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Surface modification of porous silicon microparticles by sonochemistry

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Electronic Supplementary Information

The porous silicon microparticles were fabricated by electrochemical etching of a highly doped, (100)-oriented, p-type Si wafers (boron-doped, 0.001-0.004 Ω.cm resistivity) (Cemat Silicon S.A) in a solution of 20% of aqueous hydrofluoric acid (HF, 50%, Carlo Erba) diluted in absolute ethanol (Sigma). The etching process was performed in a HDPE cell with a platinum electrode and a computer controlled galvanostat (Keithley 2425). A Si wafer with an exposed area of 7.065 cm^2 was etched at a constant current density of 192 mA/cm^2 for 15 cycles of 1 second of etch and 1 second of stop to avoid the formation of a depletion layer. The resulting porous layer was then lifted off by electropolishing with a current pulse of density of 353.8 mA/cm^2 for 15s. The etching and electropolishing procedure was repeated 20 times per wafer.

The porous membranes were either ultasonicated directly in pure 10-undecenoic acid (98 %, Sigma), or in pure 1-dodecene (95 %, Aldrich), or, as control, in octane (98%, Sigma) or in absolute ethanol for 2 hours. In order to compare the quality of the surface functionalization, the particles formed in ethanol were afterwards thermally hydrosilylated with neat undecenoic acid (98%, Sigma-Aldrich) at 120°C for 120 min under argon. The particles were collected by centrifugation at 4000 rpm for 5 min, the solvent in excess was removed and the particles were rinsed in sequence with acetone and ethanol.

The microparticles were characterized by field emission scanning electron microscopy from Zeiss (ZEISS SUPRA™ 40). The acceleration voltage used for the experiment was between 0.1 and 30 kV, and the nominal resolution was 1.5 nm at 10 kV and WD = 2 mm. The optical parameters (thickness, porosity and refractive index) were obtained performing a best-fit calculation of the reflectance spectrum, by the means of a commercial software (SCOUT, obtained from M. Theiss Hard- and Software, Dr -Bernhard-Klein-Str. 110, D-52078 Aachen,

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Germany, Copyright Wolfgang Theiss, www.wtheiss.com) that is based on the transfer matrix method {Born M., 1999 #1610}.

The software generates the theoretical reflectance spectrum of the porous Si film, calculating its effective refractive index using the dielectric function of bulk silicon and assuming a Bruggeman effective medium approximation {D. A. G. Bruggeman, 1935 #1611}. Porosity and thickness are the two free parameters of the model; their value is adjusted in a least-squares algorithm in order to obtain the best fit between the experimental and the calculated spectra.

After modification of the surface of the pSi particles, the sample were characterized by Fourier transform infrared spectroscopy using a Bruker Tensor 37 Micro-Fourier Transform Infrared in IR-transmission mode. Few particles were deposited on a silicon wafer p-type (3-6 Ωcm), transparent to the IR, for the measurements. For all the spectra, a 15x objective was used. Opus© was used for spectrum acquisition, processing, and analysis. After the acquisition the baseline was corrected.