

# Nucleation Study of CVD Diamond Film on Ti Film-304 SS Substrate System by Microwave Plasma Source

R.A. Vieira<sup>1,2</sup> and M.C.A. Nono<sup>2</sup>

<sup>1</sup>Department of materials engineering (DEMAR), Faculdade de Engenharia Química de Lorena (FAENQUIL);

<sup>2</sup>Associated Laboratory of Sensor and Materials (LAS), National Institute for Space Research (INPE), 12245-970, C. P. 515, São José dos Campos, SP, Brazil, E-mail address: ravieira@las.inpe.br

## Abstract

CVD diamond films have been produced on the surface of 304 stainless steel (304 SS) with a titanium interlayer by using microwave plasma source. In this work, the previous surface modification of the 304 SS with adherent titanium films and subsequent deposition of CVD diamond films are presented and discussed. The titanium intermediate layer was obtained by vapor phase deposition produced by electron beam device. The CVD diamond films were obtained by microwave plasma source, using several CH<sub>4</sub> and H<sub>2</sub> gas mixtures. It was employed the two-step method for diamond deposition. These films were characterized by scanning electron microscope (SEM) and energy dispersive spectrometry (EDS). The results showed nucleation of CVD diamond films with low adherence.

**Keywords:** CVD diamond, titanium interlayer, two-step method, thin film.

and growth of these films. This way the necessary carbon species for the nucleation of diamond crystals is partially consumed [2]. Nucleation of the CVD diamond films can be improved with the creation of a barrier that difficulties the diffusion of the carbon ions through intermediary films that have low diffusion for this element [3-5]. CVD diamond films deposited on titanium substrate has been showed excellent tribology characteristics and good adherence [6,7]. However, the adherence of Ti films in most of the commercial steels should be improved for tribology applications.

Therefore a better understanding of the formation and development of surface and interface during growth is necessary to provide insight into the requirements for successful application of high quality diamond coating on titanium substrate.

The purpose of this research work is to investigate the formation and development of surface and interface after CVD diamond growth on steel surfaces.

In this work, the nucleation of CVD diamond films on 304 SS surfaces previously modified by linear defects and titanium adherent film are showed and discussed.

## Materials and Methods

The substrates used for the Ti thin film deposition were 304 stainless steel (304 SS) with 13 mm thick and 15 mm of diameter. They were polished with a sequence of SiC sandpapers down to 1200# grid, then ultrasonic cleaned in acetone for 20min. These surfaces were previously modified by linear defects to increase nucleus of diamond on Ti film-304 SS system.

Ti films with 3µm thickness were deposited in 304 SS surfaces by electron beam process, at 210°C in vacuum, to make a barrier against carbon diffusion. The cross-section of these samples with Ti film were analyzed by EDS and the results indicated a interface

## Introduction

Technological applications of materials, mainly metals, covered by CVD diamond films are vast. However, these films do not present good adherence in iron based metallic alloys, as it is the case of the steels. This limits the growth potentialities for the applications and the technological innovations of these materials [1,2].

The nucleation rate of the CVD diamond on the steel surfaces is very low, due to the high diffusivity of carbon ions at temperatures used to obtain nucleation



formation promoted by the thermal activated diffusion process [4,8,9].

The CVD diamond film was nucleated on the Ti surface using microwave plasma assisted CVD reactor. The gas used was a mixture of CH<sub>4</sub> and H<sub>2</sub> utilizing the two-step method [10,11]. The conditions used for the nucleation were: 37.5 Torr of pressure, the substrate temperature was about 600°C and 7 hours of growth time. The first growth time were utilized a mixture of CH<sub>4</sub> and H<sub>2</sub> in percentages of 10.0 vol.% and 90.0 vol.% respectively. The rest time were utilized a mixture of 2.0 vol.% and 98.0 vol.% respectively. This nucleation was characterized by SEM and EDS.

## Results

The observations by SEM of the CVD diamond deposited on Ti film surface with 3µm of thickness showed the existence of a film with characteristic of diamond-like carbon, as can be seen in Figure 1a and b. The nucleation was homogeneous but with low adherence (Figure 1b). As can be observed in Figure 1c, after unstuck, the sample surface present signs that showed existence of CVD diamond nucleated. It is possible to observe that CVD diamond showed preferential nucleus on surface linear defects. The nucleation characteristic showed to be homogeneous.

