

The influence of pore size and oxidising agent on the energetic properties of porous silicon

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ABSTRACT

The explosive properties of porous silicon (pSi) provide an alternative to existing carbon-based explosives. Here, an investigation into these explosive properties is conducted, by introducing an oxidiser onto freshly etched pSi films with varying pore sizes as determined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Explosions are triggered via the application of an electric spark. Light output and spectral data are collected to characterize the explosion. Measurements of light output indicate that sodium perchlorate produces the most brilliant explosions, however spectral data indicate that aluminium and gadolinium nitrates produce hotter explosions. The energy output is measured by means of Differential Scanning Calorimetry (DSC), indicating that almost 9 kJ/g is released, double that released by the detonation of TNT. The energetic material cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX) is added to investigate a method of improving explosive performance of pSi, with disappointing results.

Keywords: Porous silicon, explosive, energetic material, RDX

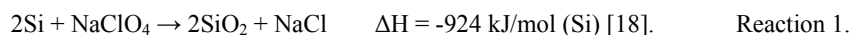
1. INTRODUCTION

The discovery of pSi originated from the attempts of establishing a method of electrochemical polishing techniques for monocrystalline silicon by Uhlir in 1956 where, “matte black, brown, or red deposit” was produced at low current densities [1]. The formation of pSi is achieved by the anodic electrochemical etching of monocrystalline silicon wafers using electrolytes based on hydrofluoric acid (HF). This produces cavities in the surface of the wafer but leaves the overall dimensions of the wafer unchanged [2]. These cavities can vary from disordered sponge-like voids to highly uniform and ordered channels [2], and can range in size from 2nm – 2µm. The most uniform and reliable pSi surfaces are produced when etching is carried out under conditions of constant current density [2, 3]. The porous structure allows for a vast internal surface area (up to 800m²/cm³), and it is estimated that 20% of silicon atoms in the porous layer are on the surface of the pores [4]. Freshly prepared pSi surfaces are covered with a monatomic layer of hydrogen atoms bound to the surface silicon atoms [5], however, when left to age in ambient air, the surface will oxidise interspersing oxygen atoms into the sub-surface Si-Si bonds at depths of up to 1 nanometre [6].

As a semiconductor, porous silicon has many potential uses in the field of nanotechnology, including, drug delivery, biosensors, and micro-reactors [1]. However, it was the accidental discovery in 1992 [7] that a drop of concentrated nitric acid (a strong oxidising agent) onto a freshly etched pSi surface reacted violently, producing a flash of light and an “audible pop,” that first sparked interest in pSi as the basis for an energetic material.

In 2001, Kovalev et al. [8] condensed liquid oxygen onto fresh and aged pSi surfaces. They found that on freshly prepared surfaces, the explosive reaction would occur spontaneously at temperatures as low as 4.2 K. On aged pSi, the surface was stable enough to not explode spontaneously, but could be initiated as a result of mechanical impact or heating by an ultraviolet laser. Research has accelerated since these works, and numerous groups have found that various oxidising agents are capable of producing the strong explosive reactions [8-17]. These agents typically include sodium perchlorate, aluminium or potassium nitrates, sulphur, potassium permanganate and potassium dichromate, with different groups reporting varying degrees of success with these agents. These oxidisers are typically loaded into the pSi surface from a solution in a low molecular weight solvent (e.g. methanol, ethanol, carbon disulphide) and seep into the depths of the pores as a result of capillary action. The solvent is then evaporated.

In all cases, however, it is the very high internal surface area of the pSi surface which permits a highly intimate mixture between the oxidising agent and the silicon. The oxidation of elemental silicon is highly exothermic, much more so than if carbon was used as the fuel instead of silicon. For example, the basic reaction of silicon and sodium perchlorate proceeds as follows:



This equates to an energy yield of 10.35 kJ/g of reactant mixture [18]. Substituting carbon for silicon in Reaction 1 yields an enthalpy of reaction of only -407.5 kJ/mol (5.56 kJ/g) [18]. For comparison, the detonation of the high explosive TNT yields 4.564 kJ/g [19a]. Reaction 1 is only a theoretical approximation of the energetic reaction. The presence of both crystallisation water associated with the perchlorate, bound hydride species on the silicon surface, and the differences in stoichiometry able to be achieved due to varying pore geometries will complicate matters [20]. Under these circumstances, the reaction will likely proceed with incomplete combustion, producing substances which can undergo further combustion with atmospheric oxygen due to the high temperatures present. These secondary reactions will often occur over a significantly longer time scale than the initial reaction, and will tend to support or enhance the effect of the explosive rather than contribute directly to it [21].

