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Mechanical properties of porous silicon and oxidized porous silicon by nanoindentation technique

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ABSTRACT

A study of mechanical properties of mesoporous silicon (PS) is presented in this article. PS was prepared by electrochemical etching of a heavily doped P^{++} silicon wafer in a hydrofluoric acid electrolyte. The mechanical properties of PS and oxidized PS obtained by thermal treatment, were characterized by the nanoindentation technique associated to the continuous stiffness measurement option. The morphology of PS and oxidized PS were both characterized by scanning electron microscope. It is shown that the Young's modulus and hardness are related to the PS preparing conditions and decrease with increasing porosity. In particular, oxidation improves the mechanical properties of the mesoporous silicon. Surprisingly, modulus and hardness decrease with penetration depth, whereas a compaction could be expected resulting in a rising modulus and hardness. These results are mainly attributed to micro cracks formation, highlighted by focused ion beam cross section.

1. Introduction

Due to its interesting physical properties, porous silicon (PS) is one of materials that attracts more attention since its discovery. Numerous studies have been proposed to characterize it [1–3]. This material has found applications in many devices such as electromagnetic interference filtering radiofrequency (EMIFRF) circuits [4], nanoelectromechanical systems (NEMS) [5], emitters [1], micro-hotplates [2], rechargeable Li-ion battery anode [6] and photonic crystal sensors [7]. PS can be prepared as thin layers or multilayers systems [8–11]. The thermal conductivity of PS is lower than that of mono-crystalline silicon and can be used as thermal insulator in microsystems [12]. This characteristic can find an application in a micro flow meter, where a large periodic thermal conductivity to the substrate surface is needed [3–14]. Monocrystalline PS is mainly classified according to the pore size as nanoporous ($\leq$ 2 nm), mesoporous (2–50 nm) and macroporous silicon ($>$ 50 nm). Thermal properties are primarily related to the porosity [15]; in addition, the dielectric constant of PS can be reduced with increasing porosity [16]. However porosity considerably weakens the structures. Consequently, it becomes essential to characterize precisely the mechanical behaviour of such structures to optimize the manufacturing process in order to reach mechanical requirements. The first studies on the mechanical characteristics of PS were performed in 1997 [17,18]. Numerous researches have been proposed to improve understanding of the influence of morphology and structure of PS on its mechanical properties, such as micro indentation [19]. These authors examine numerous issues related to the mechanical properties of the mesoporous silicon, such that, the variation in hardness of aged porous silicon low dielectric constant. Nanoindentation has been used to show influence of both PS microstructure and porosity on the mechanical properties [20,21].

In the present study, mechanical properties are investigated using the nanoindentation technique associated to the continuous stiffness measurement method (CSM) [22]. This method has shown its reliability for determining the mechanical properties of materials at the micro- and nanoscale. This article analyzes the evolutions of hardness and Young's modulus of PS and oxidized PS regarding the compaction of PS and the rupture of the sponge-like structure.

2. Experimental

2.1. Sample preparation

The mesoporous silicon is obtained by electrochemical anodization of heavily doped P^{++} type silicon (resistivity $\rho = 0.009 \pm 0.01\ \Omega$ cm, thickness about 380 ± 25 µm and {100} crystal orientation) in a double-tank cell (AMMT®). The concentration of the electrolyte (by volume) is 27% of hydrofluoric acid, 35% of pure ethanol C_2H_5OH and

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Table 1
Manufacturing parameters and description of PS and oxidized PS.

Sample		Current density [mA/cm^2]	Anodizing time [s]	Porosity [%]	Thickness [µm]	Oxidation temperature [°C]
As-prepared PS	PS_1	20	5400	44.44	110	Unoxidized
	PS_2	40	5400	58.17	190	Unoxidized
	PS_3	60	3600	59.66	170	Unoxidized
	PS_4	80	3600	68.00	206	Unoxidized
	PS_5	20	1800	38.16	41.17	Unoxidized
	PS_6	20	3600	41.86	78.7	Unoxidized
	PS_7	40	1800	45.71	57.07	Unoxidized
	PS_8	40	3600	52.55	113	Unoxidized
Oxidized PS	PS_1O	20	5400			550
	PS_2O	40	5400			550
	PS_3O	60	3600			550
	PS_4O	80	3600			550

