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Minimizing the Adhesion Effects in Food Packages Forming by the Use of Advanced Coatings

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Abstract

The metal packaging industry used for food application has undergone drastic changes in the demands of its final consumers. The raw material for these packages, is a low carbon steel coated with a thin layer of tin (2,0 g/m^2), also known as tinplate. The stamping process of these packages occurs at room temperature and is critically influenced by the tin transfer from the steel surface to the tool surface, mainly due to the tin softness. This problem is easily solved using lubrification but the purpose of this study will be the reduction or even absence of lubricants during the process in order to comply with costumers' requirements. A successful way to minimize the consumption of lubricants is to use tools which are coated with PVD (Physical Vapour Deposition) advanced coatings deposited with unbalanced magnetron sputtering technique. Thin WC (Tungsten Carbide) and CrCN (Chromium Carbonitride) coatings were deposited using PVD on tool stamping steel – AISI D2. Block on ring tribological tests were performed on the coatings against tinplate counterface in order to investigate their wear performance, with particular emphasis on the material transfer (tin) phenomena during the sliding tests. The results allowed for selecting the best coating tested with a view to avoid the tin adhesion to the die.

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Keywords: Tinplate; Advanced Coatings; Physical Vapour Deposition (PVD); WC; CrCN; Stamping

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1. Introduction

The food packaging industry has evolved over time, along with environmental concerns and production costs. Some food packages may consist thoroughly of metal or have metal components. These packages can be need for: beer and soft drink cans, food cans, drums and pails, aerosol containers, lids, etc. [1]. One of the most common metal used for food package is tinplate [2], which is cold-rolled low carbon steel between 0,13 and 0,5 mm thick, tin coated with 2,0 to 22,4 g/m2 in one or both surfaces. Tin coating is applied in a continuous electrolytic operation. This material is 100% recyclable, considered an eco-friendly packaging material, thus combining ductility with mechanical strength, which allow stamping operations. Due to tin properties, there is good solderability and corrosion resistance [3]. These characteristics have a detrimental effect on stamping process, causing unwanted wear phenomena due to the friction between sheet metal and tool surface caused by, among others, tin adhesion to the tool surface. Studies have been developed on the improvement of tribological and wear behavior through advanced coatings application [4,5]. Sergejev et. al. [6] performed industrial trials of PVD coated punches in fine blanking operation, studying punches wear regarding the punch geometry, position in the die and surface roughness, measured after a maximum of 100,000 cycles at high loads. Silva et. al. [7] studied laboratorial and experimental wear behavior of a PVD multi-layered film. The film was deposited with three different layers: CrCN as a bottom layer, CrCN as central layer and at the top a DLC (diamond-like carbon) layer. Experiments were conducted at industrial process allowing to conclude the sustainability of this film set used as coating for mold cavities applied for polymer injection molding process, which consists of the injection of automobile parts in polypropylene reinforced with 30% (wt.) glass fibres, causing severe abrasion on molds surface. Studies have led to conclude that after 135,000 injection cycles, the multi-layered film improved significantly the mold surface performance comparatively to the uncoated samples. Fernandes et. al. [2] identified the main wear mechanism developed on the stamping tools, promoted by the tin coated steel sheet used in the packages. Authors tested two advanced PVD coatings (B4C and Mo), leading to the improvement of the punch and die wear behaviour under these working conditions. The obtained results confirm that it is possible to minimize the tin transfer from tinplate to the die and punch.

2. Methods

For the purposes described above, and after previous studies pursuing this goal, CrCN and WC films were chosen. In this way, diverse tribological tests were carried out to study the wear behavior between these films and tinplate sheet. Tribological tests were performed in a block-on-ring tribometer using medium loads and measuring the friction force evolution over the tests. The used block-on-ring tribometer was developed inhouse and previously calibrated and used by several researchers [8,9].

Metal sheet used for food package, also known as tinplate, is a soft steel coated with pure tin 2,0 g/m^2 thin layer deposited by electrolytic processes. The average sheet thickness is ~0,15 mm, with an average hardness of 70 HR30T (~170 HB). Tinplate has a four-layer structure. The inner layer is composed of low carbon steel and it's composition is controlled according to the grade chosen, so that several types of steels with capacity to suffer mechanical deformation are produced. The inner layer is coated with a tin layer, providing a protective film to oxidation, having a cathodic behavior regarding the substrate. The external layer is provided with special passivation treatment in order to stabilize the surface and improve adhesion of lacquers and lithography.

