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Characterization of laboratory and industrial CrN/CrCN/diamond-like carbon coatings

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ABSTRACT

This work reports on laboratorial and experimental wear behaviour studies about a multi-layered film deposited by PVD (Physical Vapour Deposition) unbalanced magnetron sputtering. The film consists of three different layers: CrN in the bottom, CrCN as intermediate layer and DLC (diamond-like carbon) on the top. Film characterization was done using techniques such as Scanning Electron Microscopy, Energy Dispersive X-ray Spectroscopy, Atomic Force Microscopy and X-ray diffraction. Scratch-tests, nanoindentation analysis and ball-cratering wear tests were used in order to measure the adhesion critical load, hardness and wear coefficient, respectively. Experimental tests were developed letting one to realise the suitability of this film for mould cavities used on injection moulding machines that produce automotive parts in polypropylene reinforced with 30% (wt.) glass fibres, because this composite material performs severe abrasion on injection moulding which brings important challenges to surface wear resistance. Experimental tests revealed that, after 135,000 injection cycles, multi-layer coating improved significantly the performance previously revealed by uncoated samples. The good results achieved by this film can be partially assigned to DLC top layer due to its low friction coefficient. This paper discusses these results, comparing them with some other PVD coatings already tested in the same conditions.
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1. Introduction

The P20 steel is commonly used as main material by mould manufacturers for plastics injection. There are many important factors influencing the life cycle cost of the plastic injection moulds, as previously referred by Folgado et al. [1]. However, wear and corrosion are the most important issues acting on the mould surface and they are one of the main reasons that contribute to put moulds out of service. As well-known, plastic injection moulding industry has brought some different challenges to materials used in moulds due to polymers recently developed, much more aggressive for non-coated tool steel surfaces, and increased abrasion brought by the introduction of short glass fibre reinforcements on the injection moulding process. Maintenance tasks influence the process cost and cause a consequent lack of productivity due to the losing of time in these operations and, when the injected material is reinforced with hard and abrasive fibres, these aspects become particularly evident. In order to overcome this inconvenience, some solutions have been adopted, such as surface treatments or advanced coatings, ranging from chromium plating to High Velocity Oxy-Fuel WC/Co [2] films. Other metallic coatings like hard chromium or nickel–phosphorus produced by electrodeposition or electroless processes [3], as well as titanium, aluminum and other carbide/nitride layers or synthetic diamond, produced by PVD (Physical Vapour Deposition) or CVD (Chemical Vapour Deposition), are also usually used for this purpose [4–7].

In the last decade, some researchers have dedicated their attention studying different coating properties and characterizing their behaviour when submitted to injection moulding conditions or other that can be compared with those ones. However, it seems that researchers' attention is focused on machining tool improvements and it is much lesser extent explored for plastic injection moulds and metal forming dies. Some approaches were done by this way using coatings such as Cr_2O_3/CrN [5] and Ti–Ni–N [6], among others obtained by PVD techniques, and even diamond [7] was used, being synthesized by CVD technique. Some improvements were effectively achieved, increasing the adhesion between coatings and substrate, and minimizing the surface roughness, which is dramatically important for plastic injection moulding, and extending the lifetime of the mould due to better wear behaviour of the mould surface.

Taking into account the problem referred above and the solutions presented, investigations should be developed in order to find a coating system that increases the moulds lifetime, also improving their surface wear resistance. In this work, a multi-layer CrN/CrCN/DLC coating was used and investigated in both laboratorial and industrial tests, assuming that the wear mechanisms involved are quite different.

CrN film was used as bottom layer due to its good properties such as adhesion [8] and hardness, as well as tribological behaviour. The

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selected CrCN intermediate layer was produced adding C_2H_2 gas after the bottom layer deposition, and it was used in order to introduce carbon, preparing the multi-layered system to the next deposition through a higher content of carbon in this layer. As well known, this transition film also presents good tribological properties [8]. Diamond-like carbon (DLC) layer formation was feasible due to the presence of carbon in the intermediate deposit and it has been used in the top layer for acting as solid lubricant.

This paper also presented a comparative study with some other PVD coatings already tested in the same conditions.

2. Experimental details

2.1. Materials

The substrate material used in this work both for laboratorial and industrial tests was AISI P20 IMPAX SUPREME HH steel (W. nr. 1.2738) with 380 HBW 2.5/187.5/5 hardness and 205 GPa Young's modulus. The chemical composition (wt%) obtained on a mass spectrometer was C 0.35%, Si 0.29%, Cr 1.95%, Mn 1.39%, Mo 0.19%, and Ni 1.00%. This steel is provided as hardened and tempered and its hardness value is in the range usually pointed out by the manufacturer (Uddeholm), presenting a refined structure as illustrated in Fig. 1.

The plastic material used in injection moulding industrial tests was polypropylene (PP) reinforced with 30% (wt.) glass fibre, as depicted in Fig. 2. This composite presents generically 110 MPa hardness and 1.14 g/cm^3 specific weight.

