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Characterization of the temperature behavior of optimized SiC gratings emissivity

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Abstract

By exploiting experimentally measured optical properties of Silicon Carbide (SiC) at different temperatures, we recently showed based on a numerical analysis, that when SiC grating is heated, the position of its emissivity peak shifts towards higher wavelength values and its amplitude decreases. This proved the necessity to adapt the parameters of the grating to the temperature of measurements. In this previous article, we performed only numerical simulations for ideal devices. In order to check the validity of these results, we need some verifications from both the experimental and numerical points of view. In the herein study, we milled gratings on the same type of SiC for which the optical properties measurements were conducted. Then, we performed reflectivity measurements as a function of wavelength and temperature, in order to experimentally show the peak shift and to validate our optimized gratings. Hereby, this work provides guidelines for the design of optimized coherent thermal sources for different temperatures based on a combined numerical and experimental analysis.

Keywords- Thermal radiation, Coherent thermal source, SiC, Nanostructures

1 Introduction

The development of surface microstructuration techniques has allowed significant progress for the study of microscale gratings and consequently of coherent thermal sources. Indeed, it was shown that milling a grating on top of a polar material, supporting surface phonon-polaritons (SPhP), makes a thermal source, typically incoherent, spatially and temporally coherent. We have then a monochromatic and directional thermal source.

The last two decades have witnessed many studies on such coherent thermal sources. Usually, the objective is to tune the surface emissivity by designing a grating on polar materials [1–5] or metals [6–9] in order to obtain a monochromatic and directional emission peak. Simple 1-dimensional (1D) gratings were thus designed on bulk materials supporting SPhP or surface plasmon-polaritons (SPP) [2, 3, 6, 10–13] or on doped materials [14–17]. Some of these gratings were extended to 2 dimensions [4, 18, 19]. These 2-dimensional (2D) gratings produce coherent thermal emission for both directions of

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polarization: transverse electric (TE) and transverse magnetic (TM), which is more efficient for potential applications. More complex 1D gratings [16, 17, 20–22] were also developed as coherent thermal sources; these include gratings with a mix of polar and metallic materials [23–25]. Similar to simple 1D gratings, 2D complex gratings were also designed and studied [23, 24, 26, 27]. In addition, optical cavities [28, 29], nanotube antenna [30] or photonic structures [31–36] can also behave as coherent thermal sources. It is worth mentioning that gratings can also be used in complement to different periodic structures such as multilayer structures [37–40] and waveguides [41] or in association and combination with graphene [42].

Coherent thermal sources constitute potential candidates for a variety of applications, such as thermal rectification or thermal management. For example, gratings could be used in association with a multilayer structure in radiative cooling devices [43, 44]. Coherent thermal sources can also act as a source for thermophotovoltaic converters [38], be used as biochemical sensors [45, 46] or as camouflage [13].

Some of these applications are used at room temperature, other at higher temperatures. Furthermore, emissivity measurements, to test the design and evaluate the emissivity peak, usually require high temperatures to get enough signal to process. Apart from a few exceptions [27], in most of the studies [1, 2, 47], the grating design is optimized for room temperature application with material optical properties related to this temperature.

In a previous study [48], by exploiting the experimentally measured optical properties of Silicon Carbide (SiC) at different temperatures, we showed numerically that the emission peak of a SiC based coherent thermal source shifts towards higher wavelengths, when the temperature increases. This showed the necessity to take into account the temperature of measurements in the choice of the grating parameters. Considering a SiC ideal surface, we designed several gratings presenting a coherent thermal antenna behavior at different temperatures. To check the validity and consistency of the results of this first fully numerical study, we need further experimental verifications supported by a finer numerical analysis mainly to take into account any possible non-idealities (imperfections) in the geometrical shape and/or composition of the grating. In the present work, we perform reflectivity measurements in order to check our predictions, and then we adjust the theoretical model by taking into account all possible experimental constraints. We take into accounts the real geometry of the structures that can be achieved and fabricated in our lab. The analysis allows us to achieve a better match between the theoretical model and the experimental results. It is also shown how sensitive the result could be to a number of experimental factors.

