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Characterization of a Pure Diamond-Like Carbon Film Deposited by Nanosecond Pulsed Laser Deposition

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***Abstract*—**This work aims to investigate the properties and microstructure of diamond-like carbon film deposited by pulsed laser deposition by ablation of a graphite target in a vacuum chamber on a steel substrate. The equipment was mounted to provide one laser beam. The target of high purity graphite and the steel substrate were polished. The mechanical and tribological properties of the film were characterized using Raman spectroscopy, nanoindentation test, scratch test, roughness profile, tribometer, optical microscopy and SEM images. It was concluded that the pulsed laser deposition (PLD) technique associated with the low-pressure chamber and a graphite target provides a good fraction of sp3 bonding, that the process variable as surface polishing and laser parameter have great influence in tribological properties and in adherence tests performance. The optical microscopy images are efficient to identify the metallurgical bond.

***Keywords*—**Characterization, diamond-like carbon, DLC, mechanical properties, pulsed laser deposition.

I. Introduction

THE deposition of diamond-like carbon (DLC) has a wide range of application due to its high density, hardness, electrical resistivity, chemical inertia, infrared transparency and biocompatibility [1], [2].

PLD technique is highly efficient in production of DLC films with maximum content of sp3 phase [2], [3], and also by the production of free hydrogen amorphous carbon (a-C) or nanodiamonds [4].

Owing to the difficulty in determining the properties of the deposited film [2], several works in characterization and measurement of DLC films have been reported, and can be noticed that the techniques are continually evolving [5].

The films produced by nanosecond lasers (ns-DLC) are well known for high sp3 bonding obtained, and picosecond and femtosecond lasers are reported to reduce residual compressive stress in the film [2].

In this study we characterized a DLC thin film deposited by a nanosecond pulsed laser, identifying the metallurgical bond between steel substrate and DLC film to measure some mechanical and tribological properties. Through microscope images we intend to analyze the visual aspects of metallurgical bonding layer as thickness, superficial valleys on steel substrate and also to evaluate the DLC film quality measuring its mechanical and tribological properties.

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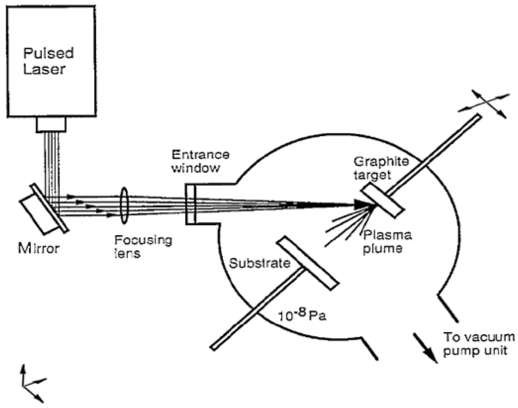
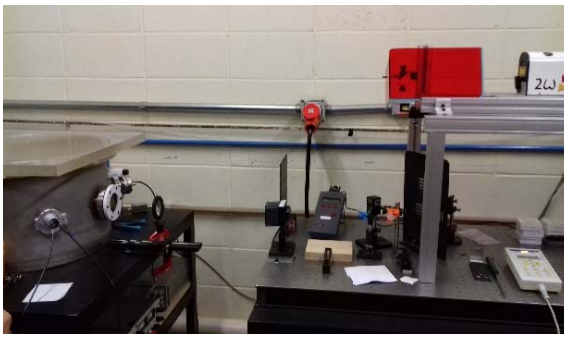
II. Experiment

For laser deposition of diamond like-carbon film under vacuum we mounted an experimental apparatus as in Figs. 1 (a) and (b), based on [6].



(a)



(b)

Fig. 1 Schematic view of apparatus for PLD of DLC film (a) and the vacuum chamber, laser and optical setup (b)

As laser source we used a nanosecond laser, λ = 532 nm, pulse duration 9 ns, repetition rate 10 Hz. By using lens, we produced a focused beam diameter 400 µm with an incident angle of 45°. The fluence was set to 190 J/cm².

DLC film was deposited on a polished SAE 4340 steel substrate by ablating a 99,998%C high purity graphite's target during 30 min at room temperature. The distance between target and substrate was 4 cm.

