

# A novel microwave source for collisional plasma for nano-crystalline diamond deposition

O. Antonin<sup>1,3</sup>, L. Latrasse<sup>6</sup>, A.A. Taylor<sup>2</sup>, J. Michler<sup>2</sup>, P. Raynaud<sup>3,4</sup>, D. Rats<sup>5</sup>, Th. Nelis<sup>1</sup>

<sup>1</sup> Institute for Applied Laser, Photonics and Surface Technologies, Berner Fachhochschule, Biel-Bienne, Switzerland

<sup>2</sup> Empa, Swiss Federal Laboratories for Materials Science and Technology, Laboratory for Mechanics of Materials and Nanostructures, Thun, Switzerland

<sup>3</sup> Université de Toulouse, UPS, CNRS LAPLACE (Laboratory on Plasma and Conversion of Energy), Toulouse, France

<sup>4</sup> CNRS, Laboratoire LAPLACE – Matériaux et Procédés Plasmas, Université Paul Sabatier, Toulouse, France

<sup>5</sup> NEOCOAT SA, La Chaux de Fonds, Switzerland

<sup>6</sup> SAIREM SA, Neyron, France

This work presents first results of deposition of nano-crystalline diamond (NCD) on a low temperature substrate using a novel microwave source, Hi-Wave. The source is designed to operate without additional impedance matching. The power transmission efficiency was measured and the conditions for optimal hydrogen dissociation were determined. Plasma density was determined to reach level of  $10^{11}$  cm<sup>-3</sup>. First results on the NCD deposition are presented

## 1. Introduction

In previous studies [1,2], elementary microwave plasma sources distributed according to a square lattice matrix configuration (two-dimensional configuration) have been employed to generate plasma with relative uniformity of plasma densities.

To increase plasma processes possibilities, two types of sources were developed: One, working at very low pressure (up to a few Pa) using a Electron Cyclotron Resonance phenomenon (ECR-type source) the other without magnetic field (collisional-type source), working at higher pressure (up to 100 Pa).

A classical microwave power supply chain is composed of a magnetron based microwave generator providing microwave through waveguide, a power divider and an impedance matching device.

In this study, we use a novel plasma source called Hi-Wave. This collisional-type source was designed to avoid power losses and to be used without any tuning system over 1 decade of pressure. The exact pressure range for which optimal power transmission depends on the plasma carrier gas. Each source is connected to its own solid-state microwave generator that produces a wave with a narrow spectral range, without distributor and impedance matching device. The narrow frequency spectrum of the microwave power produced in solid-state microwave generators allows for precise plasma power control by tuning the microwave frequency [3].

This ability to tune the microwave frequency allows compensating small impedance mismatching due to minor changes in the plasma properties.

## 2. Experimental results

In this work, we present first results obtained with a single source using this new design and then its

application in a 2×2 matrix configuration. The final objective is to deposit nano-diamond on low temperature substrates.

### 2.1 Microwave power efficiency

As a first step, a power transmission efficiency map for variable power and pressures in the range of 10-100 Pa was established, i.e. the plasma power over forward power was measured as function of forward power and total pressure. The pressure range of 50 Pa was chosen as a compromise for high 3D aspect ratio (low pressure) and high deposition rate (high pressure) for nano-diamond deposition.

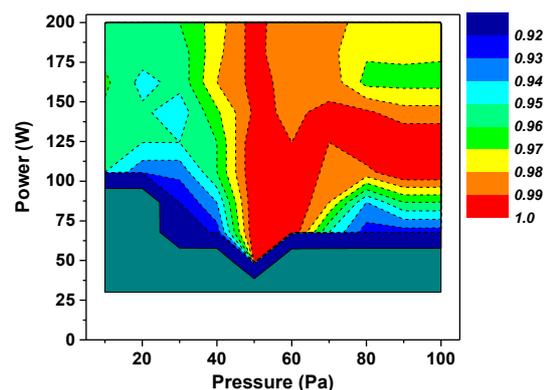


Figure 1: power transmission efficiency map in H<sub>2</sub> (97%) /CH<sub>4</sub> (3%) mixture with on Hi-Wave source.