The material used as substrate in the samples used in tribological tests, was AISI D2 steel because it is used both in die and punch production. This steel presents an average hardness of 60-62 HRC and 210 GPa Young's modulus. It is provided in hardened and tempered condition, and the mechanical properties were provided by the supplier. The AISI D2 chemical composition is (wt%): C(1.45 – 1.65%); Si(0.10-0.40%); Mn(0.15-0.50%); Cr(11.0-13.0%); Mo(0.60-0.90%); V(0.70-1.00%); P(0.030%); S(0.030%).

Minimizing friction and wear phenomena, is one important goal for reliability and extension of tool lifespan. CrCN and WC films were selected as advanced coatings for this research, being recognized as potentials coatings to solve the industrial problems between die tool and tinplate. Generally, these coatings are characterized by high hardness, low friction and promising wear behavior [13-15].

In this way, as referred previously, tribological tests were carried out using a block-on-ring configuration. Samples were shaped as blocks and rings, as presented in Figure 1.

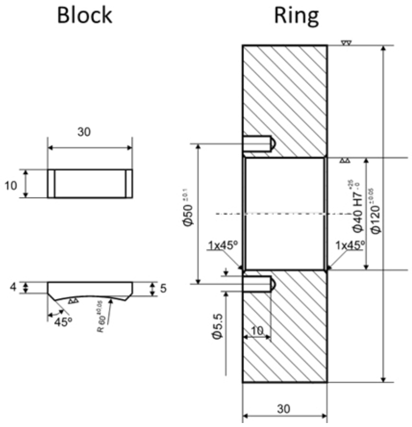

Figure 1 - Rings and blocks samples geometry

Table 1 – Tribological parameters

Description	Parameters
Normal load [N]	70
Ring rotational speed [rpm]	60
Ring linear speed [m/s]	0,38
Ring diameter [mm]	120
Contact area between block and ring [mm^2]	255
Contact pressure [MPa]	0,27
Number of cycles	100
Total distance per test [m]	~37,7

Samples assumed two different geometries: blocks and rings. These two geometries performed distinct functions: (a) ring intend to simulate the tool surface while (b) the block intend to simulate tinplate during the contact. Hence, the block was produced with an internal radius of 60.2 mm in order to further accommodate the tinplate sample and keep the stipulated radius in the corresponding value. Tinplate samples with 10 mm x 30 mm were bounded with Loctite 454 structural adhesive on both surfaces. Block-on-ring experiments were performed under dry condition. The normal load and linear speed selected were 70 N and 0.38 m/s, respectively. Concerning to laboratory tests, a load cell was connected to the tribometer arm in order to assess the tangengial force induced by the contact between the coated ring surface and the tinplate bonded on block's surface. The signal emitted by the load cell was sent to an electronic device able to filter and magnify this signal converting it in tangencial force, expressed in N. Table 1 summarizes all test parameters. This test configuration aimed to evaluate the coating's wear behavior at room temperature, simulating the industrial conditions, as well as, observing possible tin transfer. This test configuration allows as well the friction coefficient determination, coating adhesion to the substrate and pattern wear identification.

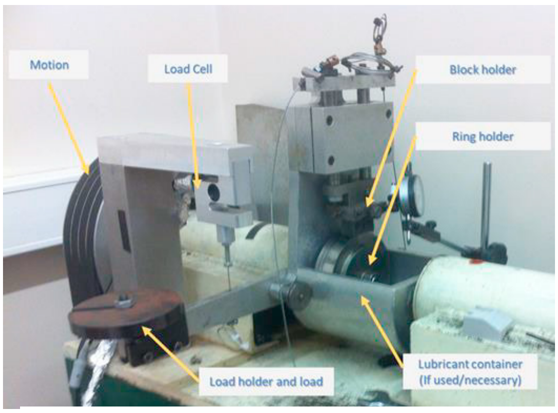

Figure 2 - Block-on-ring configuration view

Table 2 – PVD sputtering deposition parameters

	CrCN	WC
Time deposition [min]	134	110
Chamber gases	Kr – 120 mln; Ar – 180 mln; N_2 – 100 mln; C_2H_2 – 70 mln	Kr – 200 mln; Ar – 295 mln
Pressure [mPa]	500	500
Temperature [°C]	380	380
Bias [V]	-80	-90
Target Current [A]	10	10
Holder rotational speed [rpm]	1	1

