# Synthesis and characterization of ion-crafted nano/micro field ion emitters

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At the present time, interest is focused on the production of cold electron emitters and vacuum microelectronic devices [1]. The methods for manufacturing the above systems by the mechanical assembly of individual needles, directional growth of whisker crystals from gaseous media, photolithography followed by chemical etching, and directional solidification of eutectic oxide metal alloys are known [2]. An interesting alternative method for fabricating such large area field emitter arrays is the nuclear track etch technique [3–7]. In this technique, the hollow etched tracks are filled with a suitable cathode material and then a replica technique is employed. The advantage of multiple emission structures manufactured by track etch technique is that they offer a wide range of cathode materials with a broad range of the diameter of emission electrodes. In this technique, the field emitter pins are statistically distributed and quite higher areal density can be achieved. Because of the conical shape of the emitter pins generated using track etch technique, they are better emitters than those generated by microlithographic techniques. Further, the main advantage of track etching technique over conventional techniques is that several tedious processing steps involved are eliminated.

A field emitter is a metallic needle-electrode held at negative voltage with respect to a flat counter electrode. Under good vacuum conditions, the needle cusp emits a strongly bundled electron beam of high intensity. The brightness of field emitters can be several orders of magnitude higher than that of thermal cathodes. Therefore field emitters are beginning to replace directly heated tungsten filaments in many applications, for example, in scanning electron microscopy. The most important parameter of the field emitter pin is its smallest radius of curvature at the outermost tip: the maximum field strength is inversely proportional to the tip radius. Large area field emitter arrays are promising devices for many applications ranging from high quantum yield photocathodes to energy saving radar tubes. Their working depends critically on keeping tip radii as close as possible within the given specifications. Copper points arranged on copper backing are the most easily made structures and show promise for future application as explosive electron emitters. The high electrical and heat conduction qualities of the point material ensures a small loss of substance from their surface and consequently a long lifetime. Another trend in the production of emitters is the use of triode field emitting electron systems. The advantages of such cathodes is a small electrode gap and consequently low values of supply and control voltage, which can directly be controlled at the output of the microelectronic devices. The low absolute energy of the secondary ions minimizes erosion of the cathode and extends its lifetime. Cathodes of this type form the basis for display panels, which may replace cathode ray tubes.

The Template Synthesis technique entails synthesis of desired materials (metals, semiconductors, and metal-semiconductor junctions) of very low dimensions [3–11]. The underlying principle of this technique is well known. It is an electrochemical process in which metallic ions in a supporting solution are reduced to the metallic state at the cathode, which, if closely covered by a nuclear track filter (NTF) membrane, would lead to the formation of growth of plated film as an embodiment of micro- or nanostructure. The etched pores of the membrane used would act as templates. The generated structures can either be heterogeneous or homogeneous depending upon the pore size and geometry, with complete control over the aspect ratio. As is evident, the Template Synthesis is a membrane based technology. One of the types of membrane used here is known as a track etch membrane or nuclear track filter (NTF). NTFs have emerged as a spin-off from solid state nuclear track detectors—solid state materials capable of storing tracks of energetic, heavily ionizing ions which can subsequently be chemically amplified as see-through pores or channels of well defined geometry and spatial density. NTFs have been put to numerous filtration applications besides their use in the synthesis of nano/microstructures and devices. Martin [11], Chakarvarti and Vetter [7] have produced extensive reviews of the Template Synthesis technique along with morphological revelations of the structural ensembles so generated.

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The template synthesis of metals is usually carried out through a galvanic replication technique. This involves the synthesis process on a metallic cathode substrate which is tightly covered by an etched NTF as an overlay and having the custom made pores which act as templates for growth of structures. In general, a suitable cell design is required and the lay-out design of such a cell along with other relevant details of the technique have been given previously [5]. The NTF used here as template was Makrofol-KG foil (polycarbonate from Bayer AG), 10 $\mu$m thick, having average pore diameter $\sim$ 800 nm with pore density $1 \times 10^8$ $m^{-2}$. This was prepared by irradiating the foil with $^{238}$U ions (energy 13.64 MeV/n) at 90 $^\circ$ utilizing the UNILAC facility available at GSI, Darmstadt, Germany, followed by chemical amplification of the damage trails by etching in 6 N NaOH, at 60 $\pm$ 2 $^\circ$C for 15 min. In order to produce see-through pores, optimum etch time and etch conditions were preset. For the fabrication of copper nanowires in the form of columnar ensembles, the cathode (Cu tape as substrate having its base coated with conducting adhesive) of the designed electrodeposition cell, was covered tightly with the processed NTF and the electrolyte solution to be used in the bath was prepared using milli Q 10-M$\Omega$ water and ultra-high purity reagents 200 g/l $CuSO_4 \cdot 5H_2O$ (98%) + 20 g/l $H_2SO_4$. The pH of the electrolyte was adjusted to 0.90. The electrodeposition was carried out at 0.8 V, at room temperature (29 $\pm$ 1 $^\circ$C), with the anode made from pure Cu sheet. The variation of current with time during electrodeposition is shown in Fig. 1. The optimum conditions, e.g., temperature, voltage, time etc.,