Figure 1 shows the transmission efficiency map obtained with the (H<sub>2</sub>: 97%, CH<sub>4</sub>: 3 %) mixture. The minimum power required to maintain plasma in this gas mixture is 30 W - 80 W, depending on the gas pressure setting between 10 Pa and 100 Pa. The power transmission is optimal, 99 % for a total pressure of

50 Pa to 60 Pa for the entire power range available with one source from 30 W to 200 W. Here, the plasma impedance is totally adapted to the equivalent coaxial line. It is worthwhile to note, that over the entire pressure and power range the transmission efficiency is better than 90 %, without any additional impedance matching device in the microwave transmission line.

## 2.2. H<sub>2</sub> dissociation

Efficient and high quality diamond growth is known to depend strongly H<sub>2</sub> dissociation, i.e. the presence of free H atoms.

In order to monitor the H<sub>2</sub> dissociation using the Hi-Wave source in this pressure and power range we established a relative emission intensity map for the H $\alpha$  line, again as function of power and pressure.

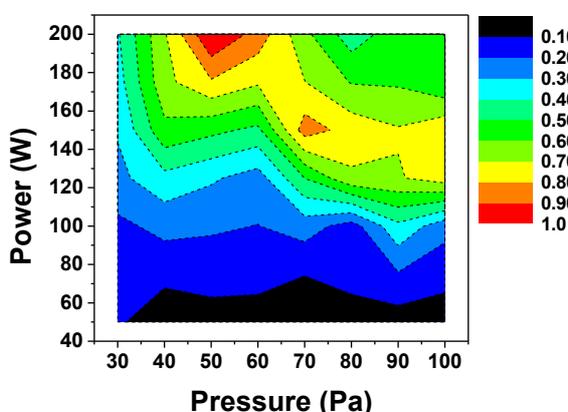


Figure 2: Relative H $\alpha$  (656 nm) emission map in H<sub>2</sub>/CH<sub>4</sub> gas mixture using the Hi-Wave source. The color code indicates the relative emission intensity.

Figure 2 is in good agreement with the efficiency map of Figure 1 and the expected result that the emission of the H $\alpha$  line increases with the power transmitted to the plasma. The strongest emission is measured for 200 W microwave power and a pressure of 50 Pa. At this pressure the power transmission is also optimal. As far as the H $\alpha$  emission can be considered as a good indicator for H<sub>2</sub> dissociation, a pressure of approximately 50 Pa and high power can be considered as optimal for this configuration.

## 2.3. Comparative study with argon as carrier gas

Figure 3 shows the transmission efficiency map obtained with argon as carrier gas using otherwise similar conditions as for the hydrogen – methane mixture. The plasma ignition is possible for lower power settings compared to the hydrogen plasma. At 50 Pa only a few Watt are required to maintain the plasma. However, in the pressure range studied here the power transmission efficiency is significantly lower for argon compared to hydrogen. In particular for a

microwave power setting above 100 W up to 25 % of the emitted power can be reflected. The power transmission does not only depend on pressure and power but also on the nature of the discharge carrier gas. The Hi-Wave source is not optimal for this pressure range (10 Pa - 100 Pa) in argon.

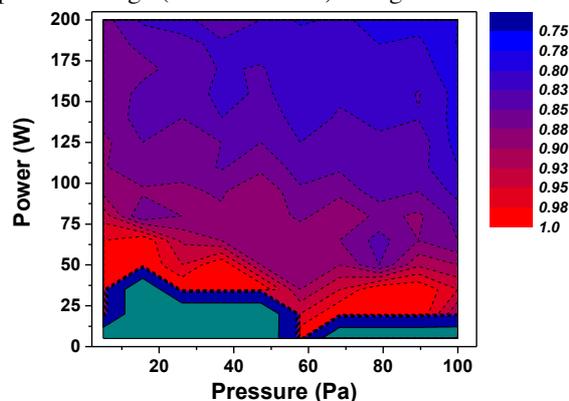


Figure 3: Argon power transmission efficiency map. The color code indicated the proportion of the power transmitted to the plasma.

Optimal power transmission in Argon can be obtained at lower pressure. At a total pressure of 5 Pa in argon the power transmission was found to be optimal, better than 99 %, for the entire power range up to 200 W. The results of plasma density measurement using a matrix of 2 $\times$ 2 Hi-Wave sources are displayed in figure 4.

