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COMBINING SCRATCH, WEAR, FRICTION AND NANOINDENTATION TESTS IN THE ASSESSMENT OF TRIBO-MECHANICAL PROPERTIES OF DLC AND DLC-Si FILMS[1]

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Abstract

This work addresses the evaluation of the tribo-mechanical performance of DLC and DLC-Si films deposited onto AISI 4340 steel by the use of a combination of tribological and mechanical tests. Polished AISI 4340 steel substrates were submitted to a plasma nitriding process prior to the deposition of DLC and DLC-Si thin films in a PECVD system using N_2, CH_4 and CH_4 + 5 %v. SiH_4 plasmas. Scratch test results showed higher critical load values for the DLC-Si films (8.7 N) when compared with DLC (5.3 N), indicating better adhesion to the substrate when Si is present in the DLC structure. Nanoindentation tests showed typical hardness values for DLC films (16 - 21 GPa) and lower hardness values for the DLC-Si films (13 - 15 GPa). Average friction coefficient was 0.13 for DLC films and 0.05 for the DLC-Si films, both much lower than the values found for the uncoated AISI 4340 steel (0.71), as expected. Wear rates obtained for DLC films (0.1×10^{-6} mm^3/Nm) were lower than the ones found for DLC-Si films (0.47×10^{-6} mm^3/Nm). Obtained data from scratch, wear, friction and nanoindentation tests are set together as a valuable tool for the study of thin films.

Keywords: Diamond-like carbon; Mechanical properties; Tribological properties.

COMBINANDO ENSAIOS DE RISCAMENTO, DESGASTE, ATRITO E NANOINDENTAÇÃO NA AVALIAÇÃO DAS PROPRIEDADES TRIBO-MECÂNICAS DE FILMES DLC E DLC-Si

Resumo

O presente trabalho aborda a avaliação do desempenho tribo-mecânico de filmes DLC e DLC-Si depositados sobre aço AISI 4340 por meio da utilização de uma combinação de ensaios de tribologia e mecânica. Substratos de aço AISI 4340 polidos foram submetidos a um processo de nitretação a plasma, antes da deposição de filmes DLC e DLC-Si em um sistema PECVD usando plasmas de N_2, CH_4 e CH_4 + 5%v. SiH_4. Resultados do teste de riscamento apresentaram valores de carga crítica maiores para os filmes DLC-Si (8,7 N) quando comparado com DLC (5,3 N), indicando uma melhor adesão ao substrato quando o Si está presente na estrutura do DLC. Testes de nanoindentação mostraram valores de dureza típicos para filmes DLC (16-21 GPa) e valores menores de dureza para os filmes DLC-Si (13-15 GPa). O coeficiente de atrito médio foi de 0,13 para os filmes DLC e 0,05 para os filmes DLC-Si, muito menor do que os valores encontrados para o aço AISI 4340 não revestido (0,71), conforme esperado. Taxas de desgaste obtidas para os filmes DLC ($0{,}1 \times 10^{-6}$ mm^3/Nm) foram inferiores aos encontrados para filmes DLC-Si ($0{,}47 \times 10^{-6}$ mm^3/Nm). Os dados obtidos a partir dos testes de riscamento, desgaste, atrito e nanoindentação são confrontados juntos servindo como uma valiosa ferramenta para o estudo dos filmes finos.

Palavras-chave: Carbono do tipo diamante; Propriedades mecânicas; Propriedades tribologicas.

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1 INTRODUCTION

The increasing demand for high technology materials with specific performance and characteristics in various types of environments may require that these materials possess near-surface properties different from their bulk properties. Technology involving thin and thick coatings and modified surfaces which alter the properties of materials is an important field of research.

In this context, hard coatings such as diamond-like carbon (DLC) have been studied for many years to be used as protective or lubricant layers for several types of applications.[1,2] DLC films have very interesting properties, such as high hardness, chemical inertness, low friction coefficient and biocompatibility. In recent years, incorporation of other elements to the DLC structure like Si (DLC-Si films) have brought improvements such as lower residual stress, higher thermal resistance and even lower friction coefficient.[3,4]

Development and characterization of hard coatings like DLC and DLC-Si require a broad range of tools that, when used together, can provide a very complete understanding of the mechanical and tribological behavior of these films. In this way, the Materials Metrology Division of Inmetro has set a combination of techniques which is expected to fulfill the need for tribo-mechanical characterization of hard coatings.

