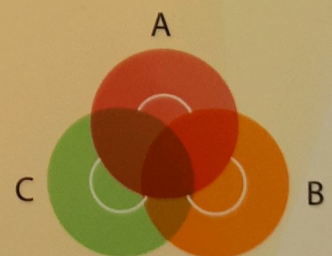


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Technologies for a Sustainable Future**
Issue 61



**MICRO AND NANO TECHNOLOGIES
ADVANCES IN BIOTECHNOLOGY**

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FOR A SUSTAINABLE FUTURE
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FEATURES OF TOTAL OPTICAL REFLECTION IN SILICON NANOSTRUCTURES OBTAINED BY METAL ASSISTED CHEMICAL ETCHING

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ABSTRACT

Total (specular+diffusive) reflection of nanostructured Si layers prepared by metal assisted chemical etching (MACE) of crystalline silicon (c-Si) wafers is investigated. The thickness of nanostructured Si layers is varied from 0.3 to 100 μm by increasing the MACE time. The time dependence of silicon nanostructured layers thickness on the etching time was found to be logarithmic. Spectra of the total reflection were measured in optical range from 0.2 to 2 μm . Depending on the thickness of sample, it was observed a low level of the total reflection near 1% in the ultraviolet (UV)-visible spectral ranges and a strong increase up to 80% in the infrared (IR) optical range above 1 μm . The low reflection in UV-visible region is explained by partial localization of light in Si nanostructures accompanied with the strong light absorption. The enhanced IR reflection is probably related to the strong scattering of light by silicon nanocrystals in the transparency region. The maximal total reflection in the IR range was found for the layer thickness of about 30 μm . An effect of doping of Si nanostructures by donor and acceptor impurities on the total reflection in the IR region was observed. The obtained results can be useful to prepare new materials with desired properties for photonic applications.

Keywords: silicon, nanostructures, total reflection, optics.

INTRODUCTION

Development of technologies of obtaining comparatively low cost nanomaterial with given stable optical properties is one of the topical issues of many related fields of technics and science [1-6]. Silicon (Si) nanostructures are exhibit unique physical and chemical properties, which are different from those of the bulk crystalline Si (c-Si) [7, 8]. Metal-assisted chemical etching (MACE) of c-Si is a simple and flexible method to prepare Si nanostructures as nanowires and porous layers with required optical properties [9]. The technology of MACE is based on the selective chemical dissolution

of silicon using noble metal particles as a catalyst. In terms of catalyst, particles of silver are preferable because of their low cost and good chemical properties. The process shows a good controllability by setting etching conditions allowing obtaining of silicon nanostructures of required thickness and given parameters [10]. Despite of numerous studies devoted to the optical properties of Si nanostructures prepared by MACE [11,12], there are many open questions related to an influence of morphology of nanostructures and their doping level on the reflection and absorption of light. Our present work is devoted to investigation of the total reflection and absorption of prepared nanostructured Si layers with different thickness. The main purpose of research was to study the features of the formation of silicon nanostructured layers on the surface of silicon single-crystal plates of the p and n types of conductivity and to reveal mechanisms of growth of structures.

EXPERIMENT DETAILS

Samples of silicon nanostructured layers of different thickness were obtained by MACE of c-Si (100) substrates with different doping level in solutions based on hydrofluoric acid. In order to study the impact of conductivity type of the initial substrate on the growth characteristics of silicon nanostructured layers, we have used double side polished c-Si substrates with equal resistivity, which was equal to $1-10 \Omega \cdot \text{cm}$. Usually MACE is carried out in three stages: 1) deposition of metal particles, 2) chemical etching in a solution based on hydrofluoric acid and hydrogen peroxide, 3) removal of residual metal catalyst particles. In our experiments, we used silver particles as the catalyst of etching reaction. Before the beginning of each experiment on obtaining nanostructures, the original silicon substrates underwent chemical treatment in a 2% solution of hydrofluoric acid, involving the removal of a layer of natural oxide.

The structural properties of the obtained samples were studied by scanning electron microscopy (SEM) using a Lyra Tescan microscope and a Quanta 200i ons. The thickness of nanostructured Si layers controlled by the MACE time was varied from 0.1 to 50 μm . Spectra of the total reflection were measured in optical range from 0.2 to 2 μm using a spectrophotometer Jasco. All measurements were carried out at room temperature in air.

