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A Study of Cr/CrN and Cr/CrN/CrAlN Multilayer Coatings for Permanent Mold Castings of Aluminum Alloys: Wear and Soldering Tendency

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ABSTRACT

Physical vapor deposition (PVD) coatings namely Cr/CrN, Cr/ CrN / CrAlN multilayers (period of μ = 4 with a Cr bonding layer 138 nm thick), have been synthesized on a quenched and tempered X38CrMoV8 steel to test their ability to avoid soldering during casting of aluminum alloys. Wear tests, optical profilemetry observations and demolding stress tests were carried out. Intermetallic compounds were formed and aluminum cast alloy soldering layer was found on surfaces of all tested pins, which were observed and quantified by SEM/EDS. Cr/CrN multilayers have been found to exhibit the best performance among all materials and coatings considered here. The results showed low friction coefficient of Cr/ CrN multilayers and the amounts of intermetallic compounds were lower than those formed on Cr/CrN/CrAlN ones.

1 Introduction

Premature failure of dies, moulds and cores is a critical issue for manufacturers in hot-working processes industry. Nowadays, thermal shock cracking is by far the first degradation mechanism. This phenomenon is due to the high temperature of the molten metal at casting and abrupt cooling of the surface by the release agent. Soldering is the second most common

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degradation mechanism of the tools in aluminum foundry. It is a very penalizing phenomenon in foundries resulting from mold / metal reactions and the formation of iron-aluminum-silicon inter metallic compounds at the mold surface. It is generally found in aluminum foundry practice and from results of physico-chemical attack of the mold by the alloy and by the formation of hard intermetallic compounds adhering to the mold surface. Al or Si elements diffusion into the die substrate can be observed [1-4].

Molten aluminum adhesion or welding to the die is often responsible for a reduction in die life with a consequent increase in process cost. There are several studies on soldering phenomena which indicate that intermetallic phase layers are formed in the steel die surface. These intermetallics are undesirable and responsible of the decrease in cast component quality. The use of die coatings (PVD, CVD) considerably reduces soldering problems, which can seriously disturb the production process and may in some cases lead to complete stop production [1-4].

The thin layers obtained by PVD methods offer a significant slowdown in the molten aluminum attack on steel [1, 5-8]. The sector of thin PVD coatings is constantly improved to respond to the growing demand for better performance of cutting tools [9, 10]. Advanced PVD coatings are designed to withstand severe mechanical and thermal stress conditions [11-13]. Chromium nitride (CrN) deposited by physical vapor deposition (PVD) technology has been identified as one of the promising protective coatings for molding applications [14]. CrN coatings have been widely investigated for their industrial significance [15-19] in many applications such as cutting tools, mechanical components and surface steels due to their good mechanical properties, better oxidation resistance and tribological behavior. Nevertheless, these binary coatings are unable to respond to the increasing needs from the industrial technology development [20, 21]. CrN coatings reveal a high hardness, chemical and corrosion resistance as well as a good resistance against abrasive and erosive wear [4] but the oxidation resistance of CrN is limited to a maximum service temperature up to 600 °C [22]. For hard protective coatings, thermal stability is the main required property as they are exposed to high temperature variations during the casting process. The ternary coating (Cr,Al)N, by incorporating Al into transition binary CrN thin coatings, has been intensively investigated in previous studies [10, 19, 23, 24]. The addition of Al to CrN raises the oxidation temperature. Indeed, CrAlN coatings have been reported by Kawate et al. [25] to be stable up to 900°C according to the Al content in the Cr0.6Al0.4 N and Cr0.4Al0.6N coatings. CrAlN coatings also exhibit higher hardness and a lower friction coefficient compared to CrN coatings [26, 27]. Therefore, CrAlN coating is a good candidate as an alternative to conventional CrN coatings, especially for high-temperature oxidation-resistance applications [28, 29]. Each of the properties of the monolayer can be combined by superimposing Cr, CrN and CrAlN to obtain multilayer coatings that provide better fulfillment of the industrial requirements. There is a great variety of studies on multilayers such as TiN/CrN [30], TiAlN/CrN [31], TiN/TiAlN [32, 33], Cr/CrN, CrN/CrAlN and Cr/CrN/CrAlN [34, 35]. However, in the case of the casting aluminum tools, like molds, pins and cores, there are few studies on the behavior of CrN and CrAlN monolayers and CrN/CrAlN multilayers.