Linescan analysis profiles showed carbon diffusion in steel substrate, as can be seen in Figure 2. This diffusion has been promoted by temperature of diamond deposition. It was not observed Fe diffusion in titanium film.

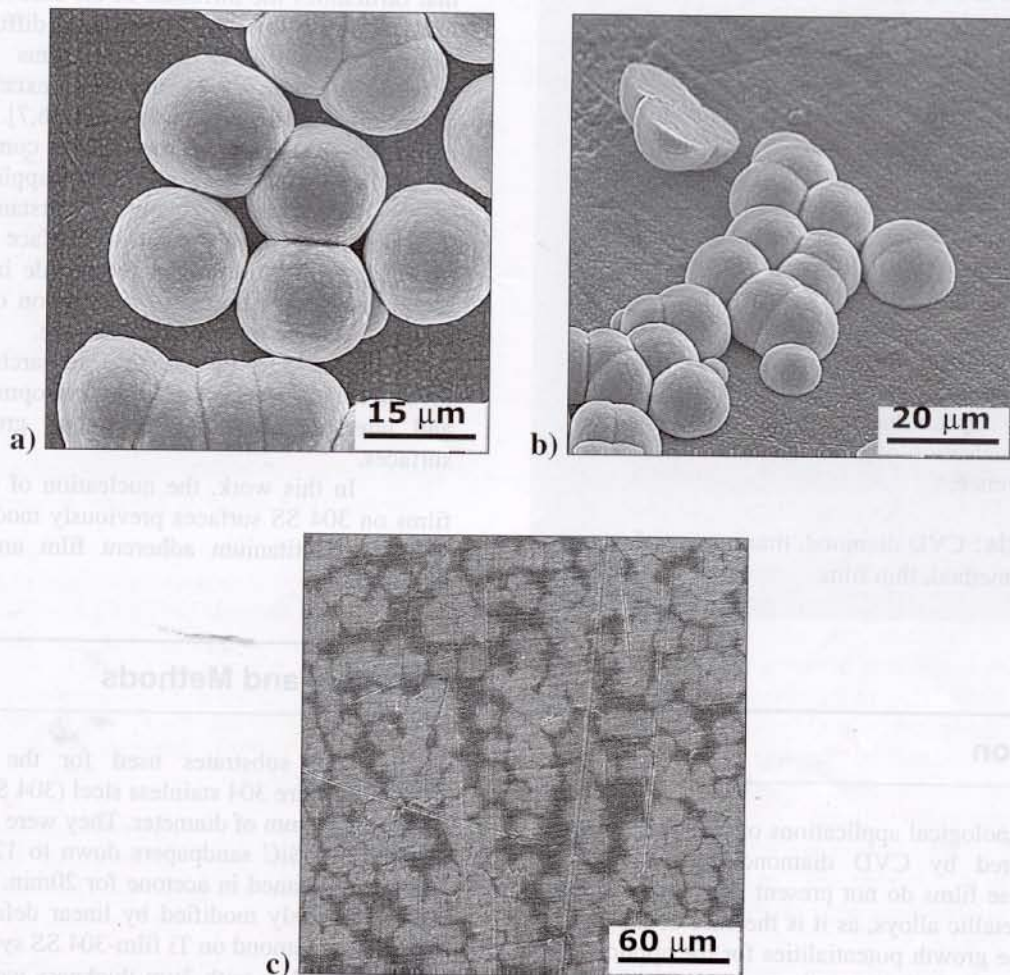


Figure 1. SEM micrographs of CVD diamond deposited by plasma assisted microwave technique on Ti film-304 SS substrate system, in the conditions of 37.5 Torr, 600°C and 7 hours of growth time utilizing two-step method