The graphite target of 30 mm diameter and 10 mm thickness showed in Fig. 2 (a) was mechanically polished with sand papers of grit 600, 800, 1000 1200, 1500, 2000 and 4000, to avoid scattering losses of incident laser beam as indicated in [7]. The steel substrates of 25 mm diameter and 5 mm thickness were mechanically polished with sand papers of grit 400, 600, 800, 1000, 1200 and 1500 and then they were finally polished with 0.1 and 0.05 μm alumina suspension. The substrates showed in Fig. 2 (b) were cleaned in an ultrasonic bath of high purity acetone during 10 min before the film deposition.

(a)

(b)

Fig. 2 Polished graphite target (a) and steel polished substrate (b)

On the surface: The film produced has been tested by Raman spectroscopy with laser of λ = 514 nm. The hardness of the steel substrate and DLC layer were measured using nanoindentation Anton Paar equipped with a diamond Berkovich tip loaded to penetrate at about 90 nm. The adhesion of the film has been tested by scratch test, within scratch length of 2 mm, test speed of 1 mm/min and normal load increased linearly from 100 mN to 1000 mN. Tribological properties have been evaluated using a standard tribometer Anton Paar mounted with a 3.00 mm hard metal ball, full amplitude set to 4.00 mm, normal load to 2.00 N and stop condition to 10.00 m. We used also a roughness profiler with cutoff set to 0.25 mm to measure the film rough.

On the film section: First, we cut small samples using a high precision cutter Buehler ISOMET 1000, with a diamond cutting disc of diameter 152 mm, thick 0.5 mm manufactured by Arotec.

To capture the images of the microstructure of the film and substrate we used optical and SEM microscopy. Samples have been mechanically polished with sand papers of grit 400, 600, 800, 1000, 1200 and 1500. Then they were polished with 0.05 μm colloidal silica suspension. And then they were attacked by 2% nitric acid solution by scrubbing with a wet cotton during 20s to reveal the grains boundaries of the steel substrate.

III. Results and Discussion

To investigate the quality of the DLC film, a Raman spectroscopy has been conducted. As shown in Fig. 3 the Raman spectrum of DLC film has a broad band at 1500 cm^{-1}, indicating a significant fraction of sp^3 bond present in tetrahedral amorphous carbon (ta-C).

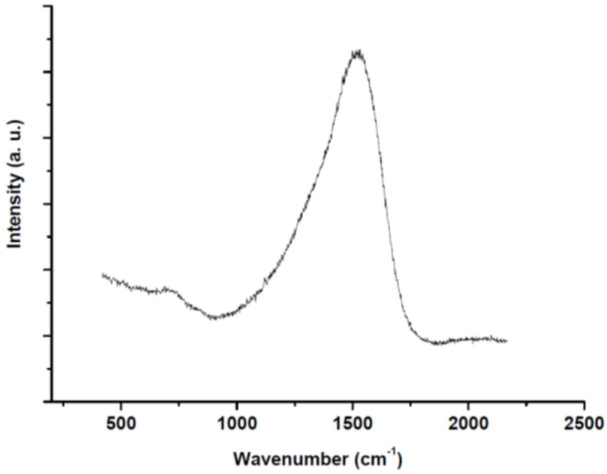


Fig. 3 Raman spectroscopy of DLC film

The hardness of the steel substrate and of the DLC layer are shown on Table I. The hardness of the film is more than two times bigger than the substrate, but the result may be considered low when compared with reported in [8].

TABLE I
NANOINDENTATION TESTS RESULTS

	max load [mN]	HIT [MPa]	HVIT [Vickers]
4340 steel	3.00	3865.5	358
DLC film	2.00	9569.4	880

The result of scratch test is shown in Fig. 4. At 102.3 mN superficial risk starts, at 453.5 mN the film starts cracking, at 776.1 mN delamination begins and at 6661.3 mN delamination is complete. These results indicate a good adhesion strength for a pure DLC film produced by a nanosecond laser if compared with another report [1].

A roughness profile of the DLC was obtained. After five measurements, the total rough mean was Rz 0.43 μm and apparent roughness mean was Ra 0.0295 μm.

The performance of steel substrate on tribometer is in Fig. 5 (a). The abrupt increasing of friction coefficient at the beginning indicates the creation of a third part in the wear scar (Fig. 5 (b)) due to crackled parts of the surface.

