DIAMOND DEPOSITION USING SURFACE WAVE SYSTEM WITH HIGH MICROWAVE POWER

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Abstract

In this work we show the option for getting thin diamond films using a technique assisted by surface wave guide microwave discharge, where the generated plasma is out of the resonant cavity. This technique itself has been demonstrated to be a powerful one for basic investigation and also for many applications. The advantages of the microwave surface guide discharges compared with the conventional microwave are the plasma shell formation, low contamination allowing diamond growth out of plasma region and the energy density inside the plasma shell that was around 30% higher than inside the conventional bell jar plasma.

The present paper provides the results of the parametric study of the diamond quality films as well as of the incident microwave power, internal pressure of the reactor, substrate position and substrate temperature. Deposition of adherent and stress free CVD diamond films with very low grain size was obtained on silicon and quartz. The main techniques used for characterization of the films were Raman Spectroscopy and Scanning Electron Microscopy.

1. Introduction

Conventional microwave systems can operate in 2.45 GHz for diamond growth, but also, at smaller or larger frequency [1]. Nowadays, 2.45 GHz is one of the most commonly used frequencies in plasma applications. The reasons for that are: a) it is the frequency authorized in all countries by International Telecommunication Union (ITU) for Industrial, Scientific and Medical Applications; b) it has been investigated in many laboratories as early as the 1950s. and it has shown efficient microwave power dealing to high density plasmas, and c) more recently, the advent of low cost 2.45 GHz magnetrons for domestic use (microwave oven, 500-1500 W) contributed to disseminate different plasma processing equipment.

Surface wave can propagate in the interface between the generated plasma column and its surrounding dielectric tube. It has been studied and tested experimentally since the 1960s. In the 1970s, the interest in such waves, shifted to their conventional uses, have been hardly studied by Moisan et al [2], introducing the first simple, compact and efficient surface wave launcher for generation of long plasma columns at microwave frequency, the Surfatron [3]. The properties of the wave sustaining such plasma columns were then investigated by Moisan et al [2] who measured its dispersion and attenuation characteristics. Borges et al used an improved Surfatron system operating at 2.45 GHz, in 1995, with a non-conventional configuration, the surface wave chemical vapor deposition (SWCVD) system to obtain plasma with hemispheric forms for diamond deposition [4]. In 1996, Trava-Airoldi et al obtained diamond thin films of high optical transparency in the spectral region from ultraviolet up to infrared, also using the Surfatron system with frequency of 2.45 GHz. [5]. Also, very low roughness diamond film has been obtained using the Surfatron system [6]. Some advantages of such new plasma reactor compared with the conventional can be remarked [7]: a) the generated plasma is completely outside of a resonant cavity, providing better conditions for " in situ" diagnosis; b) there is an increment of about 30% of the power density in the plasma shell for a given incident microwave power. This gives a higher deposition rate and low surface roughness, and c) flexibility in adjusting the distance between the susceptor and the plasma hemisphere. The occurrence of discharge outside a resonant cavity eliminates most of the interactions of the substrate with the cavity and also the power density inside the plasma shell of the SWCVD is higher than the power density in conventional microwave discharge as has been described.

Surfatron fundamental studies and its applications have been object of intense investigation and there is a great perspective for the CVD diamond deposition technology. This is the newest diamond deposition technique and for this reason we believed that the SWCVD deserve special attention. In this work, this technique has been used from 0.6 to 3.5 kW of microwave power. The grown diamond films have good quality, high adherence and very low roughness.

2. Experimental Set up

A schematic configuration of the Surfatron system is shown in Fig. 1. Basically the surfaguide set is composed of the launcher and two coaxial dielectric tubes. The external tube is made of pyrex and used as an external wall jacket for flowing the cooling liquid, the dimetylpolisiloxane (DMPS). DMPS is transparent to the 2.45 GHz microwave wavelength. The inner tube, made of quartz, is the internal wall jacket and it works as the dielectric tube of the surface wave-guide. The quartz tube, out of the launcher region has a slightly increasing cross-section that ends with an abrupt transition. This finishes the surface wave propagation and create a hemispherical plasma shell. The plasma shell thickness is around few millimeters, uniform and has high energy density.

