

Resumo

Among all the allotropic forms of Carbon, diamond is the one that attracts the most curiosity and fascination, whether for its characteristic brilliance when cut in the shape of a jewel, or for its extreme and unique properties rarely matched by other materials in nature. In a rapid rise from a technological point of view, much has been achieved in the study of obtaining this material through CVD. This work presents an analysis focused on obtaining CVD diamond films through the microwave plasma activation method (MWPCVD) in a high applied power regime using a modified substrate holder. The films were characterized using Raman scattering spectroscopy and scanning electron microscopy. The results point to optimized conditions for depositing films with growth rates of up to 8 μm/h with good structural quality and uniform microcrystalline morphology along the deposition surface.

Introduction

In this work, we employ the MWPCVD technique modified by using a substrate holder adapted to position the sample in a warmer region of the plasma, to deposit polycrystalline CVD diamond films and evaluate how this modification can influence growth, observing growth rates, crystal morphology of diamond, film quality and presence of phases of non-diamond carbonaceous materials.

The structural quality of a diamond can be studied in different ways, one of which is the Raman scattering spectroscopy which, by means of the excitation of the material with light at specific wavelengths, results in a spectrum referring to light emission by the Raman effect of the connections between the atoms of that material.

In this context, important information can be obtained from the analysis by Raman spectroscopy using the relationship between the displacement of the characteristic diamond peak compared to its naturally occurring position to obtain an estimate of residual stresses (σ) generated by inclusions of non-diamond carbon phases within and between the diamond grains that make up the film and by its thermal accommodation to the substrate (Eq. 1) [1].

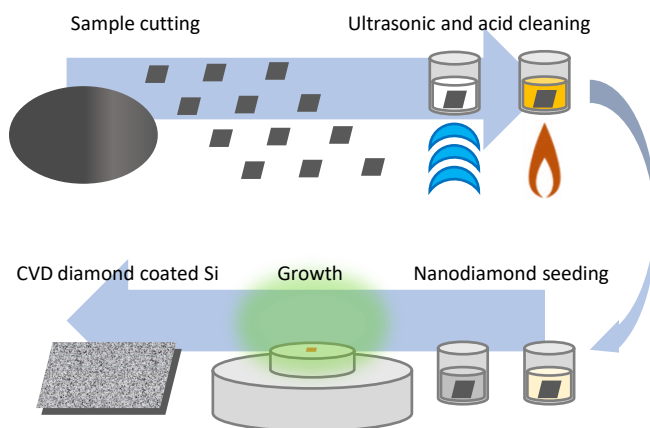
$$\sigma(\text{GPa}) = -0,567 \times \Delta\nu_m \quad (1)$$

And it is also possible to obtain a film quality factor (Q) from the relationship between the characteristic peak intensities of diamond and bands related to non-diamond carbon phases present in its structure (Eq. 2) [2].

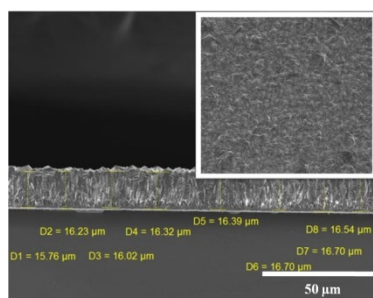
$$Q_{[514\text{nm}]} = \frac{I_{\text{diamond}}}{(I_{\text{diamond}} + I_{233})} \times 100 \quad (2)$$

Material and method

The substrates were subjected to a cleaning procedure to remove organic residues and particles adhered to the surface. Then, a seeding procedure with diamond nanoparticles was performed by the ESD method described in detail in another work [3]. The CVD diamond films were deposited using microwave power of 3.6kW, a 2% proportion of CH₄ diluted in H₂, and varying the pressure and temperature parameters from 125 to 200 Torr and from 950 to 1040°C respectively.



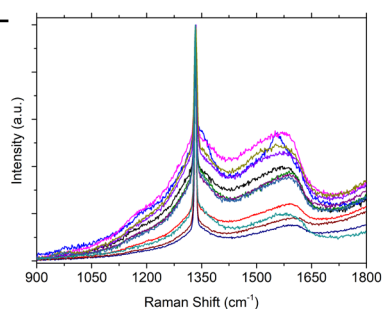
Results



FE-SEM micrograph of surface morphology and cross section.

Sample		σ (GPa)	Q
P(Torr)	T (°C)		
125	950	-0,64071	99,87601
125	1000	-0,59535	99,95674
125	1040	-0,77679	99,88093
150	950	-0,65772	99,85366
150	1000	-0,52731	99,92785
150	1040	-0,36855	99,9855
175	950	-0,85617	99,90302
175	1000	-0,40257	99,94107
175	1040	-0,36855	99,98221
200	950	-0,78246	99,88951
200	1000	-0,41391	99,91273
200	1040	-0,66339	99,94669

Stress and quality factor calculations for the obtained films



Raman spectra of all samples

It is possible to observe in all films a significant luminescence, identified from the rise of the spectra baseline. The characteristic peak of diamond centered around 1332 cm⁻¹ is also visible in all spectra. The morphology of all grown samples presents characteristics mostly of microcrystalline CVD diamond films, with grains varying in size in the micrometer scale.

Conclusion

The results showed that for the condition of higher plasma concentration obtained by the modification carried out in the substrate holder of our reactor, the temperature range above 1000 °C is more suitable for obtaining better quality films, using pressures in the range of 150 to 175 Torr, where there was a gain in the growth rate of films without significantly compromising their structure.

References

1. RALCHENKO, V. G. *et al.* Diamond deposition on steel with CVD tungsten intermediate layer. *Diamond and Related Materials*, v. 4, n. 5–6, p. 754–758, 1995.
2. VEILLERE, A. *et al.* Influence of WC-Co substrate pretreatment on diamond film deposition by laser-assisted combustion synthesis. *ACS applied materials & interfaces*, v. 3, n. 4, p. 1134–1139, 2011.
3. CAMPOS, R. A. *et al.* Development of nanocrystalline diamond windows for application in synchrotron beamlines. *Vacuum*, v. 89, p. 21–25, 2013.