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# Multifractal analysis and optical properties of nanostructured silicon layers

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ABSTRACT

For the optimization of the light trapping in semiconductor devices, especially in the solar cells, microtextured structures are commonly used. Structures with ultralow spectral reflectance were formed on flat and pyramidally textured single crystalline silicon wafers by using of the surface structure chemical transfer method (SSCT). This method is based on effective etching of treated surface in HF solution in contact with a Pt mesh. The structure of the SSCT layer is determined by the etching conditions and influences substantially its optical properties. We studied microstructural properties of the SSCT layers by the SEM and AFM methods and we analyzed obtained morphological information by the multifractal (MF) and Fourier methods. The properties of etched surface layers determine the spectral reflectance and Raman scattering. Exponential fall of the spectral reflectance is caused by the gradient of layer density and enhancement of Raman scattering is related to forming of small particle size fraction during the SSCT etching. Results of MF and Fourier analysis are used in interpretation of the optical properties of formed microtextured structures.



## 1. Introduction

Flat silicon surfaces show high spectral reflectance in the visible range of wavelengths (30–50%) due to the large difference in refractive indices of air and Si. For the optimization of the light trapping in semiconductor devices, especially in the solar cells, microtextured structures are commonly used. Commonly used methods of surface structuring employ multiple reflection of light from the pyramidal shapes formed by anisotropic alkaline etching of silicon using KOH, NaOH, TMAH and isopropanol [1–5]. The reflectance of pyramidal structure decreases to 10–20% [6]. To improve the device properties additional antireflection layer (such as silicon nitride SiN) can be formed, but this increases the production cost. Another way of production of Si structures with ultralow reflectance is based on catalytic activity of metals. Particles as Au, Ag or Pt in contact with Si surface immersed in etching solution penetrate into the Si volume and create the nanoporous structure [7,8]. Porous Si layers can be prepared also by anodic dissolution of crystalline Si [9]. This method is not suitable for treatment of larger Si surfaces because it produces nonunifom structure. The fabrication of porous structures causes also problems connected with forming of high quality pn-junctions due to the thickness of porous Si layer, which reaches 500–1000 nm.

In this work we prepared Si surfaces with ultra-low spectral reflectance by using Pt assisted etching of silicon surface. Si surface was contacted with a Pt mesh in etching solution. The mesh structure is transferred into the Si surface during the etching procedure and a nanocrystalline layer is formed (Surface Structure Chemical Transfer method, SSCT) [10–12]. We also prepared pyramidally textured Si structures etched by the SSCT method. In both cases (SSCT layers and pyramidal textures with the SSCT layer) we observed significant suppression of the spectral reflectance and changes in a Raman scattering connected with the development of the structure properties. We examined the surface structure by the scanning electron microscope (SEM) and by the atomic force microscope (AFM). Properties of the surface structure were studied by the multifractal methods (MF) and by analysis of the SEM and AFM images in the 2D Fourier domain (2D FFT). Results of this microstructural analysis are significantly connected with the optical properties, determined by the spectral reflectance and Raman scattering. Optical properties of textured surfaces are strongly related to the complexity of formed features revealed by the MF and 2D FTT analysis.

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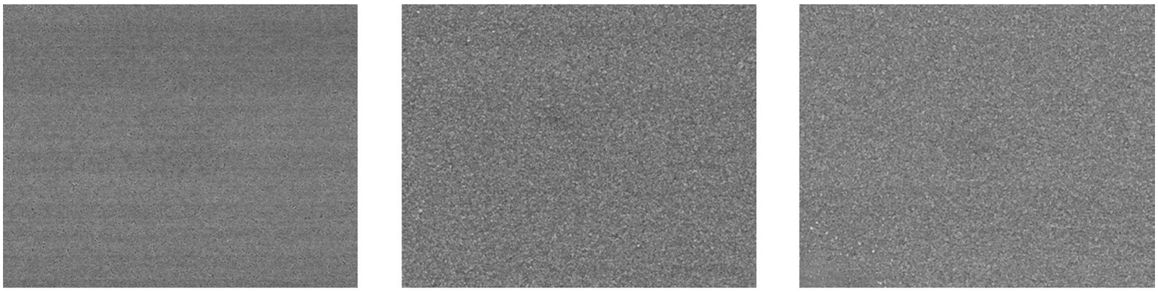
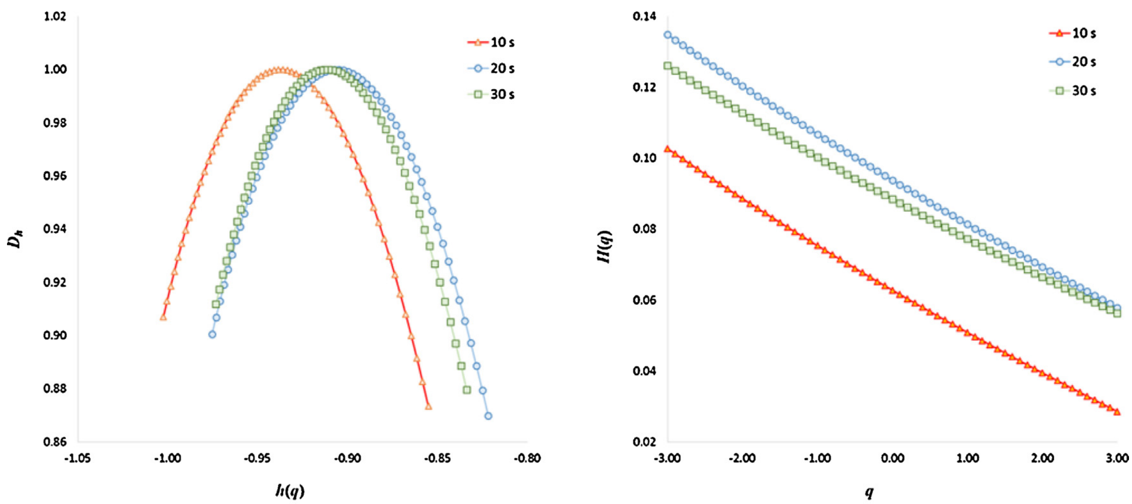