The aim of this study was to investigate the structure and properties of the CrN and CrAlN monolayers, as well as the Cr / CrN, Cr / CrN / CrAlN multilayers, as a means of improving the life of molds and dies for casting aluminum alloy”

The thin films were deposited by DC magnetron sputtering on hardened steel X38CrMoV8 for wear tests (ball-on-disc), optical profilometry and home-made demolding stress tests. SEM observations permitted to determine the surface morphology and the thickness of the layers while EDS microanalysis was realized for coatings chemical composition.

2 Experimental details

2.1 Coatings

The coatings were realized on 380 μm thick <100> Si wafers (10 × 10 mm^2) for SEM observations and also on quenched and tempered hot-working tool steel X38CrMoV8 (chemical composition (w. %) 0.38 C, 1.16 Si, 0.418 Mn, 0.02 P, 0.002 S, 4.8 Cr, 1.3 Mo, 0.45 V Fe (balance)) substrates. The steel samples are discs (4 mm thick, 20 mm of diameter) for wear tests and optical profilometry (Fig 1a) and the last type of steel samples are pins as described in Fig 1b for home-made demolding tests. Samples were grounded with 400 and 800 grades silicon carbide papers to obtain average value of surface roughness, estimated by optical profilometry (VEECO, Wyko NT-1100), Ra = 0.13 ± 0.004 μm. Thereafter, the samples were cleaned in an ultrasonic bath in alcohol and acetone for 10 min, respectively. Subsequently, they were washed and rinsed in distilled water. Cr/CrN, and Cr/CrN/CrAlN multilayers were deposited by DC reactive magnetron sputtering (KENOSISTEC KS40V system) using chromium and aluminum targets with dimensions 127 × 403 × 7 mm^3 (purity 99, 99%).

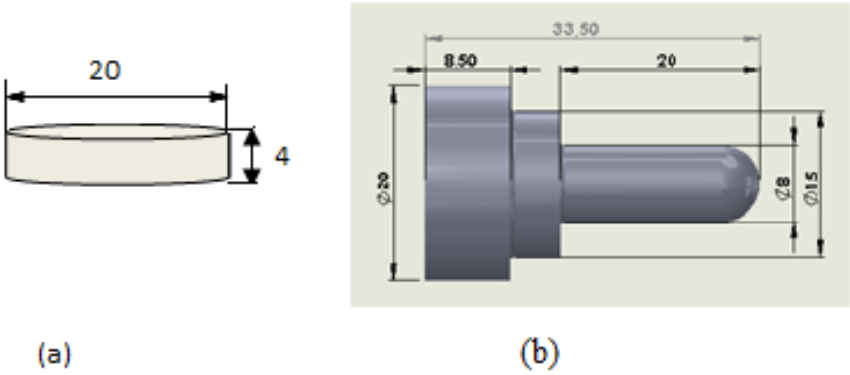

Figure 1 – Steel samples: (a) discs and (b) pins

The residual pressure is 2.10^{-5} *Pa*, the depositions were conducted in an argon and nitrogen atmosphere with 33.3 and 68.8 sccm flow rates respectively. During the deposition, the working pressure is 0.5 Pa, the power applied was 1500 W for the chromium (Cr) target and 1000 W for the aluminum (Al) target with a substrate temperature of 300°C. The bias voltage applied to the substrate- holder was -500 V. Under these conditions, coatings were deposited at rotation speeds of the substrate of 3rpm, the deposition time is 4 hours for the multilayers coatings. These parameters have already been optimized for single layer of CrN and CrAlN in a previous study [36].

A schematic illustration of Cr/CrN and Cr/CrN/CrAlN multilayers is shown in Figure 2.

