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Controlled Growth of Highly Aligned Cu Nanowires by Pulse Electrodeposition in Nanoporous Alumina

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Metallic nanowire networks are emerging as potential replacements for transparent conducting oxide coatings because of their high conductivity, flexibility and relative transparency. However, a cheap, reliable and controlled manufacturing process is required to exploit this and the surface of the copper nanowire needs to be protected if high conductivity is to be retained. In this study a fabrication method for highly aligned and densely packed copper nanowires with controlled length using pulse-electrodeposition and a nanoporous alumina template has been developed. Nanoporous alumina was obtained by anodisation of pure aluminum in oxalic acid using a two-step anodisation process. In order to provide the conductivity at the bottom of the pores, a dendritic structure at the interface was created through the stepwise voltage reduction method with a voltage reduction rate of 15 V/s followed by mild chemical etching. Highly repeatable near 100% filling of copper is achieved. Copper nanowire length was highly controllable from 100 nm to 2 μm with a fixed diameter of 60 ± 5 nm by monitoring current density during the deposition. Such controlled growth of Cu nanowires could lead towards transparent conducting layer applications but the protection of the material against oxidation remains an issue.

Keywords: Anodising, Nanoporous Alumina, Barrier Layer Thinning, Cu Nanowires.

1. INTRODUCTION

One dimensional nanostructures such as nanowires and nanorods have become a major subject of research over last few years because of their enormous potential applications in nanotechnology.^{1–3} For instance, copper nanowires have recently been suggested as excellent candidates for transparent conducting films because Cu has high intrinsic conductivity; it is a potential alternative to indium tin oxide (ITO) and silver nanowires. In addition, Cu is only 6% less conductive than Ag, very abundant (1000 times more abundant than Ag), and 100 times less expensive than Ag or ITO,⁴ and several studies have demonstrated how copper nanowire films can achieve high optical transmittance with high electrical conductivity.^{5–7} However, there remain challenges for the passivation of the copper nanowires because they are prone to oxidation under ambient conditions, and thus they cannot retain performance comparable to that of ITO.

To date, a variety of methods have been developed to fabricate nanowires.^{1,2,8,9} Controlled nanowire geometry

has generally been achieved by using lithography,^{8,9} however this technique is complex, expensive and requires specialized equipment with inherent limitations.¹ The template-based approach is an alternate method to fabricate nanowires,¹⁰ and substrates such as mesoporous silica, patterned polycarbonate and nanoporous alumina are used to create the template.^{11–13} Porous alumina produced by anodisation of aluminium is often used as its geometry can be tuned.^{11,14} Despite this only a few studies have shown that metal nanowires can be created in the pores of porous alumina using the electrodeposition method. The reason is that a non-conducting layer that exists at the interface between porous alumina and the aluminium substrate, and this is sufficiently thick that it does not allow electrons to tunnel through during electrodeposition.^{15,16}

To get highly ordered nanoporous alumina on an aluminium substrate, a two-step anodisation process can be used that was introduced by Masuda et al.¹⁷ The geometry of porous alumina (pore diameter, interpore distance and film thickness) can be controlled by controlling the anodizing parameters such as anodising voltage, electrolyte concentration, temperature and time of anodisation. In all

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cases a non-conducting dense oxide layer called the barrier layer exists at the interface between the porous alumina and aluminium.¹⁸ This oxide layer prevents electrochemical deposition of metal.^{16, 19–22} However previous studies have shown that the barrier layer can be reduced or thinned either by cathodic polarization^{23–25} or stepwise reduction in anodisation voltage/current.^{10, 15, 26–29} Cathodic polarization involves reversing the polarity in a different electrochemical bath, and electrochemically etching the barrier layer, however it not only etches the barrier layer but also etches the pore walls thickness of porous anodic alumina (PAA).²⁵ To overcome this problem without affecting the wall thickness a stepwise voltage reduction can be used.^{10, 15, 26–29} In this method, the voltage is reduced in steps immediately after the second anodisation process. Since the geometry depends on the anodising parameters (for example pore diameter is linearly proportional to the anodising voltage) the voltage reduction process results in a dendrite like pore structure at the bottom of the main pores.²⁸ After thinning of the barrier layer by such a process metal nanowires can be fabricated by filling metals into the pores.¹⁶