Regarding the goal of this work, CrCN and WC were selected. These two coatings were investigated to prevent tin transfer to tool surface and to minimize tool wear, as well as friction. Both coatings were produced by unbalanced magnetron sputtering technique using industrial Cemecon CC800/9ML PVD Magnetron Sputtering equipment. The details of the PVD magnetron sputtering deposition process are described in Table 2. Coated surfaces' topography, as well as thickness were accessed and measured using a FEI Quanta 400 FEG scanning electron microscope (SEM) equipped with EDAX genesis X-ray spectroscope. Surface roughness was measured using a VEECO multimode atomic force microscope (AFM) with a contact area of analysis of 50 x 50 µm^2, providing the selected parameters: arithmetic mean surface roughness (R_a) and maximum surface roughness ($R_{máx}$). Samples were subjected to metallographic preparation in order to enable thickness measuring.

In order to evaluate the adhesion between coatings and substrate, two different techniques were used: scratch test and Rockwell indentation. So, to evaluate the cohesive and adhesive failures, a scratch-tester CSM REVETEST, was use not provided with acoustic emission, according to the BS EN 1071-3:2005 standard [10]. The load range selected was 0-100 N and load was increased at rate of 10 N.min-1. Rockwell indentations were carried out with 1470 N (150 kgf) normal load in an EMCO M4U Universal Hardness tester, repeated eight times for each coating. Border craters results were compared with the effects shown in the VDI 3198:1991 standard [11]. Both grooves and indentations were examined by optical microscopy (OLYMPUS DP70 optical microscope) and results were compared to failure modes illustrated in the above-mentioned standards. Furthermore, hardness was determined by Vickers indentation, using a micro-hardness Fishercope H100 equipment. The selected normal load was 50 mN, which was kept constant during 30 s, avoiding this way creep phenomenon. This test was performed according to ISO 14577-1:1015 standard [12] in different areas, in order to obtain an average value provided with the required accuracy. This equipment produces values chart that allow obtaining 'load-depth' curves, conducting to hardness (H) and Young's modulus (Er /E').

3. Results

Coatings process resulted in a monolayer thin film for WC (Figure 3 (b)). Regarding the CrCN film, on the other hand it, can be distinguished two distinct layers. Energy Dispersive Spectroscopy (EDS) spectra of the two areas revealed that the inferior layer (Z2) (Figure 3) presents a higher amount of nitrogen (N) relatively to the superior one (Z1) (Figure 3). This is related to a variation of flow in the gas chamber during the deposition process.