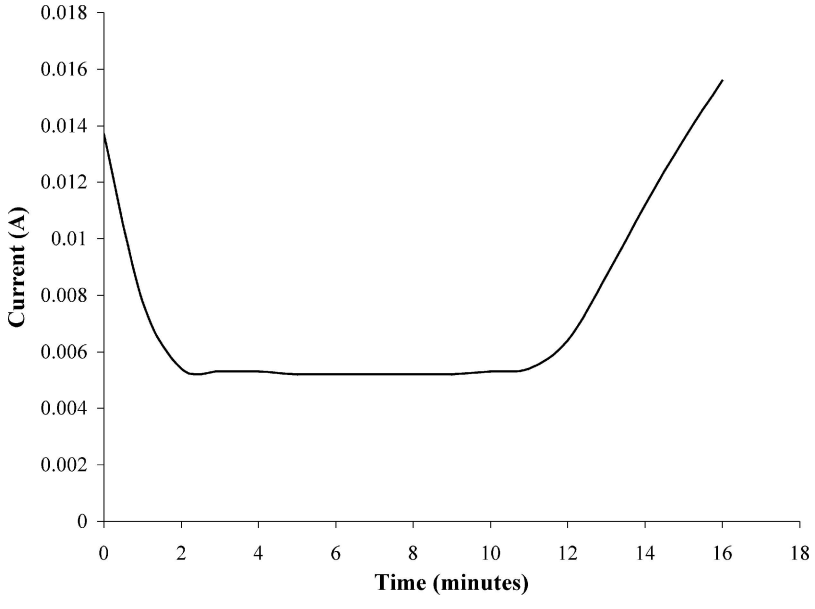


*Figure 1* Variation of current with time during Cu electrodeposition through pores in polycarbonate.

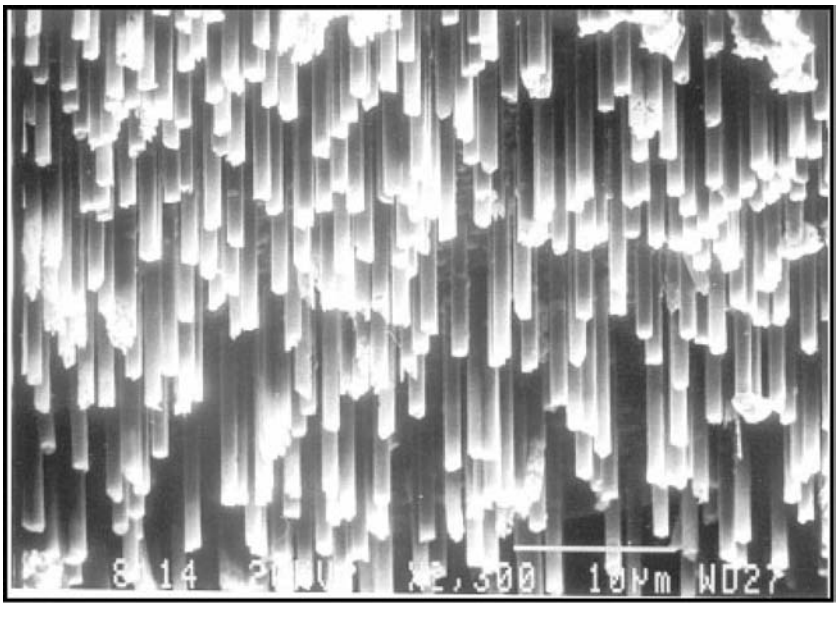

*Figure 2* SEM micrographs of copper nanostructures having diamater 800 nm and length 10 $\mu$m.

depend upon the chemistry of the process. After the plating process was completed, the NTF overlay along with the substrate were removed and the makrofol foil was dissolved carefully in $CH_2Cl_2$ so that the grown microstructures with Cu tape as substrate were revealed (Fig. 2).

In the Fowler Nordheim relation [12], field emission is described as tunneling through a potential barrier at the surface of a solid when a large electric field is applied. Knowing some of the surface properties, one can calculate the probability of an energetic electron tunneling through the potential barrier. Field emission experiments were carried out in a vacuum chamber with $2 \times 10^{-5}$ Torr at room temperature $29 \pm 1$ °C. The distance between a copper anode and the tip of a copper wire was 200 $\mu$m. The measured emission area was 30 $mm^2$. Emission current ($I$) as a function of applied voltage ($V$) was monitored with a Philips digital multimeter PM 2525 (Fig. 3). The emission current-voltage characteristics were analyzed by using the Fowler-Nordhiem (FN) equation for the field emission:

$$J = A(\beta^2 V^2/\phi d^2)\exp(-B\phi^{3/2}d/\beta V)$$

where $J$ is the current density, $A = 1.56 \times 10^{-10}$ ($AV^{-2}$ eV), $B = 6.83 \times 10^9$ (V $eV^{-3/2}$V $m^{-1}$), $\beta$ is the field enhancement factor, $\phi$ is the work function, $E = (V/d)$ is the applied field, $d$ is the distance between the anode and the cathode and $V$ is the applied voltage. Fig. 4 presents the plot between $\ln(I/V^2)$ and

