EVALUATION OF THE MECHANICAL BEHAVIOUR OF A DLC FILM ON PLASMA NITRIDED AISI 420 WITH DIFFERENT SURFACE FINISHING

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Introduction

Diamond-like-carbon films (DLC) are hard coatings with a very low friction coefficient, a high wear resistance and chemically inert. However, these coatings have adhesion problems on metallic substrates, because the high residual stresses in the DLC films cause failure of the interface between the film and the substrate. Different methods have been tested to improve the adhesion: deposition of interlayers, multilayer coatings between the substrate and the DLC film have been used as interface, chemical and physical modifications of the carbon coating or diffusion treatments of substrate surface [1-3].

Plasma nitriding is a well-established method to harden stainless steels and can act as a pre-treatment to increase adhesion and improve tribological behaviour of the DLC coating. Hopwever, the characteristics and phases of the nitrided layer can influence on the adhesion and the mechanical behaviour of the system [4-6].

In this work, the mechanical behaviour of a DLC film deposited over non-nitrided and nitrided martensitic stainless steel with two different surface finishing is studied.

Experimental

The AISI 420 steel was heat treated, but two different surface finishing were accomplished previous to the nitriding process: paper grinding until grit 1000 (named GN) and polishing with 0.5 µm diamond powder (named PN). Another group of samples was coated without nitriding (named C). The nitriding process was carried out in a DC pulsed discharge for 10 hours at temperature of 390 ºC using a gas mixture composed of 20 % N_2 and 80 % H_2. The DLC film was deposited by the PACVD process, with an asymmetrical bipolar DC pulsed discharge described in a previous work [7], using methane and hydrogen as precursor gases. Hardness on the surface was measured with a Vickers microindenter, 50 g load. Both the nitrided layer and film were observed with optical microscope and SEM. The film composition was analysed by Raman Spectroscopy. Adhesion was tested with the methods of Rockwell C Indentation and Scratch Test with loads of 1500 N and 35 N respectively.

The mechanical behaviour was analysed in both linear and rotational reciprocating sliding wear tests. The linear reciprocating sliding tests were performed in a self-made machine using a WC ball 5mm in diameter as counterpart with 12 N load. The amplitude was set in 0.5 mm and the frequency in 11.7 Hz. The rotational sliding wear tests were carried out in a pin on disk tribometer, with the same WC ball as counterpart and 5 N load. The track radius was set in 5 mm, the tangential velocity in 10 cm/s and the total wear length was 500 m.

Results and discussion

The hardness of the DLC film reached 1400 $HV_{0,05}$. This value corresponds to a composite hardness because the indentation depth exceeds 10% of the film thickness. The films had about

20% hydrogen content, typical of these CVD processes. In the Raman Spectrum for the DLC films (not shown) the two bands known as D and G bands well positioned could be observed, indicating a good quality film as already reported by some of the authors [7]. The film thickness varied between 2.6 and 3 microns (Figure 1) with a regular interface with the previous nitrided layer. The nitrided case did not seem to affect the film growth, since it had the same width on the three types of samples.
Regarding the nitriding process, in the polished samples with a roughness of 0.026 µm, the layer thickness was about 11 µm (Figure 2) and only 8 µm in the grounded ones which had a roughness of 0.033 µm. It seems that the polished sample allowed better nitrogen diffusion even though the surface hardness was almost the same for both samples, 1150 $HV_{0,05}$.