There are a number of advantages to using pSi as the basis for an energetic material. The infrastructure necessary for mass production of silicon based devices is well established, and the techniques for that production are well understood [11]. The potential exists to incorporate pSi energetic materials directly onto the surface of electronic components, enabling tiny yet complex energetic devices to be constructed [11]. Real-world industrial applications of such devices include the energy source for portable atomic emission spectroscopy instruments [13] and micro-scale electro-explosive devices (e.g. automotive air bag initiators) [13, 22-24].

The influences on the explosive performance of these systems has been shown [8-10, 16] to include the size and depth of the pores (in turn influenced by the etching variables of HF concentration, wafer properties, etching current and time), the choice of oxidising agent and ratio of Si to oxidising agent. This work will explore the effect that some of these influences have on the energetic performance of pSi.

2. EXPERIMENTAL METHODS

2.1 Materials

The silicon wafers used were p-type – boron doped, with resistivity of 3 – 6 Ωcm , <100> orientation, supplied by Silicon Quest International. Oxidising agents ($\text{NaClO}_4 \cdot \text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), hydrofluoric acid and solvents, were supplied by Sigma Aldrich.

2.2 Analytical Methods

Light output data was collected using a Centronic OSD15-5T photodiode 430-900nm (Farnell), with a response speed of 12 ns. A Twalux TW-MF2CAB photodiode amplifier board (Farnell) was used to convert photocurrents to voltage. A USB 6900 device was used to record data with National Instruments Lab view version 8.2 software. This system analyses the time-resolved light emission from the reacting energetic material at a single point on the etched area, producing a curve of amplified photodiode voltage (volts) vs. time (milliseconds), and is characterised in terms of the peak (maximum) light output and total light output (area under the curve).

Emission spectra of explosions were obtained using an Ocean Optics USB 2000 spectrometer, a fibre-optic cable, and Spectra Suite 1.4.2_09 software. Emissions were detected with a 2048-element Sony ILX511 linear CCD array detector.

Tapping mode AFM imaging was performed with a Nanoscope IV Multimode microscope (Digital Instruments). Commercial Si cantilevers (FESP7, Veeco Corporation, USA) with a resonance frequency 75-85 kHz were used for all experiments. The images were processed using scanning probe microscopy software WSxM 4.0. SEM images were obtained on a Phillips XL30 Scanning Electron Microscope (SEM).

Differential scanning calorimetry (DSC) traces were recorded by a DSC2920 TA instrument using TA instruments Universal Analysis 2000 version 3.9A software. Additional experiments were performed on a DSC Q10 TA instrument using TA instruments Universal Analysis 2000 version 3.9A software.

2.3 Porous Silicon Preparation 2.3 Preparação de silicone poroso

p-type (3 – 6 Ωcm) silicon wafers were cut with a diamond scribe into small pieces (approximately 2 cm x 2 cm). Each piece was washed with ethanol, acetone and dichloromethane (DCM) and dried under nitrogen prior to etching. The clean piece of silicon was secured into a Teflon etching cell and washed with ethanol, acetone and DCM and dried under nitrogen. The cell produced a circular etched area 15mm in diameter (1.77 cm^2). All etching was carried out using 40% HF in ethanol.

Oxidising agent stock solutions consisted of sodium perchlorate (0.6M, 0.7345g) dissolved in 10 ml AR grade methanol, aluminium nitrate (0.2M, 0.7503g) dissolved in 10 mls of AR grade ethanol, and gadolinium nitrate (0.2M, 1.8g) dissolved in 20 mls of AR grade ethanol. All oxidising solutions were discarded after one week storage. 20 μ L of solution was pipetted onto the pSi surface, and the solvent allowed to evaporate for 1 hour prior to ignition. A higher concentration of sodium perchlorate was used compared to aluminium or gadolinium nitrates in order to provide an equivalent number of oxidising anion species available for energetic reaction.

All samples were ignited using an electric spark having a potential of 3–4 kV.