In this work, DLC and DLC-Si films were characterized by a combination of these techniques: scratch, wear, friction and nanoindentation tests.

2 MATERIALS AND METHODS

AISI 4340 steel discs of 40 mm diameter were surface finished with #1200 sandpaper and diamond paste of 3 µm and 1 µm sizes before being submitted to plasma treatment. Three pre-deposition treatments were undertaken in the same PECVD system: cleaning with argon plasma, nitriding and deposition of amorphous silicon layer (a-Si:H). Argon plasma cleaning was applied on the samples for 30 min. Nitriding was conducted using N_2 plasma for 2 h at a negative self-bias voltage of 800 V and pressure of 2 Pa. An amorphous silicon (a-Si:H) layer was then produced prior to the deposition of DLC and DLC-Si films by application of a SiH_4 plasma for 3 min. Self-bias and pressure were kept the same as for the nitriding process.

Finally, DLC and DLC-Si thin films were deposited in the same PECVD system using CH_4 and CH_4 + 5 %v. SiH_4 plasmas, respectively. For both types of films a negative self-bias voltage of 800 V and a pressure of 2 Pa were used. Film thickness varied from 2.5 to 3 µm.

Scratch tests were conducted with a Rockwell C stylus by varying the load linearly from 1 to 15 N, with constant velocity of 0.05 mm/s and total scratch length of 5 mm. Critical load values (Lc_1) were obtained by comparing optical micrographs taken from the sample surface after the tests, to the measured friction force. Delamination of the film was visible and coincided with the increase in the scattering behavior of the friction force. Average value of critical load was taken from four tests of each sample material.

Wear and friction were measured in a reciprocating ball-on-plate configuration, constant load of 5 N, wear stroke of 5 mm and frequency of 10 Hz over 100,000 cycles. AISI 52100 steel balls of 6.35 mm diameter (nominal hardness of 64 HRC), were used as counter-body. The wear rate of the films was calculated by measuring

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the wear volume of the scars with a 3D profiler. Average wear volume was taken from three tests of each sample material.

Nanoindentation tests were carried out with a Berkovich stylus using loads from 7 to 50 mN in order to reach to depths from the surface of around 10% of film thickness. Oliver-Pharr method[5] was used to determine hardness values. Average hardness related to each indentation load was taken from five tests of each sample material.

3 RESULTS AND DISCUSSION

Figures 1 and 2 show the results from scratch tests of DLC and DLC-Si films, respectively. As it can be seen, the upper part of the figures shows an optical micrograph of one of the scratches and, at the bottom, the behavior of the friction (Fx) and normal (Fz) forces as a function of the running distance. A vertical line also marks the point where the first detachment of the film occurs, identified as the point of critical load, coinciding with the increase of the scattering in the Fx graph.