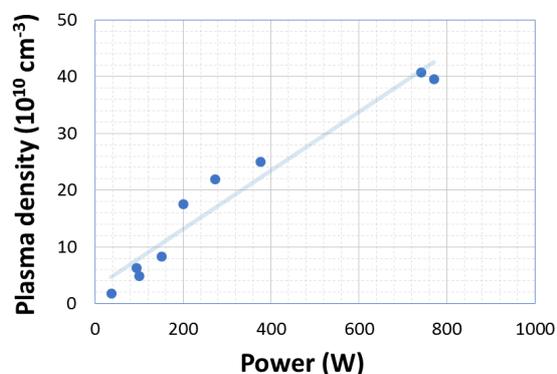


Figure 4: Electron density in a 2 $\times$ 2 Hi-Wave matrix plasma with argon at 5 Pa, measured at 160 mm from the source plane. The power indication corresponds to the total power transmitted to the four sources.

The results show that the plasma density increases linearly with the power and reaches of  $4 \times 10^{11}$  cm<sup>-3</sup> at 160 mm from the source plane.

## 2.4. Application to diamond growth

The major motivation for this work is to study and evaluate the usage of the novel Hi-Wave microwave plasma sources for growth of nano-crystalline diamond thin films [4].

A small test reactor comprising four Hi-Wave sources each connected to an independent solid-state microwave reactor. Each of the generators is capable of delivering 200 W. The four sources are situated on a square matrix and separated by 80 mm. The maximum power surface density is therefore 3.1 W/cm<sup>2</sup>. The reaction gases, H<sub>2</sub> (97%) and CH<sub>4</sub> (3%) are introduced on one side of the reactor chamber using a set of mass flow controllers. Figure 5 displays a photo of the reactor in operation.



Figure 5: Image of the discharge chamber using the 2×2 Hi-Wave matrix with H<sub>2</sub> (97 %) / CH<sub>4</sub> (3 %) 800 W and 50 Pa, 50 sccm.

Following the preliminary study on power coupling and Ha emission the total pressure during deposition was set to 50 Pa with a flow-rate of approximately 50 sccm.

Under these conditions we were able to deposit nano-crystalline diamond film on a silicon wafer. A silicon wafer of was prepared by prior seeding to initiate diamond growth. If today the diamond growths mechanisms are not clearly established, it is recognized that an environment saturated in atomic hydrogen at the surface of the substrate is a major key for it.

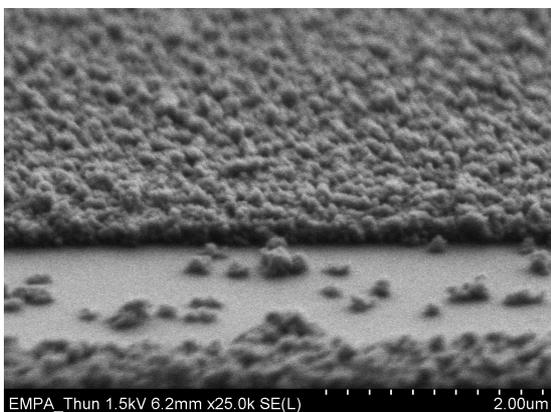


Figure 6: Tilted SEM of nano-crystalline diamond film using a 2×2 Hi-Wave matrix.

The conditions of deposition were at low pressure (50 Pa), a ratio of H<sub>2</sub> (97%) / CH<sub>4</sub> (3%) and 200 W applied on each sources. The temperature at the surface of the wafer was only induced via heattransfer from the plasma. The substrate temperature was measured to approximately 300 °C.

Blue coloration of the deposited layer indicated correct thin film coverage over the entire wafer. SEM imaging was then made ex-situ (Fig.6).

The deposition rate is approximately 5 nm/h. Raman measurements as well as TEM measurements, not shown here, revealed the nano-crystalline diamond structure of the deposited thin film.

### 3. Conclusion

The Hi-Wave sources are suitable for generating a plasma in a H<sub>2</sub>/CH<sub>4</sub> atmosphere used for nano-diamond deposition. There design allows feeding nearly 100 % of the microwave power emitted by the solid-state generators into the plasma. The lack for additional impedance matching systems and power divider will allow up scaling to an N×N matrix for deposition on larger areas.

However, in order to optimize the entire system ensuring reproducible adhesion of the deposited diamond films at high deposition rate further studies are required.

### Acknowledgement

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### 4. References

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