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Wide Band High Level Thermal Radiation Emission of Highly Doped Black Silicon

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Abstract. We present a silicon based metamaterial, Black Silicon in our case, which exhibits a very high wide band spectral emissivity in the spectral range of thermal radiation, up to 8 μm , which can be used as a broadband emitter. Black Silicon is a bottom-up nano-structured silicon surface that can be created using a variety of processes, such as wafer-level cryogenic plasma etching in our case. After demonstrating previously that the use of a heavily doped silicon wafer to fabricate Black Silicon can extend such remarkable emissivity to the mid-infrared (MIR) and touching the far-infrared (FIR) spectrum, we now report on the spectral hemispherical emissivity of BSi measured by both indirect and direct radiometric methods. The emissivity values obtained by the two methods are in good agreement.

INTRODUCTION

Emissivity characterizes the thermal radiation intensity emitted from a sample surface as compared with that from the blackbody which is the ideal emitter at the same temperature for the same spectral and geometrical conditions [1]. Emissivity measurements are of great importance for both theoretical and experimental research, in such areas as temperature measurements by radiation thermometry [2], radiative heat transfer between objects [3], thermal shield for space applications [4], energy efficiency in buildings [5] and thermal management of different systems [6]. The methods for emissivity measurements can be roughly classified into two categories: indirect methods [7] and direct methods [8]. The method is labeled direct when the emissivity is obtained by comparison between the radiation intensity emitted by the sample and a blackbody radiator. Meanwhile, the method is considered indirect when the reflectivity and the transmissivity of the sample are measured, the absorptivity and emissivity deduced from energy conservation and Kirchhoff's law, respectively. The integrating sphere is the most commonly used spectroscopic accessory in the indirect method for hemispherical spectral emissivity determination at room temperature [9]. However, this method is limited by the technical difficulty when using an integrating sphere, which is unavailable at high temperatures. Hence, direct measurements of emissivity [10] are made calorimetrically [11] or radiometrically [12]. For the calorimetric emissivity measurements, the rate of radiant heat transfer from a sample is measured in terms of the heat lost by the sample. The ratio of the measured rate of heat transfer to that of a blackbody radiator under the same conditions is the emissivity. The evaluated emissivity is usually the total hemispherical emissivity. Meanwhile direct radiometric emissivity evaluations are made by measuring the intensities of a heated sample by an IR detector, an FTIR spectrometer detector for example, and of a blackbody at the same temperature as that of the sample under the same spectral and geometric conditions, and by computing the emissivity as the ratio of the two intensities. The direct method based on the definition of the emissivity is the most common method to determine emissivity. A laboratory blackbody or possibly some other source of radiation with a known high spectral normal emissivity, is used as a reference. The radiation intensity of the reference is measured either at the same time as the sample intensity at the same temperature, or the reference is employed as a calibration radiation source for the detection system. In the second instance, the sample radiation intensity is detected after the calibration process. The ability to design metamaterials with controlled emissivity is very useful for numerous applications related to thermal radiation such as thermophotovoltaics, infrared spectroscopy, gas sensing, radiative cooling, thermal rectification, etc. In this paper, we focus on a silicon based metamaterial. The spectral emissivity of silicon (Si), investigated as early as 1967 in the work of Sato et al. [13], gained more recently a renewed interest owing to the ability to produce nano/microscale surface structurations, which led to radically different properties from those of plain flat surfaces. Micro/nano structured silicon exhibits a much higher absorptivity than flat silicon and hence, a much higher emissivity, by virtue of Kirchhoff's law. Nowadays, surfaces of silicon structured at the micro/nano scales [14] are of prime intrigue for a variety of applications such as solar cells [14], selective thermal emitters [15], radiative cooling,

thermal rectification, photodetectors [16], and black body radiation sources [17] which require high emissivity [18] materials. Nanostructured silicon surface (NS-Si) that can be acquired by various strategies including cryogenic plasma as considered in this work, shows interesting wetting and optical properties. Specifically, because of its particular morphology, it is notable for its fantastic absorption of nearly 99% of incident light mainly in the visible wavelength range and consequently its name is derived as it appears to the bare eye. BSi has been studied since 1995, when the micro-structured silicon was fabricated by reactive ion etching (RIE) with high depth-to-width ratio [19]. However, not many works have been dedicated to the arena of radiative properties characterisation of BSi for infrared applications, hence the motivation to conduct this study.

In the present manuscript, we report on indirect emissivity measurements at 30 °C and direct measurements of hemispherical spectral emissivity of moderately doped black silicon in the mid-infrared range of wavelength up to 20 μm and at a temperature range from room temperature up to 125°C.