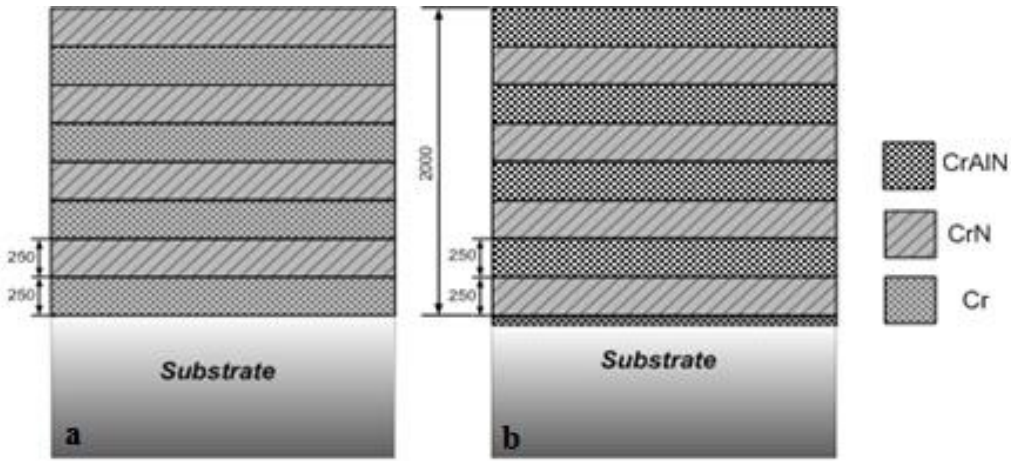

Figure 2 – Schematic illustration of the multilayer coatings: (a) Cr/CrN and (b) Cr/CrN/CrAlN

2.2 Characterizations

A Field-Emission Scanning Electron Microscope (FEG-SEM) (JEOL JSM 6400F) was used to observe the surface morphology, to verify the thickness and microstructure (observation of cross-sections) of the PVD layers. The composition of the coatings was determined by EDS Energy Dispersive Spectrometry microanalysis.

A continuous rotating tribometer (TriboX, CSM Instruments) permitted to obtain the friction coefficient and wear resistance using a 5 N load, a 3 *cm*/*s* sliding speed and 30 *m* as the sliding distance against an aluminum ball (6mm of diameter). A 3D optical profilometer (VEECO, Wyko NT-1100) was used to observe the wear profiles.

A home-made demolding test was carried out for the practical evaluation of the adhesion tendency of a cast alloy to study the material transfer from the pin to the mold and vice versa. The pins studied in this test are used as core in the die (Fig. 3a). A product of the casting process is a pin casting assembly that serves as a sample for the traction test (Fig. 3b) on the mechanical tensile testing machine.

The casting alloy employed is AlSi8Cu3 (chemical composition (w. %): Si 7; Cu 3; Zn 0.8; Fe 0.65; Mg 0.42; Mn 0.27; Ni 0.15; Cr 0.01; Pb 0.06; Sn 0.03; Ti 0.06; Al (balance)). For melting, a resistance furnace with a ceramic crucible was used.