In the next sections, we first present a brief review of coherent thermal sources and their theoretical behavior with temperature. Second, we describe our fabrication procedure and measurements setup. Then we perform a comparison between our measured data and the theoretical model. Finally, we show how imperfections in the grating geometry could influence its emissivity and how it could explain the small discrepancies that appear between the computed and the measured results.

2 Behavior of SiC grating emissivity with temperature

2.1 Coherent thermal sources

Thermal sources are usually typically incoherent [1]. In our study, we consider SiC, which is a polar material that supports surface waves, called surface phonon-polaritons (SPhP). The SPhP are evanescent waves that propagate along the material surface and exist only in the near-field. These waves fade out exponentially far from the material surface and as such do not contribute to the thermal emissivity conventionally and generally measured in the far-field dominated by the propagative waves [49].

By milling a grating on a surface of SiC material, the SPhP undergo a diffraction process which leads to coupling them to the far-field propagative waves. This constitutes the main mechanism that is responsible of creating a coherent thermal source in this configuration[48]. Coherent thermal sources occur usually only for TM polarization in the case of 1D gratings and for both polarizations in the case of 2D gratings. In the rest of the study, we often consider only TM polarization for the 1D grating. Figure 1 shows the geometry of our 1D grating with all its parameters.



Figure 1: Scheme of the grating geometry with its parameters: period d, filling factor F and depth h (d and F are in both axis for a 2D grating).

Theoretical modeling uses the Rigorous Coupled Wave Analysis (RCWA) method to calculate the emissivity of the designed gratings as a function of wavelength and incidence angle. Calculations were performed with the RETICOLO code developed by J.P Hugonin and P. Lalanne from "Institut d'optique". Further details and references about RCWA and RETICOLO can be found in [48].

2.2 Influence of temperature

We reported in figure 4 of our previous article [48], the measured temperature behaviors of both the real and imaginary parts of the dielectric permittivity of SiC for different temperatures. The resonance peak of the SiC dielectric permittivity (for both real and imaginary parts) shifts towards higher wavelengths (smaller wavenumbers) and its amplitude decreases as the temperature increases.

This behavior has a direct influence on the grating emissivity, which also shifts towards higher wavelengths and decreases in amplitude when the temperature increases. We showed this feature on Figure 5 of our previous article [48] that illustrates the behavior of the emissivity as a function of wavelength for different temperatures in TM polarization for a SiC 1D grating of parameters d=6.25 μ m, F=0.5 and h=0.285 μ m.

We previously showed that it was possible to obtain an emissivity peak with a maximum close to 1 for each temperature by adapting the grating parameters d, F and h [48]. Therefore, it is possible to adapt the grating parameters to the temperature at which the emissivity measurements are conducted. We also showed that it was possible to find a grating's set of parameters that allowed an optimum emissivity peak, not only for a single temperature, but also for all temperatures [48].

3 Fabrication procedure and measurements setup

3.1 Focused Ion Beam technique

For the fabrication of the samples, we used the Focused Ion Beam (FIB) technique. This technique exploits the focusing of an ion beam on a sample surface such that surface atoms are sputtered away under the collision force of the high energy ions. The FIB instrument, also known as dual-beam microscope, generally consists of two beam sources: an electron beam and an ion beam, which are directed and focused through separate columns. The electron beam is used for imaging via scanning electron microscopy (SEM), and the ion beam is used for milling. For the preparation of our 1D and 2D gratings, we used the Helios NanoLab G3 CX FIB instrument from Thermofisher equipped with a Ga+ ion source. With the help of the "nanobuilder" software, we were able to customize an automated structure milling with a chosen periodicity, depth and size. Due to the large surface of the gratings ($500\mu m \times 500\mu m$ for the 1D structures and 400 $\mu m \times 400 \mu m$ for the 2D structures), a preliminary study was performed to find the best compromise between milling time and accuracy of the grating geometry. The resulting operating parameters, used for both 1D and 2D structures are: an ion beam accelerating voltage of 30kV, a beam current of 2.5nA and a dose of 0.15 $\mu m^3/nC$. In such conditions, the milling of a full size grating is about

18 hours. The ion beam scanning direction was chosen to be parallel to the length of the milled channels in order to optimize the quality of the edges.