Metal nanowires can be prepared by several other methods, but pulse electrodeposition (PED) in the template is a very simple, controlled, highly repeatable and cost-effective method and it also can be applied in wide range of materials. There are several studies of fabrication of nanowires such as Ag, Ni, Zn and Co nanowires have been produced using PAA template.^{16, 30–32} Previous studies have also reported that the growth of nanowires by the electrochemical deposition method prefers certain axial crystallographic orientations which are influenced by electrochemical deposition conditions such as applied potential. The growth of metal crystallites during electrodeposition using porous materials is associated with a mobility assisted growth model that requires nucleation from the cathode substrate at the bottom of the pores.³³ To date most research has focused on the ordering of metal nanowires with uniform diameter (i.e., the control of their position and spacing), deposited in porous alumina with a regular, ordered structure. However, it is also essential to control the length and diametric uniformity along the length of nanowires for applications such as transparent conducting films. In literature, a few articles have reported the fabrication of Cu nanowires,³⁴ but some questions still remain about how to control the length of Cu nanowires, their diameters and uniformity along their length as maintaining diffusion metal ions from bulk electrolyte is more challenging.

In this work a highly controlled growth of Cu nanowires is achieved in a PAA template formed by anodisation of pure aluminum (Al) by creating a dendritic porous path at the interface of the PAA and the Al substrate during the anodisation process. Copper is then deposited by pulsed electrodeposition and after mild etching of the alumina

template this results in highly aligned Cu nanowires. The thickness of the nanowires is controlled by the anodisation process. The ability to tune the length and thickness of such ordered copper nanowires could provide a large flexibility in material choice for transparent conducting film applications.

2. EXPERIMENTAL DETAILS

Copper nanowires were fabricated by filling metal into nanoporous alumina (NPA) by the pulse-electrodeposition method. The process is shown schematically in Figure 1. PAA was obtained by anodisation of pure aluminium. Pure Al sheet (99.999%) with a dimension of 10 mm × 10 mm × 2 mm was obtained from Advent Research materials. Before anodisation the pure Al samples were subjected to a series of pretreatment processes. Mechanical polishing was carried out using silicon carbide paper with different grit size varying from 400 to 2500, followed by 0.5 μm diamond paste used on selvet cloth to get mirror finish surfaces. Subsequently, the samples were electrochemically polished in a solution of 20% perchloric acid in ethanol at a constant voltage of 10 V for 3 minutes. Initially the current density was high at 200 mA/cm². It subsequently decreased and after about 30 s it got stabilised at 70 mA/cm². After electropolishing, the samples were rinsed several times in DI (deionized) water. The average roughness value (R_a) was measured using atomic force microscopy (AFM) and it was around 10 ± 2 nm for a scan length of 90 microns. After the pretreatment process, the first step anodisation was carried out in a solution of 0.3 M C₂H₂O₄ (oxalic acid) at constant voltage of 40 V at 18 °C for 30 minutes. The current density was high (100 mA/cm²) initially, measured as soon as the samples were immersed in the electrolyte bath and it rapidly decreased to 5 mA/cm² in few second and remained constant. The non-uniform porous alumina layer which was obtained after the first anodisation, was then chemically dissolved (chemical etching) in a mixture of 6 wt% H₃PO₄ (Phosphoric acid) + 1.8 wt% H₂Cr₂O₄ (Chromic acid) at 50 °C for about 30 minutes. The dissolution time had to be precisely controlled to just etch out the porous alumina leaving dimples at the aluminium substrate. These ordered dimples formed the nucleation sites for the pores in second step anodisation.¹⁷ After chemical etching, the second anodisation was carried out under the same conditions as the first anodisation. Thereafter the barrier layer was thinned gradually by reducing the anodising voltage from 40 V to 0 V linearly in steps of 1 V every 15 seconds.³⁵ After barrier layer thinning, the mild chemical etching was carried out in 0.63 M C₂H₂O₄ solution at 25 °C for 2 hrs. The samples obtained were rinsed in distilled water several times and then dried under a nitrogen purge.