RESULTS AND DISCUSSION

Figure 1 shows SEM images of the c-Si surfaces of p-and n-type conductivity with deposited layer of silver particles within for 45 seconds. In the case of a p-type conductivity, sizes of silver particles diameters were 50-300 nm, it is clear that silver particles distributed evenly on the substrate, and in some places assembled in clusters. In the case of n-type silicon, deposited silver particles form much denser on the surface of wafer. One can see that during 45 seconds metal particles coalesce into large tenth micron scale clusters and cover almost the entire surface.

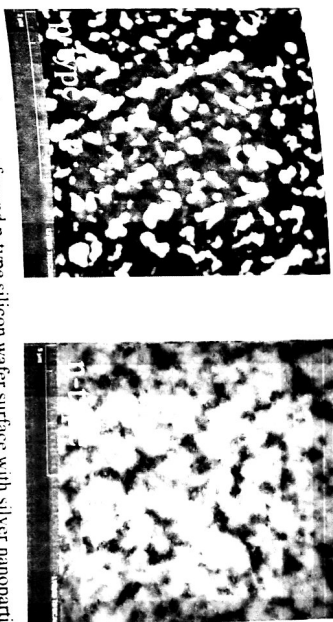


Figure 1 – SEM images of p-and n-type silicon wafer surface with silver nanoparticles on the top.

Charge carriers presence could explain such distribution and deposition rate of particles on the surface of the wafers. In the case of p-type conductivity, the surface is conditionally charged positively and Ag^+ ions, also having a positive charge, in most cases repel from the surface, forming islands, in places potentially advantageous for this. In the case of n-type substrate the surface is charged negatively, which leads to a rapid establishment of the Coulomb interaction between the surface and the silver ions. With increasing deposition time of metal particles, such clusters become much larger for substrates of both types of conductivity, which in turn leads to an increase in the risk of obtaining heterogeneous structures in the MACE process [13].

Cross section SEM images of silicon nanostructures layers grown on the surface of p- and n-type c-Si at different etching times are given in figure 2. Images shows that with increasing of etching time, the thickness of the nanostructured layer increases. For the case of p-type silicon substrate, the growth rate is on average 15% faster than for an n-type substrate, which is also due to the number of holes that determines the intensity of the rate redox reactions. The more holes, the more silicon atoms go into the electrolyte, and thus the deeper pores we get in the resulting structure.

Despite of the conductivity type of initial substrate the character of pore formation is quite common for both types. One can easily note that porosity of studied structures the porosity gradually decreases in the direction from the surface to the bulk Si during MACE.

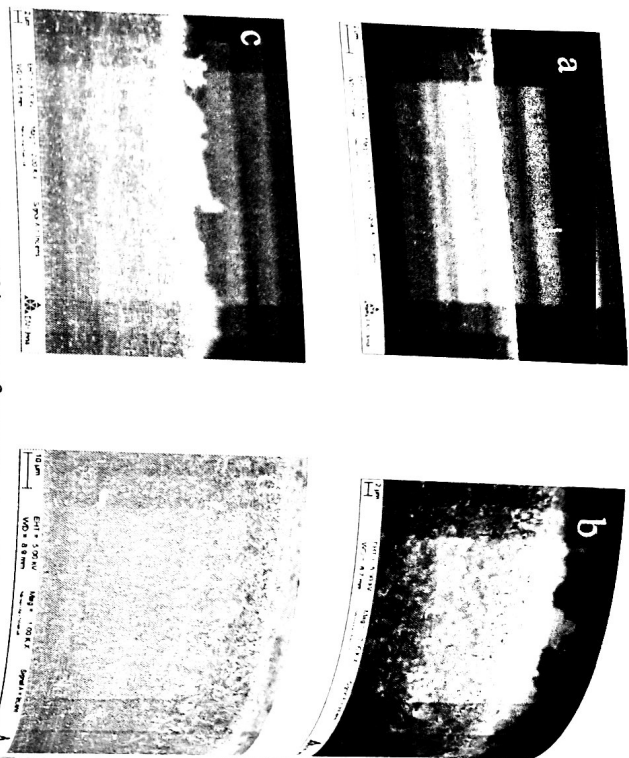


Figure 2. The cross section SEM images of nanostructured layers obtained by MACE on the p-type c-Si surface during: a) 1-hour, b) 10 hours, and the n-type c-Si surface during: c) 1 hour, d) 10 hours.