3.RESULTS AND DISCUSSION

3.1. Effect of choice of oxidising agent

Figure 1 depicts the time-resolved light output of the energetic reaction, as recorded by the amplified photodiode. Three samples of pSi were prepared by etching for 30 minutes at 40 mA (22.5 mA/cm²) and loaded with 20 μ L of aluminium nitrate oxidising solution. The light rises sharply to a peak in 2 to 3 ms, indicating a sudden initiation of the energetic reaction across the surface, followed by a broader decrease in light intensity resulting from longer term combustion reactions and cooling of energised gasses.

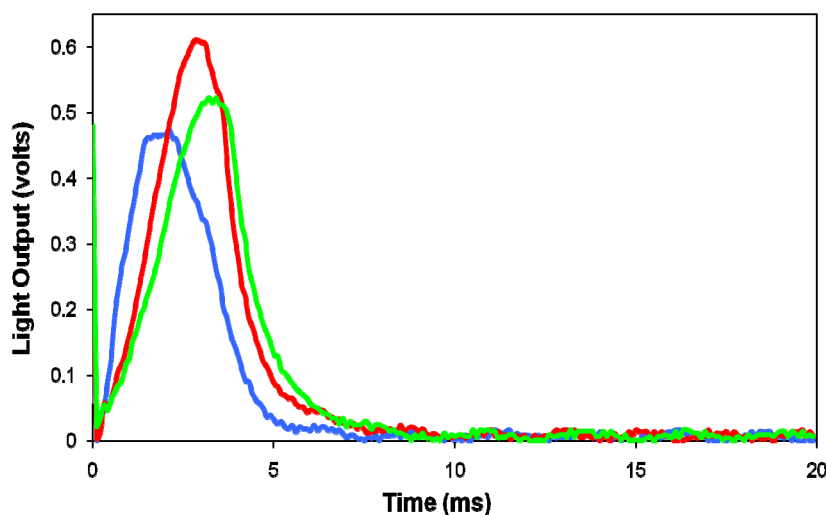


Figure 1: Recorded photodiode output of pSi loaded with aluminium nitrate. Three separate samples to indicate variance in recorded light output.

SEM analysis of samples etched under these conditions indicated that the porous layer was 15 μ m thick (Figure 2). Gravimetric analysis following the method of Halimaoui [3] was used to determine that these etching conditions produce pSi with a porosity of 55.9%, and the etched area had a mass of 4.57 mg. 20 μ L of solution equates to 1.5 mg of aluminium nitrate, with a total energetic material weight of 6.07 mg having a density of 0.880 g/cm³.

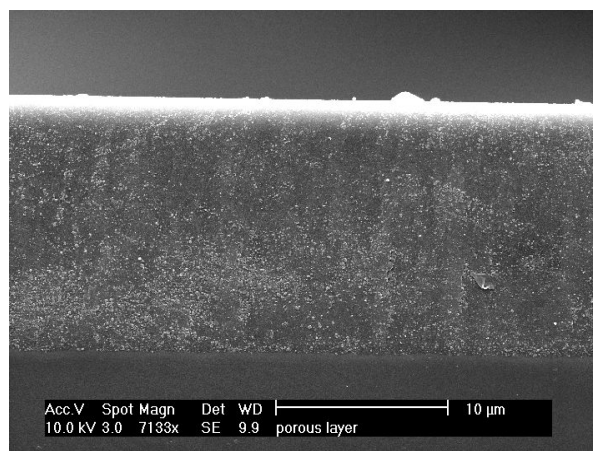


Figure 2: SEM image of pSi layer (p-type, 3-6 Ω cm, 30 minute etch, 40% HF, 40 mA). Porous layer is 15 μ m thick, average pore diameter is 12 nm.

