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# Infrared transmittance model for pyrometric monitoring of surface quality of thin diamond films

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## 1. Introduction

## ABSTRACT

The thickness and surface roughness of thin diamond films grown on quartz substrates are studied in the paper using a model of changes in the infrared transmission ratio associated with multiple constructive interference within a thin crystal film. On the other hand, the model is referred to periodic variations of the apparent temperature of the substrate measured by the two-color pyrometer. Obtained results are then compared with those of other similar studies, and the AFM measurements. Some discrepancy between the results from infrared and AFM data is explained in terms of optical absorption of the crystal, which is neglected in the transmittance model.

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A vast majority of diamond applications, such as surface acoustic wave (SAW) devices [1], and protective as well as optical coatings [2,3] use highly adherent thin films deposited onto non-diamond substrates. Among others, several oxides (e.g.:  $Al_2O_3$  (sapphire), SiO<sub>2</sub> (quartz, fused silica)) have attracted substantial attention particularly because no carbide interfacial layer is formed during the diamond growth, which preserves high transparency and low electrical conductivity of obtained structures.

Deposition of diamond onto quartz substrates suffers mainly from low nucleation density, low growth ratio, and large interfacial stress due to the mismatch of thermal expansion coefficients of diamond and substrate material [4]. To apply diamond films in optical devices, such parameters as film thickness, surface roughness, and growth rate need to remain within acceptable limits. From that point of view, the infrared pyrometry appears to be a great tool of monitoring phenomena that occur during the deposition process period (in-situ). The presented paper is aimed at extracting data on the film thickness, growth rate, and surface roughness of several diamond samples using a simple semi-empirical approach to the problem of fitting of complex model of the infrared transmittance into measured sample temperature. The model relies on the fact that in the presence of a thin film the two-color pyrometer produces an output of apparent temperature, which might be related both to the real temperature of the film and to its structural properties (thickness, and roughness in principle). Obtained results are then compared with results of similar studies carried out by others, and verified using the AFM data.

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## 2. Material and methods

Diamond samples were grown in a 3 kW ASTeX microwave plasma CVD system (AX 6560) described elsewhere [5]. A standard gas mixture containing methane diluted with hydrogen was used. Quartz substrates (1 mm thick) were ultrasonically seeded with diamond powder (diameter of seeds was 250 nm) on a vibrating plate. The deposition parameters were as follows: CH<sub>4</sub> concentration 1–5% (vol.), substrate temperature in the range from 500 to 920 °C, gas pressure 7.33 kPa (55 Torr), microwave power 3000 W, and deposition time varied from 5 min. up to 13 h. Surface morphology of the films was studied using the AFM method (Veeco Multimode 8). Growth rate and roughness of diamond films were calculated from the oscillations of the apparent temperature, which were measured in-situ by the two-color pyrometer (Williamson Pro 92-40) located at an angle of 40° from the substrate normal.

## 3. Theory/calculation

## 3.1. The thermal radiation

The intensity of black-body radiation into vacuum expressed in terms of the wavelength and temperature is described by the Planck formula [6]:

$$I_{BB}(\lambda,T) = 2hc^2\lambda^{-5}[\exp(hc/k_B\lambda T) - 1]^{-1}$$
(1)

where: h is the Planck constant, c is the velocity of light,  $k_B$  is the Boltzmann constant,  $\lambda$  is the wavelength, and T is the black-body temperature. However, if the wavelength is in the range of 2–3 µm,

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and the temperature does not exceed 1200 K, then the term hc/  $k_B\lambda T\!>\!1,$  and Eq. (1) can be reduced to the expression:

$$I_{BB}(\lambda,T) = \frac{2hc^2}{\lambda^5} \exp\left(-\frac{hc}{k_B\lambda T}\right)$$
(2)

which is known as the Wien radiation law.

In order to obtain the energy radiated by a particular material with respect to that of a black body, the emissivity  $\varepsilon$  needs to be taken into account:

$$I(\lambda,T) = \varepsilon(\lambda,T) \cdot I_{BB}(\lambda,T) = \varepsilon(\lambda,T) \cdot \frac{2hc^2}{\lambda^5} \exp\left(-\frac{hc}{k_B\lambda T}\right). \tag{3}$$

In general, the emissivity can depend on the wavelength and the temperature in a quite complex manner, which is specific to a given material.

