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Enhancement of on-chip combustion via nanoporous silicon microchannels

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ABSTRACT

Due to its high energy density and MEMS compatible fabrication methods, on-chip porous silicon shows considerable promise as an energetic material. Rapid combustion events have been demonstrated with flame propagation speeds eclipsing 3 km/s, but much is still unknown about the controlling parameters of porous silicon combustion. Recent studies show that implementation of microstructure within a nanoporous silicon film greatly increases reaction rate of a relatively slow burning system. The present work utilizes porous silicon microchannels to enhance an already rapidly-reacting system. Reactions in channeled porous silicon regions of this system propagated at speeds up to 1.2 km/s faster than similar neat porous silicon films. The fastest propagation speed was 3660 m/s, the highest reported flame speed for comparable nanoenergetic systems to date. We provide evidence that the enhancement of flame propagation rates by channeled porous silicon is mechanistically different from the convectively controlled burning of neat porous silicon. This evidence suggests the presence of acoustically aided reactions for porous silicon channel combustion where the channels more readily ignite compared to neat porous silicon. We predict this allows for propagation of the reaction by intense sound waves within the porous medium.

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1. Introduction

Energetic applications utilize many forms of silicon including nano and microparticles [1–3], porous microparticles [4], and porous on-chip films [5–19]. On-chip porous silicon (PS) is particularly unique as its production uses common microelectromechanical systems (MEMS) fabrication processes allowing for relatively easy integration as an on-chip energy source. Some of its material properties including a high specific surface area (>900 m^2/g [8]), a predicted energy yield per unit mass of 2–3 times greater than that of TNT [7,15], and nanoscale features (∼3 nm), make on-chip porous silicon an intriguing candidate for energetic materials. Many works demonstrate the energetic ability of on-chip PS/oxidizer systems [7,8,11,12,14–16], but characterization is primarily qualitative. Quantitative analysis of rapidly-reacting on-chip PS by Becker et al. shows propagation rates of over 3100 m/s [8], eclipsing the fastest reported burn rates of nanothermite materials at ∼2500 m/s [20]. This rate is the fastest reported propagation for a solid-phase, metallic-based nanoenergetic system, but is still far below that of traditional high explosives (i.e. TNT, propagation rate of >6600 m/s [21]). Despite this gap, more ways can be explored to further increase the reaction speed of PS energetics. Parimi and coworkers recently demonstrated that on-chip PS combustion speeds are enhanced through the implementation of hierarchical microstructures within the nanoporous silicon film [5]. This method yields an increase in propagation rate of ∼2 orders of magnitude, starting from a relatively slow propagation rate of ∼2 m/s. The implementation of microstructures to a porous silicon system with explosive-like combustion properties has not yet been reported, but could produce similar propagation rate increases and provide insight into the controlling mechanisms for combustion in porous solids.

Rapid combustion of solids can proceed in a number of ways. Convection is thought to be an important parameter for deflagrating or combusting systems [22–24] that propagate below the speed of sound of the combusting material. For detonations, where flame propagation is supersonic relative to the material through which the reaction propagates, the energy and heat carried by a shock wave can propagate reactions at very rapid rates. In porous media, shock waves can alter material properties by compacting and fracturing the system producing variations in porosity and lowering the effective activation energy for the system [25,26]. Korotkov et al. note that as the effect of convective heat transfer increases, so too does the intensity of compressive waves sent through the porous medium [22]. They elaborate by stating that compressive waves in a porous medium could play a crucial role in the subsonic or pre-detonation regime of combustion. The pre-detonation or rapid deflagration

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regime remains difficult to fully understand as many factors contribute to the combustion, but further knowledge on these events could help to improve tuning and enhancement of energetic materials.

As with many energetic materials, the primary reaction mechanism for porous silicon combustion is still not clear. This issue is complicated by the wide range of reported propagation rates, 1–3100 m/s [5,8], which suggest that multiple reaction mechanisms are at play. The faster burning porous silicon systems could be in a rapid deflagration, or even detonation regime as the speed of sound in porous silicon ranges from <1000 m/s to ~8500 m/s, depending on porosity [27], but predicted reaction mechanisms have generally been speculative. In comparing two rapidly reacting samples (2200 and 3100 m/s), Becker et al. [8] acknowledge the importance of stoichiometry and surface area for optimizing the reaction. They also suggest that a lower porosity may allow for more rapid travel of acoustic or shock waves that could aid the reaction. However, inconclusive evidence is provided to support the idea that such events are occurring. Plummer and co-workers [9] provide an in-depth study on the dependence of porous silicon combustion on its material properties for moderate burn speeds (1–400 m/s). They show that stand alone porous silicon films burn at a faster rate than films still attached to the underlying silicon substrate in a confined environment. It is thought that the suspended films have more surface area available for hot product gases to convectively heat the substrate at a faster rate. They also show evidence of a flame jump ahead of the primary flame front; a phenomenon that has been observed in a variety of propagating combustion systems [8,9,28,29]. They suggest this is caused by lift off of the film from the underlying silicon substrate, allowing gases to penetrate below the film and ignite the sample ahead of the primary flame front. In their work on the effect of microstructure on porous silicon combustion, Parimi et al. attribute increased performance for combustion in porous silicon microstructures to a shift from conductive to convective heat transfer [5]. This conclusion follows theory on the deflagration to detonation transition, and is thought to be the controlling mechanism for rapidly reacting nanothermites that burn at ~1000 m/s. Although convective heating is likely relevant at those reaction speeds, it is not clear what controls propagation at higher rates.

