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Meso-Structure Study of a Porous SiO_2 Thin Film on (001) Si by Transmission Electron Microscopy

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SiO_2 with nanometre-scale isolated pores exhibiting low dielectric constants (less than 2.2) and good mechanical properties may be highly desirable for future semiconductor devices [1]. The first preparation of meso-structured porous SiO_2 templated by a surfactant, so-called structure directing, was reported in 1992 [2]. Since then, a great deal of interest in the synthesis of various meso-structured porous inorganic oxides has developed. The mesoporous thin film in the present study is a special type of crystalline solid. Similar to traditional crystals, this meso-structured thin film has a three-dimensional order, but its lattice parameter of 13 nm is much larger than those of ordinary crystals (few angstroms). In the present crystalline thin film, nanometre-sized voids, instead of atoms with 1 - 2 Å (in diameter) in the usual crystals, are arranged in a cubic array and are distributed in the SiO_2 matrix, which is essentially amorphous.

In this paper, we report on the meso-structure determination and dislocation analysis of the meso-structured porous SiO_2 thin film on a (001) Si substrate by transmission electron microscopy (TEM). The details of the film synthesis were described elsewhere [3]. Combining a cross-sectional TEM sample and a scratched TEM sample prepared from the non-free standing thin film, the images and diffraction patterns of three low-index zone axes [100], [110] and [111] were obtained. Through systematical tilting of the samples, image simulation, and calculation of plane spacings and angles between two planes, the structure was unambiguously determined as a body-centred tetragonal (BCT) with a = 13.5 nm, c = 13 nm, which is slightly distorted from a body-centred cubic (BCC) with a = 13 nm. Figure 1a is a representative TEM image of this BCT film in the [110] cross-section; the corresponding electron diffraction pattern and the index are shown in Figure 1b and 1c, respectively.

Two types of dislocations were observed. One was an edge dislocation, with the Burgers vector $\mathbf{b} = a[010]$ and the dislocation line direction ξ = [100]. This dislocation was formed by the reaction of two regular dislocations in the BCC structure: $\mathbf{b_1} = (a/2)[1,1,-1]$ and $\mathbf{b_2} = (a/2)[-1,1,1]$ ($\mathbf{b} = \mathbf{b_1} + \mathbf{b_2}$). The origin of this dislocation is argued to be the tensile strain developed during the formation of the meso-structured thin film via the solvent evaporation induced sol-gel and self-assembly processes. The formation of such dislocation partially relieves the strain developed in the film; the critical thickness for the formation of this dislocation was estimated using an elastic strain energy argument. The other was a dislocation dipole with the Burgers vectors $\mathbf{b} = \pm(a/2)[-1,1,1]$ on a (0,1,-1) plane. Figure 2 shows an example of the first type dislocation in a [100] cross-section TEM image.

References:
[1] Miller, R. D. Science 286 (1999) 421.
[2] C. Kresge et al., Nature 359 (1992) 710.
[3] Yu, K.et al., Langmuir 17 (2001) 7961.