The pulse electrodeposition (PED) method in a mixture of 0.5 M Cu₂SO₄ (copper sulphate) and 0.57 M H₃BO₃ (boric acid) solution was used for filling Cu into the pores

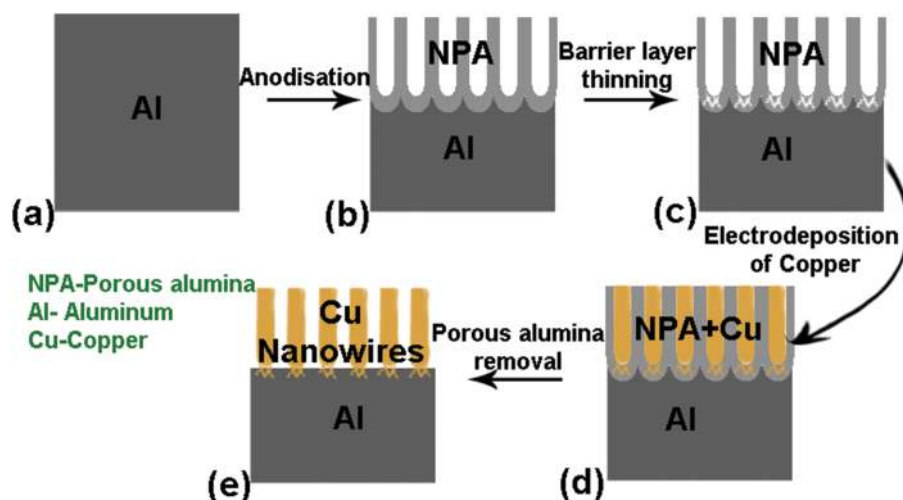


Figure 1. Schematic representation of the formation of copper nanowires (CuNWs) using a nanoporous alumina (NPA) template: (a) Electropolished pure aluminium sample (b) two-step anodisation in oxalic acid to obtain highly ordered porous alumina, (c) barrier thinning performed by reducing the voltage stepwise, (d) Cu filling into the porous alumina by pulse electrodeposition and (e) porous alumina removed to get CuNWs.

of the porous alumina template. Before electrodeposition the sample were kept in the solution for 2 hrs. Electrodeposition was carried out into the nanoporous template using Pt as a counter electrode. A positive pulse of 7 V for 3.2 ms and a negative pulse of 17 V for the duration of 3.2 ms were applied to deposit copper followed by a delay of 50 ms. The delay time (electrode off) allowed for compensation of metal ion depletion near the cathode. In order to liberate formed Cu nanowires from the PAA membrane chemical etching in 0.1 M NaOH was performed for 2 minutes at 30 °C.

The morphology of the porous alumina and Cu nanowires was analysed using field emission scanning electron microscopy (FE-SEM). To obtain reliable data at least a hundred nanowires were measured using Adobe Photoshop CS5 software in order to get quantitative diameter and length of nanowires. The crystal structure was characterised using X-ray powder diffraction (XRD) with Cu K α radiation ($\lambda=0.1542$ nm). XRD was conducted with sample support surface perpendicular to the length of the nanowires.

