INFLUENCE OF THE SILICON INTERLAYER DEPOSITION PROCESS IN THE ADHESION OF THE DLC COATINGS DEPOSITED ON NITRIDED MARTESNITIC STAINLESS STEEL

Eugenia L. Dalibon[1]*, Dubrazkha C. Lugo[2], Marco A. Ramírez[2], Vladimir J. Trava-Airoldi[2], and Sonia P. Brühl[1]

[1]*Universidad Tecnológica Nacional, Facultad Regional Concepción del Uruguay, Argentina.*
[2]*Instituto Nacional de Pesquisas Espaciáis, Brasil.*

1. Introduction

The DLC coatings are characterized by good wear resistance, low friction coefficient and chemical inertness [1]. However, they have adhesion problems when they are deposited on metallic substrates. For this reason, a thin interlayer of different materials, such as silicon, is deposited previous to the coating in order to improve the adhesion. There are a few studies about the influence of the conditions of the silicon interlayer deposition on adhesion or properties of the coatings [2,3]. In this work, the adhesion of DLC coatings deposited on nitrided and non-nitrided martensitic stainless steel, modifying the silicon interlayer bias potential deposition, is studied.

2. Experimental

AISI 420 martensitic stainless steel was used as base material. The samples were nitrided in a DC pulsed discharge for 10 h at a temperature of 390°C using a gas mixture composed of 20% N_2 and 80% H_2. The DLC (Diamond Like Carbon) coatings were deposited by means of a modified PACVD (Plasma Assisted Chemical Vapor Deposition) process at INPE (Brazil) [4], using acetylene as precursor gas at 150 °C for 50 minutes. The total gas pressure was 6.10^{-4} Torr. Prior to the coating deposition, an amorphous silicon layer was deposited using silane gas for 10 minutes and different bias voltages: –5 kV, –8 kV and –10 kV. The coatings and interlayers were deposited on nitrided and non-nitrided stainless steel (named Duplex and Coated samples, respectively). The DLC coatings were characterized by means of EDS and Raman spectroscopy. The microstructure was analyzed by OM, SEM-FIB, and XRD. Adhesion was evaluated by means Scratch test using 10 N, 15 N and 20 N loads and Indentation Rockwell C.

3. Results and Discussion

The Raman spectra for DLC films presented two overlapping bands known as the D and G bands. The positions and intensity ratio of the D and G bands (I_D/I_G), FWHM G band for different samples are shown in table 1. Taking I_D/I_G ratio and the G band position into account, the percentage C–C sp^3 bonds should be about 20% according to the three stage model proposed by Ferrari [5]. The hydrogen content was about 20%, which was calculated from the slope of the fitted line to the base of the original Raman spectrum. It can be indicated that the growth voltage variations of the silicon interlayer did not affect the D and G bands position, FWHM or I_D/I_G ratio (Table 1).

Table 1. Summary of Raman results for different samples

Samples	Bias Voltage (-kV)	D Band (cm^{-1})	G Band (cm^{-1})	FWHM (G) (cm^{-1})	I_D/I_G
Coated	5	1388.70	1545.38	171.50	0.69
	8	1387.66	1541.20	176.26	0.66
	10	1385.96	1550.36	169.67	0.74
Duplex	5	1382.59	1548.17	174.92	0.69
	8	1389.34	1547.49	176.88	0.65
	10	1392.00	1551.76	166.84	0.75

The DLC coatings thickness was of about 1 µm with a well-defined interphase with the substrate. The nitrided layer thickness of 10 µm. The expanded martensite peaks, i.e. nitrogen expanded martensite, were detected by XRD in the duplex sample because the film is amorphous, transparent to x-ray radiation.
With respect to the adhesion, the duplex sample had better adhesion than the only coated sample as it could be observed in SEM image indentation Rockwell C (Figure 1, 2, 3 and 4).

*dalibone@frcu.utn.edu.ar

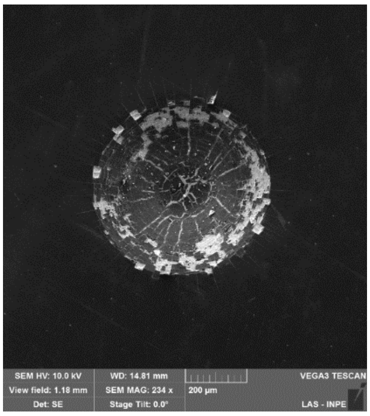

Fig. 1. SEM image of Indentation Rockwell C in duplex sample for -10 kV bias potential silicon interlayer.