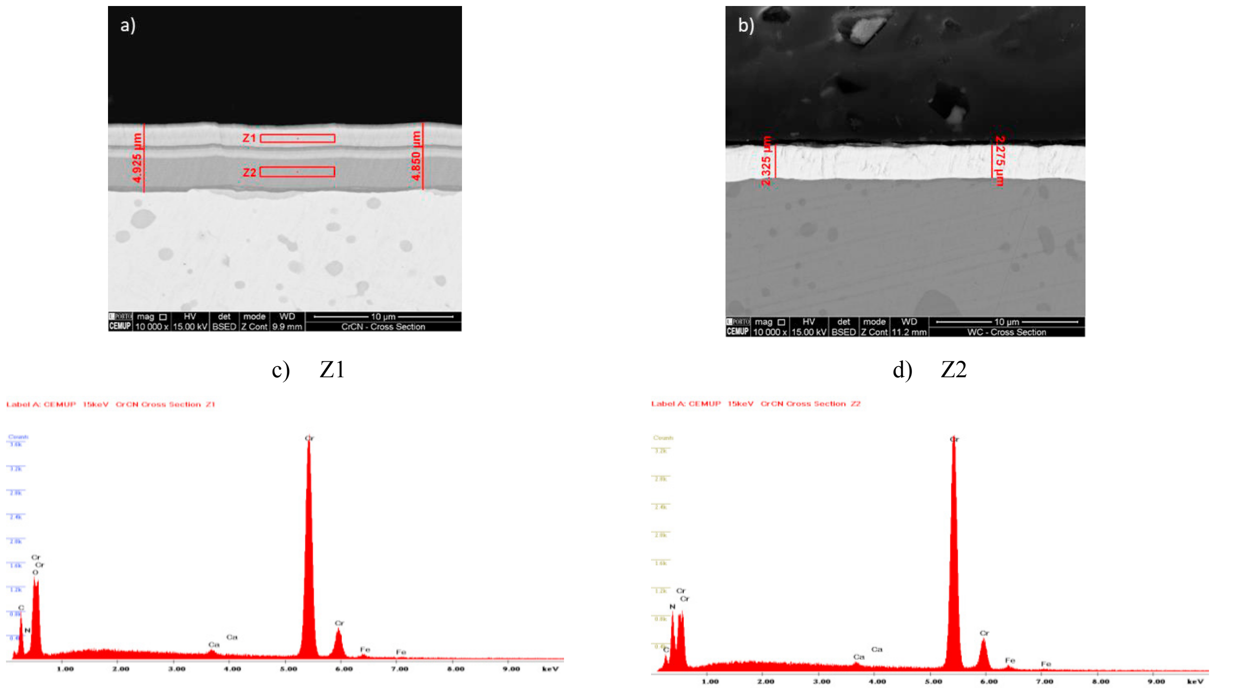

Figure 3 - SEM cross section views of the coatings (a) CrCN and (b) WC, with embedded coating thickness measurements in the figure and (c) CrCN and (d) WC EDS spectra collected during SEM observations

The AFM (atomic force microscopy) allow for access to roughness'main parameters and obtained values from substrate and coatings are presented in Table 3. Considering the values pointed out to the sample surface before coating, there is a significant decrease of the mean average roughness and maximum roughness, denoting that deposition parameters selected for PVD process promote surface smoothness. Despite this fact, some little aggregates were observed in the surface of both coatings by SEM observation, which can contribute negatively to tribological performance of the films.

Samples	Ra (µm)	$R_{máx}$ (µm)
Substrate	0,349	3,618
CrCN	0,109	1,175
WC	0,155	1,223

Table 3 – Substrate and coating roughness values

Films morphology was analyzed by SEM, as previously referred, which can be seen in Figure 4, showing a very textured surface. It is possible to observe that WC has a predominantly large globular structure, whereas, CrCN tends to small globular structure arranged in a columnar form. Hence, CrCN is most suitable for tribological purposes.

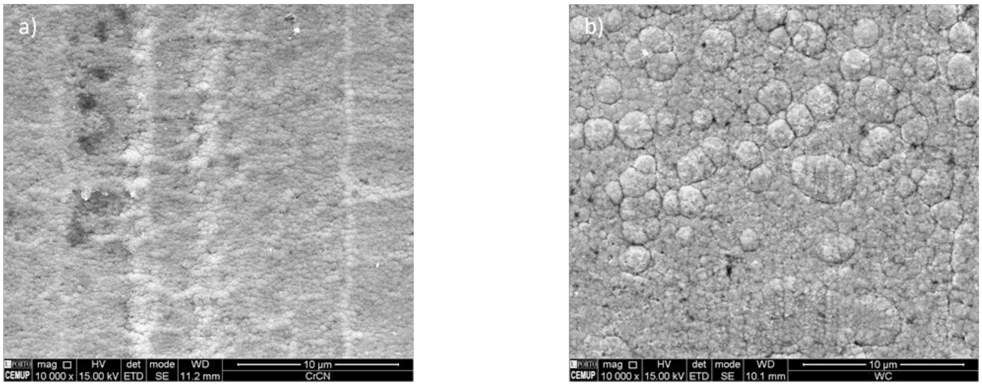

Figure 4 – Coating morphological characterization (a) CrCN and (b) WC (top view)

Scratch test measurements were performed to quantify the normal load that corresponds to the initial failure, allowing assessing the adhesion force between the deposited films and the substrate. At the end of the tests, the scratch grooves were carefully observed by optical microscopy. The critical load Lc1, corresponding to cohesive failure, measured in CrCN and WC were 29 N and 26 N, respectively. The critical load Lc2, corresponding to adhesive failure, of CrCN and WC, were 55 N and 33 N, respectively. These values are in line with other analysis made in similar films [13,14].

The adhesion quality of the coatings to the substrate was also assessed through Rockwell indentations following the VDI 3198:1991 standard [11]. Indentations were carried out at 150 kgf in each coated sample, as can be seen in Figure 5. It is possible to observe a larger number of small cracks in the indentation border of CrCN film than in the WC film. According to the VDI standard, these results can be classified as 'HF1' behavior. No border spallation was verified. The indentation results can be more accurate than the scratch-test, due to the lower influence of the surface roughness, film hardness and coating thickness, as well as the slower increasing of the normal load. Therefore, it can be concluded that both coatings have good adhesion to the substrate.

