Porous silicon surface feature size estimation using the reflectance spectrum

Christopher Lowrie*, Susan Earles, M. de Fernandez
Florida Institute of Technology, 150 W. University Boulevard, Melbourne, Florida 32901
*Corresponding author: clwindsurf@aol.com

ABSTRACT

In this paper we excite the surface of porous silicon with incoherent, broad band white light and observe the spectrum of colors reflected from the surface. Using an atomic force microscope images from red and green porous silicon samples are collected. In this paper we relate the optical color of the surface to the size of scattering features on the textured surface. From image segmentation using the watershed transform the height distributions of the optical scattering features are determined. The heights of these surface features are then used as input variables to a computer simulation of a reflective grating. The computer predicted color is compared to the measured color. In this manner, by inspection of the reflected color from the textured porous silicon surface the physical size of the surface features can be estimated.

Keywords:
Watershed Transform, surface roughness, atomic force microscopy, light scattering.

1.) INTRODUCTION

Porous silicon (PSi) can be used as smart transducer material in sensing applications and in the detection of vapors, liquids, and biochemical molecules. Upon exposure of chemical substances, several physical quantities, such as refractive index, photoluminescence, and electrical conductivity, change drastically. A key feature of a physical transducer, being sensitive to organic and biological molecules, either in vapor or liquid state, is a large surface area: PSi has a porous sponge-like structure. PSi is an available, low-cost material, compatible with standard IC processes, which can be employed in the realization of smart sensors and microsystems.

Recently, experimental work has been reported concerning the use of porous silicon in optical sensors for chemical and biological sensing [1]–[4]. In environmental monitoring, optical readout techniques are of particular interest because they do not require electric contacts that may cause explosions or fire in dangerous environments. Also optical sensors are not affected by electromagnetic interference.

Photo luminescent (PL) properties of porous silicon (PSi) have been widely reported [5]-[7]. However in this paper we are not concerned with the PL, but rather, the reflected optical spectrum from white light illumination. In this paper the surface of the PSi is excited with incoherent, broad band white light (from a microscope lamp source) and observe the spectrum of colors reflected from the surface. Using an atomic force microscope (AFM) atom force microscope images in non-contact mode of the PSi surface are taken and correlated to the color of the surface. Light scatters from the textured PSi surface producing a spectrum of colors similar to light scattering of the water droplets in a rainbow. We present statistical quantities of the surface structure and match the quantities to color. Finally statistical quantities of the surface structure are matched to surface color.

2.) PROCEDURE

Silicon Photonics III, edited by Joel A. Kubby, Graham T. Reed
Proc. of SPIE Vol. 6898, 68981F, (2008) · 0277-786X/08/$18 · doi: 10.1117/12.759323

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx

The porous silicon samples are made using [100] p-type silicon wafer with 4 ohm-cm resistivity. The silicon wafer is anodically etched in 15% HF and methanol alcohol solution at a current density of 6 milliamps per square centimeter for 15 minutes.

The samples are analyzed using an XE-100 SPM manufactured by Park Systems (formally PSIA). Images are made of the PSi in the AFM non-contact mode. The PSi images are from samples that are red or green in color. These SPM images record the topography of the samples surface. The reflected optical spectrum is measured using a Filmetrics spectrometer.

3.) EXPERIMENTAL RESULTS

All images used in this paper are 2x2 microns in size which allows many of the textures surface features to be present in one image. Also, this scan size is several wavelengths long in visible light thus allowing one to detect light scattering from PSI structures on the order of a wavelength in size. Figure 1 shows the images of typical samples of red and green PSi images, all 2x2 microns in scan size.