Three groups of ten samples of pSi etched for 30 minutes at 40 mA (22.5 mA/cm²) were loaded with sodium perchlorate, aluminium nitrate and gadolinium nitrate and the light output recorded. The maximum and total light output of these samples is depicted in Figures 3 and 4 respectively. These figures show that pSi loaded with sodium perchlorate produces the most brilliant explosions in terms of maximum intensity and total light output. This is in good agreement with results presented by other groups [9, 16]. This observation may indeed be due to the greater intensity of the explosion of this particular system, however it may be due to the fact that sodium has a strong spectral emission peak at 589 nm (within the optical range of the photodiode) whereas neither aluminium nor gadolinium have strong emission peaks in this range (see Section 3.4). A similar result was observed by Mickulec et al. [13]. It must be remembered that the voltage generated by the photodiode is passed through an amplifier prior to recording. Variances in adjustable gain settings within the amplifier mean that the measured light output (expressed as volts) is somewhat arbitrary. These results may not be directly comparable to results from other groups, but will be comparable with other results in this research group. The reader should endeavour to use appropriate control samples when conducting their own testing.

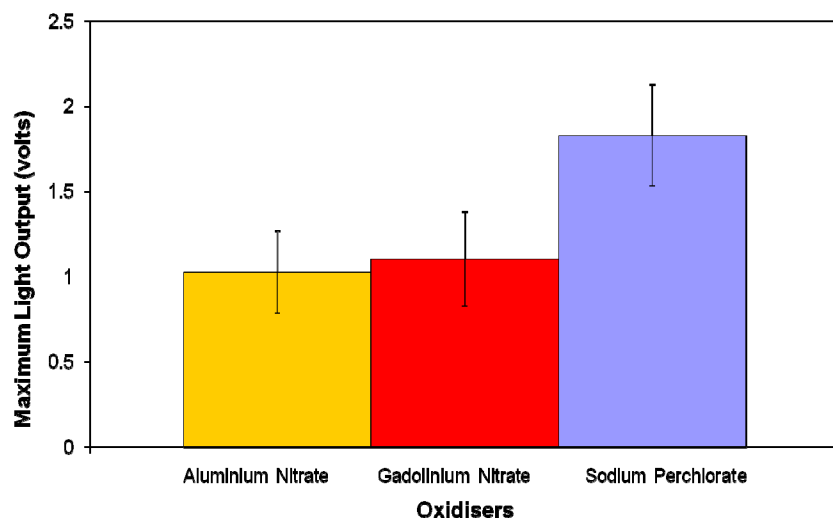


Figure 3: Maximum light output of pSi (p-type, 3-6 Ω cm, 30 minute etch, 40 mA, 40% HF) loaded with various oxidising agents.

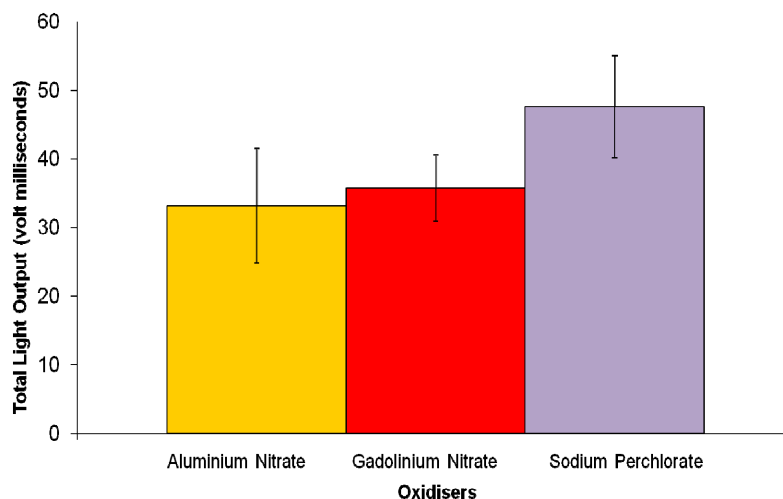


Figure 4: Total light output of pSi (p-type, 3-6 Ω cm, 30 minute etch, 40 mA, 40% HF) loaded with various oxidising agents.

3.2 Effect of etching current on pore size and subsequent energetic performance

Samples of pSi were prepared at etching currents of 20 mA (11.3 mA/cm²), 40 mA (22.5 mA/cm²), 60 mA (33.9 mA/cm²) and 80 mA (45.2 mA/cm²). Samples were etched for 60, 30, 20 and 15 minutes respectively, to maintain a constant charge of 72 coulombs. AFM was used to determine the average pore size at these etching currents, as depicted in Figure 5.