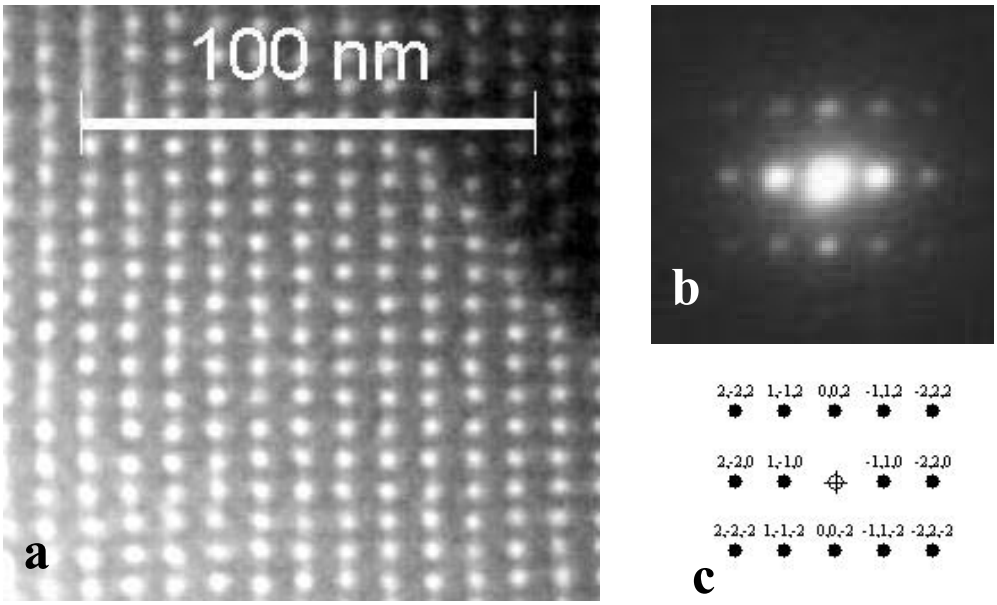

Figure 1: [110] zone axis TEM image of the meso-structured SiO_2 film with isolated pores (a), the corresponding diffraction pattern (b), and the index of the diffraction pattern (c).

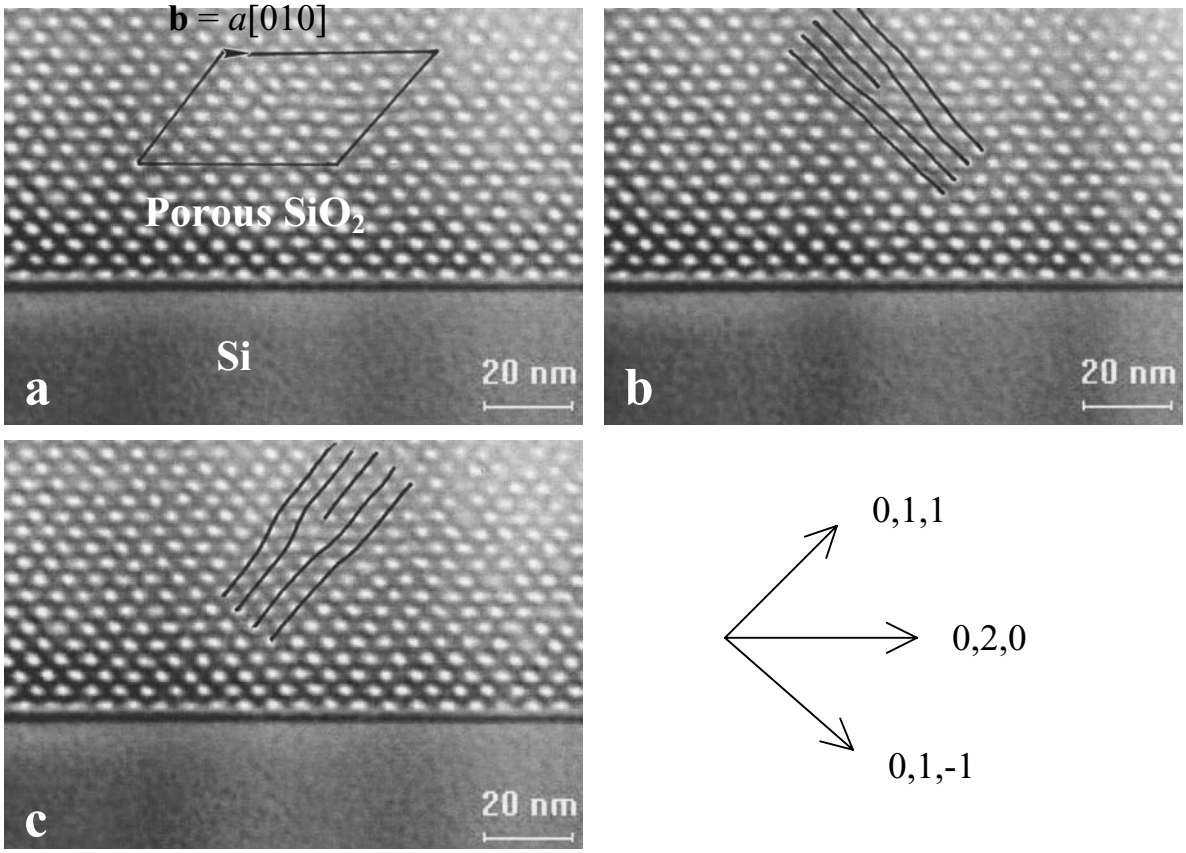

Figure 2 [100] cross-section TEM bright field image showing the first type dislocation in the film: (a) The Burger circuit shows that the dislocation is **b** = *a*[010]; (b) Guide to show the position of the extra (011) plane of dislocation $\mathbf{b_1}$ = (*a*/2)[1,1,-1]; and (c) Guide to show the position of the extra (0,1,-1) plane of dislocation $\mathbf{b_2}$ = (*a*/2)[-1,1,1].

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