3. RESULTS AND DISCUSSION

A uniform NPA coating was formed using the two step anodisation procedure introduced by Masuda et al.¹⁷ The variation in current density during the anodisation using the two-step process is shown in Figure 2(a). The current density is approximately constant during the anodisation stage. The corresponding SEM images of the top surface and cross-sectional view of the porous alumina taken after 10 minutes of anodisation are also included in the figure. The dimensional parameters of the structure were calculated from the top surface and cross-sectional SEM images. The porous alumina template possesses nearly equal pore diameter of 45 ± 5 nm and interpore distance

of 100 ± 5 nm along the pore length (Fig. 2(a)). The two-dimensional ordered honeycomb porous structure can be seen from the bottom of pores. The porous oxide layer and substrate are separated by a compact oxide barrier layer.

This non-conducting barrier layer in-between the porous alumina and the aluminium substrate is then reduced using the stepwise reduction of voltage just after the

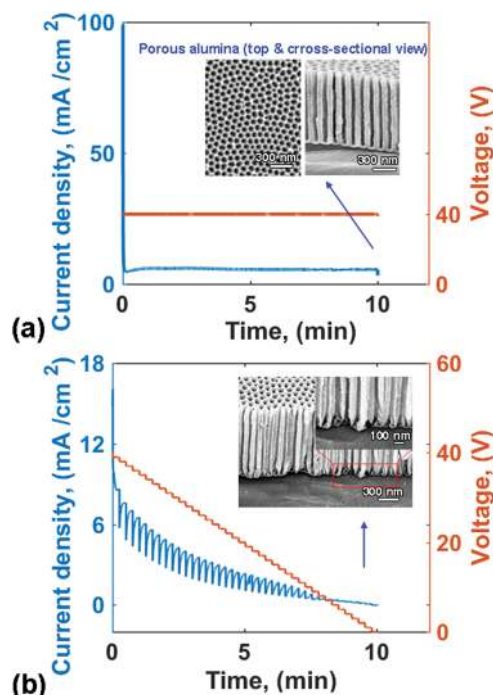


Figure 2. Current density and voltage versus time graph (a) during anodisation and corresponding SEM top and cross-sectional view of the porous alumina (b) during voltage reduction at the rate of 1 V/15 s after the anodisation and SEM cross-sectional view after creating dendritic path at the bottom of the pores of the porous alumina template.

second anodisation in the same electrolyte as shown in Figure 2(b). Yadav et al.³⁵ had found that the dendritic structure formed by reducing the voltage stepwise by 1 V/15 s resulted in more than 95% of pore barrier layers being removed over a 10 mm × 10 mm area. Choosing a linear reduction of voltage offers fine control over the resulting dendritic structure at the pore base.³⁵ In order to remove the irregularities in the dendritic structure a mild chemical etching was done in 0.63 M C₂H₂O₄ solution at 25 °C after the voltage reduction. This process is different from the one reported by Nielsh et al.¹⁶ wherein the pore widening is carried out before barrier thinning. We found that our procedure helped in controlling uniform deposition of Cu in all the pores distributed over 1 cm × 1 cm area. The SEM cross-sectional view (Fig. 2(b)) confirms the thinning of the barrier layer at the bottom of the pores of the porous alumina. The shape of the thinned structure depends on the steps of potential applied during the voltage reduction. However, the kinetic control of the metal growth does not depend on the number of branches.³¹ It is worth noting that the diameters of the pores at the bottom are smaller, generally by few nanometres, than the diameter of the pores in rest of the coating which is due to reduction of voltage. This does not affect the subsequent metal deposition.

After the two-step anodisation and creating the conducting path at the bottom of the pores copper was filled into the pores of PAA using the pulse electrodeposition (PED) method.^{16,30,36} The current density measured in a single pulse during the deposition was shown in Figure 3(a). The positive pulse was applied first which resulted in copper growing uniformly in the pores.³⁵

Initially, during the dendritic filling the current fluctuated for about 30 s, and then the growth of CuNWs started with very little variation in the current pulse. It was observed that the current increased slightly at the start of the positive pulse and then exponentially decayed to a value of about ~250 mA/cm² after less than a millisecond (Fig. 3(a)). This was, probably, because any uneven growth of the nanowires was dissolved favorably at the start of the positive pulse.^{16,37} During filling the deposition current remained at steady state (~270 mA/cm²) and then suddenly increased in magnitude (~500 mA/cm²) that indicated overgrowth on the surface (Fig. 3(b)).

