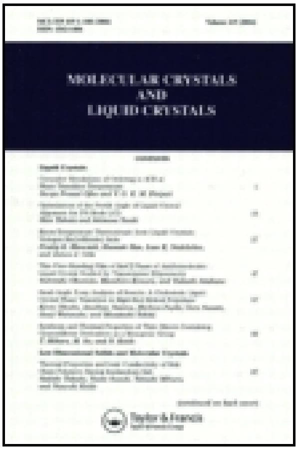

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# An Atomic Force Microscopic Study of Pore Wall Structures of p-Type Macroporous Silicon

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Atomic force microscopy (AFM) has been used to study the pore wall structure of p-type macroporous silicon. Nanometer size particles are found to distribute on the pore wall of the luminescence macroporous silicon samples. Combided with the analysis of photoluminescence (PL) spectra, it can be concluded that the PL phenomenon of macroporous silicon should be due to the quantum confinement in these nanoparticles distribution on the pore wall.

*Keywords:* atomic force microscopy; macroporous silicon; photoluminescence

## INTRODUCTION

Since the discovery of visible PL in porous silicon (PSi) layers[1], many workers have suggested models to explain this phenomenon. Although the exact origin of PL is still under debate, the proposed mechanisms prevalent in the literature have been attributed to quantum confinement effects[1], arising from isolated, nanometer Si features produced during the etching process[2]. Furthermore, the nanometer particles have been separated from thin sections of PSi by ultrasonic dispersion in organic solvents. High resolution TEM and FTIR have also been used to establish the size range and surface structure and composition of these particles[3]. However, the distribution of these nanometer particles on the pore wall of PSi hasn't been well observed. At present, we combine PL emission spectra with AFM images to study the microstructure of the pore wall and try to give an explanation about the relationship between these nanoparticles in depth profile of macroporous Si and PL phenomenon.



## EXPERIMENTAL

A <111>-oriented crystalline silicon wafer (p-type) was used with a thin aluminum film deposited on its back side to form an ohmic contact. It was immersed in a mixture of HF (49%) and $C_2H_5OH$ (98%) (1:1, V/V) with 45 $mA/cm^2$ anodization current density for 5 minutes. A platinum wire serves as the counterelectrode. After anodization, the wafer was rinsed with ethanol and dried in flowing nitrogen, then characterized immediately by TEM and AFM. This procedure resulted in uniform porous layers with exhibition of visible (red/orange) PL under ultraviolet lamp irradiation (254nm, wavelength).

## RESULTS AND DISCUSSION

Bulk Si can be made microporous (pore width ≤ 20 Å), mesoporous (pore width 20-500 Å) or macroporous (pore width > 500 Å) strongly depending upon substrate resistivity and anodization conditions. In this paper, we chose macroporous Si as an example to have the observation of the pore wall structures clearer and more manipulable.

The morphology of the macroporous Si layers produced by the above process was examined with a X-650 SEM (Hitachi). Figure 1A reveals an array of holes 1-3 μm across, often having circular shapes. The remaining silicon skeletal framework is single crystal with interconnecting columns that mostly have cross-sectional diameters of > 200 nm. The SEM micrograph only gives a survey of the morphology of the PSi layers, so atomic force microscopy has to be used for further exploration for the nanostructure of the pore wall.

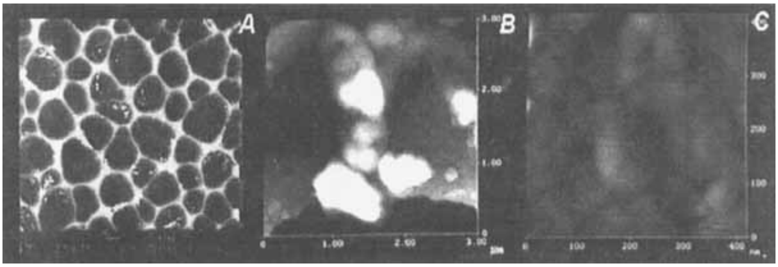
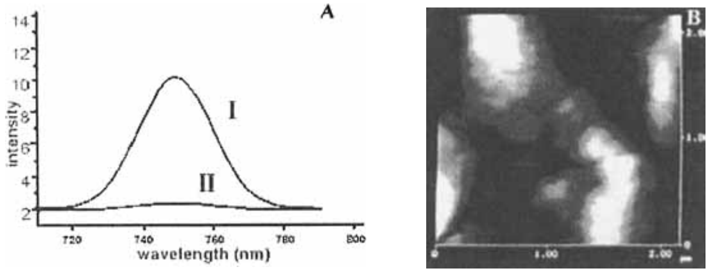



FIGURE1 Microstructure of PSi: (A) SEM micrograph and (B) AFM image (3 μm×3 μm) of PSi; (C ) AFM image (400 nm×400 nm) of the pore wall.

AFM measurements were carried out using a commercial system (Nanoscope IIIa, Digital Instruments, Senta Barbara) in the contact mode. As shown in Figure 1B, the pore wall of the macroporous Si consists of nanometer size particles whose diameters vary from 40 to 100 nm, and the pore is about 1-2 μm across. In the profile of the pore wall (Figure 1C), we can see the structures in detail. Xie et al.[4] observed the similar pore wall structures of macroporous Si with aperture 200-400 nm by TEM. We presumed that these nanometer particles should be responsible for the PL phenomenon of macroporous Si due to the quantum confinement effects. To testify it, we must do some further experiments. After the anodization, the macroporous Si was treated by supersonic wave in 98% ethanol solutions for 5 minutes, then analyzed with a spectrofluorophotometer (Shimadzu, RF-5000). The luminescence characteristic peak at about 749 nm in the PL spectra (excited at 250 nm) has dramatically decreased from 10.2 to 3.0 (Figure 2A) after the treatment. And it is also found that the nanometer size silicon particles on the pore wall have been removed by ultrasonic dispersion, while the average pore size is still about 1-2 μm (Figure 2B). Both the AFM images and the PL emission spectra analysis results suggest that the luminescence property of macroporous silicon should be due to the quantum confinement in these nanometer particles distribution on the pore wall.




FIRURE 2 Great changes taken place after treated by supersonic wave in both (A) PL spectra [before (I) and after (II) the supersonic treatment] and (B) AFM image.

## CONCLUSION

We present a method, atomic force microscopy, to observe nanoparticles on macroporous Si walls clearly with nanoscale resolution. It is found that the average size of nanoscale particles in PSi is about 70 nm in diameter with distribution range from 40 nm to 100 nm. Our result shows that the information about the size distribution of nanoparticles on walls will hold much promise for elucidation of the visible light emission mechanisms of PSi.

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