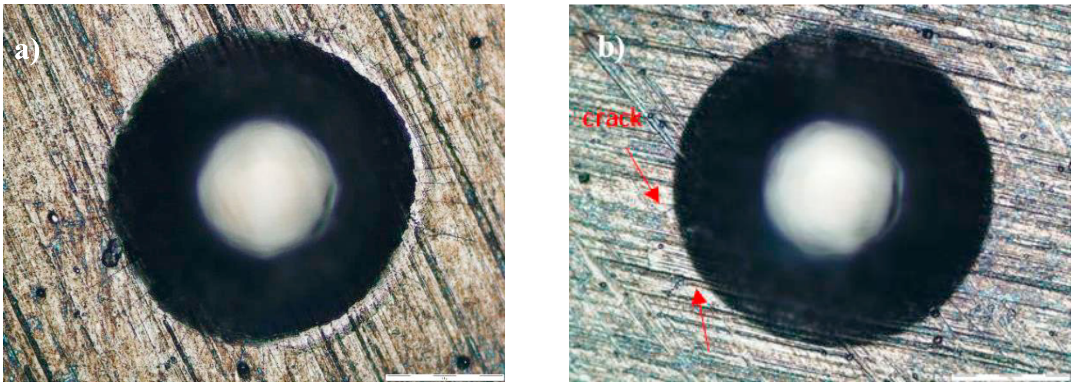

Figure 5 -Optical microscope images of the coating indentations at 150 kgf normal load in: (a) CrCN and (b) WC

Due to the reduced coating thickness, some care must be taken in the hardness assessement. In order to overcome this problem, low load was selected (50 mN), minimizing the indenter penetration depth and the consequent substrate deformation. In literature, indentation depth should not surpass 10% of coating thickness, avoiding substrate influence [17,18]. Problems with elastic recovery and creep were avoided, keeping constant the maximum load during 30 s. The hardness value obtained for CrCN coating were 14.1 GPa, being lower than others registered by other authors (20 to 30 GPa) [20-22]. Otherwise, about WC coating, hardness value obtained were 37.5 GPa, slightly higher than wide range of values referred in literature (14 to 22 GPa), leading to concluding that deposited film has a dense layer structure [23-24]. Effective Young's modulus is presented in Table 4.

Table 4 – Coatings Young's modulus

Coating	Effective Young's Modulus - Er [GPa]
CrCN	235,4 ± 20
WC	311,1 ± 16

Wear tests were performed in the block on ring tribometer using the entire width of the ring. Figure 6 shows the SEM micrographs of the wear scars corresponding to CrCN and WC coatings after sliding tests. Quantitative evaluation of mass loss was not important to this study, once it was expected mass transfer between surfaces, increasing the weight on the harder surface. Furthermore, tin transfer from tinplate surface to the coated ring was considered more important than weight loss. SEM micrographs reveal no great difference between the two coatings regarding the tin transfer pattern. Indeed, adhesive wear can be observed, as well as, many scratches following the sliding direction. Both coatings present tendency towards to creation and anchoring of oxide films on their surfaces, formed during contact. The oxide films show different shades due to the different density of the coatings, being the shade a relative tonality.

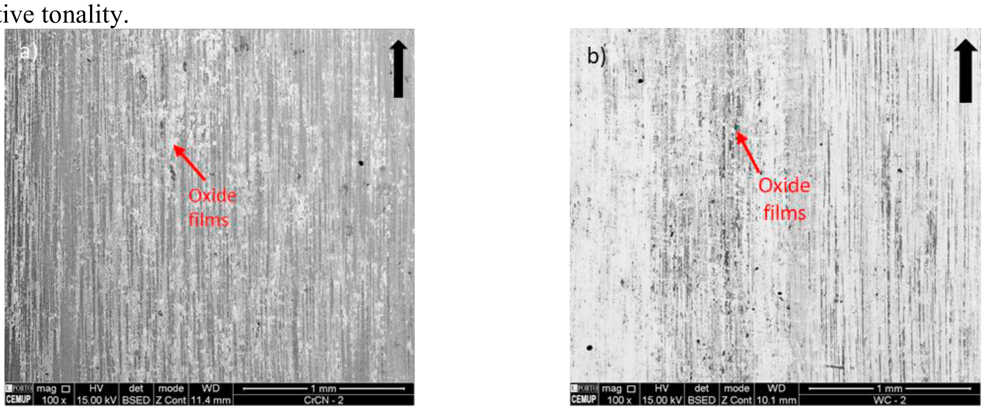

Figure 6 – SEM micrographs on coated ring surface: (a) CrCN and (b) WC. Black arrows indicate sliding direction.