The substrate is placed close to hemispherical plasma shell. The susceptor area, made of molybdenum, is around 20 cm^2. An optical pyrometer placed on the top of the quartz tube was used for measuring the substrate temperature.

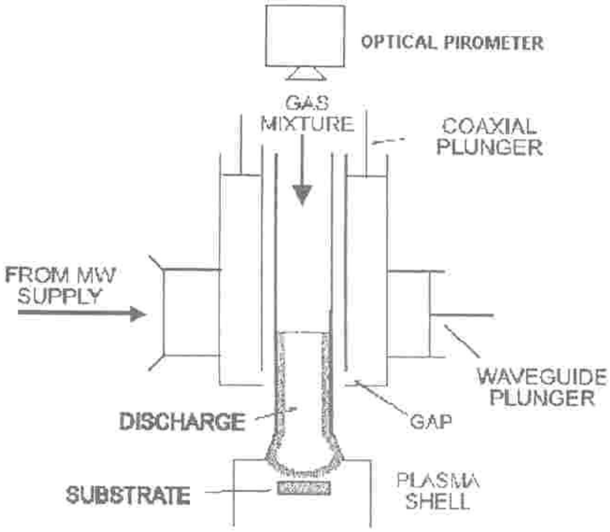

Figure 1: Schematic diagram of a SWCVD reactor for CVD diamond

3.Results and Discussion

One of the parameters for surface wave propagation is the oscillation mode (m=0), that assists the best energy transfer process from the wave launcher to the excited wave. This mode was obtained by tuning the coaxial and wave-guide plunger.

Another parameter is the behavior of the reflected power as a function of the incident power. It was made a plot of this dependence for an incident power up to 5.5 kW, as is shown in Fig.2. For incident power until 2.0 kW was observed that the reflected power increases, then it decreases to acceptable ratio of 2.3% for to the incident power of 5.5 kW.

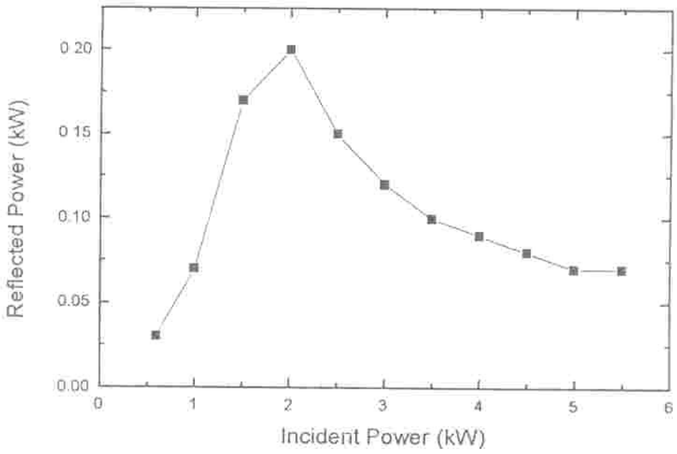

Figure 2: The dependence of incident and reflected microwave power

In this work, the experiments focused the search for diamond films of small grain size, good quality and high nucleation rate, due to its importance for the applications in optical and tribology areas.

First of all, two different substrates were prepared: quartz and silicon. They were kept for one hour in ultrasound bath using 0.25 μm diamond powder dispersed in hexane solvent. After that, the samples were cleaned in ultrasound with water and acetone bath for more 5 minutes.

The parameters used for diamond growth in the initial studies were 1.0% vol. of methane in hydrogen, 100 sccm total flow rate, substrate temperature kept constant at 850°C, growth time about 1 hour, incident microwave power of 2.5 kW and inside reactor pressure of 40 torr.