Due to the anodizing process, vertically oriented large pores possessed a regular hexagonal pattern of cylindrical pores spread over the entire area of the porous template.¹⁸ However, during deposition, some nanowires in the template grew faster and reached the top of the template creating uneven growth on the surface. The unfilled or half-filled pores were blocked which might have been due to hydrogen bubbles and, as a result, further electrolyte transportation into the unfilled pores was terminated. Incomplete, uneven filling of pores may have happened because of the high growth rate of the

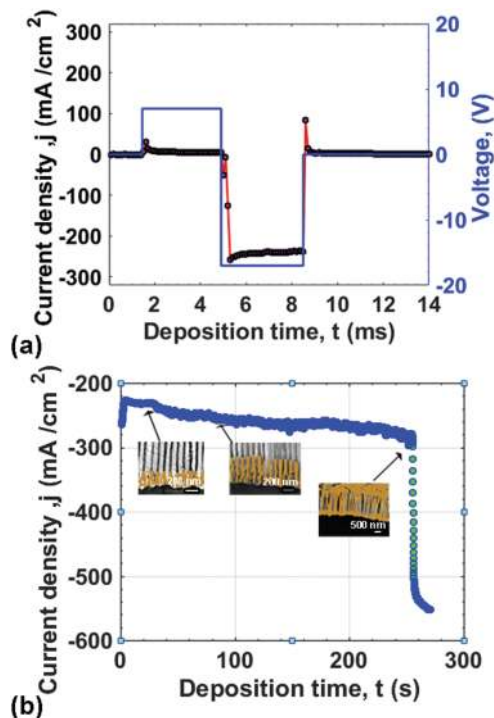


Figure 3. Current density versus time during pulsed electro deposition: (a) Current for individual pulse. (b) Variation in current density of the deposition pulse during electro deposition. In the initial pulses all the dendrites were filled and the sudden increase in the magnitude of current indicated that the Cu had been filled inside the pores and overgrowth on the surface began.

nanowires. Therefore, in order to fabricate uniformly distributed nanowires and prevent uneven growth, it was essential to optimize the positive pulse during the deposition. For instance, we have found that same pulse duration with slightly lower potential than the negative pulse, could prevent the uneven growth.³⁵ Once the porous template was filled with CuNWs, the current increased suddenly as shown in Figure 3(b). This can be used to define the end point of the nanowire deposition. By using optimized pulse conditions, it was ensured that almost all the pores were filled by Cu. The total deposition time controls the length of the CuNWs.

Selective dissolution of alumina in NaOH allowed the production of free standing nanowires. Figure 4 shows SEM images of the top surfaces of CuNWs with different length obtained in this manner. All CuNWs has approximately the same diameter which did not vary systematically along the fibre length. It was observed that the diameter of the freestanding nanowire was slightly larger than the initial mean pore diameter of the template. The measured mean diameter of the CuNWs was 60 ± 5 nm, which was about 5 nm larger than the mean diameter of the pores. This was due to the widening of pore diameters of the porous alumina as it was immersed in a solution of pH ~ 4 before and during the electro deposition process. The 10 nm discrepancy was needed to be accounted for if

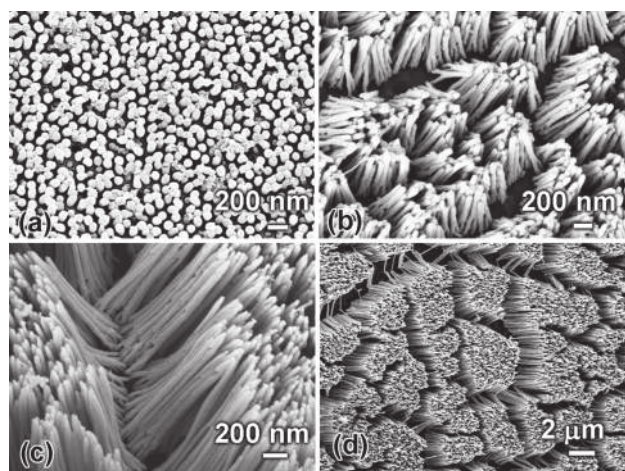


Figure 4. SEM images of CuNWs after removing the porous alumina, deposited with various time of (a) 5 sec, (b) 1 minutes, (c) 2.2 minutes, and (d) 4 minutes.

