



## Feedback control of the substrate surface temperature in a laser-induced plasma CVD process

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### Abstract

A laser-induced plasma chemical vapour deposition (LPCVD) process is used for the deposition of CVD diamond coatings. For the CVD diamond deposition, the surface temperature of the substrate has to be between 900 °C and 1200 °C, but already small temperature variations influence the crystal structure and the growth rate. To ensure the reproducibility of such a deposition, a feedback control of the temperature was developed. The surface temperature, measured by a pyrometer, and the temperature of the backside of the sample, measured by thermocouples, were used for the input to the control. For the feedback control, a combination of the two temperature measurements was used to regulate the laser power.

**Keywords:** CVD diamond, feedback control, temperature measurement, pyrometer

### 1 Introduction

Ever since 1998, a laser-induced plasma chemical vapour deposition (LPCVD) process has been used for research at the BIAS institute [1]. The *in situ* measurement of the surface temperature while the plasma flame is in contact with the substrate has been a challenge the whole time. In 1998 the temperature was measured by thermocouples on the edge of the substrate. The measuring position was later changed to the backside of the substrate directly underneath the area of deposition [2]. The laser power was adapted by hand according to this measured temperature to achieve the desired temperature. For better reproducibility of the deposited CVD diamond coatings, a feedback control needs to be integrated into the process system, to automatically adapt the laser power to the required temperature.

In addition to the thermocouples, a narrow-band pyrometer will be integrated, to measure directly the surface temperature through the plasma flame. In this way, the thickness of the substrate does not have an impact on the measured temperature, in contrast to using thermocouples for the measurement.

The temperature of the molybdenum substrate at the beginning of the deposition process will be measurable by the pyrometer. After that the CVD diamond coating starts to grow on the substrate of which the heat radiation

is measured. That leads to a continuously varying emissivity, as explored by Choi et al. [3] for another coating process. Through that, a changing temperature can be expected until the substrate is covered by a closed CVD diamond coating, after which constant emissivity conditions will obtain. It is also possible that the heat radiation of the molybdenum substrate has an influence over the whole process because of the transparency of the CVD diamond, so that constant emissivity conditions cannot be reached.

### 2 Method

CVD diamond coatings were deposited by a LPCVD system. A CO<sub>2</sub> laser with a nominal power of 6 kW was used in the process. Molybdenum plates with dimensions of 25 x 35 x 1 mm<sup>3</sup> were taken as the substrates for the deposition. The deposited CVD diamond coatings covered a round surface of 10 mm<sup>2</sup>. The diamond growth rate was about 30-50 μm/h.

A detailed description of the research facility and the process parameters were published by Schwander et al. [2]. Figure 1 shows the LPCVD process. The process setup has been modified by installing a narrow-band pyrometer (IMPAC-Pyrometer IGAR 12-LO) into the wall of the ignition chamber of the plasma flame. In this way, the position of the pyrometer is fixed in relation to the plasma flame, even when the substrate is moved

relative to it. Hence the pyrometer always measures the thermal radiation at the process position (shown in Figure 1 (right)).

Thermocouples (type K) monitor the sample temperature on the backside of the substrate. They always measure the temperature at the same spot relative to the substrate (Figure 1 (right)).

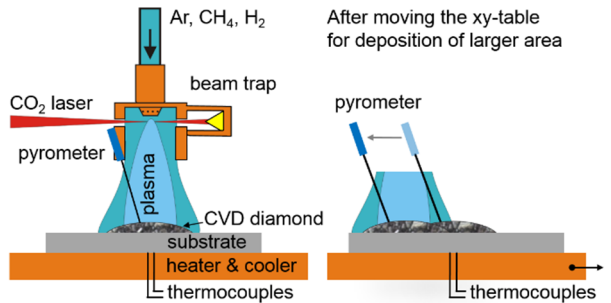


Fig. 1: Schematic construction of the LPCVD process (left). For the temperature measurement, thermocouples and a narrow-band pyrometer are used. The thermocouples move together with the xy-table, but the pyrometer moves together with the plasma flame (right).

It is expected that measuring reliably the substrate surface temperature with the pyrometer at the beginning of the deposition process is not possible. Hence, two feedback control loops shall be introduced, one referring to the pyrometer and the other to the thermocouples. The output of the controllers is the desired laser power in kW. The controllers are followed by the process control line (see Figure 3). The output value of the process control line is the substrate temperature to be influenced. To identify the process control line, its step response to an abrupt rise in laser power was measured. The CVD diamond coating is usually deposited at 880 °C as measured by the thermocouples. To avoid damaging the substrate or the CVD diamond coating, the laser power was switched from 3.19 kW to 3.72 kW, resulting in the backside temperature rising from 787.8 °C to 883.0 °C. This rise of approximately 100 K also seems reasonable, as it is larger than the expected measurement uncertainty of the thermocouples.