The present study investigates the rapid reaction of on-chip nanoporous silicon strips with etched microscale channels. The starting porous silicon material was similar to that used by Becker et al., with the exception of channels. With these microchannels, significant enhancement of flame propagation was observed, resulting in the fastest reported propagation speed for metallic fuel based energetic materials to date. In the literature, the most common controlling mechanism attributed to rapid combustion of nanoenergetic materials is convection, but it was not apparent that convection was responsible for enhancement in the microchannels. In previous work [8] we alluded to the presence of shock or compressive acoustic waves, which can alter media and enhance reaction rates [22,25,26]. However, for rapid combustion of porous silicon, evidence for this hypothesis has not been reported. Here we provide experimental evidence that reaction propagation in microstructured porous silicon correlates to flame "jump" events in neat porous silicon combustion, and also to the speed of sound through porous silicon. These results strongly suggest that shock or acoustic propagation can drive porous silicon combustion in these microchannel structures.

2. Experimental setup

2.1. Sample preparation

The porous silicon in this study was formed through a galvanic etch process that is detailed elsewhere [6,8,17,30,31]. The starting material was a p-type, boron-doped silicon wafer with resistivity of 1–10 Ω-cm, thickness of 525 μm, and <100> orientation. The wafer front-side had a low pressure chemical vapor deposited (LPCVD) silicon nitride (Si_3N_4) coating with an average thickness of 6078 Å, and a back-side layer of 170 nm sputtered platinum by LGA Thin Films, Inc. (Santa Clara, CA) that acted as the cathode during the etch process.

The Si_3N_4 was selectively removed via reactive ion etching to reveal strips of silicon for subsequent porous silicon etching. Channels of 50 μm wide and 50–60 μm deep were formed prior to porous silicon formation by deep reactive ion etching (DRIE). A sample prior to porous silicon formation is shown in Fig. 1 with both channeled and un-channeled sections of silicon. Scanning electron microscope (SEM, Hitachi S-4500) images of the ion etched channels are also shown in Fig. 2-I.

Channels were parallel to the direction of flame propagation in some samples, and perpendicular in others to observe the effect of direction on combustion. Channeled structures were also placed in various regions of the porous silicon strips, and integrated with un-channeled sections of PS as shown in Fig. 3A. Figure 3B depicts the transition of the neat porous silicon into the channeled region for perpendicular channels. For each set of experiments with one channel type, parallel or perpendicular, all samples came from a single wafer. For the longest test samples with channeled and un-channeled sections shown in Figs. 1 and 3, the bulk dimensions of the nanoporous silicon strip were 6 cm long and 3 mm wide, with the 3 sub-sections alternating between channeled and un-channeled measuring 2 cm in length.

After implementation of the channels, a porous silicon forming galvanic etch process was performed by placing the sample in an etch solution consisting of hydrofluoric acid (HF, 49% in H_2O), ethanol (EtOH), and hydrogen peroxide (H_2O_2, 30% in H_2O) for 10 min. The concentration of each component of the etch solution affects the structure of the porous silicon, and thus the combustion event. The solution primarily used in this study was a 3:1 volumetric ratio of HF:EtOH with 2.4% by volume hydrogen peroxide, similar to that used by Becker et al. [8]. It should be noted that the porous silicon film did not obviously crack or fragment as observed in other studies [5,18].

2.2. Material characterization

Characterization of porous silicon films was primarily done through gas adsorption porosimetry using a Micromeritics Tristar II 3020 surface analyzer. This system used the Brunauer–Emmett–Teller (BET) theory [32,33] to determine surface area and the Barrett–Joyner–Halenda (BJH) theory [33,34] to determine average pore size and total pore volume of the porous silicon. Porosity was derived through a gravimetric technique where the

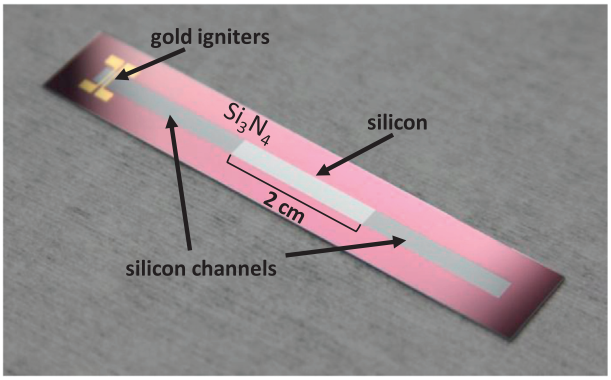

Fig. 1. A channeled silicon/neat silicon test strip prior to porous silicon formation.

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