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Combustion rate of powdered porous silicon with limited space

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The method of determination combustion rate of powdered porous silicon with limited space is presented. The values of the combustion rates of porous silicon are close to the values of the rates of explosives.

Keywords: porous silicon, combustion rate

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Porous silicon with pore sizes ranging from several nanometers to several tens and hundreds of nanometers is presently regarded as one of the most promising materials for application in such fields of science and engineering as pharmacology, electronics and optoelectronics, manufacture of explosives [1–3], etc. The fabrication of self-destructing chips is one of the possible practical applications of porous silicon [4]. These chips may get destroyed either by combustion or by explosion. In the latter case, a certain layer with a specific thickness impregnated with an oxidizer is needed. Exploding, this layer should trigger the destruction of the chip itself. Self-destruction in the process of combustion may well be achieved without impregnating the layer with a specialized oxidizer, since it was reported [5] that the supply of oxygen from air is sufficient for porous silicon to combust.

A correct estimate of the rate of combustion of porous silicon in a confined space (i.e., with oxygen in silicon pores being virtually the only oxidizer available) is needed in order to determine whether self-destruction of a chip by combustion is feasible. Unfortunately, the overwhelming majority of studies either contain no data regarding the rate of combustion (explosive transformation) of porous silicon or present data corresponding to porous silicon impregnated with various oxidizers [6,7]. It is hard to estimate experimentally the rate of combustion of a porous silicon slice with a thickness below $100\ \mu\text{m}$ (i.e., thickness falling within the range that is actually of practical interest). This is attributable to the fact that one cannot simultaneously establish air-free conditions outside of pores and perform digital recording of the combustion process to obtain numerical rate values.

Therefore, the following algorithm of experiments on determining the rate of combustion of porous silicon was used in the present study:

1. Porous silicon layers were obtained by subjecting a single-crystal silicon (grade KDB-100) wafer with a thickness of $\sim 250\ \mu\text{m}$ heavily doped with boron (*p*-type) to $1.1 \cdot 10^{19}\ \text{cm}^{-3}$ to double-sided galvanostatic electrochemical etching. A mixture of equal volumes of hydrofluoric

acid (48% HF) and ethanol was used as the electrolyte. Electrochemical etching allows one to produce homogeneous mechanically stable wafers of *p*- and *n*-type heavily doped silicon with a porosity up to $\sim 75\%$ (through pores 20–35 nm in diameter). The structure of such layers is formed by a system of branching pores extending primarily along crystallographic directions $\langle 100 \rangle$. Thus, according to the classification of the International Union of Pure and Applied Chemistry (IUPAC), the studied porous silicon samples were mesoporous. The process of fabrication of porous silicon samples has been discussed in more detail in [2,5,8,9].

2. The obtained porous silicon wafer was ground manually into powder with a particle size of 30–40 μm . The image of a powder particle obtained using a scanning electron microscope (SEM) is shown in Fig. 1.

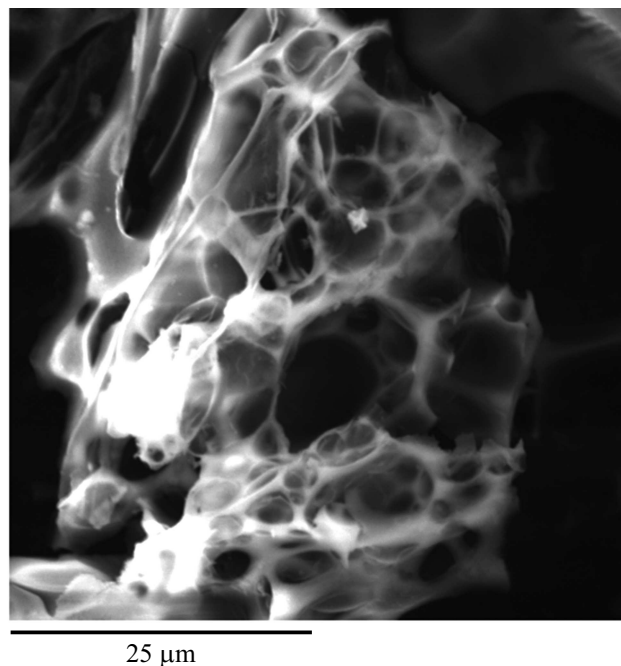


Figure 1. SEM image of a particle of porous silicon powder.

