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Short communication

Ion emission properties of indium nanowires grown on anodic aluminum oxide template

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ABSTRACT

Indium (In) nanowires were synthesized on self-ordered hexagonal porous anodic aluminum oxide (AAO) template by electrodepositing $In_2(SO_4)_3$ in the pores of AAO template. In nanowires obtained are very straight and have uniform-diameter about 45 nm. The ion emission properties of In nanowires were investigated. The onset field of ion emission is observed at about 1.6 V/μm, which is much lower than that for a conventional liquid metal ion source (LMIS) consisting of a sharpened metal needle. The *I-E* curves follow nearly a linear relationship with a slope of about 6.0 μA/(V/μm), indicating a conventional ion emission phenomenon in LMIS. It is proposed that the unique geometry of nanowires may be the reason for their advantages in LMIS.

Liquid metal ion source (LMIS) has attracted much attention due to the applications in focused ion beam (FIB) [1–3], radiation materials science [4,5] and field emission electric propulsion (FEEP) thrusters [6–8]. Field emission of ions is a key principle used in LMIS, which refers to the process of using a strong electric field to produce a spray of charged ions and droplets from emitters [9,10]. It has been presented experimentally that electric field is extraordinarily high with a value level of 10^9 Vm^{-1} to extract ions [11]. The geometry of the emitter is extremely important in LMIS [12]. To enhance the local electric field on the emitter and reduce the applied voltage, usually a sharp needle, such as tungsten tip, wetted with low melting point metals is used as emitter in LMIS [2,3,6–8]. The radius of curvature of the needle which is fabricated out of bulk metal is about one micrometer to several ten micrometers. But how to achieve an emitter with much smaller radius of curvature and reduce the applied voltage further is still a challenge.

Nanotubes or nanowires have high aspect ratios and small radius of curvature. It has been proved that nano-materials have excellent electron field emission properties [13,14]. They may have good ion field emission properties, too. In this paper, the In nanowires grown on anodic aluminum oxide (AAO) template were used as ion emitters, the ion emission properties of which had been studied. It shows that metal nanowires also present good ion emission properties and can emit ions under very low applied electric field. It is proposed that metal nanowires may be potential candidates for the application in LMIS.

The schematic of the process for fabricating In nanowires is shown in Fig. 1. Firstly, the self-ordered hexagonal porous AAO template were prepared by two-step anodization method as reported before (Fig. 1 a) [15,16]. Secondly, one side of the AAO template was coated with a layer of Au film by sputtering deposition (Fig. 1 b). The Au layer would be used as a working electrode in the next step. Thirdly, In nanowires were electrodeposited in the pores of AAO template by a similar electrodeposition method as reported before (Fig. 1 c) [17,18]. Briefly, the electrodeposition was carried out in a common two electrode plating cell with a piece of graphite as the counter electrode. The electrolyte for the deposition of In nanowires contained a mixture of $In_2(SO_4)_3$ (13 g/L), H_3BO_3 (30 g/L). The pH of the electrolyte was adjusted to about 1.5 by adding H_2SO_4. The electrodeposition was carried out under the current density of 1.0 mA/cm^2. Finally, the surface of the AAO template was mechanically polished away to exposed the tips of the In nanowires (Fig. 1 d).

The morphologies of In nanowires were observed with field emission scanning electron microscope (FE-SEM), and transmission electron microscope (TEM). The crystal structures of In nanowire arrays were characterized by X-ray diffractometer with Cu $K_{\alpha1}$ radiation ($\lambda = 0.154056$ nm). For TEM observations, the AAO template

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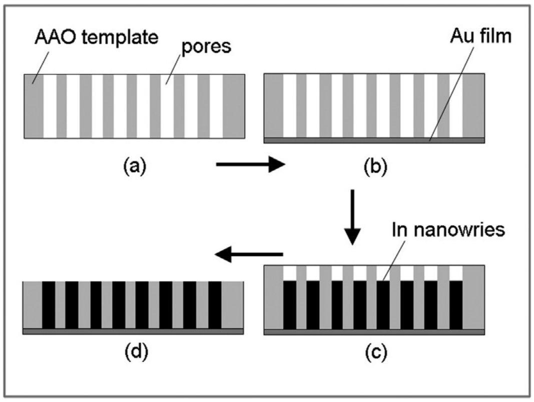

Fig. 1. Schematics of the process for fabricating In nanowires on AAO template.

was completely dissolved with 1 M NaOH solution to get rid of alumina and then the In nanowires obtained was rinsed with absolute ethanol.

Fig. 2(a) shows the typical FE-SEM image of In nanowires embedded in the pores of AAO template. It can be seen that high filling, ordered and uniform In nanowires are obtained. The corresponding TEM image is shown in Fig. 2(b), indicating that In nanowires are very straight and have the uniform-diameter about 45 nm. Fig. 2(c) shows the morphologies of the surface of the samples. Each white point represents a tip of In nanowire, which means the tips of In nanowires were exposed to vacuum during the *I*–*E* test. Fig. 2(d) shows the XRD pattern of the as-prepared sample which confirms further that the sample is In nanowires with polycrystalline structure (JCPDF 05-0642). A peak marked with star symbol may be indexed as aluminum oxide (Al_2O_3) (JCPDS 82-1467).