3.2 Gratings design

In order to compare the theoretical response with measurements, we have fabricated two gratings: a 1D grating and a 2D grating. We milled them on the same type of SiC material that was used for measurements of the optical properties. We first numerically designed some gratings that could easily be fabricated and which presented some interesting qualities. For the 1D grating, we designed a grating that has an optimal emissivity for all temperatures with parameters d=6.25 μ m, F=0.41 and h=0.65 μ m. Figure 2 (a) illustrates its numerically predicted emissivity as a function of wavelength for different temperatures. For clarity of the figures, we choose only four temperatures (which correspond to the temperatures for which we have optical properties data) for the numerical simulation to show the evolution of the emissivity peak as a function of temperature. The maximum of the emissivity peak is above 0.85 for each temperature and reaches the blackbody limit for T=686K. For the 2D grating, we designed a grating that is optimal for T=295K, but with a fairly high emissivity for the other temperatures as well. For example, for T=1460K, we have an emissivity peak maximum of 0.8 (Figure 2(b)). The parameters of the 2D gratings are the following $d_x=d_y=6 \ \mu$ m, $F_x=F_y=0.9$ and h=0.6 μ m.



Figure 2: Evolution of the emissivity as a function of wavelength for different temperatures of the optimized 1D SiC grating (a) (d=6.25 μ m, F=0.41 and h=0.65 μ m) and 2D SiC grating (b)($d_x=d_y=6 \mu$ m, $F_x=F_y=0.9$ and h=0.6 μ m).

The resulting gratings, obtained experimentally with the FIB, are presented in Figure 3. In order to view the profile contour of the structures, and in order to measure their depth, we created a small cross-section cut at the bottom of the structures by using the "cleaning cross-section" (CCS) ion milling method. This method is ideal for creating clean (without redeposition of material) cut inside the sample. With the help of the "tilt correction" option that transforms the y-axis into axis of depth, we were able to measure accurately the depth of the milled structures.

3.3 Reflectivity measurements

Reflectivity measurements have been performed with a Hyperion 3000 microscope equipped with 15_x Cassegrain objective and coupled to a Vertex 80_v infrared spectrometer working under vacuum. The optical configuration selected for the signal acquisition included a globar source, a DLaTGS/KBr beam-splitter and a MCT detector cooled with liquid nitrogen. The spectra were acquired between 2 and 20μ m with an instrumental resolution of 4 cm^{-1} using a gold mirror as a reference. Polarized measurements



Figure 3: Images of the milled 1D SiC grating (a) and 2D SiC grating(b).

were also performed by introducing a mid-infrared polarizer in the optical path. A Linkam Sientific THMS600 heating and freezing microscope stage was used to heat and control the temperature of the samples. In the spectral range and for the sample thickness investigated in the paper, the samples are opaque. Neglecting light scattering, the emissivity of the samples is simply the complement to one of the reflectivity, in virtue of the Kirchhoff's law.

4 Comparison between measurements and theory

We first performed reflectivity measurements at room temperature for both gratings and both polarizations. Then, we recorded the reflectivity spectra at different temperatures in TM polarization up to 610K for the 1D grating, and with no polarization and up to 400K for the 2D grating.

As the analyzed SiC samples are sufficiently thick to be opaque, in the long wavelength range there is no transmission, so it is possible to plot the emissivity directly from the reflectivity measurements to facilitate the comparison. Due to the large numerical aperture of the microscope objective (NA=0.4), the measurement setup does not allow to make pure directional measurements; the signal is collected for angles varying between 0 and 24 degrees. Nevertheless, it is worth mentioning that the designed two gratings are isotropic in this angle range, i.e. they have an emissivity peak at the same wavelength for all angles.

