TEMPLATE-BASED SYNTHESIS OF Al/Ni NANOCOMPOSITE ON SILICON SUBSTRATE

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Introduction

Since Masuda and Fukuda [1] first reported the highly ordered porous alumina membrane in 1995, porous alumina membrane (PAM) has been intensively used as a template in nanomaterials fabrication [2-4]. Nickel (Ni)1D nanomaterials, especially on rigid substrate, fabricated using PAM are attracting a great interest, because of their large potential application in magnetic [5], field emission [6], and sensors [7] devices, and so on.

However, there are nearly no reports about preparation of Al-coated Ni nanorods/nanowires on substrate as nanoenergetic materials. Due to their large contact area and rigid substrate, free-standing Ni nanorods on silicon substrate followed by Al deposition can be applied to fabricate a new nanoenergetic material, which holds great promise to be integrated into small energetic devices with MEMS scale fabrication methods.

Experimental

A 200 nm thick Ti layer was deposited by the radio frequency (RF) sputtering on a cleaned p-type (111) silicon wafer. Al film was subsequently deposited on top of the Ti layer with a thickness of 600 nm. PAM was prepared following the two-step anodization procedure, which was carried out in 0.3 M oxalic acid solution at room temperature under the constant voltage of 40 V. After that, the anodization was continued for 60 min to remove the barrier layer in situ [8] .

Before electrodeposition Ni nanorods, the PAM/Ti/Si sample was immersed in 5 wt% H_3PO_4 solution at 30 ℃ for a few minutes to completely remove the remaining barrier and widen the pores. The electrodeposition system consists of the PAM/Ti/Si as cathode, Pt wire as anode, $Hg/HgSO_4$ as the reference electrode and the electrolyte (a mixture of 100 $g \cdot L^{-1}$ $NiSO_4 \cdot 6H_2O$ and 30 $g \cdot L^{-1}$ H_3BO_3, with 4.0 in pH value). Ni nanorods were deposited into the PAM by an electrochemical workstation (CHI1140A, Chenhua, China). The pulse mode consists of -5 mA for 0.08 seconds and 1 mA for 0.02 seconds. Each pulse was applied of 0.1 seconds, and the total time was 90~150 seconds. After electrodeposition, the PAM template was removed in 2 wt% NaOH solution. A film of Al was deposited on the surface of the Ni nanorods to forming the Al/Ni nanocomposite.

The morphology of PAM, Ni nanorods and Al/Ni nanocomposite was examined by field emission scanning electron microscope (FESEM). Also energy dispersive X-ray (EDS) studies were done to verify the presence of elements we need.

Results and Discussion

Fig. 1 shows FESEM image of the PAM after pore-widened for a few minutes. It can be clearly seen that the PAM pores vertically stand on the substrate. And at the PAM/Ti interface, the TiO_2 conductive channels are formed, which can be utilized routeways for ions transferring in the electrodeposition. Inset shows the top view of the PAM, the average pore diameter is about 50 nm and the pore density is about $10^{10}/cm^2$.

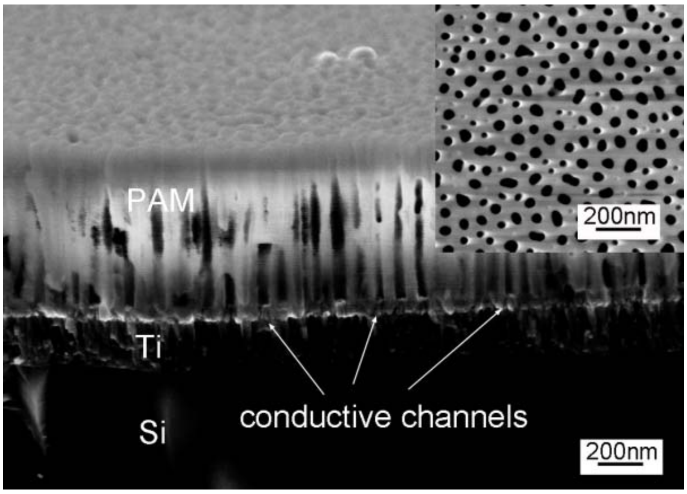

Fig. 1 FESEM image of PAM formation under 0.3 M oxalic acid.

A FESEM image of grown Ni nanorods is shown in Fig. 2, inset is the surface morphology of the free-standing Ni nanorods. It can be observed from the figure that the Ni arrays have been grown in a relatively large area even though there are a few regions where the nanorods are absent. The Ni nanorods are self-supporting and vertically stand up to the Ti/Si substrate surface.

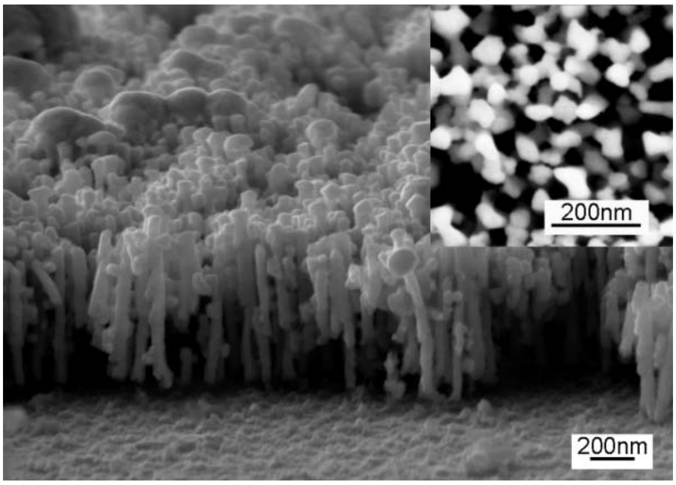

Fig. 2 FESEM image of Ni nanorods on Ti/Si substrate.