## 3.2. The pyrometric ratio

The two-color pyrometer has two identical, narrow-banded optical channels centered at wavelengths  $\lambda_1$ , and  $\lambda_2$ , and therefore, it is actually the intensity ratio (hereafter called the pyrometric ratio (PR)) which is measured by the two-color pyrometer:

$$PR(\lambda_1,\lambda_2,T) = \frac{I(\lambda_1,T)}{I(\lambda_2,T)} = \frac{\varepsilon(\lambda_1,T)}{\varepsilon(\lambda_2,T)} \left(\frac{\lambda_2}{\lambda_1}\right)^5 \exp\left[-\frac{hc}{k_BT}\left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2}\right)\right].$$
(4)

Given the wavelengths:  $\lambda_1 = 2180.5$  nm, and  $\lambda_2 = 2350.5$  nm (Williamson Pro 92-40), the pyrometric ratio can be written, as:

$$PR(\lambda_1, \lambda_2, T) = 1.455 \cdot \frac{\varepsilon(\lambda_1, T)}{\varepsilon(\lambda_2, T)} \cdot \exp\left[-\frac{T_0}{T}\right]$$
(5)

where  $T_0 = hc/k_B(1/\lambda_1 - 1/\lambda_2) = 478$  K is the reference temperature.

The pyrometric ratio can be further simplified within the frame of a gray body approximation. In such a case, even if the target emissivity varies with the temperature, it is assumed to be independent of the wavelength, which leads to the following equation:

$$PR(T) = 1.455 \cdot \exp\left[-\frac{T_0}{T}\right].$$
(6)

Eq. (6) states that the pyrometric ratio depends only on the target temperature.

## 3.3. Apparent temperature vs. real temperature

The above considerations apply for systems, in which the radiation passes directly from the target to the detector. Let us assume, however, that the path of the infrared beam crosses another body, which transmits the incident light, but hardly emits its own thermal radiation. In such a case, the transmittance Tr needs to be taken into consideration, which defines the fraction of incident light passing the sample:

$$Tr = \frac{I(\lambda, T)}{I_0(\lambda, T)}$$
(7)

where  $I_0$  is the intensity of incident light, and I is the intensity of passed light. The measured pyrometric ratio can now be expressed in the form:

$$\frac{I(\lambda_1,T)}{I(\lambda_2,T)} = \frac{\mathrm{Tr}_1 \cdot \mathrm{I}_0(\lambda_1,T)}{\mathrm{Tr}_2 \cdot \mathrm{I}_0(\lambda_2,T)}$$
(8)

where  $I(\lambda,T)$  is the intensity of light measured by the pyrometer,  $I_0(\lambda,T)$  is the intensity of light emitted by the target, and  $Tr_1$ , and  $Tr_2$  are the transmittances of the intermediate body at the wavelengths  $\lambda_1$ , and  $\lambda_2$ , respectively. Eq. (8) can be then written as:

$$PR_{meas} = \frac{Tr_1}{Tr_2} \cdot PR_{real}$$
(9)

where  $PR_{meas}$  is the pyrometric ratio measured with the intermediate body, while  $PR_{real}$  is the pyrometric ratio emitted by the target (i.e. measured without the intermediate body). Substituting Eq. (6) into (9), the transmittance ratio can be expressed as:

$$\frac{\mathrm{Tr}_{1}}{\mathrm{Tr}_{2}} = \exp\left[\frac{\mathrm{T}_{0}}{\mathrm{T}_{\mathrm{real}}} - \frac{\mathrm{T}_{0}}{\mathrm{T}_{\mathrm{meas}}}\right] \tag{10}$$

in which the measured (apparent) temperature is distinguished from the real (target) one.

Using a model of the infrared transmittance presented in Section 3.4, Eq. (10) can now be used for fitting some properties of the intermediate body into time-dependent experimental data, i.e. the real, and the apparent target temperature.

## 3.4. Optical transmittance of a thin film

Several studies were performed to extract information on the structure of deposited films from pyrometric measurements, although they suffered from such problems as: (1) lack of data on the real emissivity of the sample (single-color pyrometry) [7–9], (2) questionable assumption that the apparent temperature must be equal to the real temperature for rough surfaces [9,10], (3) questionable assumption that the deposited film is optically flat (no scattering of transmitted light) [11,12], and (4) complicated fit procedures [10–12].