3.2 Efeito da corrente de corrosão no tamanho dos poros e desempenho energético subsequente

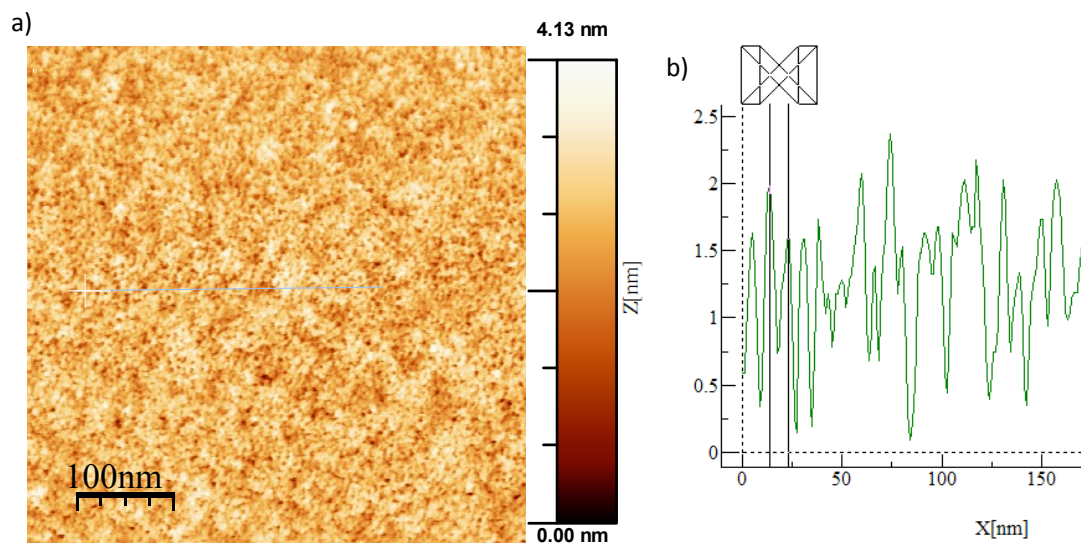


Figure 5: (a) AFM image of a pSi surface etched at 60 mA. Blue line indicates the path used to determine the surface profile depicted in (b).

These etching currents produced pores with average diameters of 9.7, 11.8, 12.2 and 14.7 nm, respectively. Each of these surfaces was loaded with 20 μ L of aluminium nitrate solution and initiated with the piezoelectric spark. The total light output of these samples (Figure 6) was measured using the photodiode. This figure shows that whilst the most consistent results were obtained at a pore size of 12.2 nm (60 mA), a critical point exists above which energetic performance decreases (11.8 nm / 40 mA for this study). Du Plessis et al. found a similar result when investigating a sodium perchlorate loaded pSi surface, concluding that the optimum was achieved at a pore size of approximately 3 nm [9, 16]. For a given number of pores per unit area, increasing the pore size will increase the internal specific surface area, but more silicon will have been etched away, reducing the amount of

fuel available for energetic reaction. There is obviously an optimum balance of pore size suited to individual systems, indicating the care that must be taken when designing a system for a particular industrial application.

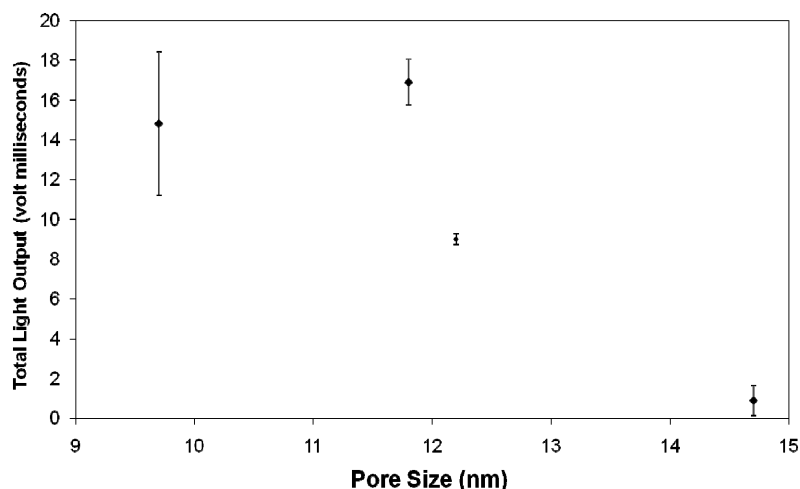


Figure 6: Total light output of pSi etched at different etching currents loaded with aluminium nitrate.