MATERIALS AND METHODS

Experimental Setup

We have fabricated two wafers of BSi having n-type doping of $1 \times 10^{15} \text{cm}^{-3}$ (moderate doping) and $5 \times 10^{19} \text{cm}^{-3}$ (high doping). More detailed information on the fabrication of the samples can be found in our previous works [20, 21]. In the indirect emissivity measurement method, we measured the transmittance as well as the specular and diffuse reflectance of BSi using a Perkin Elmer Spectrum 3 FTIR spectrometer in a spectral range from 1 to 25 μm at room temperature. We then calculated the total absorptance A_t as follows :

$$A_t = 1 - R_t - T \quad (1)$$

where, R_t and T is the total reflectance and transmittance, respectively. According to Kirchoff's Law, the absorptivity A_t is equal to the emissivity ϵ . In addition to the indirect method experimental setup, a first version of a direct hemispherical spectral emissivity measurement setup has been realized to enable the measurement of the emissivity at temperatures larger than room temperature. Here, the radiation intensity from a heated sample is compared with that of a blackbody emitter at the same temperature to calculate the sample spectral hemispherical emissivity. The experimental setup used in the the present work is inspired from [22] and comprises of a FTIR spectrometer, a sample heater, a reference blackbody (in our case, a LANDcal blackbody source, R1200P) and an optical assembly comprising of two parabolic mirrors for the IR beams collimation and a plane mirror that enables switching between the reference source and the sample under consideration. A schematic of the experimental setup is shown on FIGURE 1. The FTIR is a PerkinElmer Spectrum 3 spectrometer capable of measuring spectral intensity from 1 -25 μm was configured to operate with an external source. For this purpose, the FTIR internal source was removed and replaced by an IR reflector to redirect an IR beam coming from the spectrometer external port to the spectrometer detector. The LANDCal R1200P Blackbody source, has a working temperature up to 1150°C. The sample heater is made of a PCB of FR4 designed to ensure there is temperature homogeneity and maximum temperature reached is 125°C. Two polished highly reflecting aluminium tubes are attached to both the sample and the blackbody source to account for a) equal surface area of radiation, b) to maximize the radiation going from the source to the detector (the tube made of an IR reflective material acts as a hemispherical collector and concentrator) and c) also to reduce ambient radiation share in the detected radiation. This also ensures equal optical path lengths for the emitted radiation from the two sources. The rotation of the plane mirror is limited to only two locations and is capable of rotating 90° to switch between the sample and the blackbody source. This also ensures that no source is displaced, and the spectra of the two sources may be recorded solely by manipulating this flip mirror.

Calibration

The mirrors' alignment was tested first with a visible light source and then with two distinct infrared sources to guarantee that there is no optical path discrepancy. When working with the visible source, the spot size and spot position of the visible source from two locations: one at the blackbody position and the other at the position of the sample source, was checked to ensure identical placement on a white screen attached to the external port of the FTIR spectrometer along with collimation at every individual optical component. The alignment was then checked for IR

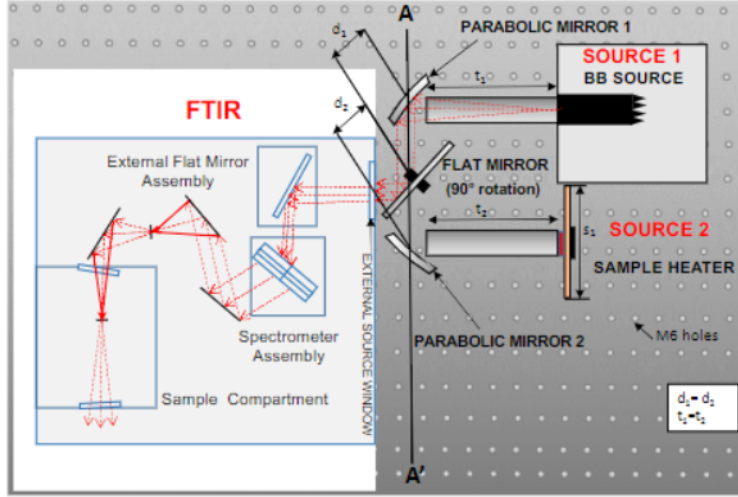


FIGURE 1: Top view of the experimental setup for direct emissivity measurement inspired from [22] highlighting different constituent elements.

radiation using two different IR detectors (S401C and S405C thermal power sensors from Thorlabs) positioned at the FTIR external port. We checked that the same source heated at the same temperature placed at any of the two sources locations- one at the sample heater position and the other at the blackbody position, provides equal values of detected IR radiation for the same detector, thus ensuring equal losses in the two optical paths. In order to quantify and minimise the losses due to the optical assembly, we measured the spectral intensity emanating from both the (1) blackbody source and the (2) sample sources first without the mirrors by placing the sources directly at the external port of the FTIR spectrometer and then using the constructed optical assembly of mirrors to direct the IR beams to the external port of the spectrometer. The maximum intensity is expected when the sources are located at the spectrometer external port. It is then compared with the intensity detected using the optical assembly to quantify the losses.