Figures 3.a and 3.b show the electron scanning micrography and Raman spectrum of a diamond thin film deposited on quartz and Fig.4.a and 4.b, the same for diamond film on silicon. The grain size is quite small and the quality of the film is very good, for both substrates. The Raman spectra show the presence of diamond peak at 1332 cm^{-1} and non-diamond carbon phase peak around 1550 cm^{-1}.

Considering that the surfatron system is being investigated for the first time at high incident microwave power (up to 6.0 kW), preliminary data about other behavior of the diamond growth parameters are studied. These parameters are substrate temperature, distance between substrate and plasma shell, microwave power and pressure inside the reactor. Grain size, quality of the diamond film and nucleation rate were measured for incident microwave power higher than 1.5 kW.

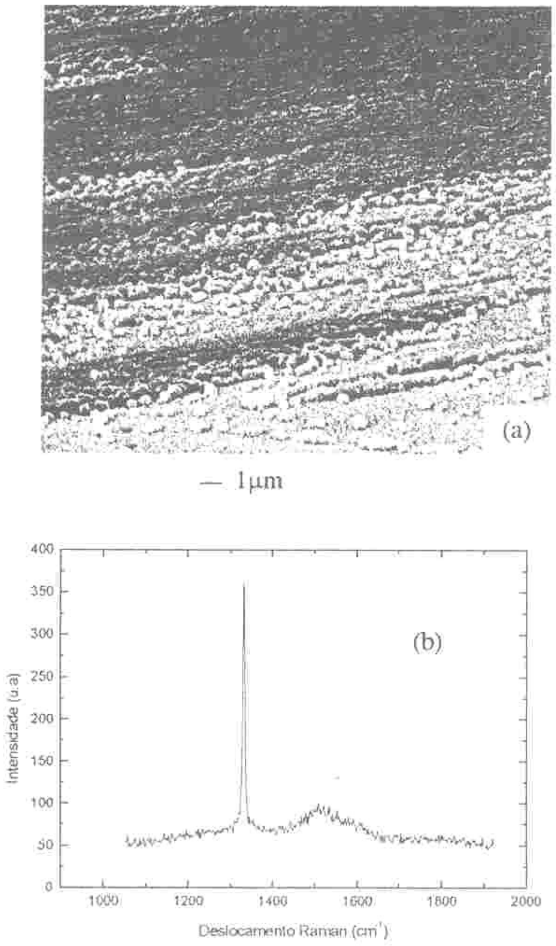

Figure 3. (a) Scanning Electron Microscopy and (b) Raman Spectrum for diamond on quartz

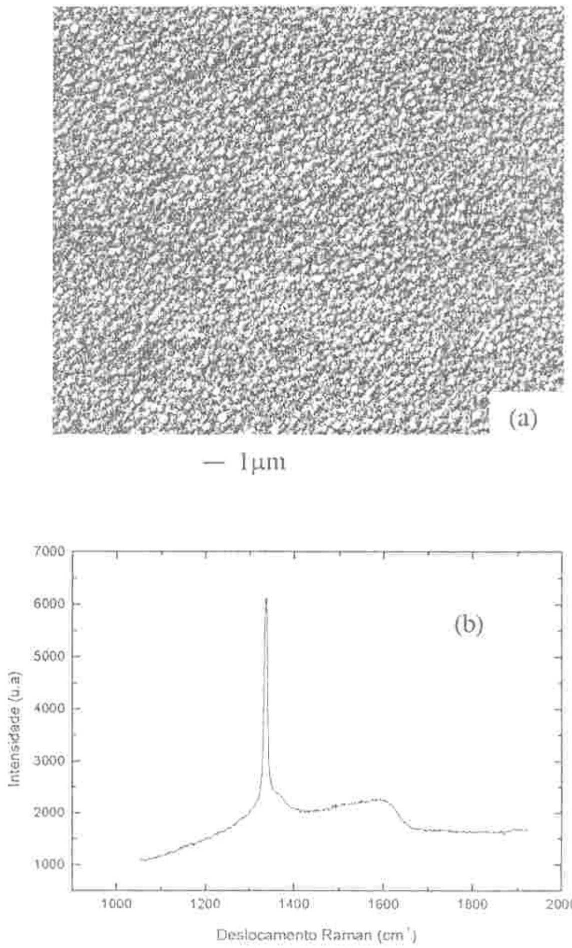

Figure 4. (a) Scanning Electron Microscopy and (b) Raman Spectrum for diamond on silicon