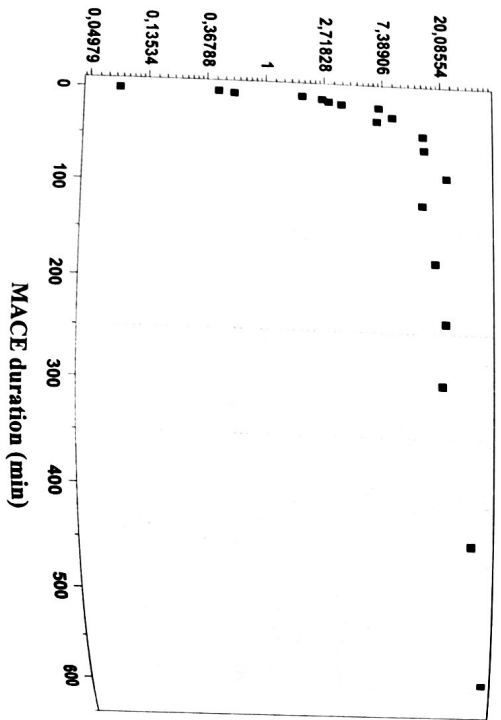


Figure 3. Dependence of the thickness of Si nanostructured layers on the MACE duration.

Further analysis of the thickness data of silicon nanostructure layers as a function of the etching time results the empirically established time dependence, which can be expressed as:

$$L = B \ln t + C \quad (1)$$

where L is the thickness of the silicon nanostructured layer, t is the time of MACE duration, B and C are constants.

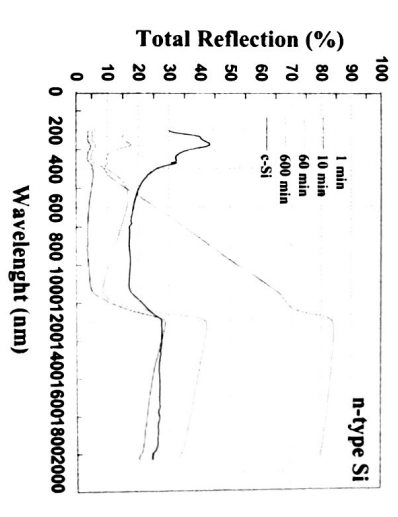
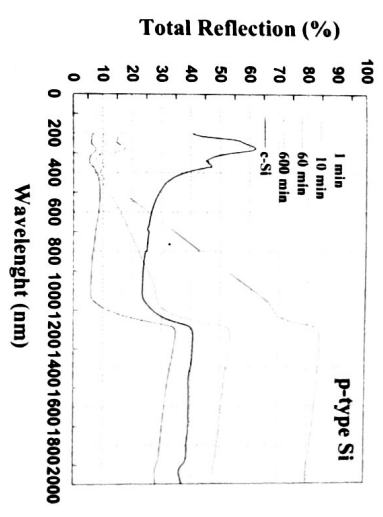


Figure 4. Total reflection spectra of silicon nanostructures layers obtained at different MACE times on the surface of p- and n-type c-Si.

The decrease in etching rate with etching time can be explained by the diffusion-controlled transfer of fluoride ions from the electrolyte solution to the nanostructured layer/crystalline silicon interface.

Spectra of total reflection of samples were measured in the wide optical range of 200-2000 nm. Spectra of total reflection of p- and n-type c-Si are shown in the figure 4. For different MACE times on the surface of p- and n-type c-Si we observe the effect of extremely low reflection structures of both conductivity types, we observe the effect of extremely low reflection down to 1% in the UV-visible parts and its increasing up to 80% in IR part of optical range depending on the thickness of sample. Extremely low reflection at the left period of range is due to effect of strong localization of light inside the structure and increasing of reflection probably could be explained by strong scattering of light on silicon nanocrystals. Since the thickness of samples start to exceed 30 μm we observe that transition, observed from total reflection spectra.

CONCLUSIONS

We presented the results of experimental studies of the formation and optical properties of nanostructured silicon layers prepared by metal-assisted chemical etching of crystalline silicon wafers of p- and n-types of conductivity with specific resistivity about 1-10 $\Omega\cdot\text{cm}$. The thickness of silicon nanostructured layers was found to a logarithmic function of the etching time. Depending on the thickness of sample, it was observed very low reflection near 1% in the UV-visible spectral ranges and a strong increase up to 80% in the IR optical range. The low reflection in UV-visible region is explained by partial localization of light in Si nanostructures accompanied with the strong light absorption. The enhanced IR reflection is probably related to the strong scattering of light by silicon nanocrystals in the transparency region. The doping of Si nanostructures was found to influence on the total reflection in the IR region. The obtained results can be useful to prepare new materials with desired properties for photonic devices in the IR spectral region.

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