Porous Silicon Templates with High Aspect Ratio for Nanocomposites Formation

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The paper describes the fabrication process of templates with high aspect ratio suitable for formation of nanomposites with enhanced anisotropy. The templates are thick ordered porous silicon layers formed with the pulsed galvanostatic mode. Characteristic feature of the developed mode is a short reversed polarity pulse of certain amplitude. In the present paper the achieved thickness of porous silicon templates is 50 μ m with pore diameter 80 nm. The aspect ratio of those structures 1:600. The maximum thickness of formed silicon layers is not principally limited allowing obtaining nanostructures with even higher aspect ratio.

deionized water.

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was cleaned in a hot RCA solution for 10 min. After each treatment step wafer was thoroughly rinsed in the

mode in a HF:H₂O solution with components ratio 1:4,

respectively. Current cycle included forward and re-

versed polarity pulses. Pulses configuration is presented

on the Fig. 1. The current density of a forward polarity

peak is 100 mA/cm² (1 second), reversed polarity -

 5 mA/cm^2 (0.5 second). Total cycle length is 10 seconds.

Anodization was carried out in a pulsed galvanostatic

1. INTRODUCTION

Nanocomposite materials are of high demand in the present-day technology of micro- and nanoelectronic devices manufacturing because of the unique combination of their characteristics and properties. One of them is the anisotropy. Structuring of nanocomposite at the formation stage with the secondary template is the most common way to achieve the anisotropy [1]. In this case, aspect ratio as a ratio of two geometrical dimensions of the given structure, for example diameter and height, becomes the most prominent parameter influencing the anisotropy.

Porous silicon is considered to be a very good template material because it can be formed using inexpensive electrochemical methods which are technologically compatible with current microelectronics industrial equipment and processes. Moreover, dimensions of pores and silicon crystallites forming porous silicon skeleton can be easily varied in the wide range [2]. Nevertheless, formation of porous silicon with high pore length/diameter ratio is the nontrivial task. The major obstacle in the traditionally used constant current mode is the dissolution of the pore walls at the bottom part during anodization due to solution depletion build-up as the pore length/diameter ratio increases. This leads to lowering of the mechanical strength of porous silicon under formation. This in turn often leads to formation of cracks during anodization or drying step [3, 4] especially for nano- and microstructured porous silicon. Among known techniques of reducing depletion and its effects is the introduction of stirring and surface wetting agents, or using of the potentiostatic mode [5].

In the present work we propose the formation method of nanostructured porous silicon in the pulsed galvanostatic mode with the aspect ratio up to 1:600 and more.

2. EXPERIMENTAL

N+-type antimony doped silicon wafer with resistivity 0.01Ω cm and crystallographic orientation (100) were used as the initial substrate. The native silicon oxide was removed from the wafer surface in a 4.5 % HF solution before the anodization step. Then wafer

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Fig. 1 – Current density vs. time plot during pulsed galvanostatic anodization process

It was experimentally determined that 17 cycles is needed for each 1 um of porous silicon layer depth. Thus, for obtaining 50 μ m of porous silicon the total process duration was calculated to be 8500 s (approximately 2 h 21 min). No stirring or agitation was used. After anodization the sample was rinsed and dried after anodization in open air for 24 hours.

3. RESULTS AND DISCUSSION

On the Fig. 2 and Fig. 3 the cross section and top view of the silicon wafer anodized in a developed regime is presented. Pore depth is $48.9 \ \mu m$. Pores have pronounced vertical structure. No branching, typical to porous silicon obtained in a constant current mode, is observed.

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b

b

Fig. 2 – Cross section of the porous silicon template obtained in a pulsed galvanostaic regime: middle section at 30k magnification (a), and the same region at higher magnification (100k) (b)

At higher magnification, one can see that pores have corrugated walls (see Fig. 2b). The corrugation step is roughly 60 nm which corresponds to the porous silicon thickness formed during single cycle (considering 17 cycles per 1000 nm).

Average pore diameter in the middle section of porous silicon layer is 80 nm, silicon crystallites diameter approximately 20 nm. It was determined that pore diameter dispersion is less than 30 %. The distance between pore centers remains virtually the same across sample surface.

Aspect ratio of obtained nanostructure is 1:600.

After preparation, drying and storing for several days under atmospheric exposure sample did not show any signs of cracking.

We suppose that switching to the pulsed anodization mode assist to ion transfer along the pores providing more fluoride anions at the bottom part of the pores and preventing thereby electrolyte depletion. The reversed polarity pulse helped to additionally refresh the surface of the pores. Thus more uniform and better defined pore structure was obtained. Fig. 3 – Top view of the porous silicon template obtained in a pulsed galvanostaic regime: overview (a) and the same region at higher magnification 200k (b)

4. CONCLUSION

In the developed galvanostatic anodization regime thick porous silicon layers having constant diameter of pores throughout the full thickness with high aspect ratio of 1:600 were obtained. Those structures can be used as templates for nanostructured composites with high anisotropy. Since pore width can be varied in a wide range by modifying current density and silicon substrate resistivity there are no obvious obstacles on the way for obtaining nanostructured templates with higher aspect ratio in developed anodization regime. We suppose that porous silicon templates with thickness up to 200 μ m and higher can be formed with the developed approach.

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