A Novel Route to Prepare Nano-Hg_2Cl_2 Arrays

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Recently, we find a novel route to prepare nano-Hg_2Cl_2 in the pores of alumina membrane. To the best of our knowledge, it is first time for preparing nano-Hg_2Cl_2 arrays. The nano-Hg_2Cl_2 has a potential application as nanoelectrode.

The ordered nanostructure materials have been attracting attention more and more recently, because of both their fundamental importance and the wide range of potential applications in nanodevices. Anodic alumina possesses uniform and paralleled porous structures and hence they have been used as an ideal template to prepare nanoparticle arrays extensively.[1-4] In this letter, we prepared nano-Hg_2Cl_2 arrays by porous alumina template.

A two-step anodization process as described previously fabricated the anodic alumina membrane (AAM).[5] In brief, the high purity aluminum foil was anodized in oxalic acid, then the specimen was immersed in a mixture of H_3PO_4 and H_2CrO_4 to remove the alumina layers. Then, the aluminum foil was anodized again under the same conditions. At the bottom of the alumina membrane existed the remaining aluminum layer and a barrier layer of alumina existed between porous alumina and aluminum layer. The aluminum layer was removed by solution of $CuCl_2$. The barrier layer of alumina was removed by floating the alumina membrane on surface of 5% H_3PO_4.

The porous alumina membrane with ordered hole arrays was prepared (Figure 1). A layer of Au was sputtered onto one side of the membrane served as an electrode in a standard three-electrode electrochemical cell. The electrolyte contained 80g/L $ZnSO_4{\cdot}7H_2O$ and 20 g/L H_3BO_4. The electrodeposition was performed at 1.25 V relative to the Ag/AgCl reference electrode. After electrodepositing, the Zn nanowires were embedded in AAM[6].

The alumina membrane with Zn nanowire arrays was immersed in a saturated $HgCl_2$ solution at the room temperature. In the pores of alumina membrane, the $HgCl_2$ was reduced to Hg_2Cl_2 by metal Zn and the Hg_2Cl_2 arrays were prepared.

The final sample was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). All peaks in XRD pattern (Figure 2) were indexed to the tetragonal phase of Hg_2Cl_2 with a = 4.480 Å, c = 10.906 Å, in good agreement with JCPDS No. 26-312. The TEM image (Figure 3) indicates that the nano-Hg_2Cl_2 constituted of spherical crystallites and the maximum diameter was about 70 nm, in agreement with the pore diameter of alumina membrane.

In reaction process, the formation of Hg_2Cl_2 is associated with the following

$$Zn + 2\ HgCl_2 = Hg_2Cl_2 + ZnCl_2$$

Here the metal Zn is a reductant, and the pores of alumina membrane are used to limit the size of nano-Hg_2Cl_2. The growth of Hg_2Cl_2 particles can be controlled by the pore diameter of alumina membrane.

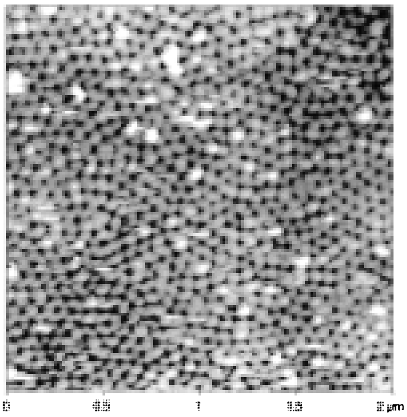

Figure 1. AFM image of surface of alumina. The aluminum was anodized in 0.3 M oxalic acid. The anodizing voltage was 20 V and temperature of electrolyte was kept constant at 5 ℃.

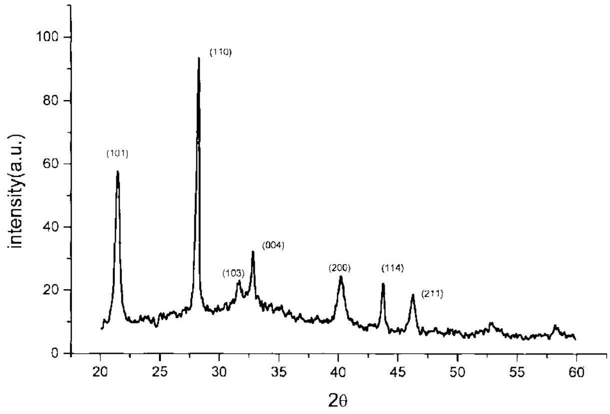

Figure 2. The XRD of Hg_2Cl_2 embedded in pores of porous alumina, operating on a Japan Rigaku DMAX-γ A X-ray diffraction with graphite-monochromatized Cu Kα radiation.

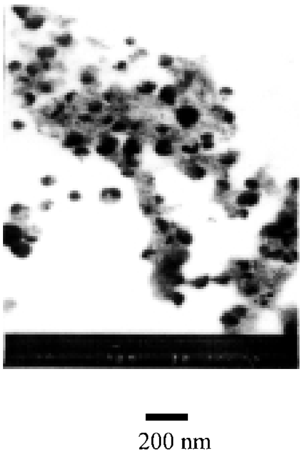

Figure 3. TEM image of Hg_2Cl_2, Hitachi-800 transmission electron microscope using an accelerating voltage of 200 kV.

Further studies will be extended toward the following two directions: one is to change the pore diameter of alumina membrane for preparing the smaller nanoparticles in the pores of alumina membrane; the other is to utilize Zn to reduce the another compound to nanoparticles arrays in alumina membrane.

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