Additionally, the EDS analysis of the wear scars of both coatings are shown in Figure 7. The purpose of conducting the EDS analysis was to detect the type of elements present in both surfaces (coated rings and tinplate bonded to block).

For both coatings, it is noteworthy sliding ploughed grooves following the motion direction, bordered of accumulated debris layer composed of wear particles released by the tinplate and oxides formed during the contact. The fluctuating of the friction coefficient during the steady wear stage, registered during monitoring, was probably induced by the accumulation of debris on the wear scars and carrying the imposed load caused from debris at the contact interface. Moreover, a stick-slip phenomenon is expected due to the hardness difference between surfaces.

As revealed by the EDS analysis (Figure 7), besides coating particles and oxides, the main elements found on debris layer were iron together with transferred tin, suggesting that steel transfer from the tinplate substrate to the ring surface had occurred.

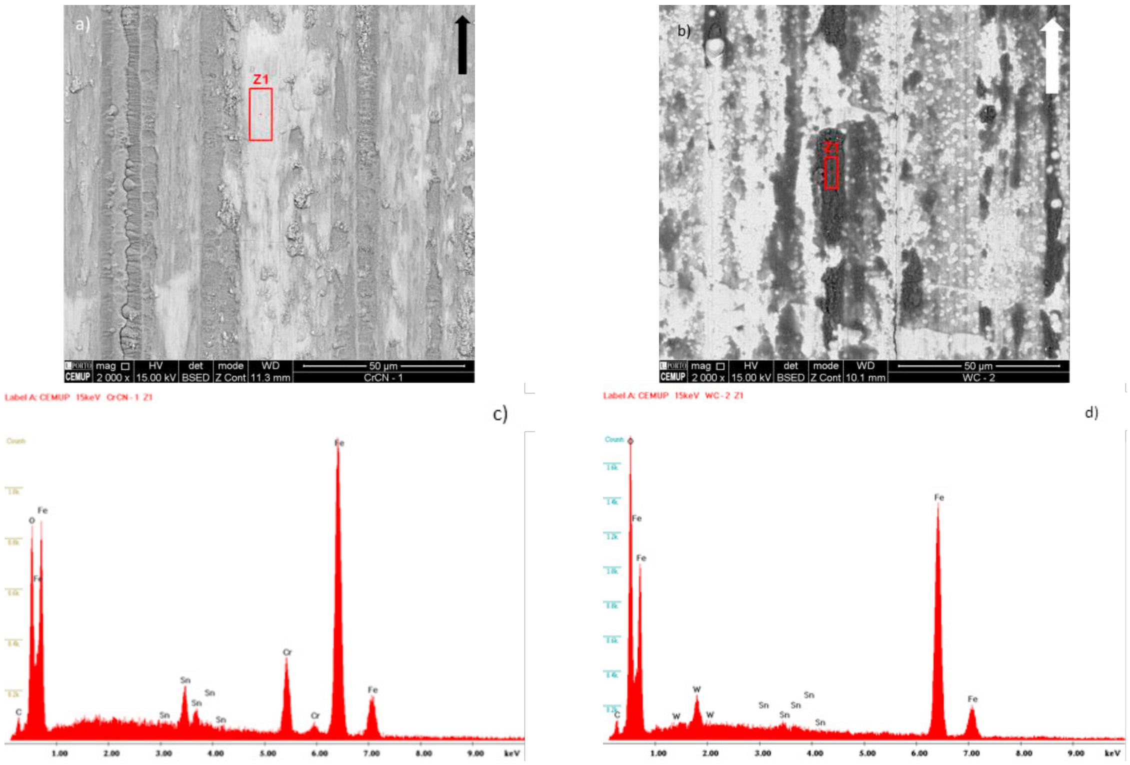

Figure 7 -SEM micrographs and spectra of transferred tin on ring coated surface (a)(c) CrCN and (b)(d) WC.. Arrow indicate sliding direction.