2.2. Samples dimensions and geometry

Two different kinds of samples were used in this work, regarding the laboratorial and industrial tests, keeping in mind the same substrate material pointed out above. Square samples with 25 × 25 × 2 mm^3 have been used for laboratorial tests while slight pyramidal rectangular samples have been employed for industrial tests, allowing inserting and adjusting them into the mould, keeping the main sample face in the same plane of the internal mould surface, ensuring the polymer truthful flow inside the mould. The geometry and dimensions of the industrial sample are shown in Fig. 3. Both laboratorial and industrial sample surfaces were mechanically ground using Struers LaboPol-21 twin wheel grinding machine provided with LaboForce 3 head and using sandpapers sequentially from F220 to F1200 mesh, crossing the grinding direction between each sandpaper changing, followed by polishing operation using 3 μm diamond slurry, until obtain 0.060 μm mean roughness (R_a).

2.3. Film preparation

The deposition of CrN/CrCN/DLC was performed in an industrial CemeCon CC800/9ML PVD Unbalanced Magnetron Sputtering machine.

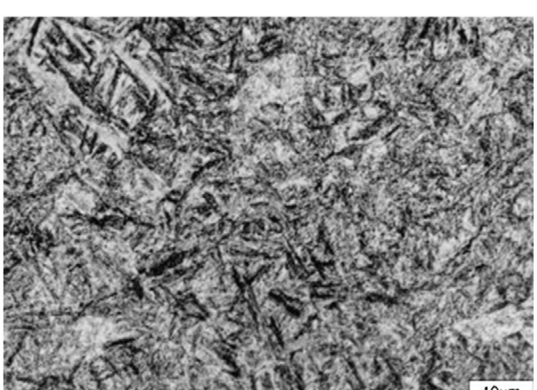

Fig. 1. AISI P20 (IMPAX SUPREME HH) steel microstructure used as substrate.

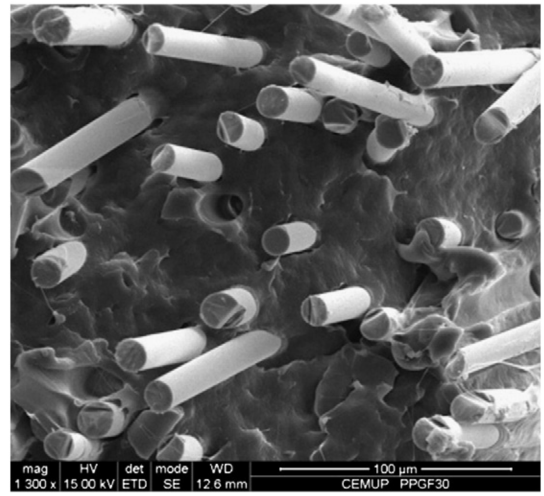

Fig. 2. SEM image of the polypropylene reinforced with 30% (wt.) of glass fibres.

The CrN bottom and intermediate layer were generated from two Cr targets using N and Ar$^+$ feed gas for the bottom layer (CrN), being added C_2H_2 for the intermediate layer (CrCN). For the top layer (DLC), Cr targets were hidden behind shutters and two graphite targets were exposed. The nanostructure shown in Fig. 4a corresponds to different CrCN phases, being some of them more carbon-rich than the others, which were obtained providing C_2H_2 periodically and controlled in time.

The main deposition parameters used for all the layers were: gas pressure 500 MPa, temperature 500 °C, target power density 16 A cm^{-2}, bias of −70 V and deposition time 4 h. Sample holder was animated by rotational motion of 1 rpm leading to obtain better homogeneity on the film composition.

2.4. Coating characterization

Uncoated and coated surfaces' topography as well as the three layers thickness (CrN, CrCN and DLC) were assessed and measured using a FEI Quanta 400FEG scanning electron microscope (SEM) equipped with EDAX genesis X-ray spectroscope. The roughness was measured with a VEECO multimode atomic force microscope (AFM) (7 nm tip radius) in contact mode with two different analysis areas: 10 × 10 μm^2 and 50 × 50 μm^2. For the thickness measuring, a metallographic preparation was done; samples were partially cut on the coating reverse side and then, they are submerged in liquid Nitrogen during 15 min in order for it to become brittle. At that time, samples were taken and mechanically broken, minimizing by this way plastic deformation close to the cutting edge. Thus, they were embedded in resin with cross section turned to the work face. Further, the assembled set was ground with F1200 sandpaper until main grooves disappear and then, it was polished with diamond grit solutions of 3 and 1 μm during 5 min each one.

The phase of the sample surface was already assessed by X-ray diffraction (XRD), using Cu Kα radiation, and was performed via a Seifert equipment provided with a 4-circle goniometer, using the Bragg–Brentano configuration. The diffracted radiation was collected by a CCD Germanium detector, which presents high energy resolution.

2.5. Adhesion tests

Longitudinal and transversal scratches on the multi-layered film surfaces were performed using CSM REVETEST scratch-tester provided with a diamond indenter of 200 μm tip radius. Scratch-tests were done in two orthogonal directions avoiding problems related to surface texture resulting of substrate grinding process. The critical load (L_c),

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