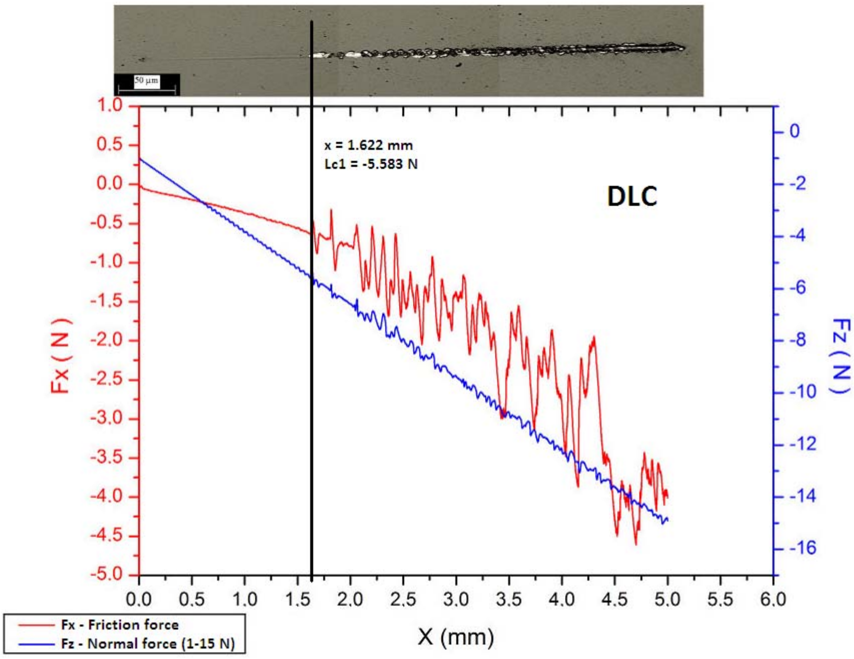

Figure 1. Scratch test results for DLC films: optical micrograph of the scratch and normal (Fz) and friction (Fx) forces as a function of the running distance (X).

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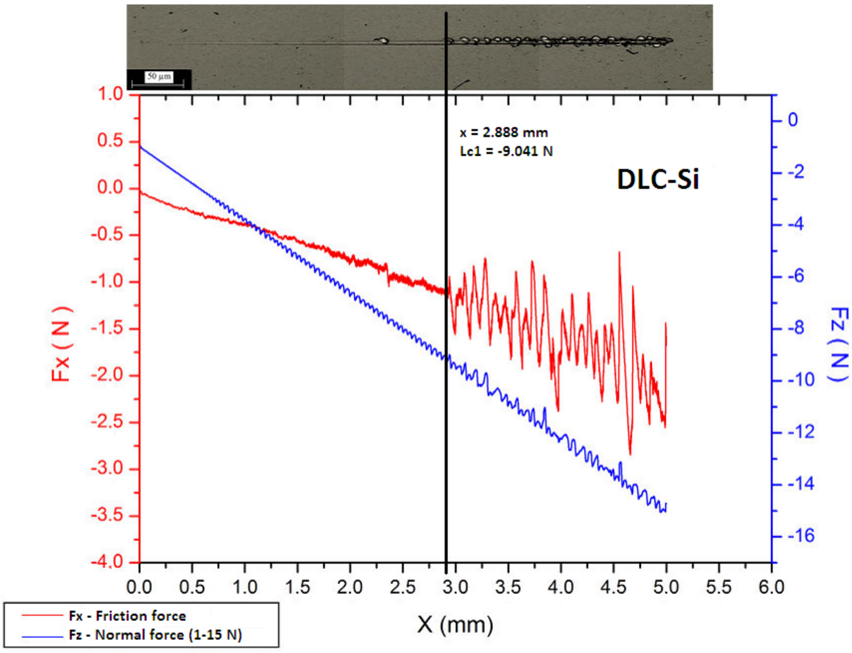

Figure 2. Scratch test results for DLC-Si films: optical micrograph of the scratch and normal (Fz) and friction (Fx) forces as a function of the running distance (X).

Figure 3 shows all the critical load values (Lc_1) obtained from the scratch tests, as well as the average and standard deviation (error bars) for each type of film. As one can notice, higher critical load values for the DLC-Si films (8.7 N) were obtained when compared with DLC (5.3 N). This result indicates better adhesion to the substrate when Si is present in the DLC structure. This could be correlated with a decrease in residual stresses caused by the inclusion of Si to the DLC carbon network.[(4)] Less stressed films generally present better adhesion to their substrates than the stressed ones.

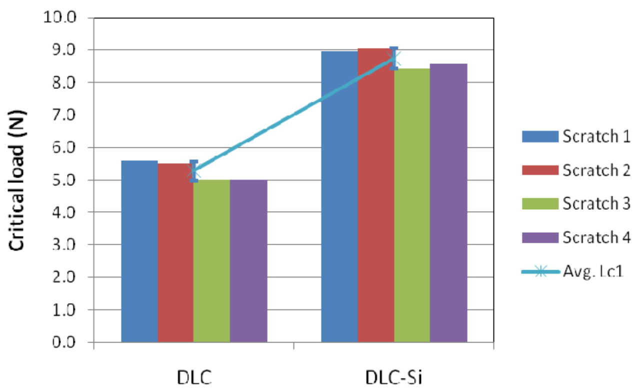

Figure 3. Critical load (Lc_1) values for all scratch tests as a function of type of film: DLC or DLC-Si.