In order to establish the model of the infrared transmittance of diamond onto the quartz substrate, let us assume that: (1) the intermediate body is a thin, transparent diamond film with refraction index  $n_1 = 2.40$  placed on a thick quartz substrate with  $n_2 = 1.48$ , and surrounded by the vacuum with  $n_0 = 1$ , (2) the diamond-quartz interface is perfectly flat, whereas the roughness of the diamondvacuum interface  $\sigma$  is considerable, but small compared to the infrared wavelengths  $\lambda_1$ , and  $\lambda_2$ , (3) both crystals are homogeneous, and (4) the absorption coefficient of diamond is negligible. Since the light emitted by the target is not polarized, and the target is observed at an angle  $\alpha = 40^{\circ}$  to the normal, one must begin with the Fresnel coefficients  $\boldsymbol{t}_{ij}$  describing amplitudes of the waves transmitted through the interfaces. However, due to possible multiple reflections within the diamond film, the Fresnel coefficients r<sub>ii</sub> for waves reflected at the diamond-quartz, and diamond-vacuum interfaces also need to be taken into account [13]:

$$t_{10p} = \frac{2\sin\alpha\cos\beta}{\sin(\alpha+\beta)\cos(\beta-\alpha)}, \ t_{10s} = \frac{2\sin\alpha\cos\beta}{\sin(\alpha+\beta)}$$
(11)

$$r_{10p} = \frac{tg(\beta - \alpha)}{tg(\beta + \alpha)}, \ r_{10s} = -\frac{\sin(\beta - \alpha)}{\sin(\beta + \alpha)}$$
(12)

$$r_{12p} = \frac{tg(\beta - \gamma)}{tg(\beta + \gamma)}, \ r_{12s} = -\frac{\sin(\beta - \gamma)}{\sin(\beta + \gamma)}$$
(13)

where  $\alpha = 40^{\circ}$  is the viewing angle of the pyrometer,  $\beta = 15.5^{\circ}$  is the incidence angle within the diamond film,  $\gamma = 26.3^{\circ}$  is the incidence angle in quartz, whereas s, and p denote the waves polarized, respectively, perpendicular, and parallel to the incidence plane. Both incidence angles,  $\beta$  and  $\gamma$ , correspond to the viewing angle using the Snell law.

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Diamond film of thickness d gives rise to a phase shift of the passing wave:

$$\varphi(\lambda, \mathbf{d}) = \frac{4\pi n_1 \mathbf{d} \cos(\beta)}{\lambda}.$$
(14)

Finally, when the wave approaches the diamond-vacuum interface, its amplitude is diminished not only by the reflection, but also due to the scattering. According to the Beckmann-Kirchhoff theory of scattering, the amplitude coefficients of light: transmitted  $S_t$ , and reflected  $S_r$ , at the surface of the roughness  $\sigma$  are given by [14]:

$$S_{t}(\lambda,\sigma) = \exp\left\{-\frac{1}{2}\left(\frac{2\pi(n_{1}-n_{0})\cos(\alpha)\sigma}{\lambda}\right)^{2}\right\}$$
(15)

$$S_{\rm r}(\lambda,\sigma) = \exp\left\{-\frac{1}{2}\left(\frac{4\pi n_1 \cos(\beta)\sigma}{\lambda}\right)^2\right\}.$$
 (16)

The amplitude (complex) transmission coefficients  $tr(\lambda)$  for multiple reflected, and scattered waves passing the diamond thin film take the form:

$$\operatorname{tr}_{s}(\lambda,\sigma,d) = \left(\frac{t_{10s}S_{t}}{1 - r_{10s}r_{12s}S_{r}\exp(-i\varphi)}\right) \tag{17}$$

$$tr_{p}(\lambda,\sigma,d) = \left(\frac{t_{10p}S_{t}}{1 - r_{10p}r_{12p}S_{r}\exp(-i\varphi)}\right).$$
(18)

The transmittance of the system for a given wavelength can now be written as:

$$Tr(\lambda,\sigma,d) = \frac{1}{2} \left( \left| tr_s(\lambda,\sigma,d) \right|^2 + \left| tr_p(\lambda,\sigma,d) \right|^2 \right)$$
(19)

$$Tr(\lambda,\sigma,d) = \frac{1}{2} \begin{cases} \frac{(t_{10s}S_t)^2}{1 - 2r_{10s}r_{12s}S_r\cos(\varphi) + (r_{10s}r_{12s}S_r)^2} \end{cases}$$
(20)

$$+\frac{\left(t_{10p}S_{t}\right)^{2}}{1\!-\!2r_{10p}r_{12p}S_{r}\cos(\varphi)+\left(r_{10p}r_{12p}S_{r}\right)^{2}}\bigg\}.$$

Finally, the transmittance ratio is given by:

$$\frac{\mathrm{Tr}_{1}}{\mathrm{Tr}_{2}} = \frac{\mathrm{Tr}(\lambda_{1}, \sigma, \mathbf{d})}{\mathrm{Tr}(\lambda_{2}, \sigma, \mathbf{d})}.$$
(21)

Eq. (21) establishes the formula for the transmittance ratio of the thin film onto thick substrate as a function of the wavelength, film thickness, and the surface roughness.