a) b) c)

**Fig. 1.** SEM images of the SSCT structure on flat Si. SSCT etching time is (a) 10 s, (b) 20 s, (c) 30 s.




a) b)

**Fig. 2.** Results of the MFDFA analysis of SEM images of the SSCT structure. (a) multifractal spectrum $D_h$, (b) generalized Hurst exponent $H(q)$. SSCT etching time is 10 s, 20 s and 30 s. SEM magnification is 2000.

## 2. Experimental

Single crystalline boron-doped p-type Si wafers with 200 μm thickness were etched in 1.5 M KOH solutions at 80–85 °C for 15 min to remove a saw-damaged layer introduced during slicing of Si ingots. Pyramidally textured surfaces were produced by anisotropic etching using 0.25 M KOH and 0.6 M isopropanol solutions at ~80 °C for 20 min. In fabrication of the SSCT surfaces the Si wafers were immersed in 15 wt% $H_2O_2$ and 25 wt% HF solution at room temperature, and a platinum mesh of a roller shape was contacted with the wafers. During the SSCT treatment, the Si wafers moved with the velocity of 2 cm/s in contact with the Pt mesh catalyst roller. We prepared structures with the SSCT layer formed on flat Si surface as well as at pyramidally textured surface by the prolongation of the SSCT etching procedure to 10, 20 and 30 s. SEM micrographs were measured by using a HITACHI S-2150 microscope with the incident electron energy of 20 keV and AFM scans were measured by the AIST-NT SmartSPM-1000 system. Reflectance spectra were recorded by using a JASCO V-670 UV–vis spectrometer with an integrating sphere. Raman spectra were measured by the Thermo Scientific Raman DXR microscope with 532 nm wavelength.

## 3. Results and discussion

### *3.1. Properties of the SSCT layers prepared on flat Si substrate*

The surface morphology of the SSCT layers as well as the pyramidal texture treated by the SSCT method is very complex and the optimization of the structure forming procedure requires sensitive information about the morphological properties. The development of surface properties was analyzed in our approach by the MF methods in connection with the surface treatment steps based on the SEM and AFM images. The microstructure of the SSCT layers was studied at various magnifications of the SEM images in range from 100 to 40,000. Fig. 1 shows SEM images of the SSCT structure prepared on flat Si surface by various etching time – 10 s, 20 s and 30 s. SEM magnification is 10,000.

Morphological information from these images was analyzed by the multifractal detrended fluctuation analysis (MFDFA) methods and these methods were also used for the evaluation of the AFM images. MFDFA analysis provides information about scale invariant content in analyzed data structure [13,14]. Scale invariance of data structure $x(s)$ is expressed by the power law $x(ps) = p^H x(s)$. Multifractal structures are particular kind of such structures, described by the presence of spatial variations in the scale invariant structure, showing local fluctuations with very small as well as large magnitudes and indicated by a spectrum of power law exponents $H$ (Hurst exponents). In our approach the sequence of rows of the SEM image are transformed into vector $x(s)$. This vector is detrended and the local fluctuations represented by the root-mean-square (RMS) are evaluated. From this RMS analysis the $q$-order Hurst exponent $H(q)$, the $q$-order singularity exponent $h(q)$ and singularity dimension $D_h$ (called multifractal spectrum) is computed. Results of the MFDFA analysis of the SEM images with magnification 2000 (the development of the multifractal spectrum $D_h$ and generalized Hurst exponent $H(q)$ with the SSCT etching time) are shown in Fig. 2. Mul-