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LETTER TO THE EDITOR

Analyses of microstructures in porous silicon by means of HREM and digital image processing

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Abstract. High-resolution electron microscopic images of porous silicon films have been obtained and their microstructures have been analysed by means of digital image processing.

Since Canham [1] reported that highly porous silicon could emit visible light under photo-excitation, much investigation has been carried out in this material. Many models, for example, the quantum wire model [1,2], the attached molecule model [3] and the surface contamination model [4], have been suggested to explain the light emission property. However, the microstructure of porous silicon was still not clear.

Recently, two main microstructure models have been proposed. On the one hand, porous silicon was considered to be comprised of tiny columns or wires of crystalline silicon [1,2,5]. On the other hand, it was thought of as being high-porosity silicon in an amorphous state [3]. By using JEM-4000EX high-resolution electron microscope (HREM) with a top entry stage operated at 400 kV, we obtained HREM images of porous silicon. Furthermore, evident information on the microstructure of porous silicon was obtained by means of digital image processing. It should be found that porous silicon consists of tiny crystalline columns or wires with diameters of a few nanometers spread on an amorphous background.

p-type (100) silicon wafers with resistivity in the range of 2–5 Ω cm were anodized in aqueous 48% HF with a current density of 10 mA cm^{-2}, which formed porous silicon films on the surface layer. Under the excitation of an Ar^+ laser or purple light, the stronger red light emitted from the surface of prepared porous silicon samples can be seen. The peak of the photo-luminescence spectra (PLS) lies in the range of 620–680 nm with a full width at half maximum (FWHM) of about 0.3 eV. The porous layers of samples used for HREM observation were peeled off the surface, and thinned by Ar^+ ion milling. The HREM observation was performed in a JEM-4000EX HREM operated at 400 kV. The image processing was carried out by using a Legend LX-486/33W microcomputer.

Figure 1 is an HREM image taken from a porous silicon film. Although the HREM image, at first glance, seems to be disordered completely, some preferential orientations in the image and some tiny ordered regions on the disordered background are still visible when a careful look is taken.

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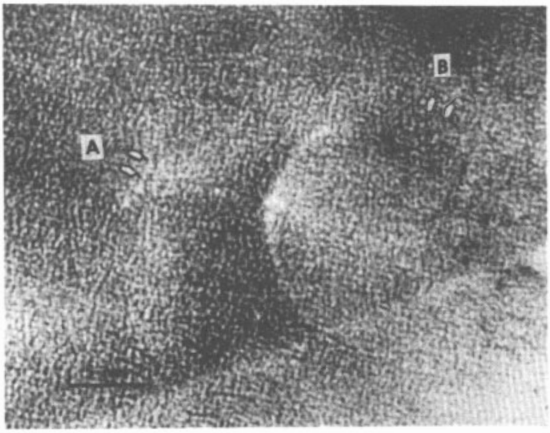

Figure 1. A high-resolution electron microscopic image of porous silicon. The directions of the arrows marked A and B are preferential orientations.

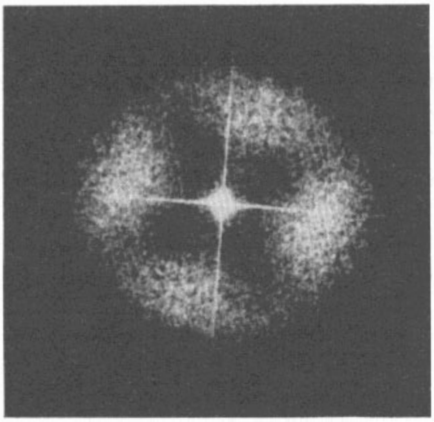

Figure 2. A diffraction pattern obtained from digital processing of the HREM image. It should be noted that the character of the diffused spots is fourfold symmetry.

In order to pick out these crystalline clusters from the disordered background, we applied a digital image method to process the HREM image. By importing the HREM image of porous silicon into a computer with an HP-SCAN scanner and cutting it, we obtained an image with 256 × 256 image elements (see figure 3(a)) and processed it with a fast Fourier transformation (FFT) calculation, so that the corresponding diffraction pattern was gained (see figure 2). The diffraction pattern consists of four diffused spots which have fourfold symmetry. It is necessary to determine whether the diffraction pattern is strictly in accordance with the HREM image. To do this, we rotated the HREM image by a few degrees and processed it with the FFT. It was found that these diffraction patterns also rotated through the same angles as the HREM image. The diffraction pattern indicates information on the microstructure of porous silicon as follows: first, there exist preferential orientations in the porous silicon;

second, from the size of diffused diffraction spots, it can be inferred that crystallites with a size of 10–30 Å exist in the porous silicon due to the uncertainly principle [7].

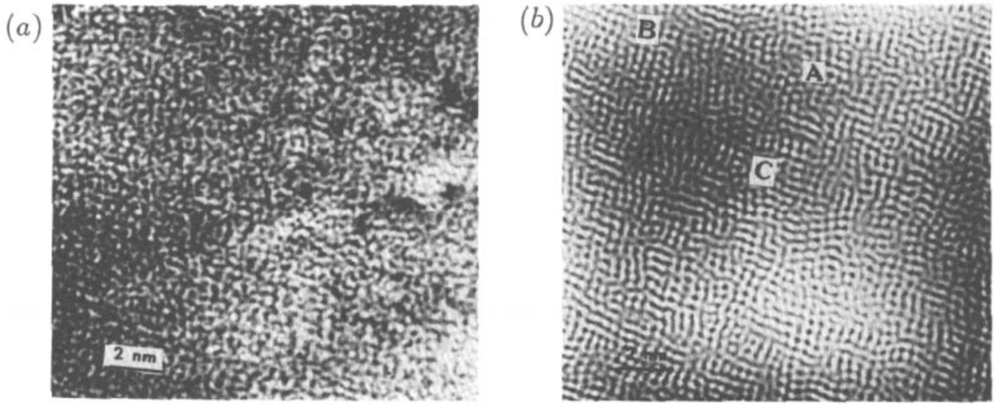

Figure 3. (*a*) Part of the image of figure 1 (256 × 256 image elements); (*b*) a filtered image of (*a*): the typical ordered regions are marked A, B and C.

With the image processing technique, we masked the diffraction pattern, using circular and ring masks. By means of an inverse Fourier translation calculation, we obtained a clearer image as shown in figure 3(*b*), where high-frequency noise and part of the disordered background were deleted out. From figure 3(*b*), it can also be seen that many tiny ordered regions of about 20 Å are spread on the disordered background. Although they have a common [001] direction perpendicular to the imaging plane, none of the crystallites, the ordered regions in figure 3(*b*), are strictly parallel to each other. This effect may be the result of the high porosity and degree of disorder in the porous silicon sample.

According to the principle of HREM imaging [6], a clearer HREM image can be revealed only when the imaged atomic columns have enough length along the electron beam. It may be concluded that the tiny ordered regions are crystalline columns or wires along the [001] direction. The structure of porous silicon in our observations coincides with the first microstructural model suggested by some authors [1, 2, 5]. It is favourable for the luminance mechanism of quantum size effect [1], i.e. the visible emission of porous silicon could be generated for crystalline columns with a diameter smaller than 3.5 nm [1].

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