ion beams can be focused to diameters down to about 5 nm, a limit imposed by the broad energy spread of the ions leading to considerable chromatic aberrations inherent to electrostatic ion lenses.<sup>1</sup>

In a good solid electrolyte the mobile ions can move almost as freely as in a liquid.<sup>2</sup> If the solid electrolyte was given the shape of a sharp tip and contacted to a reservoir of bulk metal, ions of the mobile species might be field emitted from the apex and replenished from the reservoir [Fig. 1(a)]. The nonmobile constituents of the material, however, would serve as a rigid frame that fixes the shape of the tip.

Out of the large number of known solid electrolytes, amorphous  $(\text{AgI})_{0.5}(\text{AgPO}_3)_{0.5}$  was chosen for our investigation. Its room temperature conductivity is one of the largest known ( $\sigma \approx 10^{-2} \text{ S/cm}$  at 25 °C),<sup>3,4</sup> its fabrication is easy, and, as it turned out, it can readily be shaped into a sharp tip, as shown in Figs. 1(b)–1(d). All tested tip geometries provide sufficiently high electric fields at the apex to allow ion emission into vacuum. The data presented here were generated with the micropipette filled structures, as shown in Fig. 1(c). They provide most stable and long term ion emission. Glass micropipettes are readily available with openings in the sub-micron regime. The pulverized solid electrolyte is filled into the pipette and heated to the melting temperature. The mol-

ten electrolyte has excellent wetting properties to glass. As a result, it moves towards the very end of the pipette, where it forms the desirable shape of a spherical calotte. Contacting the tip to a silver wire or fixing it with silver paste to the source holder provides a silver reservoir required for continuous operation.

For investigating the emission properties of such ion sources, a field ion microscope (FIM) is employed.<sup>5</sup> When the solid electrolyte tip is put on high potential with respect to a microchannel plate (MCP),  $\text{Ag}^+$  ions are field emitted from the solid electrolyte and accelerated onto the MCP.

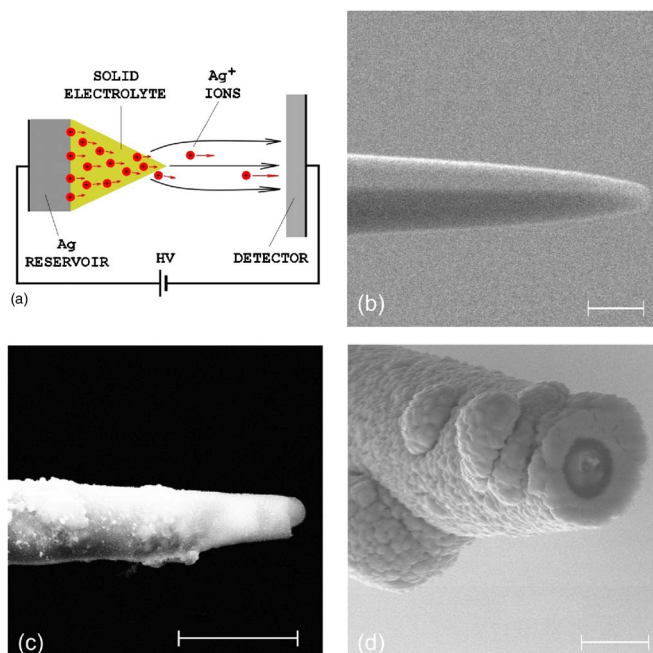


FIG. 1. (Color online) Schematic of the solid electrolyte ion source and realizations of it. (a) Principle of the solid electrolyte ion beam source. Different fabrication methods have been explored, ending up with sharply pointed tips. (b) Shaping of a tip by drawing a heated fiber of the solid electrolyte material. (c) Filling the solid electrolyte material into a glass capillary. (d) Evaporating silver onto the glass capillary followed by using a focused ion beam to cut off the very end of the tip. Although this structure is more complicated to fabricate, it has the advantage of the silver reservoir being in immediate contact to the emitting region of the solid electrolyte. The bars in the scanning electron micrographs correspond to 1  $\mu\text{m}$ .