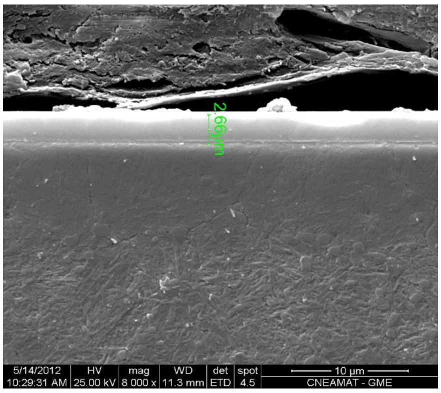


Figure 1. SEM micrograph of the coating on the GN sample

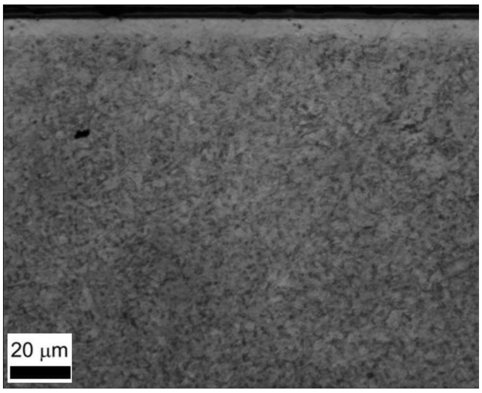
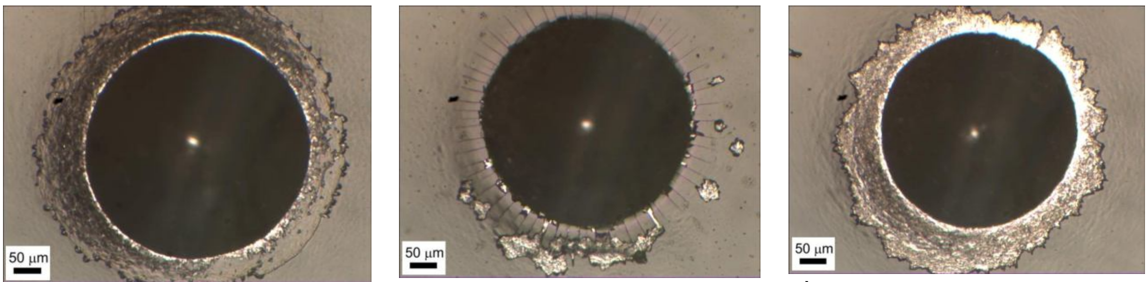


Figure 2. Optical micrograph of the nitrided layer in the PN nitrided sample

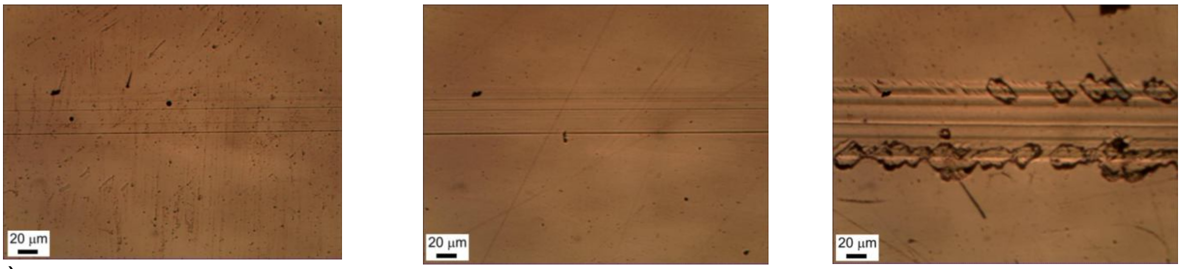
The adhesion quality of the DLC film on the PN sample resulted better than on the C sample and the GN sample (Figure 3). In the PN sample, the adhesion was acceptable according to the VDI standard, eventhough radial cracks can be observed in the coating around the indentation and the film was detached in small regions. This was expected because the nitrided layer acts as a hardness gradient between the soft substrate and the hard film. The reason for the bad adhesion on the grinded and nitrided sample -almost as bad as the non nitrided and coated sample- could be related to the minimization of interfacial energy on sharp convex surfaces, or the presence of surface oxides and contaminants which were removed after polishing, according to other researchers [8].



a) *b)* *c)*

Figure 3. Optical micrographs 100x of the Rockwell C indentation on the samples: (a) C, b) PN, c) GN.

In the scratch test, with a minor load, the film on both GN and PN samples showed better adhesion than on the only coated sample (Figure 4). In the C sample, the failure mode could be described as recovery spallation. This mode is associated with the elastic recovery that occurs when the indenter is moved over the coating surface, and it depends on the plastic deformation in the substrate and the cracking that appears in the coating [9].



a) b) c)

Figure 4. Optical micrographs 200x of the scratch test tracks: a) PN, b) GN and c) C samples.

In the linear reciprocating sliding wear test, the wear loss was smaller in the coaetd samples (PN, GN and C) compared with a nitrided sample without coating, as it can be seen in the wear track profiles in Figure 5. But the two duplex samples PN and GN had a slightly better wear behaviour than the C sample. As it is well known this is because the nitrided layer improves the load capacity of the coating and thus its wear resistance. Anyway, there is no difference between the nitrided samples grounded until grit 1000 (GN) and the others polished with 0.5 µm diamond powder (PN). This can be explained by the fact that in the region next to the surface, the hardness profile of the two nitrided layers is similar, as it is shown in Figure 6.