The performance of DLC film on tribometer is in Fig. 6 (a). As expected, friction coefficient had a better performance when compared to steel substrate; in Fig. 6 (b) the DLC coating suffered insignificant wear.

The DLC film can be seen on Fig. 7. The images were obtained from an optical microscopy. Using this technique, the measured thickness was about 6 μm. The image on Fig. 8 shows the film surface and the indentation mark from scratch test.

The film images from SEM are on Fig. 9. Using this technique, the measured thickness was about 2.4 μm. It can be seen a thin connection layer between the substrate and the DLC film, where it is expected that metallurgical bonding occurs.

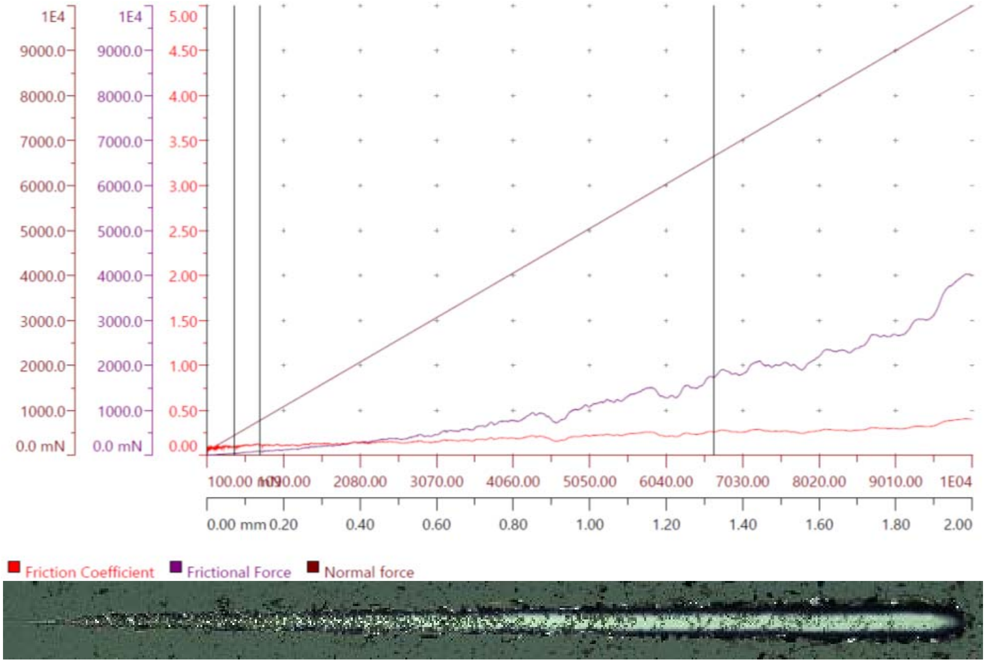
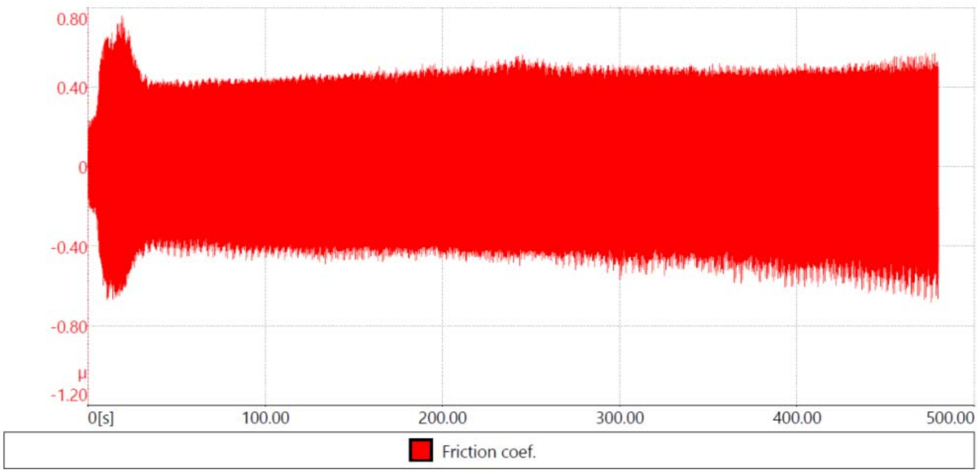