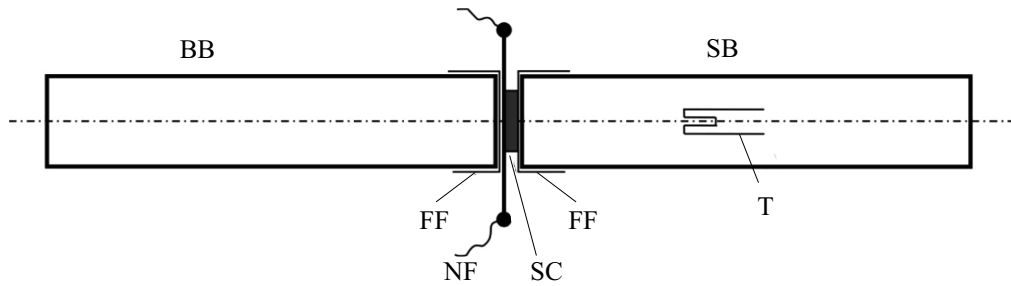


Figure 2. Schematic image of the sample secured between two measuring bars of the tension sensor. SB — measuring bar with tension sensor T ($d = 8$ mm), BB — bearing bar ($d = 8$ mm), SC — cap with porous silicon, FF — fluoropolymer film with a thickness of $100\ \mu\text{m}$, NF — nickel-chromium foil (thermal initiator).