The results from nanoindentation tests are shown in Figures 4a and 4b. Figure 4a presents the average hardness values obtained for DLC and DLC-Si as a function of contact depth. Typical hardness values were found for the DLC films (16 to 21 GPa). On the other hand, the DLC-Si films presented relatively lower hardness values (13 to 15 GPa). In fact, the addition of Si to the structure of DLC films can cause hardness decrease.[(6)] Anyhow, the hardness values for DLC-Si are still in a

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good range for tribological applications of hard films, being greater than the hardness of the steel substrate (approx. 7 GPa).

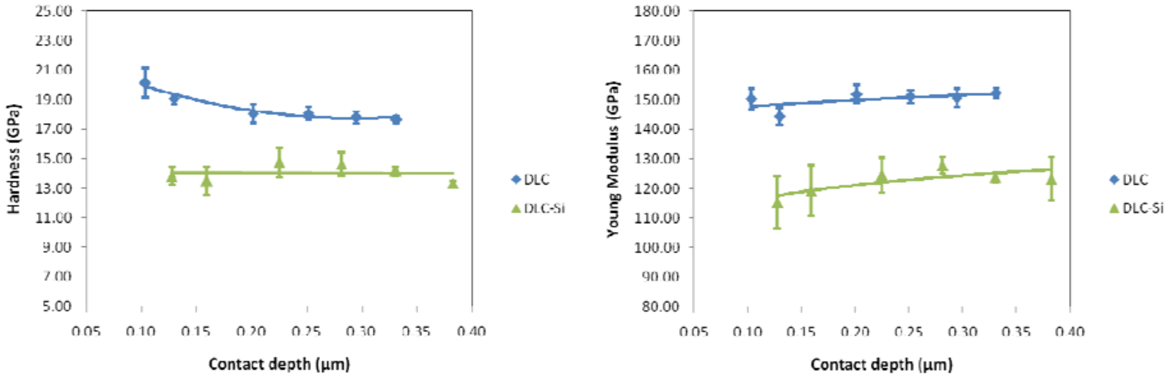

Figure 4. (a) Hardness and (b) Young modulus as a funtion of contact depth for DLC and DLC-Si.

Figure 4b shows the average values for Young modulus found for DLC and DLC-Si films as a function of contact depth. Young modulus showed similar behavior as for hardness values, with the DLC films presenting higher values (145 to 155 GPa) than the DLC-Si films (115 to 130 GPa).

Figure 5 shows the results for friction coefficient as a function of test time along the 100,000 cycles. Figure 6 shows the friction coefficient behavior for the first 1,000 s time. As it can be noticed, the average friction coefficient was 0.13 for DLC films and 0.05 for DLC-Si films, which are much lower than the values found for the uncoated AISI 4340 steel (0.71). This reflects the high applicability of these coatings as solid lubricants for steel parts. DLC-Si coatings presented very low (0.05) friction coefficient, lower than for DLC films (0.13), similarly to what was found by Oguri and Arai.[7]