The *I*–*E* characteristics of In nanowires grown on AAO template were carried out in a vacuum chamber at a pressure of 1×10^{-4} Pa. The experimental setup is shown schematically in Fig. 3. Two parallel aluminum sheets were used as anode and cathode. The In nanowires grown on AAO template were set on one side of the anode and a heater was attached on the other side to melt down the In nanowires in the pores during the test. The gap between the In nanowires and the cathode was about 1 mm.

Fig. 4 shows the *I*–*E* characteristics for In nanowires with diameters grown on AAO template at an operation temperature of about 200 °C. There is a key parameter in LMIS called onset voltage U_0 below which no ions emission will occur. The corresponding field E_0 ($E_0 = U_0$/h, h is the distance between the emitters and the extractor electrode) named onset field have been analyzed. No ion current has been observed until the extraction voltage is about 1600 V. The corresponding onset field E_0 is about 1.6 V/μm. The *I-E* curves follow nearly a linear relationship, indicating a conventional ion emission phenomenon in LMIS [2,12,19–22]. The slopes of the curves are about 6.0 μA/(V/μm).

To extract charged droplets from liquid metal, a high electric field should be applied on the surface of liquid metal. It is obvious that a sharp tip can greatly enhance the electric field [13,14,22,23]. For a single emitter in LIMS, E_0 which is related to the geometry of the emitter has been given as [22,23]:

$$E_0 = \ln\left(\frac{2\mathrm{h}}{\mathrm{r_c}}\right)\sqrt{\frac{\mathrm{r_c}\sigma}{\varepsilon_0 \mathrm{h}^2}} \quad (1)$$

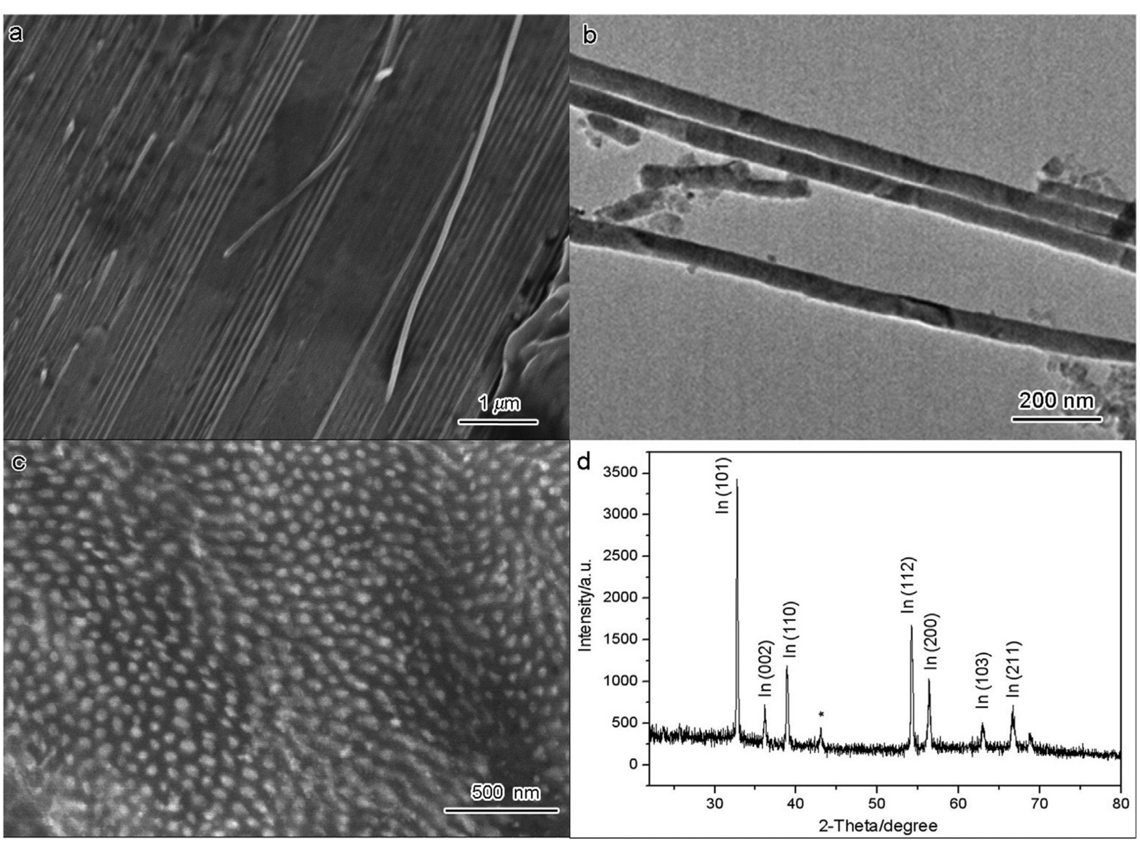

Fig. 2. Morphologies and crystal structures of In nanowires grown on AAO template. (a) A FE-SEM image taken from the side of the sample after breaking AAO template; (b) A TEM image of In nanowires after completely etching away the AAO template; (c) A FE-SEM image of the surface of the sample; (d) XRD pattern of the sample.