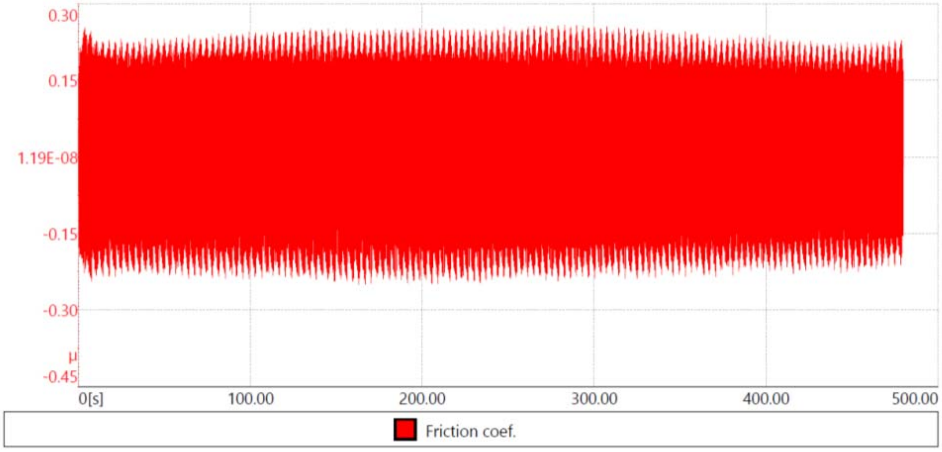
Fig. 4 Scratch test of DLC film



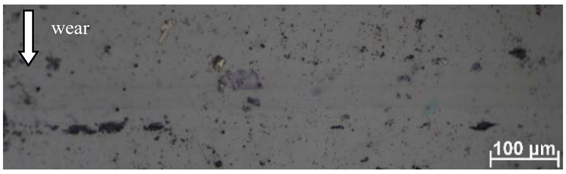
(a)

(b)

Fig. 5 Tribological test of steel substrate (a) friction coefficient performance(a) and optical microscopy image of the wear scar (b)



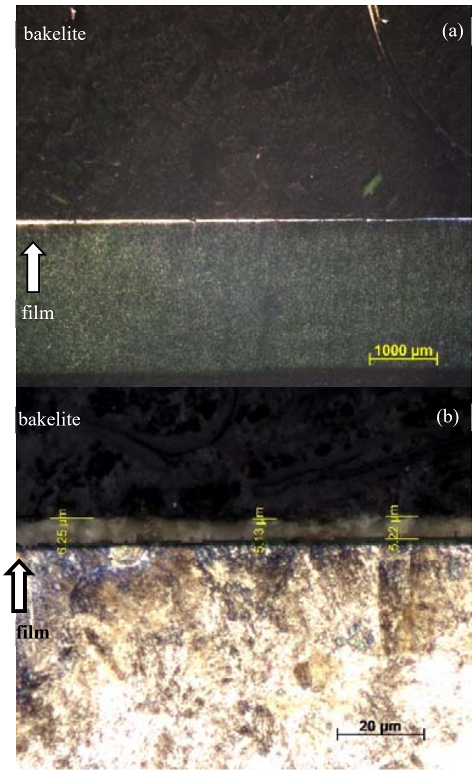
(a)



(b)

Fig. 6 Tribological test of DLC surface friction coefficient performance (a) and optical microscopy image of the wear (b)

Despite the PLD technique was efficient to fill superficial valleys in the substrate; during the polishment of the samples some parts of the film were cracked.