4.1 Room temperature

Figure 4 reports a comparison at room temperature between the computed emissivity peak in red and the measured one in blue for the 1D grating in TM polarization. The theoretical peak lies within approximately 0.1μ m of the experimental one. In addition, the measured peak appears to be larger than expected. There, it is necessarily to precise that the way we measured the reflectivity peak of our gratings is very precise for the wavelength of our peaks but less precise for the amplitude of the peak. Furthermore, the difference between the experimental results and the numerical results could also be explained by the fact that the numerical results were obtained with a 1D grating considered as ideal. The consideration of the different non-idealities or imperfections of the structures allow us indeed to explain the widening of the experimentally observed peak.

On the other hand, for the 2D grating, emissivity peaks appear for both polarizations. We plot in Figure 5 the computed and measured emissivities of the 2D grating as functions of the wavelength in TE (red), TM (blue) and both (black) polarizations. We can make the same observations as for the 1D grating: the measured and computed peaks are located at almost the same wavelength but with a shift



Figure 4: Computed (red) and measured (blue) room temperature emissivities of the 1D SiC grating in TM polarization as function of wavelength.

of 0.1 μ m and the measured peaks appear to be larger. We can also observe that theoretically, the peaks in TE and TM polarizations are at the same wavelength, hence they superimpose with the computed peak in the case of no polarization. However, the measured ones are not located exactly at the same wavelength. We will see later that this could be explained by an asymmetry in the grating geometry. Besides, the amplitude of the computed peaks is almost twice that of the measured ones. Here also and as we shall see later, the different imperfections of the fabricated gratings could explain the differences between the first numerical and experimental results.



Figure 5: Computed and measured room temperature emissivities of the 2D SiC grating as functions of wavelength in TM (blue), TE (red) and both (black) polarizations.

4.2 Different temperatures

We heated the two gratings to observe their behaviors with temperature. We report on Figures 6(a) and 6(b) the behavior of the measured emissivity as a function of wavelength for different temperatures, of the 1D grating in TM polarization and of the 2D grating with no polarization, respectively. As predicted

for both designs, when the temperature of the grating increases, the emissivity peak shifts to higher wavelengths. This is clearly visible in figure 6(a) for the 1D grating. For the 2D grating, the shift is less pronounced, but this is mainly due to a smaller interval of temperature measurement (figure 6(b)).



Figure 6: Measured behaviors of the emissivity as a function of wavelength at different temperatures for the designed and tested 1D (a) and 2D (b) SiC gratings.

To confirm this, a comparison of the temperature dependences of the measured emissivity peak position and computed one are displayed in Figures 7(a) and 7(b) for the 1D and 2D gratings, respectively. For the two gratings, measured and computed evolutions exhibit similar increasing linear trends versus temperatures. Theoretical slopes and experimental ones are close but the curves do not overlap, a gap of about 0.1μ m is also observed for instance for the 1D grating as we pointed out earlier. Such deviations cannot only be explained by experimental uncertainties, we need thus to question the validity of the geometrical shape and of the material nature used in the theoretical evaluation of the optical response of the gratings.



Figure 7: Comparison of the computed (red) and measured (blue) positions of the maximum of the grating emissivity peak as a function of temperature for the designed and tested 1D (a) and 2D (b) SiC gratings.

5 Adjustments to the real nature and geometry of the milled gratings

In this section, we explore all different possibilities that could and would explain the discrepancy between the measurements results and the theoretical predictions. In particular, we study the influence of the real geometry of our milled gratings, the effect of heating, the presence of an azimuthal angle as well as the impact of a possible amorphous layer.

5.1 Ideal versus real geometry

After we milled the gratings with the FIB technique, we acquired SEM images and performed measurements of their geometrical parameters. We show on Figure 8(a) the profile of a cross-section of the 1D grating. It is obvious in this image that the edges of the grating are not exactly vertical as expected. They are more trapezoidal than rectangular in shape. This is mainly due to the conical shape of the ion beam of the FIB. This type of tapered edges can also occur with traditional way of grating fabrication. On Figure 8(b), we show a schematic representation of the trapezoidal shape of our 1D grating, with averaged values for the top and bottom widths of the grating. The taper is somehow small, but there still is a difference of 135 nm between the top and the bottom of the grating.