Red colored PSi   Green colored PSi

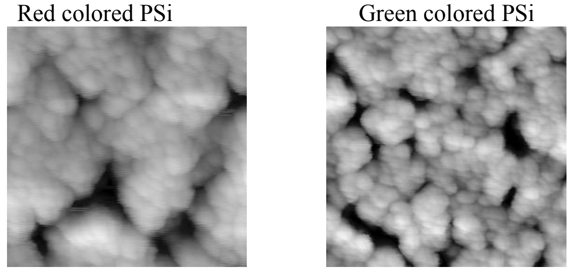

Figure 1. AFM images of red and green porous silicon, 2x2 micron scan size.

Figure 2 shows the reflectance of white light normal to the surface of a typical PSi sample. The optical reflectance of the PSi sample was measured at a normal incidence using a Filmetrics spectrometer. There are reflection peaks at 470 nm, 550 nm, and 675 nm. The absorption peaks are at 495 nm, 600 nm, and 780 nm. The reflectance peaks correspond to the blue, green, and red colored areas reflected from the PSi surface.

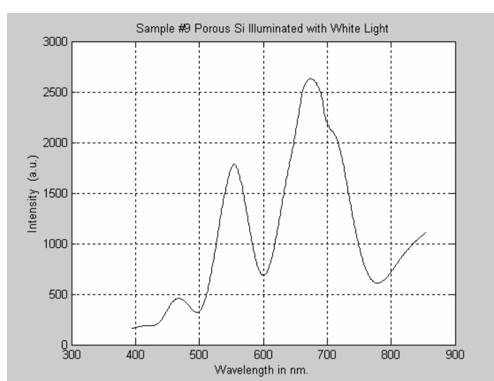

Figure 2.) Measured Reflectance Spectrum of PSi sample.

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx

4.) WATERSHED TRANSFORM

To determine the size of the surface features on the PSi samples, segmentation using the watershed transform is implemented [8]-[9]. A watershed is a ridge that divides areas drained by different river systems. The grayscale image from the AFM produces a topological three dimensional surface, where the values of the function f(x,y) are the topographical heights. If we imagine rain falling on the surface water would collect in catchment basins. A catchment basin is a area that drains into a reservoir or river. The watershed transform finds these catchment basins and ridge lines in the SPM image. It segments the image into areas depending on the size of the catchment basin. The size of the basins is determined by a watershed filter parameter. The larger the size of the watershed filter parameter the larger the size of the segmented catchment basin. It is desirable to apply a smoothing filter to the image before the detection algorithm starts. The higher the filter level, the more the image will be smoothed before grain detection, this will prevent excess grains created by noise.

Figure 3a shows an AFM generated image of a green colored PSi surface and Figure 3b is the segmented Watershed Transform of the image using filter level 2.

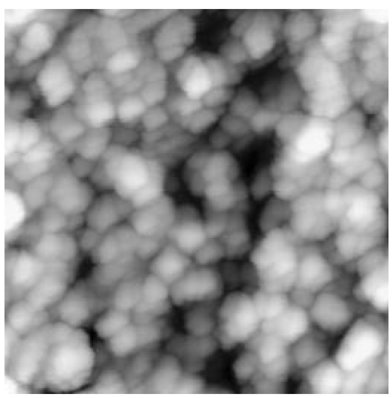

Figure 3a.) SPM Image of PSi Sample, 2x2 micron scan.

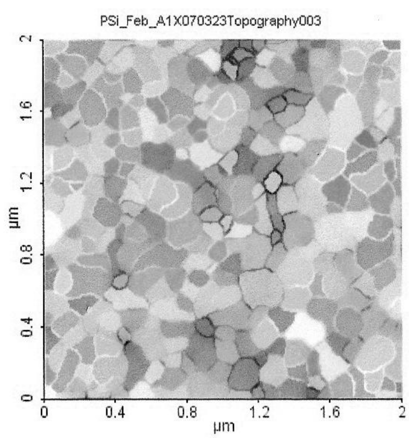

Figure 3b.)Watershed Transform of Image.

