BEHAVIOUR OF SI CONTAINING DLC FILMS IN INTERNAL COMBUSTION ENGINE COMPONENTS

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Abstract. The increase of energy demand combined with the no renewal of oil sources demands that internal combustion engines are each time less pollutant and at the same time economic. In the other hand, the development of more compact and powerfull engines causes higher mechanical efforts of the engine working parts. An alternative to improve tribological behavior of surfaces is the application of new materials with interesting properties in severe systems as engines. In this work, we have investigated tribological behaviour of a DLC-Si film deposited on steel substrate by a DC PACVD method. Linear reciprocating tribometer tests have showed that there is a great decrease in friction coefficient for coated samples. The lifetime curve has demonstrated the destruction of the film for values above 875000 cycles. It was observed the increase of hardness for coated samples in 3 times. SEM immages suggest a mechanism of transfer film, which is responsable for the low friction of DLC films. Raman spectroscopy curve shows peaks D and G in shifts 1333cm⁻¹ and 1531 cm⁻¹, which are typical values for DLC.

Keywords: DLC, engine components, tribology, Si interlayers

1. Introduction

New ambiental rules, together with the eminence of petroleum lack in the world and the technologic exigencies for more efficient, compact and powerful engines, have provoked many studies in search for improvements that attend all these aspects. Becker (2004) shows is his work that, inside a internal combustion engines, 15% of the fuel energy is mechanically lost as friction. Of these, 10% occurre in the valve train, in the mechanical contact between the cams of the command axle and the superior surface of the valve lifters. Becker (2004), Podgornik and Vizintin (2003) and Taylor (1998) realized studies about viable alternatives to enhance the efficiency of these engines, as coatings in certain components, in trying to prevent friction and wear between them. According to Podgornik (2001), these coatings must present hardness, wear resistant surface, good friction properties, and, still, mechanical resistance. DLC (diamond-like carbon) is the most likely coating now used, which is mentioned in several studies as an option to internal combustion engines components, because of their great tribological behavior in severe environments, as showed in Taylor (1998), Podgornik (2001) and Malaczynski (1997). DLC have already been mentioned in the studies of Kodai et al. (2001) and Yasuda et al. (2003) as applicable coatings in valve lifters, obtaining good results for friction and wear. However, coatings like DLC present failures as lack of adhesion and precocious breakings, as showed by Grill (1998) and Robertson (2002). However, elements as Si help to prevent breakings because they diminish film's internal tension, as showed by Kim et al. (1999) and Wu et al. (1998). The introduction of interlayers also helps to enhance adhesion of film and substrate, as mentioned by Silva et al. (2004) in their study.

It was created a coating formed by 3 layers: a first layer of amorphous Si, followed by a second layer formed by a a-c:H film doped with Si, (a-C:Si:H), and, finally, a last layer of a a-C:H DLC film.

This coating was tested in a reciprocal linear tribometer, with a speed of 5cm/s and a contact pressure of 1,42 GPa. Curves for friction and wear were gathered. The results of the tribological characterization are discussed in next.

Although we are leading with a engine component, the realized test does not intend to simulate the real conditions, because it is not possible to obtain high frequence and temperature. The main objective of this work is to validate the coating deposition process and compare the tribological behaviour with non-coated surfaces.

2. Experimental

The thirteen samples tested in this study were the lifters themselves, supplied by an automotive industry. The lifters are cylinders, with diameter and height of 35mm. The used material is a 16MC5 DIN 17210 steel. The top side is cemented, quenched and tempered and it presents a hardness between 6,8 and 8,2 GPa and maximum surface roughness of $Rz = 0.3\mu m$. The samples were polished and an average roughness Ra was 0,06 μm . Ten samples were coated. The films were deposited by a DC PACVD (direct current plasma-assisted CVD). The internal pressure in the chamber was of $10^{-3}mbar$, the Bias Volt was 500V, the substrate temperature 180 °C and the precursor gas was Acetylene. The nano-mechanical properties were measured by nanoindentation method, using a Berkovich indenter and maximum load of 0,405N. It was also realized Scanning Electron Microscopy (SEM) to obtain some pictures showing the film's relief and details of the groove wear.