Figure 8: Profile image of the 1D SiC grating (a) and its schematic representation (b).

We adapted our grating model to include these new geometry and parameters. We plot on Figure 9 the new behavior of the emissivity as a function of wavelength at 295K for a TM polarization. A comparison between the measured emissivity in blue and the computed one with the ideal rectangular shape in red and with the non-ideal trapezoidal shape in black is also reported.

The peak of the black curve is shifted by about 0.1μ m by comparison to the red one and is now centered on that of the measured response. It is obvious that a change in the geometry affects directly the wavelength position of the emissivity peak. A significant contribution to the observed shift between theory and measurement is now understood, but this still does not explain why the measured peak has a larger shape.

5.2 Effect of heating

The thermal expansion coefficient of SiC is about 4.10^{-6} K⁻¹ [50]. Hence, within the temperature interval used in our experiments, this effect should not influence significantly the SiC geometry and the position of the predicted emissivity peak computed at the different temperatures above the room one.



Figure 9: Comparison of the measured (blue) and computed emissivity behaviors of the 1D SiC grating in the TM polarization as functions of wavelength with an ideal rectangular profile(red) and non-ideal trapezoidal profile (black).

In order to detect defaults or inclusions that could melt at high temperatures, we acquired images of the grating before and after heating. No hange in the grating was detected between the two stages. Thus, we could assume that the influence of the temperature on the grating geometry is negligible.

5.3 Asymmetry and azimuthal angle

The asymmetry aspect and the azimuthal angle are two characteristic features of the 2D grating. By measuring our 2D grating, we observed that the studs don't have the same size on both axes. It is about 5.342μ m on the x-axis and 5.531μ m on the y-axis, which makes a difference of 189 nm between the two axes. Thus, we have a period of 6μ m for both axes, but a filling factor $F_x=0.883$ in the x-axis and $F_y=0.915$ in the y-axis. This small variation could explain the difference we have between the TE and TM polarizations in the measurement of the 2D grating emissivity as shown in Figure 5. We introduced this asymmetry aspect in the computations and the results are reported on Figure 10. On Figure 5, the TE and TM emissivity peaks with no asymmetry considered, were theoretically superimposed. Nevertheless, in Figure 10, we clearly see that the TE and TM peaks depart from each other and are not located at the same wavelength. This difference is the same feature that is manifested by both theory and measurements in the TE and TM emissivity peaks. It is important to note that there still is a shift between experiment and theory because we did not include in these computations the trapezoidal shaped geometry in order to study the impact of each phenomenon independently.

In the next step, computations for the same asymmetry on the 2D grating were performed, but with gradual hange of the azimuthal angle. Figure 11 reports the results of the behavior of the emissivity as a function of wavelength at T=295K for azimuthal angles of 0, 15, 30 and 90 degrees with TE polarization in plain line and TM polarization in dotted line. Up to 30 degrees, the azimuthal angle seems to have a small effect on the general trend. On the other hand, once the azimuthal angle gets higher, its influence grows gradually until this angle reaches 90 degrees where we have an exchange of the two peaks in TM and TE polarizations.

5.4 Impact of the amorphous layer

Milling gratings by FIB technique can also have some structural consequences; the major one is the gradual evolution of the grating's top layer from a crystalline state to an amorphous state. This effect could potentially explain the increased width of the measured emissivity peaks as compared to the



Figure 10: Computed and measured emissivities of the 2D SiC asymmetrical grating as functions of wavelength in TM (blue), TE (red) and both (black) polarizations.



Figure 11: Computed emissivity of the 2D SiC grating as a function of wavelength at T=295K for different azimuthal angles in TM and TE polarizations.

theoritical prediction based on a full crystalline structure. Indeed, this amorphous layer could somehow alter the expected signal.



Figure 12: Schematic representation of the SiC grating coated with an amorphous layer (yellow).

