

# Surface Roughness as a Function of the DLC Thickness Coating

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## ABSTRACT

The evolution of surfaces roughness coated with DLC as function of the film thickness is analyzed. We have used substrates with three different roughness, between hundreds of nanometers and just few nanometers. The surface roughness shifts as a function of the DLC thickness coating have been obtained. For the substrates with original roughness 393 and 278 nm, the roughness shift increased with the DLC thickness up to a maximum value and then decreased. For the substrate with original roughness around 4 nm, the roughness shifts oscillates but have no systematic tendency to decrease or increase.

**Keywords:** DLC Coating; Thin Films; Roughness.

## INTRODUCTION

Diamond-like carbon (DLC) is a superhard material containing both graphitically bonded carbon ( $sp^2$ ) and diamond-bonded carbon ( $sp^3$ ). The higher the  $sp^3:sp^2$  ratio, the more diamond-like the material performs, quite generally for all its properties. The material may be hydrogenated, however the highest  $sp^3:sp^2$  materials are hydrogen-free. Properties of DLC and methods for forming this important thin film material have been widely discussed in the literature [1].

DLC is a very interesting material to be used as a protective coating since it is hard, it has low thermal expansion, high thermal conductivity, low friction and chemical inertness. In the case of MEMS, DLC coating is especially important due to the possibility to deposit very thin coatings, avoiding changing significantly the surface morphology. But a new aspect, not yet explored, is the surfaces roughness ( $\Delta$ ) modification for a large range of original substrate roughness ( $\Delta_0$ ).

In this work we have analyzed the evolution of surfaces roughness coated ( $\Delta_c$ ) with DLC as function of the film thickness. This

study explores substrates with initial roughness (rms)  $\Delta_0$  between hundreds of nanometers and just few nanometers.

The results are discussed taking to account the thin films growth dynamics [2].

## MATERIALS AND METHODS

In this work we have used three substrates, two of polycrystalline diamond films and one of silicon. Our intention is to have substrates with three different roughnesses  $\Delta_0$ .

The diamond films were deposited by microwave plasma assisted chemical vapor deposition (CVD). The equipment used is described in reference [3]. A silicon substrate 17 x 17 mm<sup>2</sup> was polished by diamond powder, then it was cut in pieces and washed in acetone using ultrasonic bath. The silicon pieces were placed on the CVD substrate holder and a diamond film was grown using following parameters: 300 sccm hydrogen flow rate, 4.5 sccm methane flow rate (0.5-vol% methane in hydrogen),  $1.0 \times 10^4$  Pa chamber pressure, 1128 K substrate temperature, with a nominal 700 W microwave power. The substrates, obtained in this way, have  $\Delta_0$  about hundreds of nanometers.

The silicon substrates were wet etched, using a room temperature mixture of hydrofluoric acid, nitric acid and acetic acid. The silicon pieces were immersed in the acid solution during about 5 minutes. The silicon samples in this way have  $\Delta_0$  about few nanometers.

Each sample was characterized by atomic force microscopy (AFM) in contact mode. The microscope used was a Scanning Probe Microscope, NanoScope IIIA, Digital Instruments. A silicon nitride tip was used, with a highest measurable angle of 65°. In our AFM images the measured angles are much smaller than 65° and the smallest pixel size used was about 30 nm, so it was not necessary to take into account the convolution effect of the tip shape and surface profile. For each sample, three images were taken and an average roughness was obtained.

For each piece of the three substrates, different DLC thicknesses were deposited. The equipment used for the DLC deposition was a metal plasma immersion ion implantation and deposition (MePIIID) system [4]. This technique is highly effective for producing high quality DLC films, and has been described in detail elsewhere [1]. In this approach, a carbon plasma is formed from a vacuum arc plasma gun [5]. Hydrogen-free DLC films have been made that are ion stitched to the substrate. In this work, the parameters used for the DLC deposition were: 200 A for the arc current, with 5 ms for arc duration and the frequency of the pulses was 1 Hz.

Finally the DLC films on the samples were characterized by AFM. For each sample, three images were taken and an average roughness was obtained.

## RESULTS

The purpose in using the substrates describe above was to have substrates with  $\Delta$  varying from hundreds of nanometers up to few nanometers.

Figures 1, 2 and 3 present the surface roughness shifts ( $\Delta_C - \Delta_O$ ) as a function of the DLC thickness coating. As one can see, for the substrates with  $\Delta_O = 393$  nm and  $\Delta_O = 278$  nm, the roughness shift ( $\Delta_C - \Delta_O$ ) increased with the DLC thickness up to a maximum value and then decreased. For the substrate with  $\Delta_O \approx 4$  nm, the roughness shift fluctuated without any systematic tendency to decrease or increase.

These results can be interpreted using some of our previous papers [6, 7]. In one of these paper [6] we have coated silicon AFM tips with DLC, with different film thicknesses. We verified that in the tip extremity there was a higher DLC deposition rate than on the lateral surfaces of the tip, as illustrated in the scheme presented in figure 4. In this way, for pointed structures, as we have for the diamond films, the DLC films introduce broadening of the sharp edges of the faceted crystals, increasing the roughness. This explains why the  $\Delta_C$  increases for small DLC film thickness. On the other side, for thicker films, the  $\Delta_C$  begins to decrease (see figures 1 and 2).

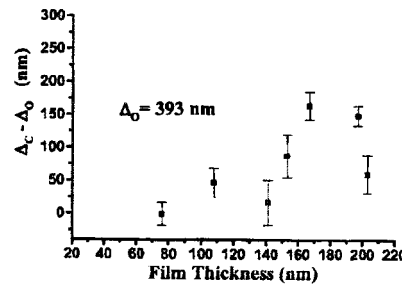


Figure 1: Plot of the roughness shifts ( $\Delta_C - \Delta_O$ ) as a function of the DLC thickness coating, using as substrate CVD diamond, with  $\Delta_O = 393$  nm.

