INFLUENCE OF TiW INTERLAYER IN ADHESION OF THE DLC COATINGS ONTO AISI 316L STAINLESS STEEL

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1. Introduction

Diamond-like carbon (DLC) films have attracted attention in the last years because of their properties, such as low friction coefficient, high hardness, high elastic modulus, chemical inertness, biocompatibility, and high degree of wear resistance [1]. Nevertheless, the DLC coatings not have good adherence on metallic substrates. This behavior is basically due to difference of the coefficients of thermal expansion between the coatings and the substrates ($0.8x10^{-6} <\alpha<4.5x10^{-6}$ K^{-1}, for 293,15<T<1073,15 K for diamond, and approximately $11x10^{-6}$ K^{-1} for steels) [2]. On the other hands, carbon presents a very large solubility in most ferrous materials and the Fe vapour pressure affects the plasma [3]. Due to this fact, several researches have been made to increase the adherence of DLC coatings onto metallic surfaces. The most common process is to place an interlayer between the substrate and the DLC film [1], [2], [4]–[7].

Another authors have studied as influenced the diffusion in the adhesion [8], [9]. They calculated the diffusion of diverse materials in the Fe and it was suggested that the interlayer should serve as a diffusive barrier between carbon and iron with a mutual diffusion with the substrate. Spinnewyn et al. [8] concluded that is better to use W, Ta, and Cr as interlayer due to their excellent diffusion barriers for the iron from the steel substrates. They obtained that Si and Ti would require very thick layers and they decided to use W and Mo as interlayers, but low adhesion was observed. On the other hands, another authors [9] ensured that Fe has a low diffusion in Cr, Ta, and W; average in Cu, Au, and Ag; and high in Ti. Therefore, a good adhesion was expected between the steel substrate and the titanium interlayer. From that point of view, the interlayer of Ti does not seem to be adequate in comparison to W interlayer. However, at the beginning of diamond growth the formation of TiC and diamond nucleation can form quickly and if the deposition temperature is low, the carbon diffusion may be reduced. Thus, they selected titanium as an interlayer material getting good adhesion.

In order to reduce the coefficient of thermal expansion gradually multi-layers were deposited by several authors [10], [11], but the use of the interlayers made with alloys are not been reported. For this reason, in this work an alloy of TiW was used as interlayer in order to improve adhesion of the DLC coatings onto AISI 316L stainless steel substrates. The influence of the interlayer thickness was studied, using 100, 200, and 300 nm. The TiW interlayers were deposited via R.F. magnetron sputtering, while the DLC coatings using a modified pulsed-DC PECVD system with an additional cathode.

2. Experimental

Samples of AISI 316L stainless steel (0.03 C wt%, 16.7 Cr wt%, 9.5 Ni wt%, 2.9 Mo wt%, and Fe balance) with sizes 1.5 cm x 1.5 cm x 0.6 cm was used as substrate. The substrates were prepared metallographically and cleaned ultrasonically in an acetone bath for 15 minutes before of deposition. A R.F. magnetron sputtering system (13.56 MHz) and $Ti_{10}W_{90}$ target (99.99% pure) were employed to TiW interlayer deposition. The interlayers were synthesized using a pressure in the chamber of 0.19 Pa ($1,9X10^{-3}$ mbar) and applying power of 200 W, while Ar was used as precursor gas for plasma with flow of 20 sccm. The deposition times were selected in order to obtain thicknesses of 100 nm, 200 nm, and 300 nm, respectively (see figure 1). After the interlayer deposition, the substrates were put into a pulsed-DC PECVD reactor with an asymmetrical bipolar pulsed-DC power supply and an active screen for the DLC coating depositions. Before DLC film deposition, the substrates were sputter cleaned in an argon atmosphere for 15 minutes in order to remove any contamination on the surface. DLC films were deposited using methane as precursor gas, a pressure of 5 Pa (gas flow of 5 sccm), and applying a negative voltage of -700 V for 3 hours.

The crystal structure was determined by X-ray diffraction (XRD) using an X-pert Pro Panalytical device operating in beam mode at 5°, with monochromatic line Kα of copper (1.540998 Å) working at 45 kV and 40 mA. The surface morphology was observed using a high-vacuum FEI QUANTA 200 scanning

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electron microscope (SEM), operating at a voltage of 30 kV. The chemical composition of the interlayer was determined via energy-dispersive X-ray spectroscopy (EDX) with energy of 4 kV to 6 kV using a SEM MIRA3. Raman spectroscopy allowed determining the microstructure of the DLC films using a HR Evolution spectrometer and a laser of 514 nm. Scratch test was employed to calculate the critical load using a Rockwell C indenter and varying the applied force, also test of indentation was used.

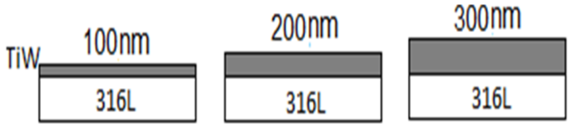

Fig. 1. *Interlayer scheme.*

3. Results and Discussions

A TixWy interlayer was deposited on AISI 316L substrates in order to improve DLC coating adhesion. The composition of the interlayer's were 56 W wt% and 44 Ti wt% for the film with 300 nm of thinness, while in the film with 100 nm 49.7 W wt%, 41.7 Ti wt%, and 8.6 Fe wt% were determined. Surface morphology showed that the coatings did not grow homogeneously, observing zones with high content of W and Ti. X-ray diffractograms are shown in figure 2, presenting a cubic structure and with space Pm-3n group, while a change of the preferential direction for the different thicknesses was detected. Raman spectra showed that DLC coatings with a content of hydrogen between 20 and 30% were deposited. The adhesion strength did not change significantly when different interlayer thicknesses were used, showing a critical load of about 30 N. This preliminary work allowed the deposition of DLC films with good adhesion on surfaces of AISI 316L substrates. Other investigations are being carried out in order to grow these coatings using lower pressures (less than 1 Pa) and other interlayer's.

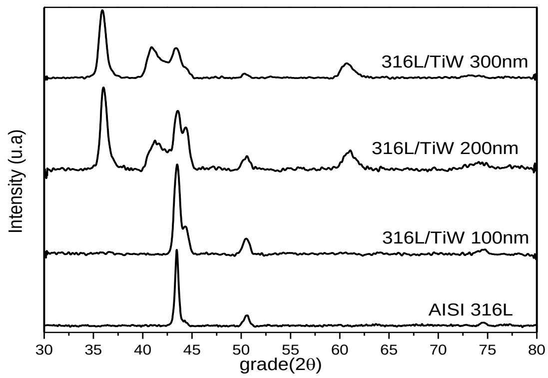

Fig. 2. *X-ray diffractograms.*

4. References

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