In order to verify whether we have actually an amorphous layer on top of our SiC gratings, we extracted a lamella from our 1D grating sample. To avoid further damage or pollution of our grating, we deposited a platinum layer on the top before the extraction. However, due to the thinness of the amorphous layer, which should be a few nanometers thick and its inhomogeneous nature on the entire surface, it was not clearly visible on the extracted lamella. Furthermore, the thickness of this amorphous layer tends to vary gradually which makes it very difficult to model exactly. We tried to model this layer in our computations as shown in the schematic representation of Figure 12. The crystalline SiC grating is represented in dark blue and the amorphous layer is shown in yellow. We did not have the exact optical properties of the amorphous SiC, thus we considered generic amorphous SiC data in [51], as the main aim is to check whether the amorphous layer influences the width of the emissivity peak shape and not specifically to reach an exact match.



Figure 13: Comparison of the 1D SiC grating measured emissivity as a function of wavelength in TM polarization at T=295K to the theoretical one with a 10nm thick (dotted red line) and without (plain red line) amorphous layer.

We plot on figure 13 the behavior of the emissivity as a function of the wavelength for the 1D SiC grating at T=295K in TM polarization; the measured curve (blue), the computed one without the amorphous layer (plain red line) and with a 10nm thick amorphous layer (dotted red line). We also considered the taper in the grating edges for this computation. By comparing the dotted and plain red curves, we can clearly see that the presence of an amorphous layer has a real and crucial influence on the width of the emissivity peak. Indeed, the computed emissivity peak becomes larger and tends to reproduce fairly well the measured one.

5.5 Sensitivity

Our computations have shown that the emissivity of the 1D SiC grating tends to be very sensitive to the taper size and the presence of the amorphous layer.

The size X of the taper, as represented on Figure 14(a), influences significantly the position of the grating emissivity peak. We plot on Figure 14(b) the behavior of the emissivity as a function of the wavelength for the 1D grating at T=295K, in TM polarization. We vary the taper size X from 0 to 500nm. We can see that even 10 nm of the taper induces a small shift in the emissivity peak position. When X increases, the emissivity peak shifts further towards smaller wavelengths. For X=500nm, the shift reaches up to 250nm compared to the one without the taper (X=0).



Figure 14: Schematic representation of the profile of the 1D SiC grating with the size X of the taper (a) and the computed behavior of its emissivity as a function of wavelength for different values of X (b).

As shown earlier, the amorphous SiC layer on top of the grating is mainly responsible of the enlargement or widening of the measured emissivity peak. The size of this amorphous layer can also influence the position of the emissivity peak and decrease its amplitude. We plot on Figure 15 the behavior of the emissivity as a function of the wavelength for the TM polarization of 1D grating with a taper at T=295K, for a 10nm- (blue) and a 50nm- (red) thick amorphous layer. For both sizes, we can see a widening of the emissivity peak, confirming that the existence of an amorphous layer can constitute a factor responsible of the observed larger shape. We also see that the amplitude of the emissivity peak decreases when the amorphous layer gets thicker, as the thicker layer tends to attenuate the resonance from the crystalline SiC grating. Another possibility that could explain a widening and such kind of distortion of the emissivity spectra may be the presence of non-stoichiometry in a thin layer at the sample surface [52].



Figure 15: Computed behavior of the 1D SiC grating TM polarization emissivity as a function of wavelength at T=295K and for two different thicknesses of the top amorphous SiC layer.

6 Conclusion

We performed reflectivity measurements at different temperatures on 1D and 2D gratings milled on the same type of SiC, on which optical properties measurements at different temperatures were conducted. We found back and therefore confirmed, the theoretically predicted shift of the emissivity peak towards higher wavelengths, when the temperature increases. We also showed that :

- a non-ideal grating geometry can influence the position of its emissivity peak.

- an asymmetry of the grating can lead to different positions of the emissivity peak for TM and TE polarizations.

- an azimuthal angle has little influence on the emissivity behavior of the grating, when the angle is small.

- the method chosen to fabricate the grating and the probable presence of a top amorphous or non-stoichiometric layer could impact significantly both the position and width of the emissivity peak.

These results shall therefore serve as guidelines for the design of optimized coherent thermal sources at different temperatures by arefully considering the impacts of the different geometrical and structural parameters.

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