INFLUENCE OF ZIRCONIA NANOPARTICLES SUSPENSIONS ON DIAMOND LIKE CARBON FILMS PROPERTIES PRODUCED BY DC-PECVD

Jesús Manuel Gutierrez Bernal[1]*, Thalita Sani Taiariol[2], Gil Capote Rodriguez[3], Vladimir Trava-Airoldi[4].

[1]Facultad de Ingeniería: Universidad Nacional de Colombia, Bogotá.
[2] Instituto de Ciência e Tecnologia. Universidade Federal de São Paulo (UNIFESP), São José Dos Campos, Brasil
[3]Facultad de Ciencias: Universidad Nacional de Colombia, Bogotá.
[4] Laboratorio Associado de sensores y Materiais (LAS), Instituto Nacional de Pesquisas Espaciais (INPE), Brasil.

1. Introduction

Research and development of Diamond Like Carbon films (DLC) with nanostructures is a new field for applications on different industries as automotive, aerospace, biomedical and electronic. DLC´s films have attracted attention due to their low friction coefficient, chemical inertness, high hardness and biocompatibility [1]. Zirconia is one of the most corrosion resistant and refractory oxides, having become a material used for thermal barrier coatings, gas sensors [2], [3] and also in applications for biocompatibility. Lastly, the use of nanoparticles for new applications has increased, especially in DLC films [3]–[6]. However, the use of zirconia nanoparticles in colloidal chemistry with dispersant is not very clear [6]. These nanoparticles are materials with very good properties which could help to improve the DLC films properties, not only decreasing the friction coefficient and stress but increasing the hardness, and biocompatibility [7]–[11].

2. Experimental

Thermal functionalization for zirconia nanoparticles (Sigma-Aldrich, size particle <100 nm) was made in a closed oven at 420°C for oxidizing surface functional groups to carboxyl group (-COOH). Fourier Transform Infra-Red (FTIR) technique was employed to find superficial functional groups and analyzing their changes before and after of functionalization process. Suspensions made from zirconia nanoparticles in different solvents as hexane and methanol were obtained by agitation with ultrasound (Vibra-Cell 750®, amplitude 60%). Times from 15 to 60 minutes were used. Some films were deposited on a flat polish silicon (100) substrates from drops of the suspensions obtained and solvent evaporation in air. Particle sizes on the films were measured using a High Resolution Field Emission SEM (TESCAN) and image digital treatment with software (MIRA3®, TESCAN) for making histograms as show in figure 1. An amorphous silicon interlayer was synthesized using gas silane (SiH_4) as precursor in order to improve the film adhesion. DLC films doped with nanoparticles were deposited using a pulsed-DC Plasma Enhancement Chemical Vapor Deposition (PECVD) system with an active screen as additional cathode [1], [12] and zirconia nanoparticles suspensions. Hydrogen content and total compressive stress in the films were determined by Raman spectroscopy (LabRam HR evolution) and Stones equation (Optical perfilometer), respectively. Coefficient of friction (COF) in DLC films was determined employing a CETR UMT2 tribometer with the ball on disc technique in linear reciprocated mode with a ZrO_2 ball. A constant force of 10N was applied on the samples at room temperature and under 40% relative humidity. The adhesion test was made using a CETR UMT-2 tribometer with a Rockwell C indenter with a 200 μm which increased over time from 0 to 50N, while maintaining a constant displacement rate of 0.1 mm/s. The critical load was determined as the value of the applied load at which film began to be pulled from the substrate.

3. Results and Discussions

Ultrasound helped to improve the deagglomeration process in zirconia nanoparticles. There were no changes in the superficial organic groups, fact corroborated by the analysis of these nanoparticles with FTIR. Suspensions showed decrease in size particle after ultrasound treatment as it is observed in fig 2. It would be possible because there were changes in electrical forces in the suspension. These electrical forces are the responsible for agglomeration process. DLC films with nanoparticles showed good friction coefficient (around 0.07), low hydrogen content (15-17%) and small total compressive stress around 0.6 GPa.