precise control of copper nanowire diameter was required. It was seen that almost all growth over a large area contained CuNWs but these were conglomerated into separate bundles. This occurred as a consequence of surface tension effects of the solution/distilled water as nanowire had been drier after the etching.

Figure 4 illustrates that the vertically aligned CuNWs lengths vary from a few nanometres to microns in size and could be controlled by monitoring the current during deposition. By varying deposition current application from 5 seconds to 4 minutes the length of CuNWs can be tuned from 50 nm to 2 μm . This confirmed that the time of deposition was controlling the filling of Cu into the pores of alumina.

Figure 5 shows the XRD patterns of the CuNWs on the aluminum substrate formed by pulsed electrodeposition. The CuNWs have the expected fcc structure with a $\langle 111 \rangle$ preferred fibre axis orientation. It has been reported that the most predominant distinct reflection in XRD pattern in CuNWs is Cu (111) when grown perpendicular

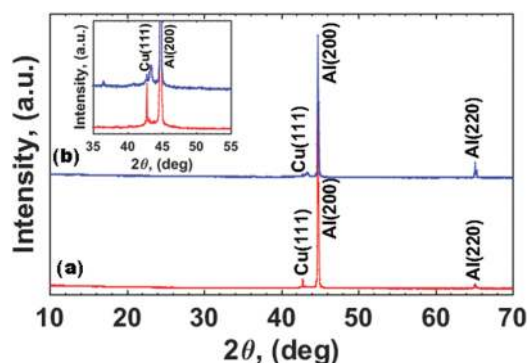


Figure 5. XRD patterns of CuNWs deposited in the porous alumina template (a) after completely removing the porous alumina (b) after partially removing the porous alumina.

to a substrate surface.³⁸ However the preferred orientation depends on the deposition conditions.³³ In the case of the electrodeposition technique it has been reported that the crystallinity depends on electrodeposition parameter like deposition potential and temperature.³³ By controlling the temperature and potential, single crystal of Ni nanowires has been produced.³³ It has been also reported that the electrodeposition method can allow accomplishing the uniform preferred growth orientation by varying the applied potential.³³ The same result was observed here for Cu. Figure 5(b) shows the XRD patterns of the partially etched Cu NWs on the aluminum substrate. The XRD results confirmed that there was no significant difference which arises due to the alumina etching process. The large peak at 2θ value 44.7° was characteristic of (200) aluminium,³⁹ whilst the intense (111) peak of the aluminium substrate is not visible in the data due to the texturing. The major peaks of Cu and Al therefore had different orientations in Figure 5(b).

4. CONCLUSION

Highly aligned copper nanowires with controlled length can be fabricated by pulsed electrodeposition of Cu into the pores of NPA template obtained by anodisation of pure aluminum. Uniform deposition is obtained after creating the dendritic structure at the bottom of the pores through the stepwise voltage reduction method. A uniform almost 100% filling of copper is achieved after optimizing the total time of deposition. The CuNWs length was highly controlled from 100 nm–2 μm with the diameter of 40 nm by monitoring current density during the deposition. XRD patterns confirm that the Cu NWs are highly oriented with axes in Cu [111] direction. The growth of CuNWs with controlled length and uniform diameter was obtained using porous alumina templates. This can have implications towards various nanoelectronic applications such as transparent conducting films. However, the passivation of the nanowire surface to prevent oxidation remains an issue to be solved.

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