<sup>a)</sup>Electronic mail: hwfink@physik.unizh.ch

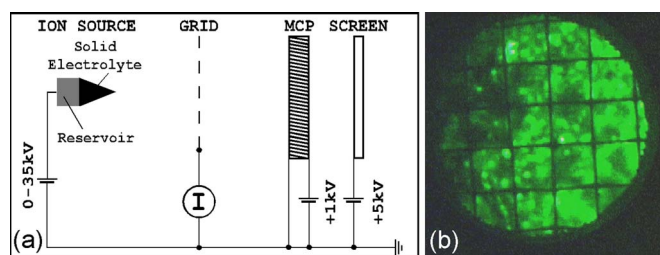


FIG. 2. (Color online) Schematic setup and field ion pattern. (a) The silver ions are field emitted from the tip and accelerated towards the MCP screen detector from which the ion emission pattern is recorded with a video camera. A grid placed in the path of the ion beam is used to measure a certain fraction of the current. (b) A field ion pattern as observed at the MCP screen detector.

There they provoke an electron avalanche, which is finally transferred into a light spot on a phosphorous screen. As the detector is approximately 10 cm away from the source, a highly magnified image of the emission sites at the tip apex is observed on the screen and recorded by a video camera. It is seen that the emission occurs in little spots representing the ends of migration channels.<sup>6</sup> A grid between the source and the detector is incorporated into the FIM [Fig. 2(a)]. It enables us to measure the ion current while *in situ* observing the dynamics of the emission patterns on the screen [Fig. 2(b)]. Since the grid blocks just 5% of the beam, only a fractional current is measured. However, when scaling it up to the total beam current, it agrees well with an independent measurement using a Faraday cup to capture the entire beam.

The maximum current amounts to almost  $1 \mu\text{A}$ , as shown in Fig. 3(a). The plot of Fig. 3(b) indicates that the emission currents are well reproduced with changing voltages applied to the source. Moreover, long term stability of the source current has been monitored during operation for a few days. It appears conceivable that the lifetime of the source is essentially limited by the volume of the silver reservoir. The plot in Fig. 3(c) shows the time dependence of the current for the first 20 min of operation. Except for the first few minutes the emission current from the solid ion source proves to be very stable.

The findings presented above, considering the large number of known solid electrolyte materials, open up the possibility for generating ion beams from a variety of chemical elements.<sup>7</sup> Focused ion beam tools can profit from the use of  $\text{Ag}^+$  ions (or  $\text{Cu}^+$ ) where implantation of Ga is to be avoided. Possibly, even direct conductor path writing with linewidths of just a few nanometers might be envisioned. Other advantages of the solid electrolyte source compared to the liquid metal ion source might be higher mechanical stability, smaller energy spread, and, as a result, better ion op-

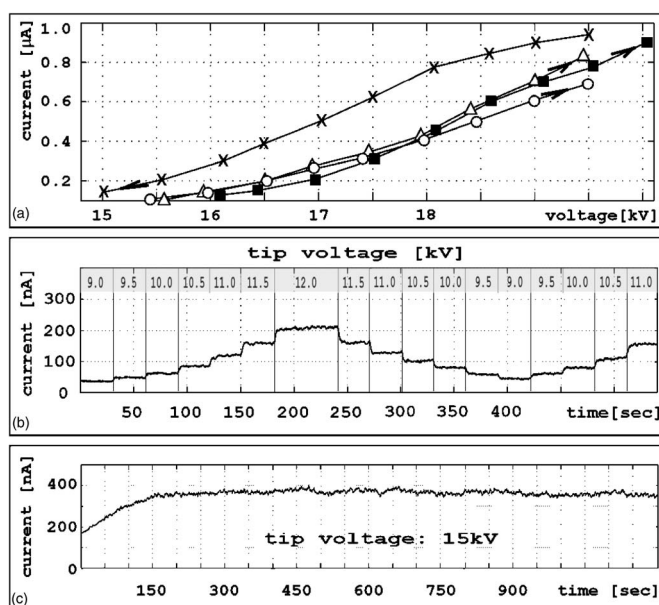


FIG. 3. Ion current measurements. (a) Four current-voltage characteristics. The data marked with circles, triangles, and squares are taken as the voltage gradually increased from 15 to 20 kV, whereas the data marked with crosses are taken as the voltage decreased from 20 to 15 kV. A slight hysteresis is apparent. (b) The current vs time response of a silver ion source to various bias voltages between 9 and 12 kV shows rather stable behavior. (c) The first 20 min of a long term current measurement at constant voltage is depicted.

tical performance. Last but not the least, the extension to solid electrolytes with conducting species different from metals will open up possibilities for alternative focused ion beam applications, such as etching with reactive ions such as  $\text{F}^-$ ,  $\text{O}^{2-}$ , and  $\text{H}^+$  and, relevant for integrated optics devices, doping with rare earth ions.

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