## 4. Results and discussion

## 4.1. Changes in apparent temperature

Fig. 1 brings an evidence to prove the deposited diamond quality as it shows typical Raman spectrum of samples under study. Prominent peak at around 1332 cm<sup>-1</sup> is the main feature of tetrahedrally bonded carbon atoms that form the diamond structure. According to the formula proposed by Vorlicek et al. [15], the volume fraction of non-diamond phase is less than 2%, and that fact confirms the diamond quality. Additionally, the AFM image of the sample with the surface roughness of 90 nm is shown in the inset in Fig. 1.

Fig. 2A shows an example of the transmittance ratio calculated using a set of time-dependent apparent temperatures measured by the pyrometer, and substituted into Eq. (10). It follows from this figure that the transmittance ratio oscillates due to interference



**Fig. 1.** Raman spectrum of the diamond sample deposited on quartz substrate at 920 °C with the predominant peak at 1332 cm<sup>-1</sup> assigned to sp<sup>3</sup>-hybridized carbon clusters (i.e. diamond structure). (Inset) AFM image of the sample surface exhibiting the topological roughness of 90 nm.

phenomena within the growing film. Using Eq. (21) it is possible to compute a series of curves of the transmittance ratio as a function of the film thickness, and the surface roughness. Fig. 2B shows a plot of the transmittance ratio calculated using Eq. (21) for the film thickness increasing from 0 to 7000 nm, with the surface roughness fixed at 0, and 100 nm, respectively. The fringes seen in Fig. 2B can serve as reference points for calculation of actual film thickness. Note that these points occur at fixed locations independent of the surface roughness. If so, the interference maxima and minima can be referred to the measured apparent temperature oscillations, which in the following yields information on the changes of the film thickness, and the growth rate.

On the other hand, Fig. 2C shows a set of curves of the transmittance ratio calculated using Eq. (21) for the roughness varying from 0 to 300 nm, with the thickness fixed at 141, 385, 580, and 848 nm, that is at four consecutive interference minima and maxima seen in Fig. 2B. Note that each plot grows asymptotically, and the oscillations are damped with increasing surface roughness. Previous models incorrectly explained such an effect in terms of increasing real temperature of the growing surface, and hence they overestimated the growth rate [9,10]. In fact, the target temperature remains constant as the other growth conditions are kept constant.

## 4.2. Fitting procedure

To establish the growth rate and the roughness of the deposited films, the following procedure is carried out in the current work. The first step involves calculation of the time-dependent transmittance ratio using apparent temperature data measured during the deposition process, which are substituted into Eq. (10). Then, obtained oscillations are linked to those in the transmittance ratio calculated according to Eq. (21). Finally, the surface roughness of the film is determined at extreme points of the interference patterns by comparing the values of the time-dependent transmittance ratio with that provided by Eq. (21). To end up with the best possible agreement, the charts similar to those in Fig. 2C can be helpful.

## 4.3. Growth rate and surface roughness

Fig. 3A and B gives example results of the above procedure. In Fig. 3A, the growth rate of the film vs. deposition time is shown, which remains constant at  $(3.5 \pm 0.8) \mu$ m/h by the time of the deposition process at the temperature 920 °C. In contrast to the

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**Fig. 2.** (A) The transmittance ratio calculated using Eq. (10) and the measured temperature oscillations, (B) plots of the transmittance ratio vs. film thickness calculated for the surface roughness fixed at 0 nm (dotted line), and 100 nm (solid line), (C) plots of the transmittance ratio vs. surface roughness calculated for the film thickness fixed at (respectively): 141, 385, 580, and 848 nm.

films deposited on carbide-forming substrates (e.g. silicon, titanium) [9,12], there is no initial period observed, in which the growth rate of diamond is suppressed due to formation of an interfacial carbide layer. Such a result leads to the conclusion that the saturation of the quartz substrate with carbon atoms as a beginning stage of the diamond deposition process does not occur in this case, which gives rise to faster growth rate (no nucleation period), and the higher purity of the deposit (no interfacial carbide layer).