As the substrate temperature can be measured with two different devices, there are in fact two separate process control lines. Only the controller design for the regulation of the surface temperature is presented here. The increase of the laser power was performed after the substrate was covered by a closed CVD diamond coating. The recorded step response for the pyrometer is shown in Figure 2. The exponential fit represents the step response of the chosen model for the process control line. It is modelled as a first-order lag element (PT<sub>1</sub>), with a gain P<sub>M</sub> of 252.17 K/kW (normalized to the step height of 3.72 kW - 3.19 kW = 0.53 kW) and a time constant τ<sub>M</sub> of 0.38 min.

In consideration of the model, an appropriate controller structure was selected. A proportional-plus-integral (PI) controller with a proportional gain P<sub>C</sub> of 3.96 W/K and an integral gain I<sub>C</sub> of 5.80 min (represented in parallel form) was used. The complete feedback control loop is shown in Figure 3. The stability of the loop's ideal model was verified with Nyquist's

criterion [4] for the general case, where the values of the gain and time constants of the process control lines and the controller model are irrelevant.

The feedback control loop for the regulation of the backside temperature was carried out in the exact same manner, merely the values for the constants are different.

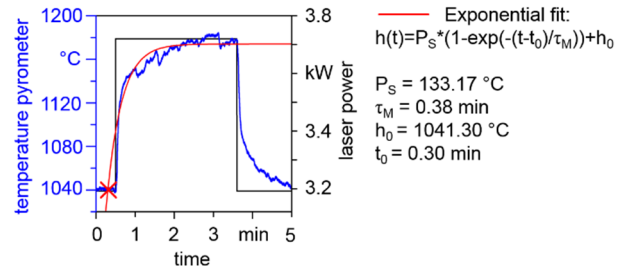


Fig. 2: Step response of the system (process control line) when the desired laser power is abruptly raised. Shown is the recorded surface temperature of the substrate, measured with a narrow-band pyrometer, and a corresponding exponential fit. The origin of the fit is marked by the red cross.

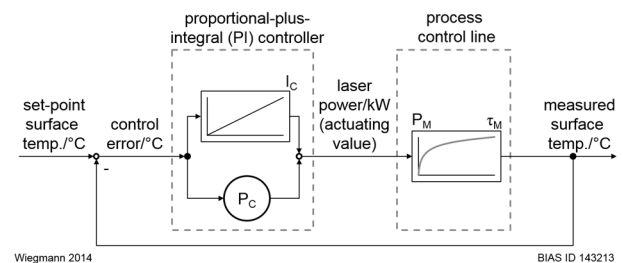


Fig. 3: Feedback control loop for the regulation of the substrate surface temperature. The laser power is given by a proportional-plus-integral (PI) controller with a proportional gain P<sub>C</sub> of 3.96 W/K and an integral gain I<sub>C</sub> of 5.80 min. The system (process control line) is modelled as a first-order lag element (PT<sub>1</sub>) with a gain P<sub>M</sub> of 252.17 K/kW and a time constant τ<sub>M</sub> of 0.38 min.

### 3 Results

As was shown by Wild et al. [5], the surface temperature of the substrate has an enormous influence on the deposition of a CVD diamond coating. By means of the employed LPCVD process, three CVD diamond coatings with different surface temperatures were deposited for 40 minutes. In the middle of the deposited diamond coatings a measurement with a laser microscope (Keyence VK 9710) was done, which is shown in Figure 4. The temperature was measured by thermocouples underneath the middle of the substrate. The diamond structure is visible as well as the influence of the surface temperature during the deposition.

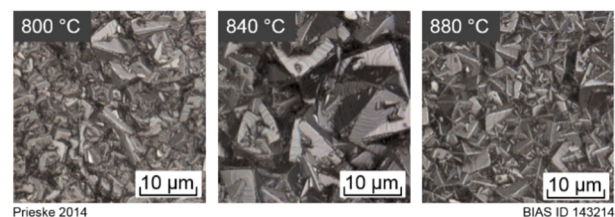
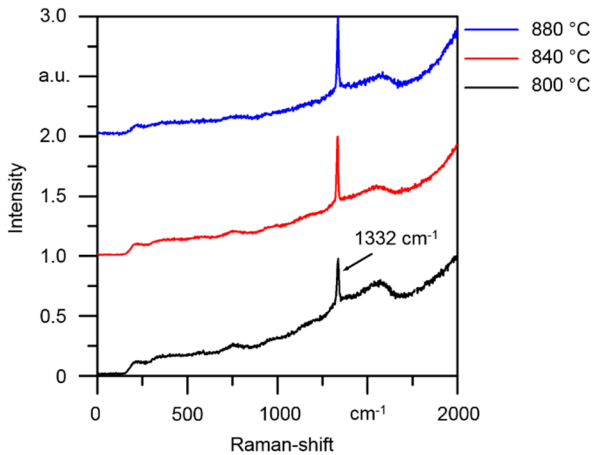


Fig. 4: The influence of the surface temperature on the structure of the CVD diamond coating is easily visible in the three laser microscope measurements. The temperature shown was measured underneath the middle of the substrate by thermocouples.