At this filter level (2) this 2x2 micronAFM image has 305 segmented surface features, where 141 of these surface features are "bumps up" and the remaining 164 surface features are "bumps down". For this green colored PSi sample, the average area of a surface feature is 1.19 e-2 square microns with a standard deviation of 7.8e-3 square microns. The average length of a surface feature is 160 nm with a standard deviation of 54 nm, and the average volume of a surface feature is 6.14e-4 cubed microns with a standard deviation of 4.4e-4. Assuming optical scattering from "bumps up" surface features, and noticing that the scatterers are hemispherical in shape, the average height of the bumps is 72 nm. Similarly, the "bumps down" have and average height of 62 nm, resulting in a total scattering height of 134 nm.

5.) HOLOGRAPHIC GRATING, A simple model for surface scattering:

In this section the heights of the surface features are used in a holographic grating to simulate colors generated from white light scattering.

The textured surface of PSi has random surface features. However, to simplify the problem, a periodic reflective grating with dimensions equivalent to the feature sizes on the PSi surface is assumed. For example, for a grating of all the same size, optical scattering gives a single color at normal incidence. For

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx

the porous silicon samples, the size of the surface features determines the colors reflected from the surface. The light's angle incidence and reflection are normal to the surface. In order to relate the rough surface topography to the color, a set of gratings of known size is used to model the optical scattering following the work of Angelsky et al [12].

A rough surface can be represented by a set of gratings [12]. The first order approximation for the reflective scattered component is give by equation 1.

$$R_r = \prod_m J_o^2(q_m) \qquad \text{(equation 1.)}$$

Here $q_m = \frac{2\pi}{\lambda}(n - n_o) * H_m$ is the phase component. Where H is the height of the grating and $(n - n_o)$ is the index of refraction of the silicon dielectric minus the refractive index of air. The variable m is the number of gratings used in the simulation. Each grating with it's own height.

For the green colored PSi sample the average height of the 'up bumps' is estimated to be 72 nm. Similarly, the "bumps down" are estimated to have and average height of 62 nm, resulting in a total feature height of 134 nm. A computer generated simulation using Equation 1 is shown in Figures 4 and 5. The scattering component from a white light source of a holographic dielectric grating is shown for green reflectance in figure 4. In this figure the Green reflectance spectrum from the PSi surface is simulated using a mean feature height of 134 nm. Seven gratings each with different heights (m=7) were used in the computer simulation shown in Figure 4 with a grating height standard deviation of 10 nm. Figure 4 shows a scattering peak at 543 nm which correlates well with the measured green peak at 550 nm as shown in Figure 2.

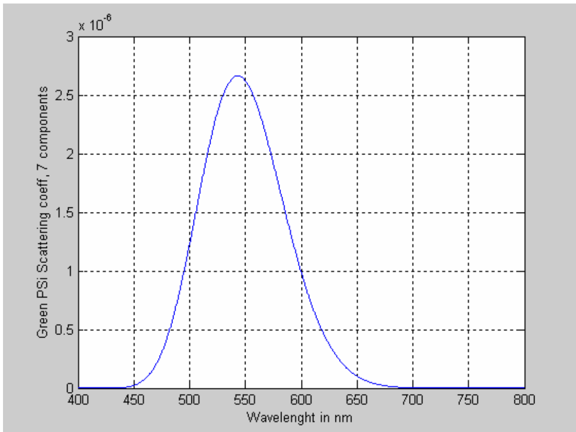

Figure 4.) Holographic Grating computer simulation of Green colored PSi

In a similar fashion to the green colored PSi sample the feature heights from the watershed transform images are calculated for the red PSi sample. For red PSi a feature height of 170.5 nm is calculated. In Figure 5 the Red reflectance spectrum is simulated using a mean feature height of 170.5 nm and seven gratings (m=7), with a grating height standard deviation of 10 nm. Figure 5 shows a scattering peak at 675 nm which correlates very well with the measured red peak (also at 675 nm) as shown in Figure 2.