3.3 Differential Scanning Calorimetry (DSC)

DSC was used to analyse the energy liberated as a result of heating loaded pSi samples in an attempt to characterise the enthalpy of reaction. Sections of pSi 2mm x 2mm were cut from the main etched sample (etched at 40 mA for 30 minutes) and loaded with 2 μ L of aluminium nitrate solution. Subsequent DSC analysis (heating from room temperature to 210 $^{\circ}$ C at 10 $^{\circ}$ C/minute, N₂ atmosphere @ 50 ml/min flow rate) recorded an ignition temperature of 75.43 $^{\circ}$ C with an energy release of 96.42 J/g (Figure 7). Considering that only the loaded pSi layer is reacting energetically, the mass of the silicon backing layer interferes with energy calculations. For a 2 mm x 2 mm section of pSi of 15 μ m thickness and 56% porosity, the actual weight of the pSi is calculated to be 42 μ g. From this, the energy released by the reacting pSi is calculated to be 5.08 kJ/g. This is compared to the energy released by detonation of the high explosive TNT (4.56 kJ/g) [19a]. Similarly, Clement et al. [11] found that pSi loaded with calcium perchlorate would yield 7.3 kJ/g using bomb calorimetry. pSi loaded with gadolinium nitrate and sodium perchlorate were unable to be initiated on the DSC instruments used.

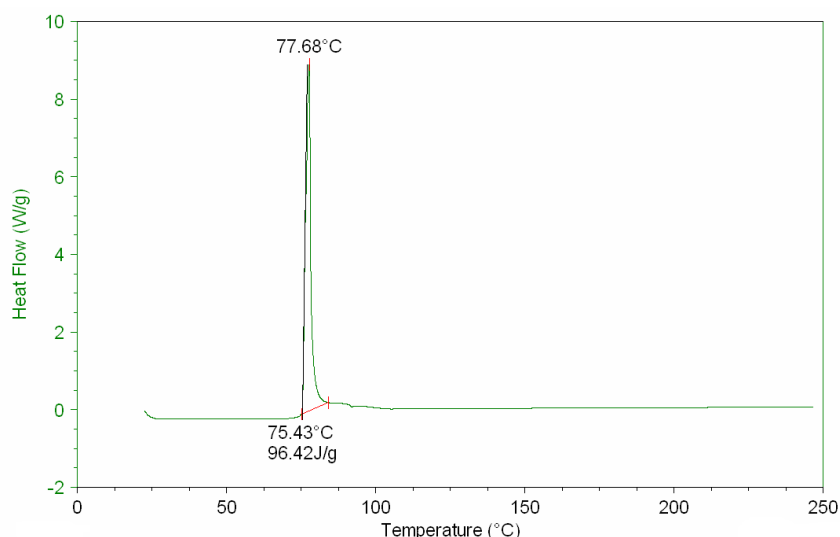


Figure 7: DSC record of a pSi film loaded with aluminium nitrate.

To account for the fact that only the pSi layer is reacting energetically, pSi samples were prepared and subsequently stripped from the backing substrate prior to loading and analysis. After the initial etching was

complete, the electrolyte was replaced with 25% HF in ethanol, and 450 mA of current was applied for 30 seconds. This secondary process cleaved the pSi layer from the backing substrate, with the resulting membranes being (very) carefully washed and dried. 5 mm² pieces of membrane were then loaded with aluminium nitrate by soaking in 20 μ L of solution and allowed to dry. The DSC trace of this material (Figure 8) gave an initiation temperature of 59.08 °C and an energy yield of 4.07 kJ/g. It is unclear as to why there is a discrepancy between the DSC results of pSi bound to the silicon substrate wafer compared to a separated pSi membrane, however the very small masses involved when testing the pSi membrane (0.03-0.05 mg) severely tested the capabilities of the available analytical balance, introducing a potential source of relative error into the calculations. Alternatively, the fact that all surfaces of the separated membrane are exposed (compared to the one free surface when bound to the backing Si wafer), may influence the dynamics of the reaction in a way that might effect the DSC data.