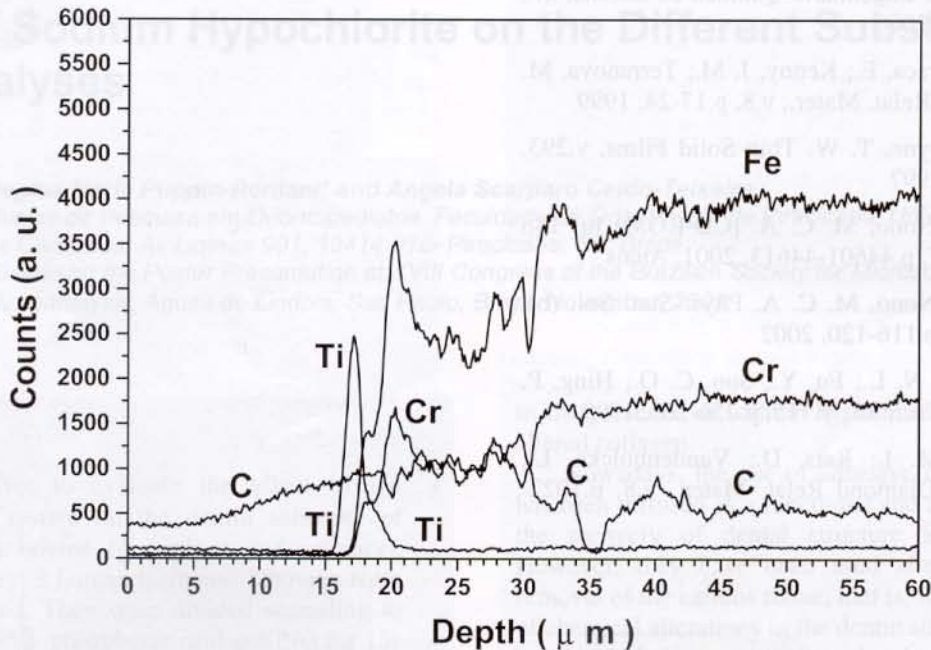


Figure 2. Interface analysis of the diamond-like carbon -Ti film-304 SS profiles by Linescan (EDS)

## Discussion

The conditions of CVD diamond nucleation on the titanium intermediary film surfaces has been studied.

Nucleation of CVD diamond deposited by microwave on 304 SS previously modified by linear defects and titanium adherent film showed to be homogeneous. These nucleus are similar with diamond-like carbon (DLC). These results showed preferential nucleus on 304 SS surface linear defects.

Interface analysis by EDS showed a carbon diffusion inside of the 304 SS substrate. This carbon diffusion must have been facilitated by temperature in CVD diamond deposition of about 600°C. It was not observed Fe diffusion in titanium film. The interface didn't suffer relative alteration by their components. The low nucleation and adherence were related to considerable amount of carbon that diffused in Ti film-304 SS substrate system.

These results showed little adherence that was associated with carbon diffusion in Ti film-304 stainless steel substrate.

## Acknowledgments

The authors would like to thank CAPES and MCT for financial support

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## Results

The observations by SEM of the CVD diamond deposited on Ti film surfaces with thin layers of thickness showed the existence of a nucleation layer characteristic of diamond film nucleation, as can be observed in Figure 1a and b. The nucleation was homogeneous but with low adherence (Figure 1b). As can be observed in Figure 1c, after nucleation, the nucleation layer was not observed. This showed existence of CVD diamond nucleation, which is possible in a variety of ways. The nucleation layer showed preferential nucleation on the nucleation characteristic of diamond nucleation. The nucleation layer was not observed in Figure 2. The nucleation layer was not observed in Figure 2. The nucleation layer was not observed in Figure 2.



Figure 2. SEM images of CVD diamond nucleation on Ti film surfaces. (a) shows a nucleation layer with low adherence. (b) shows a nucleation layer with low adherence. (c) shows a nucleation layer with low adherence.

The conditions of CVD diamond nucleation on the titanium intermetallic film surfaces has been studied.

Nucleation of CVD diamond deposited by microwave on 304 SS previously modified with Ti film and titanium adherent film showed the existence of homogeneous nucleation. These nucleation are similar to diamond nucleation (DLN). These results show the existence of nucleation on 304 SS surface. These results show the existence of nucleation on 304 SS surface.

Interface analysis by EDS showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.

diffusion must have been facilitated by the existence of CVD diamond deposition of about 60 nm. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.

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did not suffer relative alteration by their deposition. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.

low nucleation and adherence were observed. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.

considerable amount of carbon that diffused in the 304 SS substrate system. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.

These results showed little adherence that was associated with carbon diffusion in Ti film 304 stainless steel substrate.

SS film. It is important to emphasize that the nucleation of CVD diamond on Ti film 304 stainless steel substrate was not observed. This result showed the existence of titanium inside of the 304 SS substrate. This result showed the existence of titanium inside of the 304 SS substrate.