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx

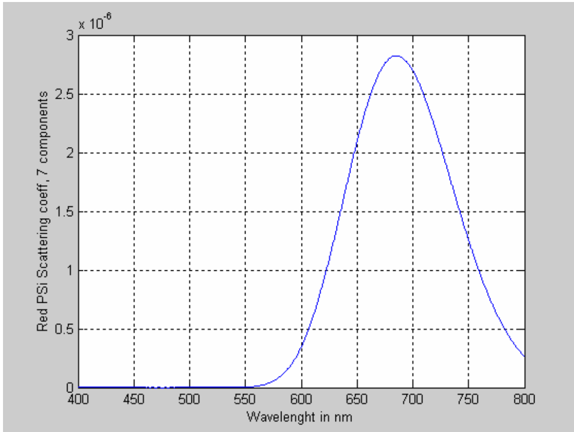

Figure 5.) Holographic Grating computer simulation of Red colored PSi

6.) CONCLUSIONS

The use of the watershed transform algorithm to determine the feature dimensions of a texture PSi surface has been demonstrated. The reflectance from a PSi surface illuminated with a white light source has been measured and compared to the results of the watershed transfrom. From the watershed transform algorithm measured dimensions of the surface features for green and red colored PSi were calculated.. The green and red peaks from the computer simulation match the measured green and red peaks from the PSi sample. These colors are believed to be a result of light scattering from a textured dielectric surface.

7.) ACKNOWLEDGEMENTS

The authors would like to thank the sponsors for the funding of this project at the Florida Institute of Technology's National Center for Hydrogen Research.

8.) REFERENCES

[1] J. Gao, T. Gao, and M. J. Sailor, "Porous-silicon vapor sensor based on laser interferometry," *Appl. Phys. Lett.*, vol. 77, p. 901, 2000.

[2] V. Mulloni and L. Pavesi, "Porous silicon microcavities as optical chemical sensors," *Appl. Phys. Lett.*, vol. 76, p. 2523, 2000.

[3] P. A. Snow, E. K. Squire, P. St. J. Russel, and L. T. Canaham, "Vapor sensing using the optical properties of porous silicon Bragg mirrors," *J. Appl. Phys.*, vol. 86, p. 1781, 1999.

[4] L. Stefano, et. Al., "Optical Senors for Vapors, Liguids, and Biological Molecules Based on Porous Silicon Technology," IEEE Trans. On Nanotechnology, vol. 3, No. 1, March 2004.

[5] LT Canham,"Silicon quantum wire array fabrication by electrochemical and chemical dissolution of wafers," Appl. Phys. Lett. 57 (10), September 1990.

[6] V. Lehmann and U. Gosele, "Porous silicon formation: A quantum wire effect," Appl. Physics Lett. 58 (8), February 1991.

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx

[7] P D J Calcott, KJ Nash, LT Canham, MJ Kane, D Brumhead, “Identification of radiative transitions in highly porous silicon,” J. Physics; Condens Matter 5 (1993).

[8] R.C. Gonzalez, R. Woods, S. Eddins,”Digital Image Processing using Matlab,” Pearson Prentice Hall, 2004.

[9] L. Vincent, P. Soille, “Morphological Grayscale Reconstruction in Image Analysis,” IEEE Trans. Pattern Anal. Machine Intell., vol. 13, no.6, pp. 583-598.

[10] XEI Imaging Software Manual Version 1.5, PSIA Corporation, 2004.

[11] LT Canham,”Silicon quantum wire array fabrication by electrochemical and chemical dissolution of wafers,” Appl. Phys. Lett. 57 (10), September 1990.

[12] O. Angelsky, P. Polyanski, S. Hanson,”Singular-optical coloring of regularly scattered white light,” Optical Express, Vol. 14, No. 17, August 2006.

Downloaded From: http://proceedings.spiedigitallibrary.org/ on 04/21/2017 Terms of Use: http://spiedigitallibrary.org/ss/termsofuse.aspx