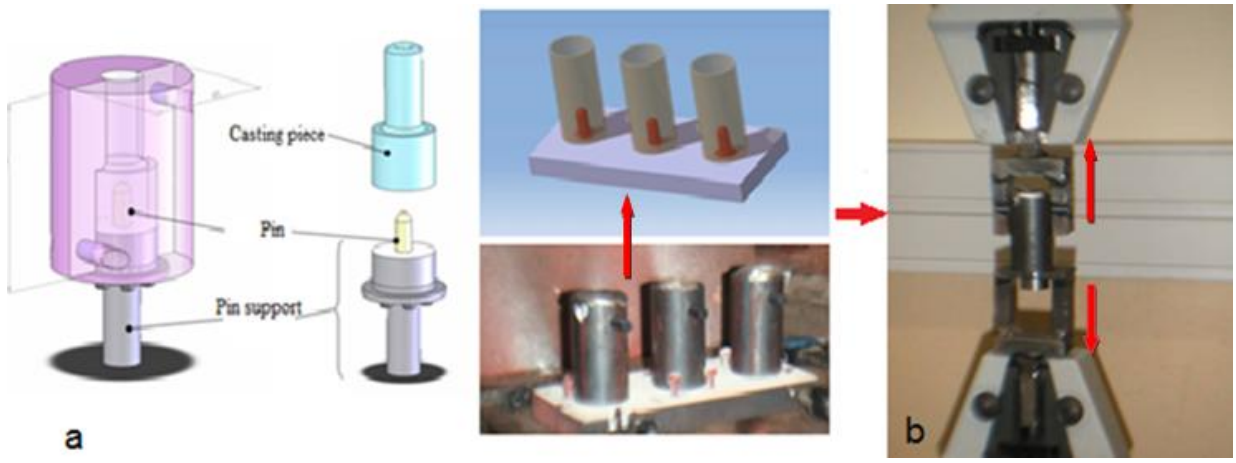

Figure 3 – (a) Casting simulation system, (b) pin-casting assembly on the mechanical tensile testing machine

Into the specially designed steel die (Fig. 3a) preheated at 350 °C, casting process was performed by gravity melt pouring of the casting alloy at 750 °C. After each casting (solidification) cycle, the process is reproduced for next sample.

Demolding tests were carried out by a 30kN mechanical tensile testing machine (LLOYD Instruments LR 30K) equipped with a suitable fastening system. The traction speed was 2 *mm/mn*. The Force / Displacement curves provide us information on the bonding and adhesion tendency between aluminum and the treated surfaces. Optical observations are realized on all the pins to study the adhesive mechanisms and material transfer.

3 Results and discussion

3.1 Structural analyzes by XRD (X-ray diffraction)

The DRX analyzes were carried out on silicon substrates (100) using a cobalt anticathode (λKα (Co) = 1.78 Å) in Bragg-Brentano configuration (θ / 2θ). Figure.4 shows the diffractograms of the films of the CrN and CrAlN layers.

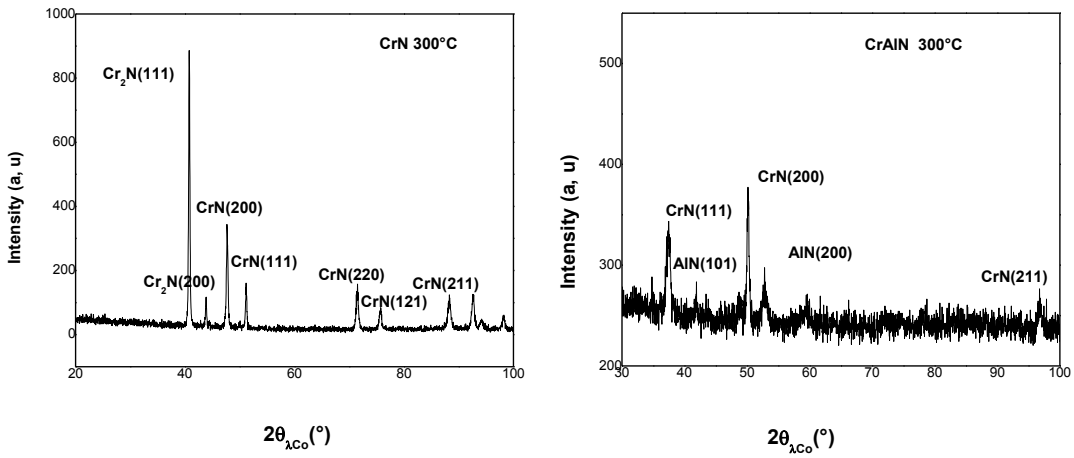

Figure 4 – XRD analysis of (A) CrN and (B) CrAlN coatings

On the diffractogram of the CrN layer, we observe a large peak showing that the layer is almost amorphous, this peak is perhaps the result of the contribution of several others such as: the Cr2N (111) observed at 40.52°, Cr2N (200) observed at 44.58° from the hexagonal Cr2N phase (hcp) and CrN (200) observed at 48.20° from the face-centered cubic phase of CrN

(fcc). The preferred orientation for CrN coating growth, the (1 1 1) direction is observed at 51.6°. We also observe crystal planes (220), (121), (211) at diffraction angles of 72.42°, 76.83 and 88.5°.