Emissivity calculation

In the present work, we follow the updated protocol proposed by [22] for a more accurate and simplified direct radiometric method of emissivity measurement. The spectral intensity coming out of the sample source can be described by a simple combination of the self-emitted sample radiation and the blackbody radiation from the surroundings:

$$L^* = \epsilon L_s + (1 - \epsilon) L_{sur} \quad (2)$$

where L^* is the effective spectral intensity, ϵ is the spectral emissivity of the sample, L_s is the blackbody spectral intensity of the sample, and L_{sur} is the blackbody spectral intensity of the surroundings, which in the present case comprises of ambient air, aluminum and the heated FR4 material. Thus the sample emissivity can be written :

$$\epsilon = (L^* - L_{sur}) / (L_s - L_{sur}) \quad (3)$$

On a final note, Eq.3 is applicable only when there are no additional parasitic intensity signals different that need to be filtered out.

RESULTS AND DISCUSSIONS

We show on Figure 2 the experimental results obtained with the indirect methods and that we described in detail in [20, 21, 23]. The quantity shown on this figure is the spectral absorptance which we assume equal to the hemispherical

emissivity according to Kirchhoff's law. We observe that the emissivity of heavily doped BSi reaches 99.5 % till 10 μm , as highlighted in inset(ii), then drops to 98 %. It is obviously larger than that of moderately doped BSi, where the absorptance level never reaches 99 % and drops from a threshold of 96% at 7 μm (inset(i)). The heavily doped BSi absorptance drops to 77% at 20 μm , while the moderately doped BSi absorptance reaches 75%. It can then unobtrusively be concluded that heavily doped BSi is a good candidate for a broadband absorber. We also show on Figure 3(a) the spectral hemispherical emissivity obtained for moderately doped BSi with the direct measurement method at 125°C which varies from 92.8% to 80% over the entire spectral range. The spectral emissivity of moderately doped BSi is then compared to that obtained by the indirect method at room temperature. We observe a significant difference in the emissivity profiles obtained by the two methods even though both of them show large emissivity values. The upper and lower limits of the emissivity obtained by the indirect method over the entire spectral range follows the indirectly measured emissivity limits within $\pm 5\%$.

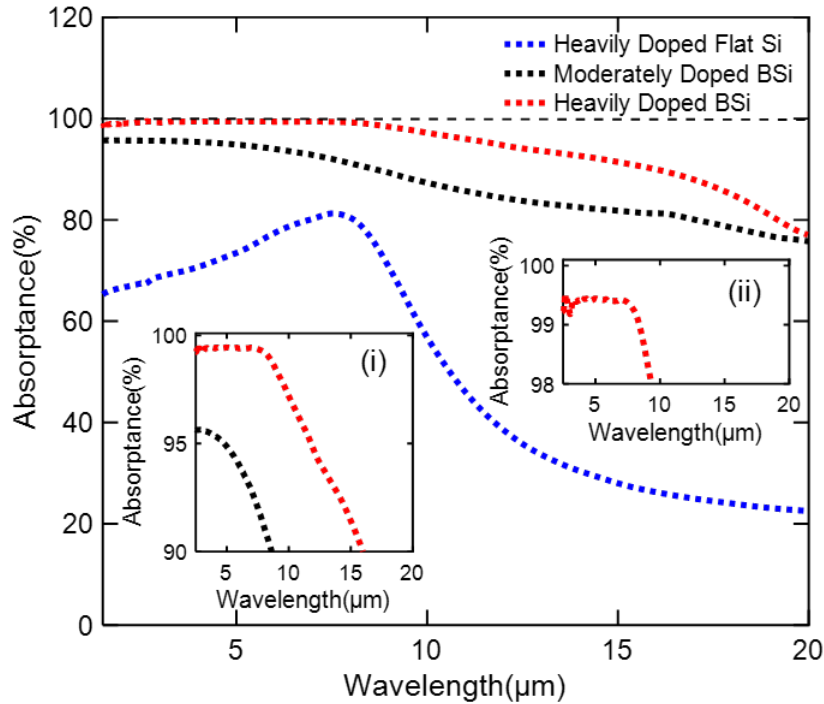


FIGURE 2: Emissivity of two samples of BSi having different levels of doping (heavily doped in red, moderately doped in black) compared with heavily doped flat Si obtained with the indirect method.