*Corresponding author: jmgutierrezb@unal.edu.co

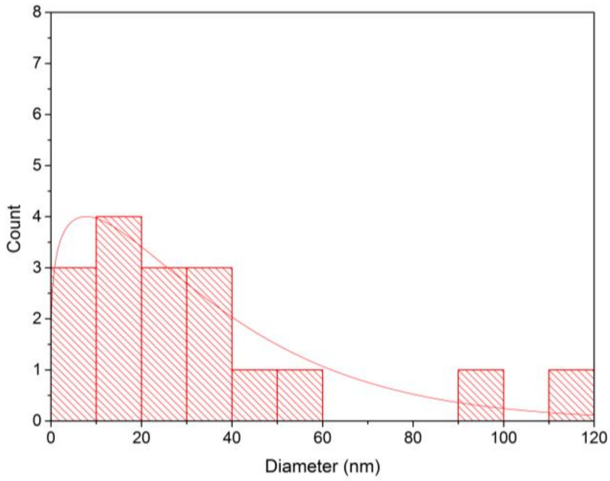

Fig. 1. *Histogram for zirconia nanoparticles after suspension process.*

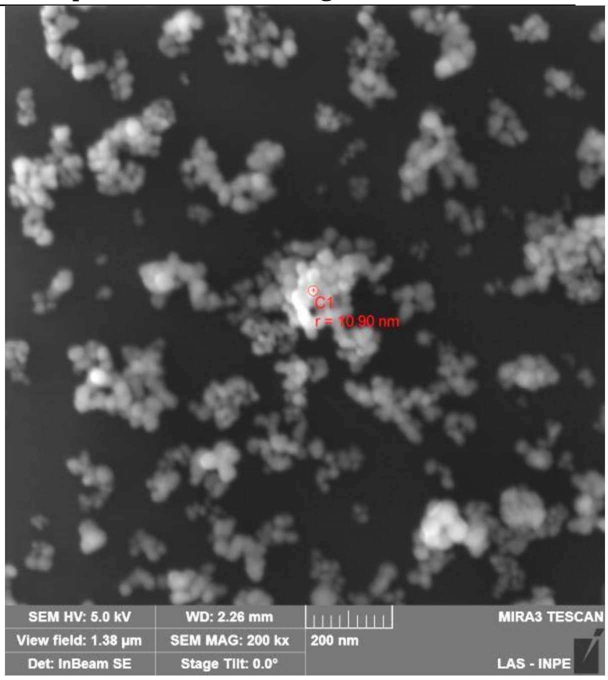

Fig. 2. *SEM image 200-Kx obtained after suspension process.*

4. References

[1] G. Capote, E. J. Corat, and V. J. Trava-Airoldi, *Surf. Coatings Technol.*, 260, 133;138, (2014).

[2] D. A. Zyuzin, S. V. Cherepanova, E. M. Moroz, E. B. Burgina, V. A. Sadykov, V. G. Kostrovskii, and V. A. Matyshak, *J. Solid State Chem.*, 179, 2965–2971, (2006).

[3] M. L. A. Guel, L. D. Jiménez, and D. A. C. Hernández, 35, 514-517, (2017).

[4] X. Liu, P. K. Chu, and C. Ding, *Mater. Sci. Eng. R Reports*, 70, 275–302, (2010).

[5] M. Stoia, P. Barvinschi, L. Barbu-Tudoran, A. Negrea, and F. Barvinschi, *J. Cryst. Growth*, 381, 93–99, (2013).

[6] T. Fengqiu, H. Xiaoxian, Z. Yufeng, and G. Jingkun, *Ceram. Int.*, 26, 93–97, (2000).

[7] F. R. Marciano, L. F. Bonetti, R. S. Pessoa, J. S. Marcuzzo, M. Massi, L. V. Santos, and V. J. Trava-Airoldi, *Diam. Relat. Mater.*, 17, 1674–1679, (2008).

[8] M. Rai, A. Yadav, and A. Gade, *Biotechnol. Adv.*, 27, 76–83, (2009).

[9] T. Tamulevičius, A. Tamulevičienė, D. Virganavičius, A. Vasiliauskas, V. Kopustinskas, Š. Meškinis, and S. Tamulevičius, *Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms*, 341,1–6, (2014).

[10] C. C. Wachesk, C. a. F. Pires, B. C. Ramos, V. J. Trava-Airoldi, A. O. Lobo, C. Pacheco-Soares, F. R. Marciano, and N. S. Da-Silva, *Appl. Surf. Sci.*, 266, 176–181, (2013).

[11] Y. Wu, J. Chen, H. Li, L. Ji, Y. Ye, and H. Zhou, *Appl. Surf. Sci.*, 284, 165–170, (2013).

[12] M. A. Ramírez R, P. C. Silva, E. J. Corat, and V. J. Trava-Airoldi, *Surf. Coatings Technol.*, 284, 235–239, (2015).

*Corresponding author: jmgutierrezb@unal.edu.co