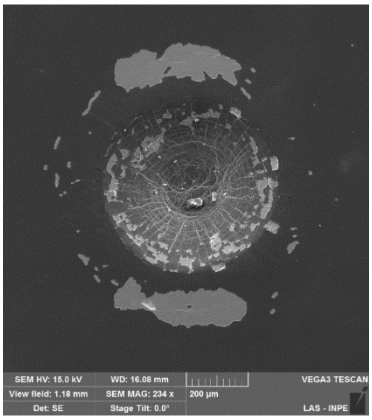

Fig. 2. SEM image of Indentation Rockwell C in coated sample for -10 kV bias potential silicon interlayer.

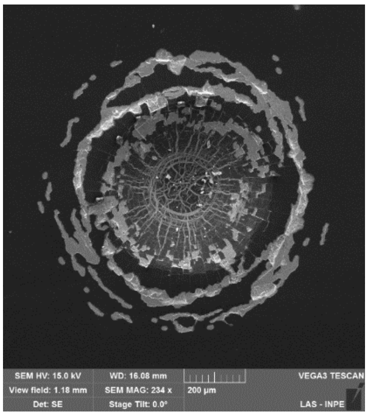

Fig. 3. SEM image of Indentation Rockwell C in coated sample for -5 kV bias potential silicon interlayer.

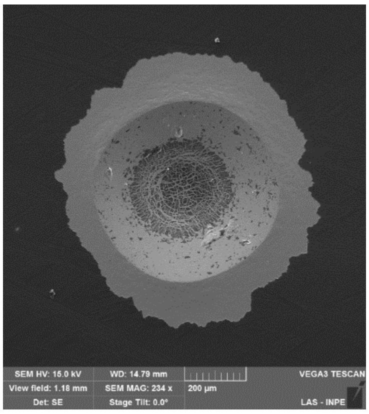

Fig. 4. SEM image of Indentation Rockwell C in coated sample for -5 kV bias potential silicon interlayer.

The same results were obtained in the Scratch Test where the coating detached with 10 N load in the coated sample. However, the coating is broken with 20 N load in the duplex sample. The improved adhesion for the duplex sample is attributed not only to the gradual transition interface between the coating and the substrate provided by the nitrided layer, but also to the affinity chemical between silicon of the interlayer and the nitrogen of the nitrided layer which can produces a chemical bonding Si-N as it was reported by some of the authors [6].
Moreover, the adhesion improved when the interlayer was deposited with higher bias voltage. The best adhesion was reached when the interlayer was deposited using –10 kV as it can be seen in Figure 1. With the bias voltage increase during silicon interlayer deposition, the ion bombardment energy increases and the adhesion improves as it was reported by some of the authors [4].

4. References

[1] J. Robertson, Mater. Sci. Eng. R Reports, **37**, 129–281, (2002).
[2] F. Cemin, L.T. Bim, C.M. Menezes, M.E.H. Maia, I.J.R. Baumvol, F. A. Figueroa, Surf. & Coat. Tech., **283**, 115–121, (2015).
[3] L.F. Bonetti, G. Capote, L. V Santos, E.J. Corat, V. J. Trava Airoldi, Thin Solid Films, **515**, 375–379 (2006).
[4] P. C. Santana Da Silva, M. Antonio Ramirez Ramos, E. J. Corat, V.J. Trava-Airoldi, Mat. Res., **19**, 882–888, (2016).
[5] C. Casiraghi, A.C. Ferrari, J. Robertson, Phys. Rev. B. **72**, 1–13, (2005).
[6] E.L. Dalibon, S.P. Brühl, V.J. Trava-airoldi, L. Escalada, S.N. Simison, J. Mater. Res., **31**, 3549–3556, (2016).

Acknowledgments

The authors are very grateful to UTN for financial support and to students from GIS–FRCU for their collaboration with the preparation of the samples.

*dalibone@frcu.utn.edu.ar