We can also observe in Figure 3(a) that the spectral emissivity of BSi houses the background spectra which contains air and other gaseous element spectra added to the radiation from the highly reflecting aluminium tube, which might be heated, as the experiment was not conducted under vacuum. Those undesired parasitic spectral contributions to the detected spectrum can be removed or at least reduced by using a vacuum enclosure and by cooling down the aluminum. Finally, to check the consistency of our results with those recently obtained by other groups, we compare the obtained spectral emissivity of the moderately doped sample of BSi with other experimental data that can be found in the literature [24] as shown in Figure 3(b).

We note similar high levels of emissivity over the whole spectral range with the deference that our sample exhibit higher emissivity for short wavelengths up to 8 μm , and a near unit emissivity below 5 μm , while the results reported in [24] show larger emissivity beyond 8 μm . The observed emissivity differences may be due to several reasons : different doping type (sulfur for [24] v/s phosphorus in our case), different doping levels as well as different BSi fabrication techniques. Over the whole spectral range, a difference of the order of 5% between the two reported results can be note while the two results show good agreement in the high level of emissivity between 1 and 25 μm . Finally, one should note here that we only show results obtained with the direct emissivity measurement method for moderately doped BSi due to the non availability in our case of heavily doped BSi samples with shapes and sizes compatible with the experimental setup for direct emissivity measurement. However, previous results obtained with

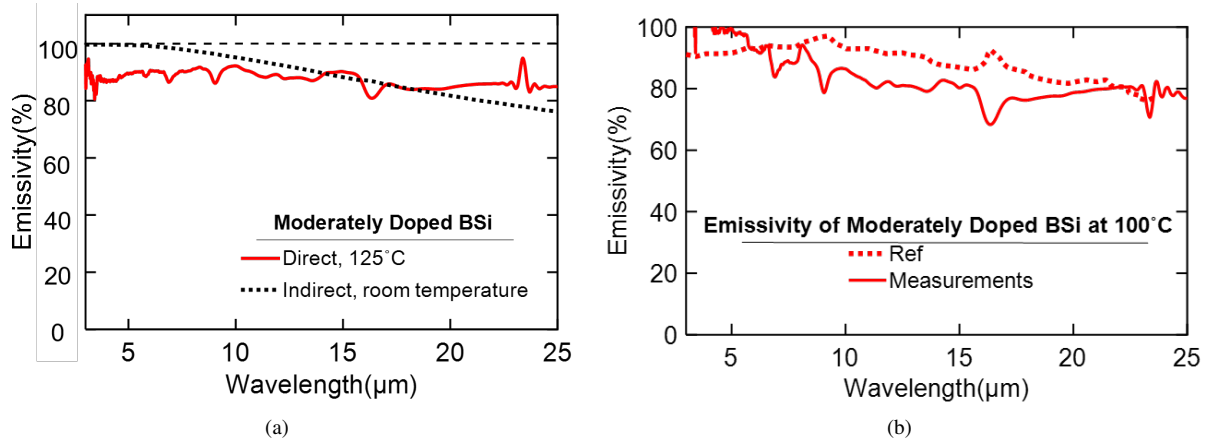


FIGURE 3: (a) Hemispherical emissivity of moderately doped BSi obtained with the indirect method at room temperature (dashed line) and the hemispherical emissivity of the same sample obtained at a larger temperature, 125° in our case, with the direct methods (plain line). (b) Measured spectral emissivity of moderately doped BSi at 100°C compared with literature [24].

heavily doped BSi with the indirect method [23] enable us to assume that heavily doped BSi will exhibit even larger emissivity at temperatures larger than room temperature.

CONCLUSION

In this work, a direct emissivity measurement setup has been developed in order to obtain temperature dependent spectral hemispherical emissivity of IR metamaterials from 1 to 25 μm . It was tested with a silicon based metamaterial, moderately doped BSi in our case, leading to a hemispherical spectral emissivity ranging from 96% to 80% over the spectral range of interest. The spectral emissivity is compared to that measured with an indirect method using the energy conservation and Kirchhoff's law, i.e. to the measured absorptance of moderately doped BSi at 30 °C. The two methods show good agreement with respect to the high level of emissivity over the spectral range of interest. The upper and lower limits of the direct emissivity spectrum are within 5% of the absorptance limits, even though a discrepancy can be observed between the emissivity and absorptivity profiles. Future works include the upgrade of the experimental setup to reach larger temperatures, in order to obtain the direct emissivity of BSi at temperatures relevant for applications such as thermophotovoltaics and to assess the potential of this metamaterial as a radiation emitter for thermophotovoltaic applications. In addition, operation under vacuum inside cooled opaque enclosures is currently being implemented in order to reduce parasitic signals and increase the experimental setup accuracy.

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