Sliding tests were performed in a reciprocal linear tribometer and the counter body was a 100Cr6 steel ball of 6mm diameter. The tribosystem studied in this work is presented on Figure 1. For each motor cycle, the counter body passes twice over a point of the sample. So, the number of cycles is the double of the number of motor cycles. The samples coated and in the original state (without polishing), were submitted to tests with speed of 5cm/s and Hertzian contact of 1,42 GPa, and the number of cycles varying from 31750 to 1 million. They were realized tests in room temperature (23°C), and relative humidity varying between 50 and 65%. The cleaning of samples and balls was realized by an ultrasonic cleaner, using acetone. It was carried out Raman spectroscopy of the film and the curves were analyzed to the shifts of peaks D and G. The hydrogen quantity in DLC film was measured by Nuclear Reaction Analysis (NRA). The film's density and thickness were measured in a Tandetron 3 MV ion accelerator. The tribometer software has calculated itself the wear rates. The perfilometry of each of groove was measured. The value of cross section area was used to this calculus.



Figure 1. Tribosystem composed by cam of the command axle in contact with the valve lifter.

3. Results and Discussion

Figure 2 shows the film hardness determined by nanoindentation method. The maximum hardness values changes between 18 and 24 GPa. The scatter can be explained by the film heterogeneity. The Modulus of Elasticity of the film was around 263 GPa. The curve shows a trend to stabilization around 8 GPa, which is the hardness of the quenched and tempered steel substrate.



Figure 2.Dependence of film hardness value with depth from the surface

The hydrogen content in film measured by NRA is of about 21,9%. The RBS analysis shows the film thickness of 0,72µm and density of 1,8g/cm³. This density value is similar to the one after Robertson (2002) at similar conditions. A SEM immage with three layers is shown in Figure 3. It was observed that the thickness of each layer is different.



Figure 3. Scanning Electron Microscopy of the multilayer film.

Figure 4 shows Raman spectroscopy curve for the DLC film. The curve deconvolution shows peaks D and G located respectively in shifts 1333 cm⁻¹ and 1531 cm⁻¹, which are values characteristics for DLC, as mentioned in Robertson (2002), Druza *et al.* (2004) and Ferrari *et al.* (2000).



Figure 4. MW Raman spectroscopy for multilayer Si and DLC film

3.1. Friction

Figure 5 shows friction evolution for different test sequences, with number of cycles varying between 125 thousand and 1 million cycles. While the friction coefficient is around the value of 0,15 for coated samples, at the same time the uncoated samples show a friction coefficient of around 0,5. The "saw" form of too many curves suggests the formation and consumption of transferred films, which are responsible by the low friction of DLC films.



Figure 5. (a) Friction curve for some number of cycles. Curve from A to E – coated, curve F – uncoated. (b) Friction curve of 1 million cycles test, showing formation and consumption of tranferred films.

3.2. Wear

Cross section areas of each groove were calculated by integrating the profiles of each groove. These values were used for calculation of wear rates. The profiles were obtained by perfilometry, as shown in Figure 6.



Figure 6. Perfilometry of groove formed while tribological test. 875000 cycles, Load = 10N, Hertzian contact = 1,42 GPa, V=5cm/s.



Figure 7. (a) Aspect of groove formed during the tribological test, (b) Detail of wear, suggesting film transference. Figure 7(a) shows an overview of 500000 cycles test wear track. Figure 7(b) shows a detail, which suggests a transfer mechanism.

The wear curve is shown in Figure 8. Values around 10^{-7} mm³/N.m are compatible with the literature to a:C-H hydrogenated alloys, after Robertson (2002), Zhang and Tanaka (2004). From 875000 cycles, it is verified a complete destruction of the film, as can be observed by the frictional behaviour in Figure 5(b). Besides that, it is a typical surface lifetime curve .



Figure 8. Wear curve for DLC + Si film

4. Conclusions

It can be observed using nanoindentation method, the increase of hardnes (3 times) in relation to uncoated sample. The results show that it can be found the hardness of the substrate for bigger displacements into the surface (8GPa).

It was also observed some heterogeneities in the film, mainly relationed to friction behaviour. A probable explanation of the results obtained would be the lack of homogeneity in thickness and chemical composition into the film.

The film utilized in this work does not present a great wear resistance because is completely destroyed after 875000 cycles. On the other hand, it was observed a significant fall in friction coefficient from 0,6 to 0,15. In an internal combustion engine component, this can mean a lower fuel consumption. The results are not conclusive, because these are preliminary tests, looking for best structure and better deposition process, trying to optimize the tribologic response.

In spite of the film does not present great wear resistance, the experimental methods employed in this work contribute to the characterization of amorphous carbon films.

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