Fig.3 is the EDS analysis result of above sample. The image clearly shows the presence of the element Ni. To verify this Ni is actually for the Ni nanorods grown on the substrate, we also have performed the EDS analysis of the PAM/Ti/Si sample. The result is just as Table. 1 shows. Obviously, the element Ni is only presence in the Ni/Ti/Si sample. Therefore, we can safely conclude that Ni nanorods are successfully formed on the substrate by pulse electrodeposition.

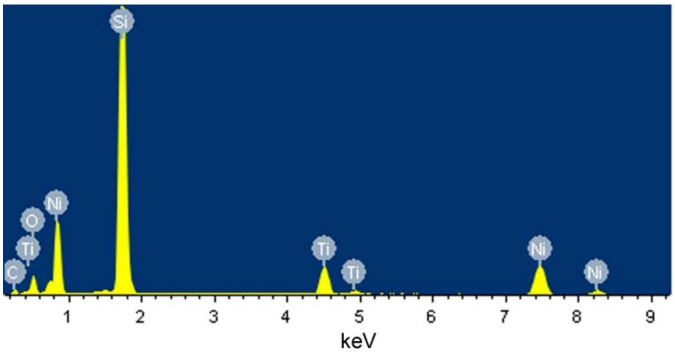

Fig. 3 Energy dispersive X-ray analysis of Ni nanorods/Ti/Si structure showing the presence of Ni.

Table. 1 EDS analysis results of PAM/Ti/Si and Ni/Ti/Si samples

Samples	Elements & Atomic percents, %					
	Si	Al	O	Ti	Ni	Others
PAM/Ti/Si	27.97	7.92	56.76	2.76	—	4.59
Ni/Ti/Si	39.26	—	20.68	4.44	11.18	24.44

Fig.4 and its inset show the respective oblique angle and top view FESEM images of a representative Al/Ni nanocomposite sample on Si substrate. About 300 nm Al is sputtered on the surface of the Ni nanorods and portion of the nanorods embed in the Al layer. It means that the Al is intimately integrated with the Ni nanorods, which results in the intimate interfacial contact between this two. The EDS analysis result (Fig. 5) of the Al/Ni nanocomposite on the Ti/Si substrate reveals that the element Al is presence in the map compared with Fig. 3, which is attributed to the sputtered Al film.

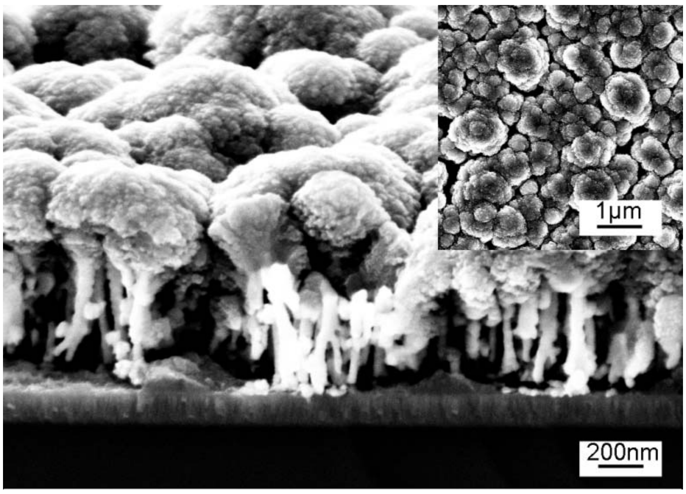

Fig. 4 The morphology and EDS result of the Al/Ni nanocomposite

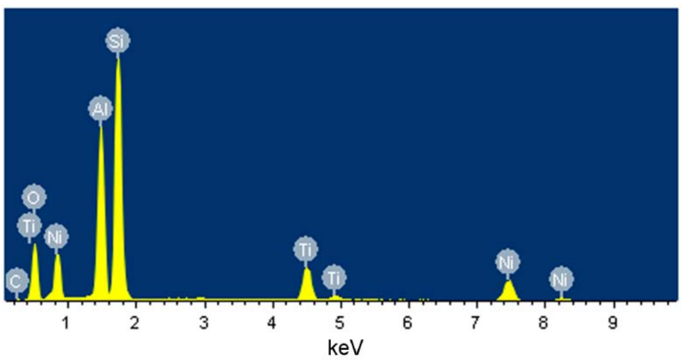

Fig. 5 EDS analysis result of Al/Ni nanocomposite.

According to our knowledge, Al/Ni system is well-studied, and it reacts according to the following equation.

Ni+Al→NiAl, △H= -118.4kJ/mol; △H, heat of reaction.

The adiabatic temperature of this reaction is more than 1911K and the initial temperature is about 425K which is below the melting temperature of Al (660℃). Therefore, the Ni nanorods prepared here with depositing an Al film have the potential application of heat source or energetic material in the silicon-based MEMS compatible device. This technique can also be used for the formation of other bimetallic nanocomposites, nanothermites, and so on.

Conclusion

Using PAM template, Al/Ni nanocomposite was successfully prepared on silicon substrate. Initially, the PAM was formed on the Ti/Si substrate via anodiazation process. Then, Ni materials were deposited into the PAM by electrodeposition. Al film is sputtered on the surface of the free-standing Ni nanorods to realize nanostructure Al/Ni layer, which has the potential application of heat source or energetic material in the silicon-based MEMS compatible device.

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