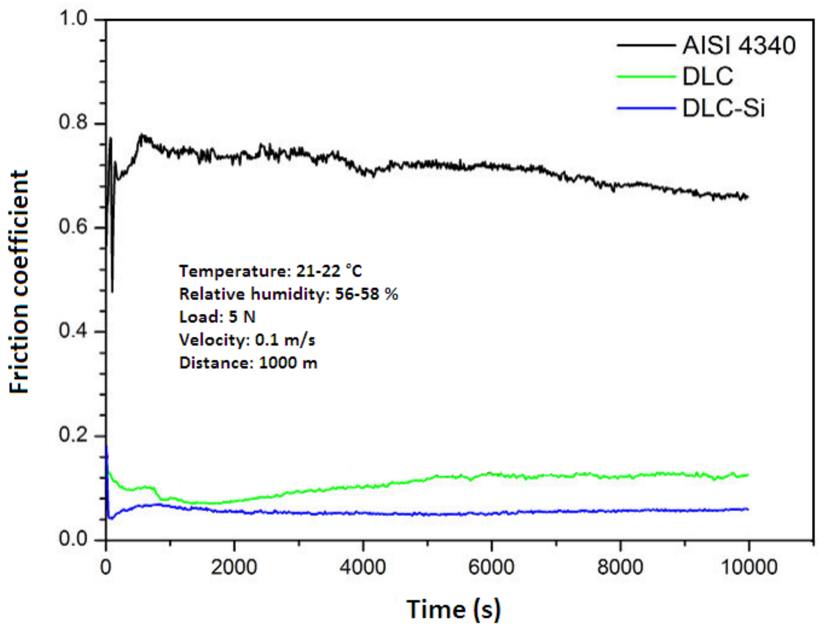

Figure 5. Friction coefficient as a function of time for AISI 4340 steel: uncoated and coated with DLC and DLC-Si films.

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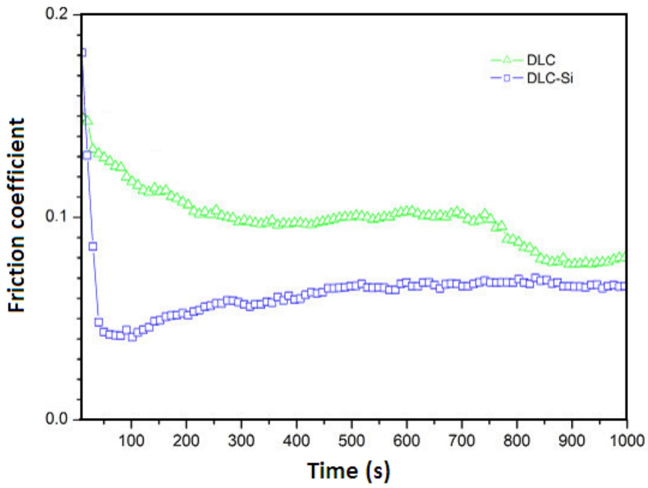

Figure 6. Friction coefficient as a function of time for DLC and DLC-Si films for the first 1,000 s time test.

Wear rates results are shown in Figure 7 for each type of film. DLC films presented lower wear rates (0.1×10^{-6} mm^3/Nm) than the ones found for DLC-Si films (0.47×10^{-6} mm^3/Nm). DLC-Si films are softer than DLC and consequently more suitable to wear, despite their lower friction coefficient. Nevertheless it is important to notice that both films performed in a very small wear range typical of lubricated systems.(8)

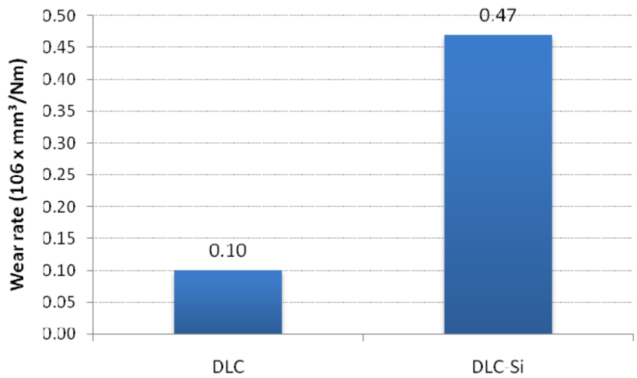

Figure 8. Wear rates for DLC and DLC-Si films.

4 CONCLUSIONS

In this work, different techniques were applied in order to assess a comprehensive tribo-mechanical characterization of thin films. Two films, DLC and DLC-Si, were compared in terms of mechanical properties, wear resistance, friction behavior and adhesion resistance. DLC film was found to be harder, stiffer and more wear resistant than DLC-Si. These characteristics make DLC a worthwhile thin film for tribological applications. In spite of this, it is emphasized that there are two additional essential characteristics that must be taken into account before making a decision on the best application. The presented results show that a film of relatively low wear resistance and inferior mechanical behavior, such as the DLC-Si film, can become more attractive if one looks at its better friction behavior and adhesion resistance, when compared to the DLC film. Therefore, this work demonstrates that a

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complete and reliable characterization of tribo-mechanical properties of thin films can only be assessed by combining results from different techniques, which can be of crucial importance for making a decision for the best tribological application.

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