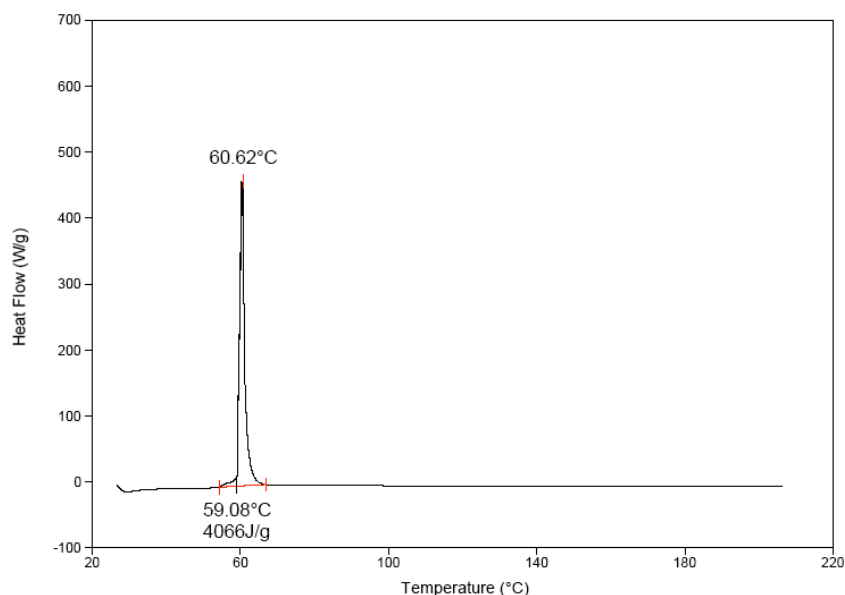


Figure 8: DSC of a free-standing pSi membrane loaded with aluminium nitrate.

3.4 Emission spectra of pSi energetic materials.

pSi samples etched for 30 minutes at 40 mA were loaded with each of the three oxidising agents, and the emission spectra of the energetic event recorded (Figure 9). Each of the three samples displayed a pronounced peak at 589 nm with intensities for sodium perchlorate, gadolinium nitrate and aluminium nitrate being 4095, 3632 and 2079 counts, respectively. This peak is to be expected for pSi loaded with sodium perchlorate, but is considered to be an artefact (and should be absent) for the other two oxidising agents. In any case, the spectra for the two nitrates are almost identical, showing a broad emission over a wide range of wavelengths. The maximum emission (ignoring the sodium peak) for these oxidisers are 687, 632 and 637 nm, respectively. Application of Wein's displacement law [25] indicates that the black body radiation equates to explosion temperatures of 4220, 4584 and 4563 K. Mickulec et al. [13] also studied the emission spectra of pSi loaded with gadolinium nitrate, and similarly found an artefact at 589 nm due to sodium contamination. This group also determined the temperature of the explosion to be only 2000 K, less than half that determined by our work. Factors such as the pore size and porous layer thickness have already been shown to affect the intensity of the explosion, one possible cause for the discrepancy.

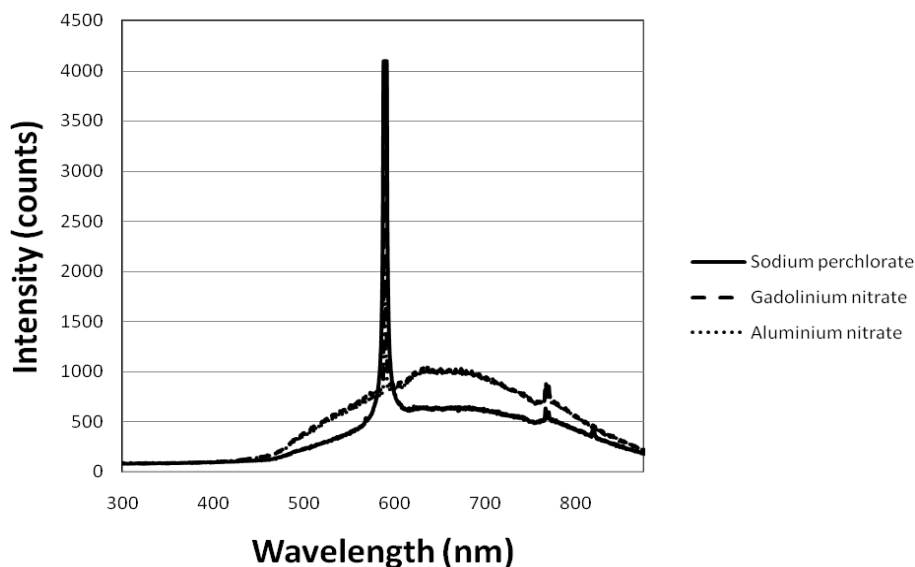


Figure 9: Emission spectra of pSi loaded with various oxidising agents.