The substrate temperature is a important parameter [8] and was investigated as a function of the distance between the substrate and the plasma shell, as shown in the Fig.5. The surfatron is provided with a system that allows moving the susceptor in relation to the plasma shell. Keeping the incident microwave power at 5.0 kW and the pressure fixed in 40 torr it was observed that there is a linear dependence of substrate temperature with distance between the substrate and the plasma shell. The position 0 mm corresponds to the shell in contact with the substrate and the negative distance values means that there is an overlapping between them. It was identified that in order to get the 850 °C diamond growth temperature, the distance between substrate and the plasma shell is around 0.5 mm.

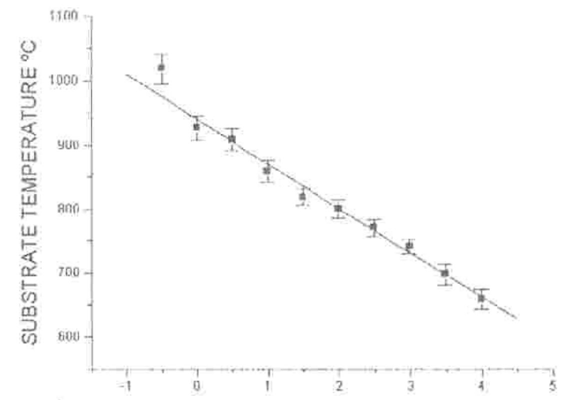

Figure 5. Substrate temperature as a function of the distance between the substrate and the plasma shell keeping the incident microwave at 5.0 kW.

The other parameter studied was the nucleation rate as a function of the pressure inside the reactor and incident power, as shown in Fig.6. This measure was done counting grains number in the specific area of micrography and diamond growth time was 1 hour. We conclude that, the nucleation rate increases for higher incident microwave power, due to the increase of the plasma shell energy. This dependence shows the necessity of choosing a set of parameters to be employed in order to get the best situation for diamond growth and good results in terms of roughness and grain size.

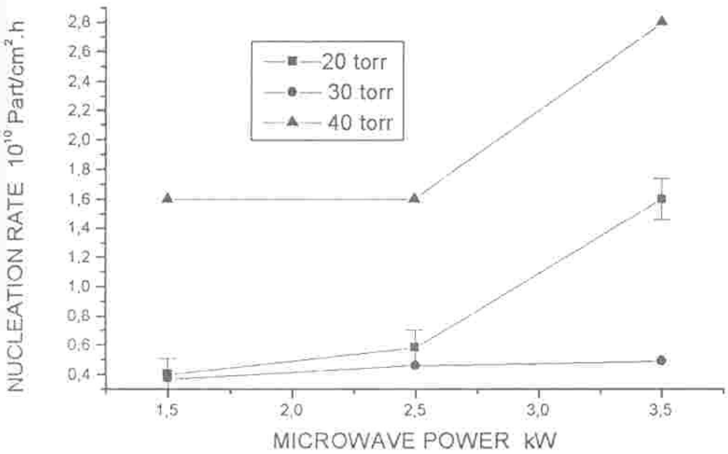

Figure 6: Dependence of the diamond nucleation rate as a function of the incident microwave power in different pressures.

Figure 7 shows the electron scanning micrography of diamond thin film on silicon surface, considering three different sets of diamond growth parameters. Keeping the pressure close to 30 torr and varying incident microwave power from 1.5 kW to 3.5 kW. It was observed that there is a significant changing in terms of grain size for higher power. Films grown at higher power presented smallest grain size. As we can see it is apparent a mixing of (111) and (100) diamond faces for low and high power and at intermediary power is more apparent the (111) face.