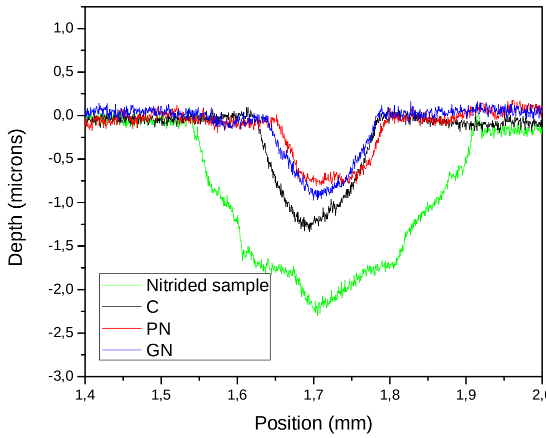


Figure 5. Depth profile of the linear sliding wear tracks.

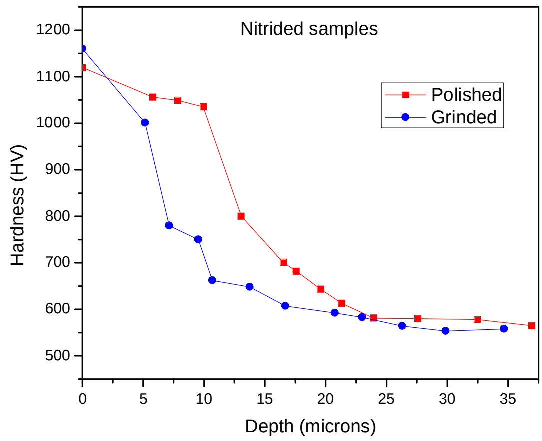


Figure 6. Hardness depth profile of the two nitrided samples.

In the pin on disk tests, with 5 N load, the wear track was almost invisible over the three coated samples, and thus the wear loss was impossible to calculate. The friction coefficient was registered during the test and it is shown in Figure 7. It can be observed that a steady value was reached after a few minutes and all the values are under 0.15. In the same experiment over the nitrided sample without coating μ_D was about 0.8. It can also be observed, the coated sample C has the lowest coefficient with a mean value of 0.08. This can be attributed to the roughness previous to the DLC deposition, which was smaller in the non nitrided sample, because the DC nitriding process always produces a rougher surface due to ion bombardment. After the CVD process it was measured that the film copied the surface roughness.

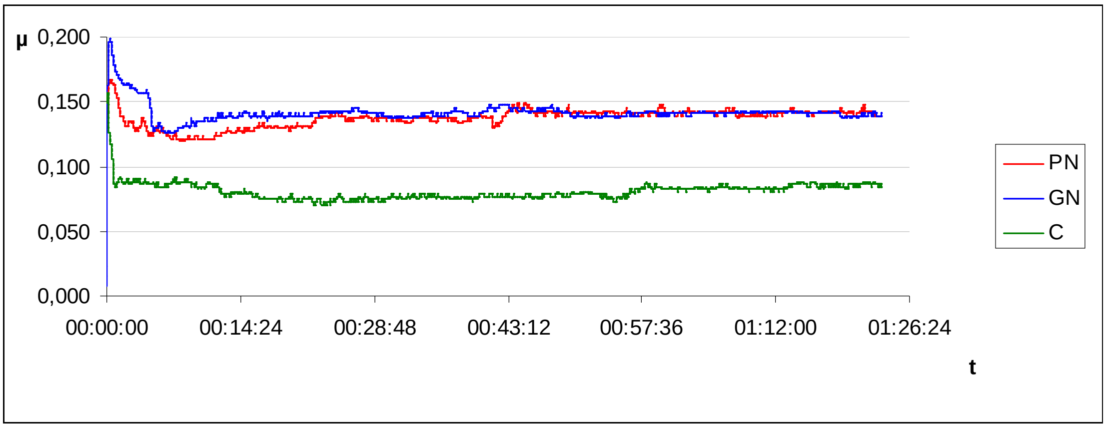


Figure 7. Friction coefficients registered in the pin-on-disk tests for the three coated samples

Conclusions

The results of the tribological tests showed that the coating improves wear resistance compared to the nitriding process alone. The friction coefficient against WC was lowered from 0.08 to 0.15. A comparison between the three coated samples showed that the nitriding pre-treatment of the steel substrates improved even more the wear resistance in the evaluated conditions. The nitrided layer proved to be a good interface that increased the substrate hardness and provided a suitable interlayer. But the adhesion quality was better in the PN sample than that in the grinded sample. The nitrided layer was also wider on the polished samples which proved to allow better nitrogen diffusion.

References

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