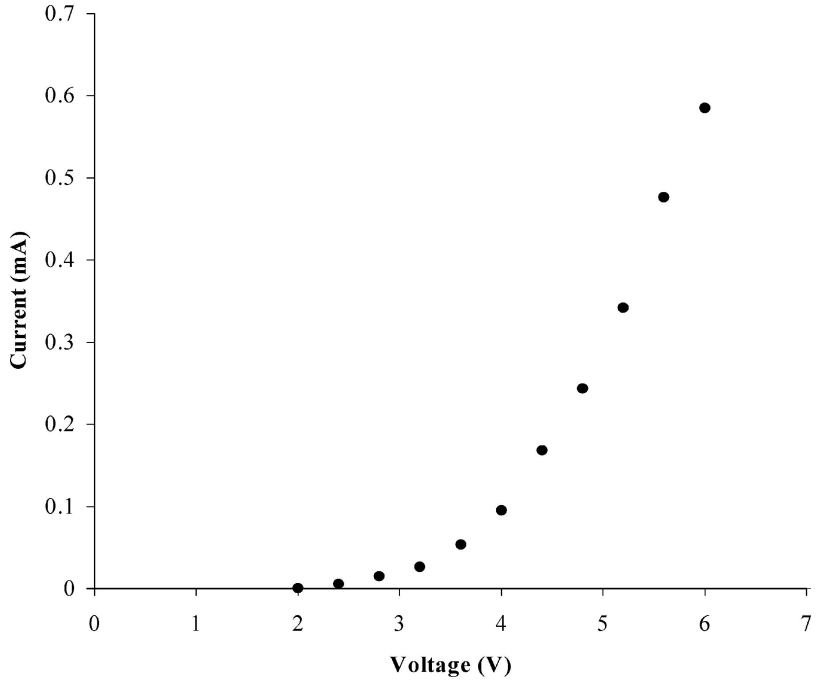


*Figure 3* Graph showing variation of emission current generated through nanostructures with applied voltage.

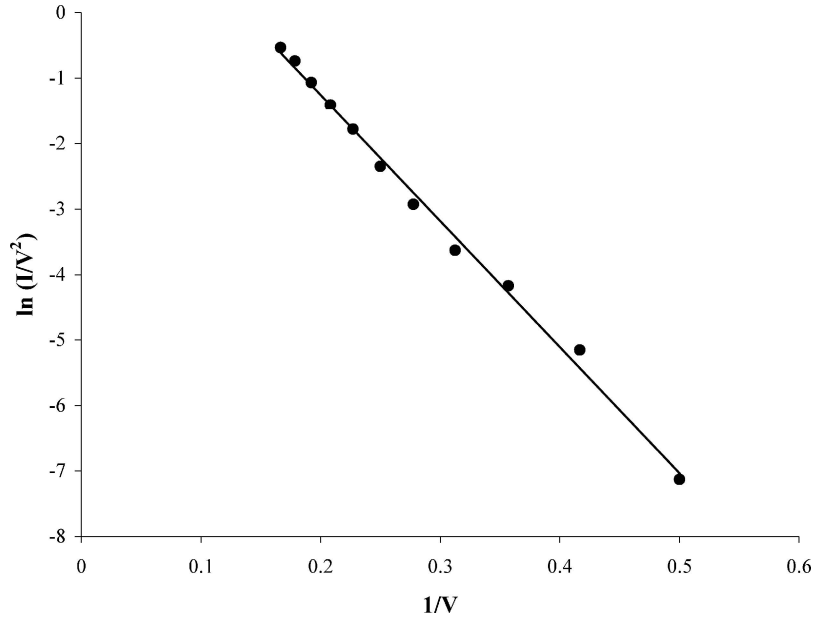


*Figure 4* Fowler-Nordheim plot satisfying emission relation.

$1/V$ (Fowler Nordheim plot). The linearity of this plot indicates that the data satisfy the F-N field emission relation.

An oriented copper nanostructure array, which was used as cathode, was stuck to a copper substrate by silver paste with the bottom end of the nanostructures facing upwards. In this configuration the copper nanostructures were placed with their long axis perpendicular to the substrate and the bottom end of the nanostructure array, which was composed of well separated and highly oriented nanostructure tips, acted as the emitting surface. A copper plate with a diameter of 1 cm was used as anode. The very low electron emission thresholds for our copper nanostructures imply that if this material were used as the cathode in vacuum microelectronic devices a voltage as low as 10 V would provide a current of more than 0.9 mA, for a gap between the anode and the cathode of 200 $\mu$m. This may be very interesting for flat panel display applications.

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