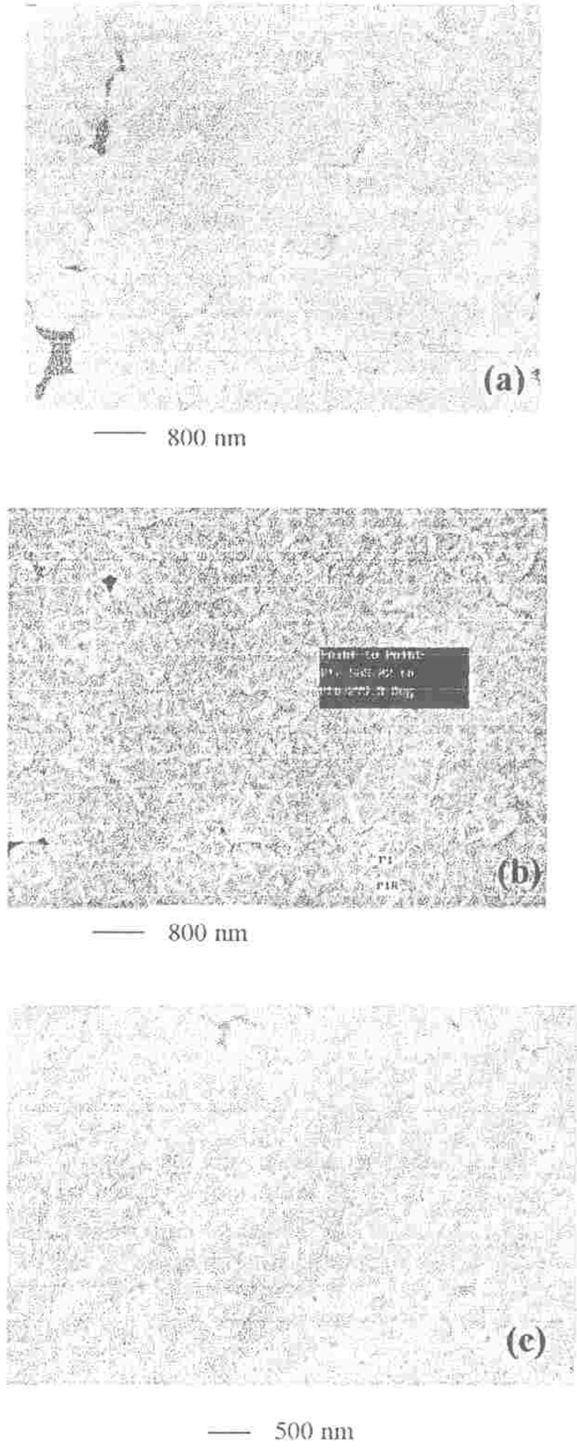

Figure 7. Scanning Electron Microscopy analysis for three different incident microwave power, a) 1.5 kW, b) 2.5 kW and c) 3.5 kW

4-Conclusion

The uses of "Surfatron" to obtain Diamond films with very small grain size and high nucleation rates, has shown quite promising. Although, it is still in initial phase of operation and identification of the best growth parameters, it is apparent the potential of this technique. It was obtained grain sizes under 200 nm, and the strong influence of the studied parameters in the quality and grain size of the film was confirmed.

The incident microwave power was explored up to 5.5 kW for identification of reactor's parameters, and up to 3.5 kW for diamond growth. The nucleation rate was examined as a function of the microwave power and the conclusion is that high nucleation rate lead to very small grain size.

It is necessary an intense work to explore the variation of the deposition parameters, such as, methane concentration, incident microwave power, temperature of the susceptor, pressure and the distance between the plasma shell and the susceptor. Also, we will intensify the studies of the cooling liquid system in order to work at 5.5 kW of incident microwave power for a long time, this is necessary to obtain good quality and very thin diamond films.

5- References

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