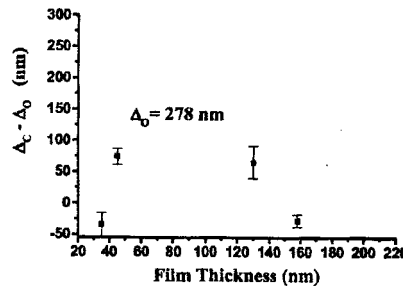


Figure 2: Plot of  $\Delta_C - \Delta_O$  as a function of the DLC thickness coating, using as substrate CVD diamond, with  $\Delta_O = 278$  nm.

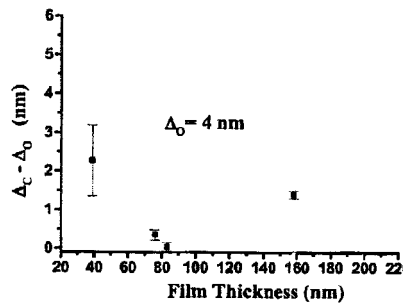


Figure 3: Plot of  $\Delta_C - \Delta_O$  as a function of the DLC thickness coating, using as substrate silicon surface etched with acid, with  $\Delta_O \approx 4$  nm.

In the other previous paper [7] flat silicon (monocrystal) have been coated with DLC, with thickness between 30 and 200 nm. In this case we verified that the DLC roughness was about 0.5 Å, corresponding to the original substrate roughness. This fact is coherent with the result, seen in figure 3, obtained here for the

silicon substrate wet etched. In this case  $\Delta_C - \Delta_0$ , due to the DLC coating, fluctuates without systematic tendencies to decrease or increase.

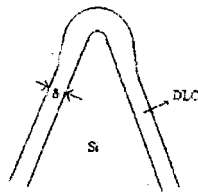


Figure 4: Scheme illustrating that in the tip extremity the DLC deposition is thicker than on the lateral surfaces of the tip ( $\delta$ ). This was observed in a previous paper [6].

## DISCUSSION AND CONCLUSION

The diamond films with different amorphous carbon concentrations have been studied using growth dynamics [8, 9]. It was verified that the ballistic deposition model (BD) is obeyed, belonging to the same universality class of the stochastic Kardar-Parisi-Zhang (KPZ) equation. The DLC films are basically amorphous carbon with diamond carbon bonds, so we expect that they also obey the ballistic deposition model. As it is extensively described in the literature [2], in the ballistic deposition the films have a vertical and a lateral growing. This last effect is generated by the capture of the arriving particles by the top of the structures on the surface. In the KPZ equation it is described by a non-linear term. This lateral growing is responsible for the broadening of the extremities of the sharp edges on the diamond crystallites, as described above. So, in the very beginning of the DLC film deposition on a rough surface like diamond with sharp edges, we have a rapid increase in the surface roughness  $\Delta_C$ . As the DLC film thickness increases, due to the lateral spreading of the deposited particles and the formation of holes in the film structure [2],  $\Delta_C$  stops to increase and tends to decrease. This is seen in figures 1 and 2. On the other hand, when DLC is deposited on a smooth surface, like the silicon with low  $\Delta_0$ , with no sharp edges,  $\Delta_C$  is practically constant with the film thickness. It does not increase and decrease, as observed in the cases of rough diamond substrates.

It is important to note that these roughness features are valid for small DLC film thickness, as presented in this paper. In these

cases the films growing have a memory of the initial substrate roughness  $\Delta_0$  [10, 11]. For thicker films this initial memory is lost and the DLC film roughness  $\Delta_C$  will tend to increase as the film thickness increases [2, 10, 11].

## ACKNOWLEDGMENTS

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## REFERENCES

- [1] A. Anders, *Surf. Coat. Technol.* **93**, 158-167 (1997).
- [2] See, for instance, Proceedings of the 10<sup>th</sup> European Conference on Diamond, Diamond-like Materials, Carbon Nanotubes, Nitrides and Silicon Carbide, in *Diamond Relat. Mater.* **9**, 231-1306 (2000) [Vol. 9, Nos.3-6, April/May, 2000].
- [3] A.-L. Barabási and H. E. Stanley - "Fractal Concepts in Surface Growth". Cambridge University Press (1995).
- [4] M. C. Salvadori, V. P. Mammana, O. G. Martins and F. T. Degasperi, *Plasma Sources Science and Technology* **4**, pp 489-493 (1995).
- [5] R.A. MacGill, M.R. Dickinson, A. Anders, O.R. Monteiro and I.G. Brown, *Rev. Sci. Instrum.* **69**, 801-803 (1998).
- [6] M.C. Salvadori, M.C. Eitz, C. Carraro, R. Maboudian, O.R. Monteiro and I.G. Brown, *Diamond and Related Materials* **10**, 2190-2194 (2001)
- [7] M. C. Salvadori, R. M. Galvão, O. R. Monteiro and I. G. Brown, *Thin Solid Films* **325**, 19-23 (1998).
- [8] M. C. Salvadori, M. G. Silveira and M. Cattani, *Physical Review E* **58**, 6814-6816 (1998).
- [9] M.C. Salvadori, L.L. Melo, D.R. Martins, A.R. Vaz and M. Cattani, *Surface Review and Letters* **9**, 1409-1412 (2002).
- [10] M.C. Salvadori, A.R. Vaz, R.J.C. Farias, M. Cattani, *Surface Review and Letters* **11**, 223 - 227 (2004).
- [11] T.J. da Silva and J.G. Moreira, *Physical Review E* **56**, 4880-4883 (1997).