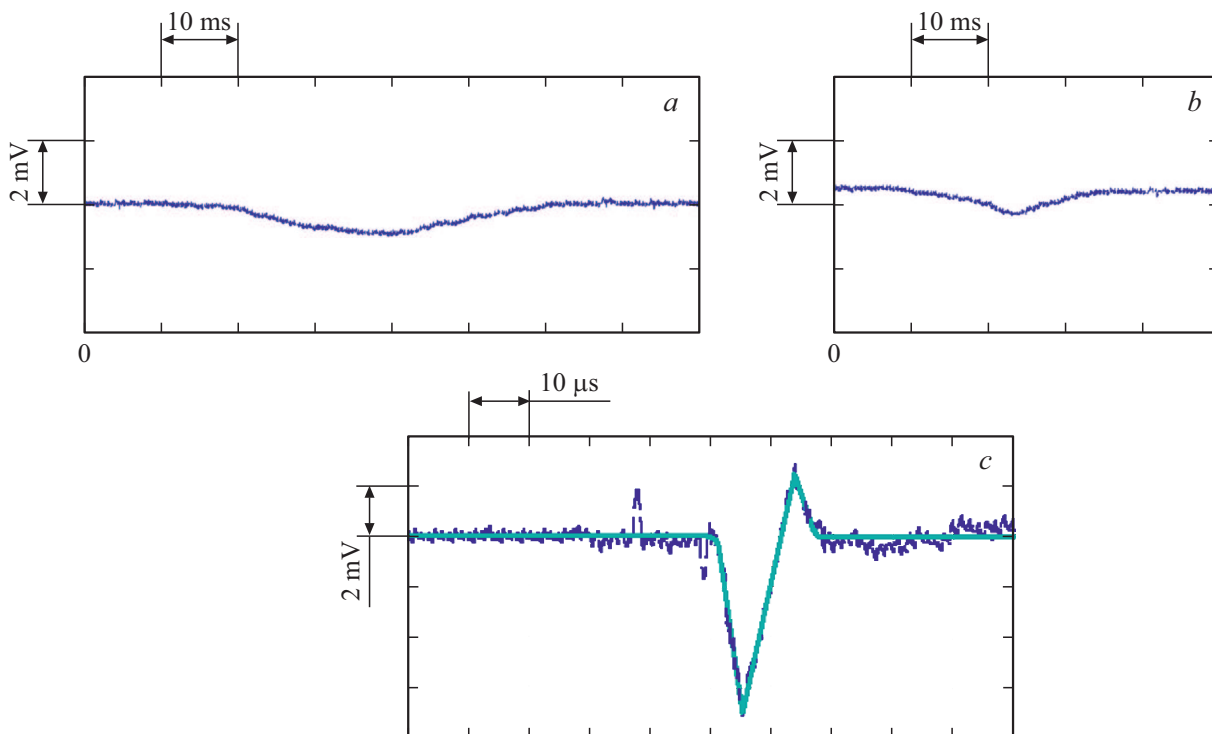


Figure 3. Examples of oscilloscope records of pressure in the process of combustion of porous silicon without an oxidizer (*a, b*) and with a calcium perchlorate oxidizer (*c*).

3. The obtained powder was introduced into a cap with outer diameter $d_s = 8.0$ mm. The cap was covered with aluminum foil with a thickness of $40\ \mu\text{m}$, and powder was pressed to a height of 1 mm through this foil. This helped minimize the gap between the powder sample and foil and, consequently, the volume of air trapped between them.

4. The cap was positioned between two bars, which were equivalent to a split Hopkinson bar [2]. A tension sensor was positioned at one of these bars (measuring bar; see Fig. 2). A nickel-chromium foil bridge (thermal initiator) was installed at the side of aluminum foil, and current was passed through this bridge. The nickel-chromium bridge was thus heated and initiated the process of combustion in pressed porous silicon powder.

5. The products of combustion of porous silicon generate pressure that was measured using the tension sensor.

Example oscilloscope records from the tension sensor are presented in Figs. 3, *a, b*. Note that an explosive transformation of a higher order than layer-by-layer combustion was initiated when silicon pores were filled with an oxidizer (e.g., calcium perchlorate). A well-pronounced peak was seen in the oscilloscope record in this case (Fig. 3, *c*).

The amplitude of mechanical stress accompanying the strain wave induced in the bar by an explosion was calculated in accordance with the following formula:

$$\sigma = \frac{\Delta U E S_1}{I R_0 k S_0},$$

where ΔU is the voltage pulse in the oscilloscope record, $I = 15$ mA is the current in tension sensors, $R_0 = 200\ \Omega$ is the resistance of tension meters, $E = 200$ GPa is the Young's modulus of the material of measuring and bearing

bars (hardened steels), $k = 2$ is the coefficient of tensosensitivity, S_1 is the cross-section area of the measuring bar, and S_0 is the area of the end sample surface.

The maximum values of pressure in the process of combustion of porous silicon were 26 MPa for Fig. 3, *a* and 22 MPa for Fig. 3, *b*. The maximum pressure of the explosive process in Fig. 3, *c* is 233 MPa.

It follows from the oscilloscope records that the rate of combustion of porous silicon in Figs. 3, *a* and 3, *b* is approximately equal to 20 and 25 mm/s, respectively. These combustion rates are close to the rates typical of fast-burning explosives (e.g., potassium picrate, lead styphnate, etc. [10]) and are more than an order of magnitude higher than the rate of combustion of nanosized copper powder (1.3 ± 0.3 mm/s) [11]. However, the obtained rates of combustion of porous silicon are several orders of magnitude lower than the rates corresponding to porous silicon impregnated with oxidizers of various kinds, which fall within the range from 0.1 to more than 3500 m/s [12]. Naturally, the highest values within this range correspond to detonation rates.

Thus, it was demonstrated that porous silicon may ignite in a confined space. Combustion may be initiated by an external heat source (hot wire). The rate of combustion of porous silicon under these conditions is 20–25 mm/s. Unfortunately, the experiments discussed above do not allow one to determine the regime of combustion (convective or conductive). Presumably, conductive combustion is the more likely option here.

Conflict of interest

The authors declare that they have no conflict of interest.

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