For all three CVD diamond coatings, which are shown in Figure 4, a Raman spectroscopy measurement was executed. The Raman spectrometer used an argon-ion laser with a wavelength of 514.5 nm for the excitation of the sample. The peak at  $1332\text{ cm}^{-1}$  in the Raman spectroscopy proves the existence of a diamond structure [6]. The peak can be clearly identified in Figure 5 for all three deposited CVD diamond coatings.

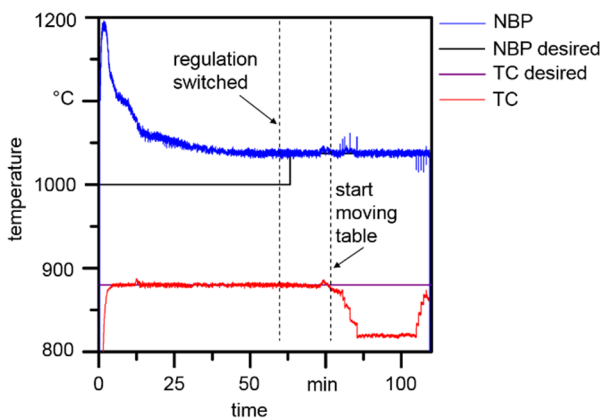


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Fig. 5: Raman shift of the three different coatings shown in Figure 4. The distinct peak at  $1332\text{ cm}^{-1}$  proves a diamond structure.

The integrated feedback control for the surface temperature of the substrate during the CVD diamond deposition was tested. The result can be seen in Figure 6.



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Fig. 6: Temperature measured by thermocouples (TC) and narrow-band pyrometer (NBP) for 110 minutes of CVD diamond deposition. The given desired temperature is used by the feedback control as goal temperature of the value measured by TC or NBP. At the first dotted line the regulation was switched to regulate the temperature measured by the pyrometer and at the second dotted line the table was moved.

At the beginning of the deposition the feedback control uses the measured temperature of the thermocouples from underneath the substrate for the temperature control over the first 63 minutes (Figure 6 first dotted line) and regulates to a desired temperature of  $880\text{ °C}$  (purple line). At the beginning of the process the centre of the deposited CVD diamond coating is exactly above the thermocouples. It can be seen that the measured temperature of the pyrometer decreases over the first 63 minutes, which can be explained by the changing surface material, as was already expected. At the start of the process the pyrometer measures the heat

radiation of the molybdenum (substrate), but after about ten minutes the diamond coating starts to grow, which leads to a changing emissivity. It is possible to change the regulation to the temperature measured by the pyrometer. This is done in Figure 6 after 63 minutes, without interrupting the process and without a change in surface temperature. The desired temperature of the pyrometer is shown by the black line and was set to  $1037.5\text{ °C}$ . When the surface temperature is regulated based on the temperature measured by the pyrometer, it is possible to keep the surface temperature constant also if the xy-table is moving (Figure 6 after 80 minutes (second dotted line)). The table was moved by five 1 mm steps to one direction. That works as long as the table is moving so slowly that the pyrometer always measures the heat radiation of the diamond coating. After 20 minutes the xy-table is moved back to the starting position and the temperature measured by the thermocouples goes back to  $880\text{ °C}$ .

#### 4 Conclusion

The new integrated feedback control, with the option of switching between two different methods of measuring the temperature, contributes to the automation of the LPCVD process. It is not possible to regulate via the directly measured surface temperature over the whole deposition process, because of the changing surface material at the beginning of the process. If it would have been possible, the temperature measurement would be completely independent of the geometry of the substrate. The integrated feedback control is based on the temperature measured by the thermocouples at the beginning of the process, which is strongly dependent on the thickness and material of the substrate.

By the research described here, the basic requirements for CVD diamond deposition on larger surfaces and on different geometries have been integrated into the LPCVD process. In this way, the preconditions for subsequent research are fulfilled, but the authors also want to call attention to the challenge of surface temperature measurement if the surface is surrounded by a plasma flame.

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