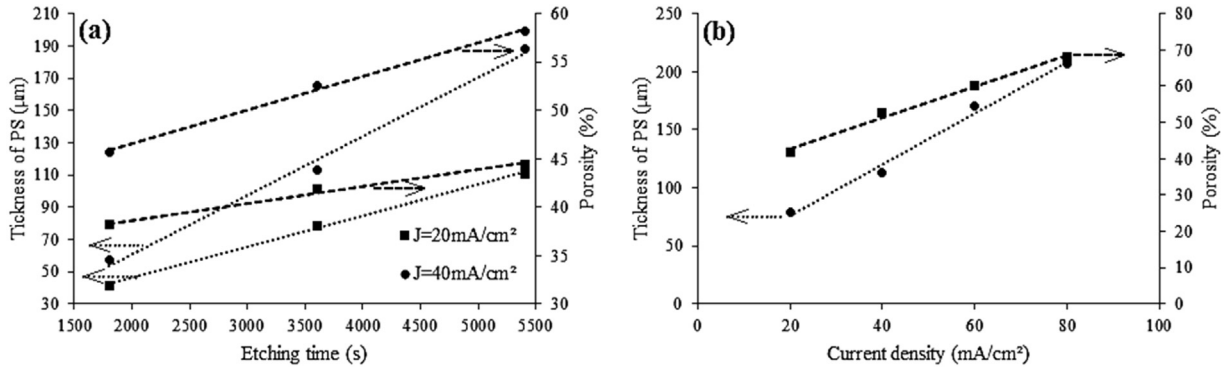

Fig. 1. Evolution of thickness and porosity of PS as a function of (a) time for two anodizing current density: 20 mA/cm^2 and 40 mA/cm^2 and, of (b) current density for a constant anodizing time of 60 min.

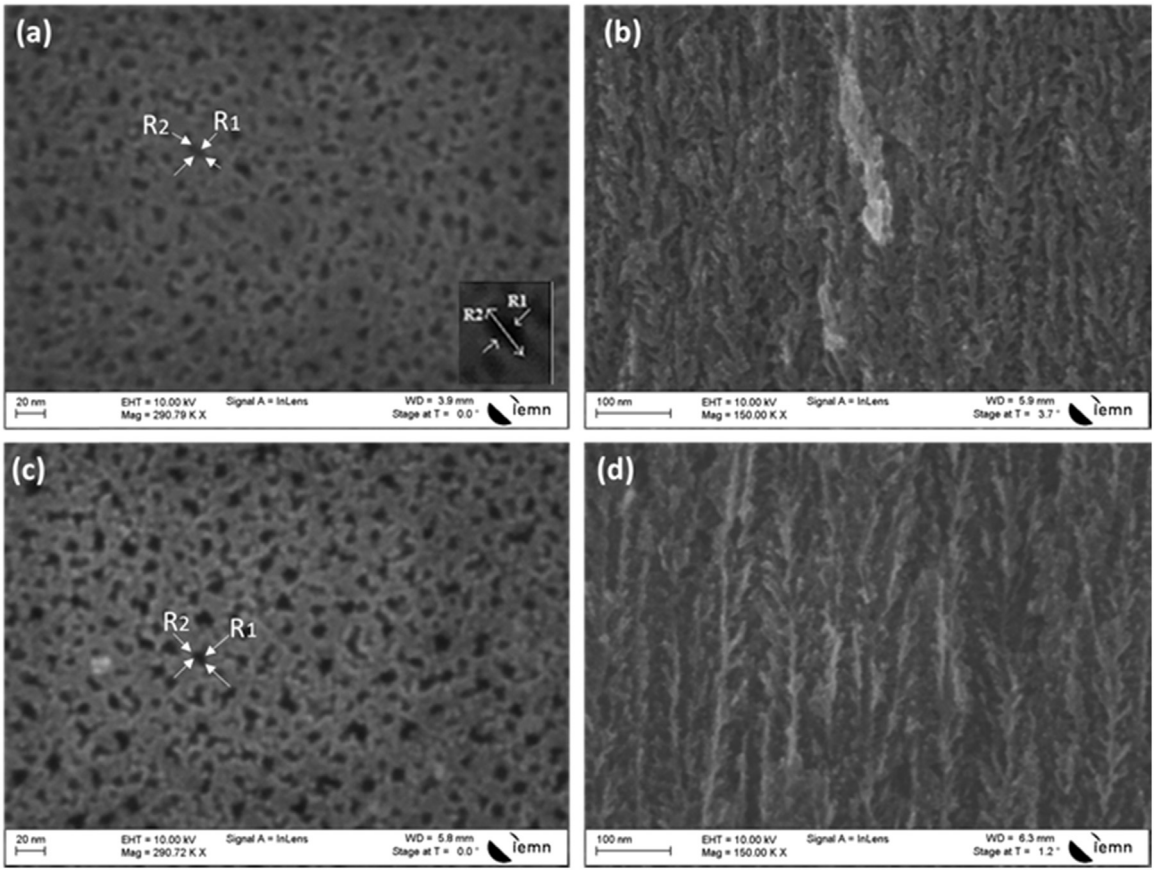

Fig. 2. SEM photographs of PS surface and PS in trench: (a, b) sample PS_6; (c, d) sample PS_4.