3.5. The use of energetic loading agents.

In an attempt to boost the explosive performance of the system, the high explosive RDX (cyclo-1,3,5-trimethylene-2,4,6-trinitramine) was added (20 μL , 0.2 M in acetone) to the pSi surface (40 mA for 30 minutes). The light output is displayed in Figure 10. The results were not encouraging. Total light output when the pSi was loaded with RDX was significantly reduced when compared to loading with aluminium nitrate. This is possibly attributable to the fact that RDX is significantly deficient in oxidising species (it requires an additional 21.8 % (by weight) of atmospheric oxygen to react completely [19b]), and whilst it may be reacting with the pSi, its performance is severely hampered.

An attempt to overcome this was made by preparing additional samples were prepared by first adding 10 μL of aluminium nitrate solution and then 10 μL of RDX solution to an etched surface, in an effort to supply an oxidising species to the mixture. A white precipitate was formed on the surface of the sample when the RDX was added, possibly as a result of leaching of aluminium nitrate from the pores due to the acetone in the RDX solution, further reducing the light output.

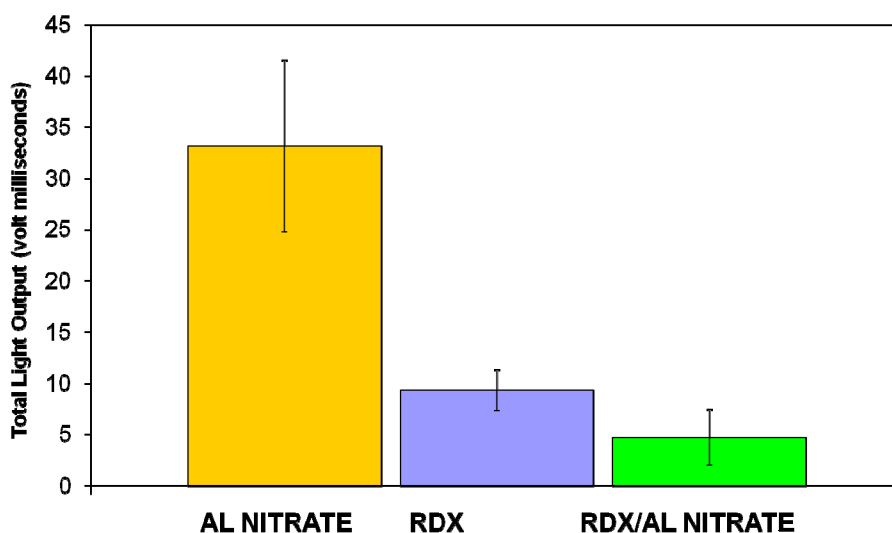


Figure 10: Performance of pSi loaded with RDX and RDX / aluminium nitrate.

4. CONCLUSIONS

The energetic performance of pSi was found to be most intense (in terms of light output) when the porous layer was loaded with sodium perchlorate. Loading the same pSi surface with aluminium and gadolinium nitrates also produced an energetic response, albeit less intense, which is further reduced when the energetic material RDX was added instead of an oxidising agent. When loaded with aluminium nitrate, the light output was found to be optimum at a pore size of less than 12 nm and a depth of 15 μm . Conversely, the emission spectra of these systems indicated that when loaded with nitrates, the explosion was hotter than when loaded with the perchlorate. It may be that the presence of significant emission spectra attributable to the oxidising agent species (sodium in this case) will tend to mask the true intensity of the explosion, leading to erroneous conclusions when different oxidising agents are compared.

DSC experiments showed that a large amount of energy (5.08 kJ/g) is released in the energetic reaction, supporting the theoretical assessment of the system, and bomb calorimetry data produced by other groups.

Future work will include a method of analysis of the explosion intensity that is independent of the spectra produced, such as the velocity of the reaction front across the surface, or the pressure generated (and hence the available work) as a result of the explosion.

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