

Photothermal Characterization of Electrochemical Etching Processed *n*-Type Porous Silicon

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The room temperature thermal diffusivity evolution of electrochemically formed porous silicon as a function of the etching time is investigated. The measurements were carried out using the open-cell photoacoustic technique. The experimental data were analyzed using a composite two-layer model. The results obtained strongly support the existing studies, indicating the presence of a high percentage of SiO₂ in the composition of porous silicon material. [S0031-9007(97)04884-9]

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Since the discovery of its room-temperature visible luminescence [1–5], porous silicon (PS) has become a subject of considerable interest, especially for its promising use as an optoelectronic device [6,7]. There are several methods [8–11] for fabricating PS from crystalline silicon wafers. The electrochemical etching [1,8] is, however, the most extensively used so far. The morphology of the resulting porous layer is strongly dependent upon the fabrication controlling parameters such as electrolyte composition, current density, etching time, etc., as well as on the type of substrate used.

In general, an electrochemically formed *n*-type PS layer consists essentially of a double-layer system on top of the silicon substrate [1,12]. The outermost thin layer, known as the microporous layer, is typically 10–15 μm thick and is responsible for the observed photoluminescence. Except for very small etching times, the inner layer adjacent to the crystalline substrate, designated as the macroporous layer, consists of a parallel array of air-embedded free-standing *n*-PS columns.

Despite the large body of literature that already exists on PS [13,14], so far there has been no reported detailed investigation of the thermophysical properties of this important system. In this Letter we apply the modern photothermal techniques to the evaluation of the thermal properties of electrochemically formed *n*-PS.

The samples used in our experiments were prepared by electrochemical etching on (100) oriented, nondegenerated, *n*-type ($2.1 \times 10^{18} \text{ cm}^{-3}$) crystalline silicon. The samples had a thickness of roughly 300 μm and an electrical resistivity of 1–5 $\Omega \text{ cm}$. The electrochemical etch-

ing was carried out following the procedure outlined in Ref. [12]. The crystalline samples, with an appropriate Pt network electrode attached to them, were immersed in a 150 ml Becker filled with HF. A current density of 40 mA/cm² was then applied to the samples using a HP-model 6206B dc power supply operating between 5–10 V. During the etching period the samples were always kept under the irradiation of a 250 W infrared lamp positioned roughly 20 cm away from the etching bath. By controlling the etching time, ranging from 10 to 83 min, we could fabricate samples with different macroporous thicknesses.

In Fig. 1 we show the side view optical micrograph of a typical *n*-PS sample, produced with 60 min etching time. The three distinct regions mentioned above, namely, the microporous and macroporous layers on top of the crystalline substrate, are clearly seen in this picture.

The room-temperature thermal diffusivity measurements were carried out using the photoacoustic (PA) technique. Of the several techniques [15] used for measuring the thermal diffusivity, we resorted to the so-called “open cell” technique described in Refs. [16] and [17]. It consists of mounting the sample directly onto a cylindrical electret microphone and using the front air chamber of the microphone itself as the usual gas chamber of conventional photoacoustics, as indicated in Fig. 2. The measurements were carried out using a 20 mW He-Ne laser whose monochromatic light beam is modulated using a variable speed mechanical chopper (SRS model 540) and focused onto the sample. The microphone output voltage is measured using a lock-in amplifier (SRS model 850).