**Fig. 3.** (A) Plot of the growth rate vs. deposition time. The growth rate remains constant at about 3.5  $\mu$ m/h during the whole process, (B) plot of the surface roughness vs. deposition time. Initially, the roughness linearly increases to the value of about 150 nm, but then abruptly falls down due to relief of thermal stresses induced at the diamond/substrate interface.

On the other hand, Fig. 3B shows the plot of the surface roughness vs. deposition time with a more complex shape. A sharp step is observed within around the first 2 min of the deposition, when the



**Fig. 4.** Arrhenius-type plot of the growth rate of diamond vs. reciprocal deposition temperature. Observed straight linear fit corresponds to single exponential decay behavior with an activation energy of 330 meV.

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**Fig. 5.** Comparison of the surface roughness calculated using the infrared transmittance model and exhibited by the AFM measurements. The pyrometric method generally overestimates the roughness, which may be due to additional optical absorption seen by the pyrometer.

surface roughness increases from initial value of about 2 nm (freshly seeded substrate) up to about 80 nm when the film thickness approaches 140 nm. Such an increase might be attributed to the freegrowth regime of the crystal taking place until the isolated diamond grains join together to form a continuous film. Beyond that point, the surface roughness linearly increases over the next 90 min of the process with the rate being equal approximately to 60 nm/h. Then, observed linear dependence abruptly breaks down, with the roughness decreasing from 150 nm to 30 nm, although in the same time the growth rate remains constant at about 3.5  $\mu$ m/h. It might be due to micro-delamination of the deposit, which on one hand releases thermal stresses in the film/substrate interfacial layer, but on the other reduces crystal distortion. It follows from Fig. 3B that the surface roughness increases again after the break down, that is, when the growth process is continued.

## 4.4. Effect of the deposition temperature

Similar analysis was performed for other diamond samples deposited at different temperatures. As seen in Fig. 4, the growth rates were found to increase from  $0.35 \,\mu$ m/h at 500 °C to  $3.5 \,\mu$ m/h at 920 °C exhibiting a correlation with the deposition temperature. To make this temperature dependence visible, observed growth rates are fitted with the Arrhenius equation in the form:

$$\frac{dD}{dt} = A \exp(-E_A/kT)$$
(22)

where: D is the film thickness, A is the pre-exponential factor, and  $E_A$  is the activation energy. As seen in Fig. 4, the growth rate follows the law with an activation energy of 330 meV, which exceeds the value of 200 meV reported for diamond grown on silicon [12]. Such a result suggests, however, that the deposition of diamond onto quartz is not only limited by the desorption of hydrogen from the growing surface, but the process chemistry is more complicated [12]. In addition, the growth rates seen in Fig. 4 are larger in comparison with those for diamond films on silicon [12]. This might stem from the aforementioned fact that the deposition process on quartz goes without any carburization of the substrate. On the other hand, the growth rates observed in the current work are at least twice as

large as those reported elsewhere for quartz substrates at similar temperatures [3,16,17], which might be due to the pre-treatment procedure that made the incubation period very short.

In order to verify the results presented in the paper, the final roughness values estimated from the transmittance ratio oscillations are compared in Fig. 5 with those measured directly using the AFM method.

## 4.5. Future work

It is found that the fitting procedure tends to overestimate the surface roughness, as the AFM method is believed to exhibit the 'true' roughness corresponding purely to topological features of the surface. If so, the observed overestimation might be caused by additional absorption of light within the diamond (neglected in the model), which contributes to diminished transmittance of light. On the other hand, 'pure' roughness values from AFM measurements could make the estimation of the absorption coefficient of diamond possible. Indeed, detailed analysis would help to distinguish the specular transmission (associated with absorption phenomena) from the diffuse one (caused by the scattering of light), but this issue will be addressed in further work.

## 5. Conclusions

The model of the infrared transmittance is presented in the paper, which explains observed oscillations in the apparent temperature measured by the two-color pyrometer. The model is used to extract information on the film thickness, surface roughness, and their variations during the deposition process. Obtained growth rate and activation energy are found substantially different than those observed for carbide-forming substrates. In turn, the surface roughness is found generally larger than in the case of the AFM measurements, which possibly suggests that the absorption of infrared light also needs to be included in the transmittance model.

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