Friction force is also very important in this case, regarding the expected application. Thus, frictional force was determined based on the values read by the load cell connected to the tribometer in order to measure the tangential force. Due to the selected magnification and functioning of the data acquisition board model NI DAQ 6036E and the LabView® v7.0 programming software, the values obtained are always multiples of 12.5 N. As can be seen in Figure 8, there is a running-in period where the tangential force is slightly higher for both coatings, but after the initial 30 cycles, the tangential force tends to decrease. CrCN coating has a relatively stable behavior between 10 and 60 cycles, subsequently a stick-slip effect in which tangential force varies between negative values and higher positive values. WC coating also presents behavior from the first 30th cycle onwards, though the tangencial force has steeper decreasing trend compared to CrCN. Observed peaks distributed during the test duration, may represent an oxide layer cleavage phenomenon implying a momentary friction decrease. The overall decreasing of the friction coefficient can be connected precisely to the presence of oxide layers formed during the test, which promote the slight decrease of the friction force.

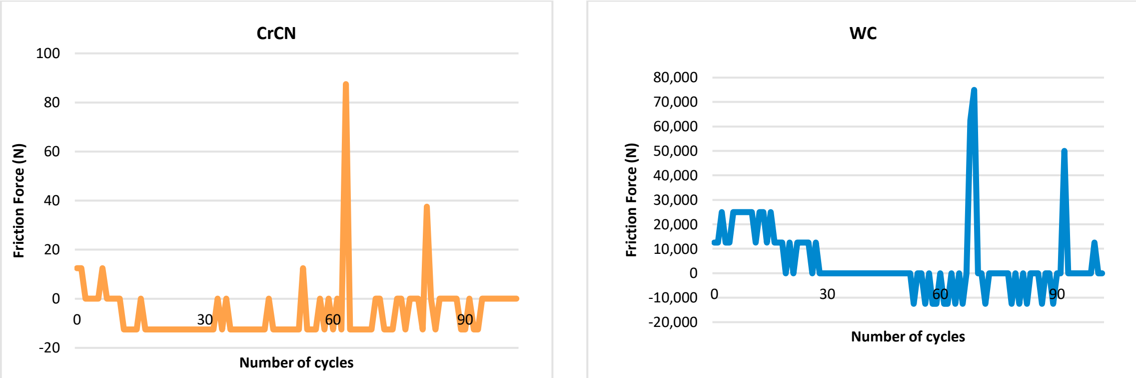

Figure 8 - Variation of the tangential force measured in the block on ring tribometer as function of the number of revolutions made by the coating ring.

Table 5 presents the friction coefficient results, showing that CrCN coating presents an average friction coefficient a little lower that the WC one. Moreover, the friction coefficient values described in the literature by other authors [20-22] for CrCN coating are usually in the range from 0.38 to 0.80. Thus, the value obtained is slightly lower, probably due to the coating morphology. Information about WC coatings was not found to compare the obtained results, but WC/C films analyzed by Wanstrand et al. [24] provided friction coefficients in the range from 0.1 to 0.4, when tested against steel. Furthermore, WC-based coatings are currently produced based on other deposition techniques, such as thermal spraying (HVOF – High-Velocity Oxygen Fuel), being difficult to compare these results with the other obtained through this work.

Table 5 – Average friction coefficient values obtained in tribological test carried out on block on ring tribometer

	CrCN	WC
Friction Coefficient (μ)	0.31	0.44

4. Conclusions

The aim of this study was to investigate the tin transfer phenomenon of two different coatings obtained by PVD technique, simulating through wear tests the contact between coated surfaces and tin coated steel sheet. The coatings tested were CrCN and WC, both obtained by Unbalanced Magnetron Sputtering technique. The coatings were properly characterized in terms of morphology, thickness, adhesion, hardness, wear resistance and friction coefficient. The coatings were tested with a view to apply them in low-deep stamping dies devoted to the plastic deformation of tin coated steel sheet usually used in food packaging. After performing the tests, it is possible to draw the following conclusions:

a. Both coatings present very goo adhesion to the steel substrate under the selected deposition conditions.
b. CrCN presents better surface morphology, being expected a better tribological behaviour.
c. Outstanding values were registered for the hardeness, comparing with the usual values reported.
d. Very good friction behaviour and tribological properties, showing low ability to capture tin from the counterface during the tribological tests.

Regarding these results, both coatings are suitable for the desired application, but the CrCN coating showed better tribological properties and lower friction coefficient, without tin transferred from tinplate to coated surface.

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