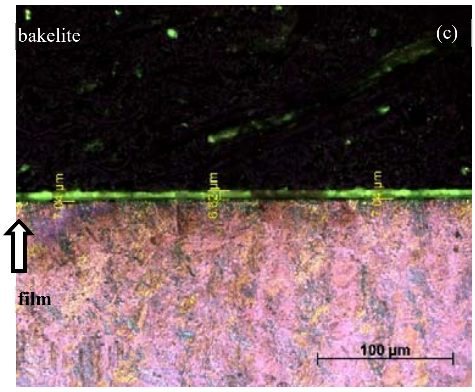


Fig. 7 Optical microscopy images of film at different magnifications

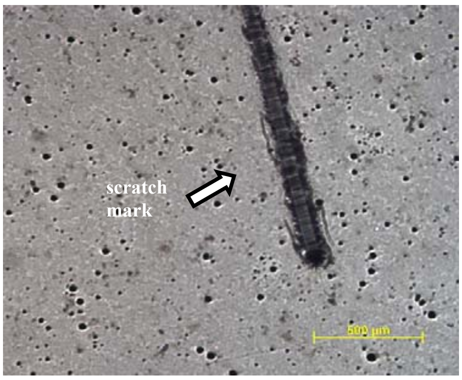


Fig. 8 Optical microscopy image of film surface

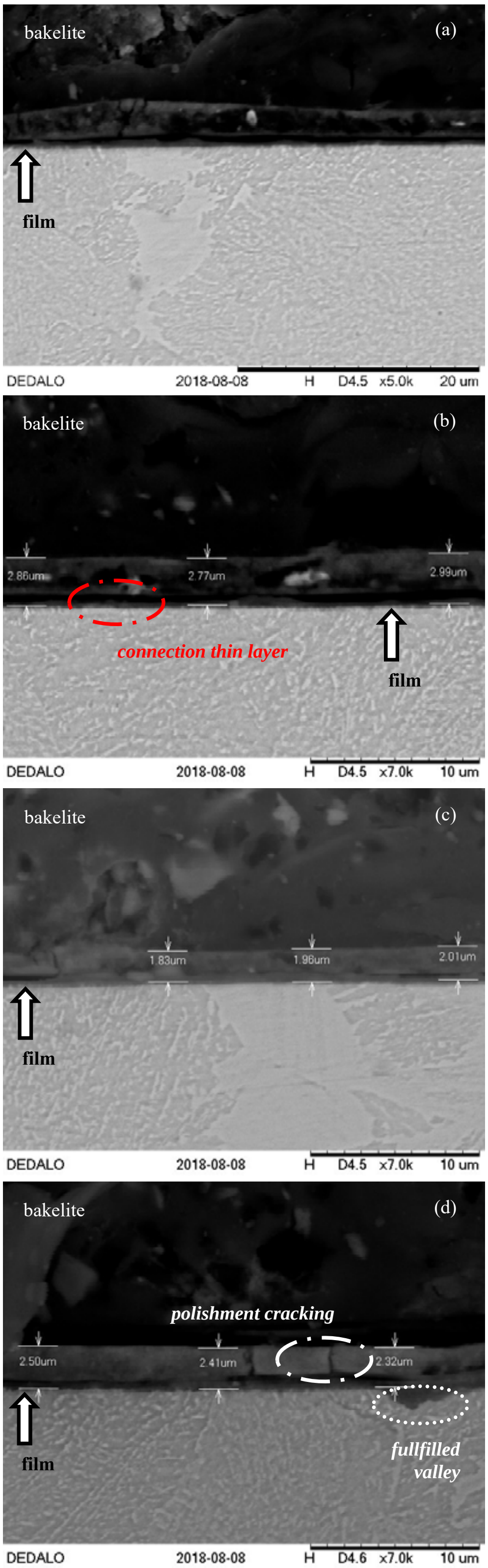


Fig. 9 Film cross section SEM images at different magnifications

IV. Conclusion

The characteristics of a DLC coating synthetized by Nd:YAG pulsed laser have been investigated. The main conclusions of this study are:

(a) The micrograph image obtained by chemically attacked sample was efficient to identify the metallurgical bond;
(b) As expected, this technique associated with the low pressure using a vacuum chamber and the purity of graphite has a great influence on fraction of sp^3;
(c) The hardness of the film improved the surface hardness achieving good results for a pure DLC, when compared with reports of doped DLC [9] and DLC with nanostructured substrate [10];
(d) The precautions adopted in this study (laser parameters and surfaces polishing) have a great influence in tribological properties and in adherence tests performance, as already reported [2], [8], [11], [13]-[15] and tribological aspects [12];
(e) The parameters of the laser, as frequency, time and fluence define the film thickness.

The investigation of laser parameters, using design of experiments methods is recommended to write an optimal curve for PLD process for specific applications, as already reported in [3].

Acknowledgment

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