For the deposition of CrAlN, it is observed that the addition of Al to the binary system (CrN) improves its crystallinity and promotes the formation of different crystalline phases. Indeed, we observe the existence at the angle 38.64° of a peak which corresponds to the cubic phase of CrN (1 1 1) and at 43.64°, either the cubic phase CrN (111) or the hexagonal phase AlN (10 1). The phase of CrN (20 0) appears at angle 51.34° with a high intensity peak. Cubic AlN (c-AlN) is observed at 52.49°, at 97° CrAlN has the same phase as CrN (the CrN phase (211).

3.2 Cross section images and surface morphology

The cross sections were observed by SEM on coated Si substrates. Figure 5 (a, c) show that all films have a columnar structure which is in agreement with previous studies [37, 38]. The Cr underlayer that improves adhesion and facilitates nucleation of the CrN layer [39], is 125-128 *nm* thick. The total thickness of the Cr/CrN/CrAlN and the Cr/CrN coating is 2.4 and 2.8 μm, respectively.

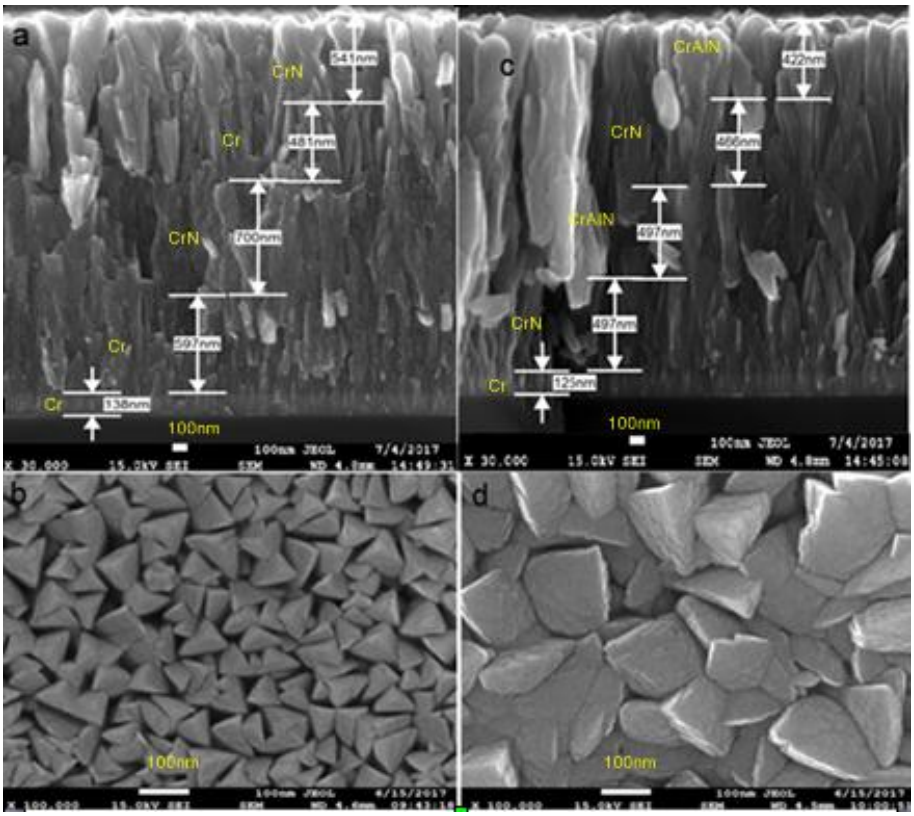

Figure 5 – SEM cross section images and surface morphology of: (a, b) Cr / CrN and (c, d) Cr / CrN / CrAlN multilayer.

Besides, one can note in Figure 5 (b,d), that the Cr/CrN multilayer coating has a denser structure and a grain size of about 50 nm, smaller than that of the Cr/ CrN/CrAlN multilayer one, around 150 nm. Similar results were obtained in comparative studies of CrN and CrAlN coatings [40, 41]. In literature, it is shown that the CrN coating has an AFM RMS roughness of 8.694 *nm* whereas CrAlN coating exhibits a roughness of about 21.853 *nm* [42]. This can explain the difference observed in the surface morphology micrographs in figure4.

3.3 EDS analyzes:

EDS analyzes of the different layers and multilayers obtained were carried out. The chemical compositions of the different films are presented in the table 1.

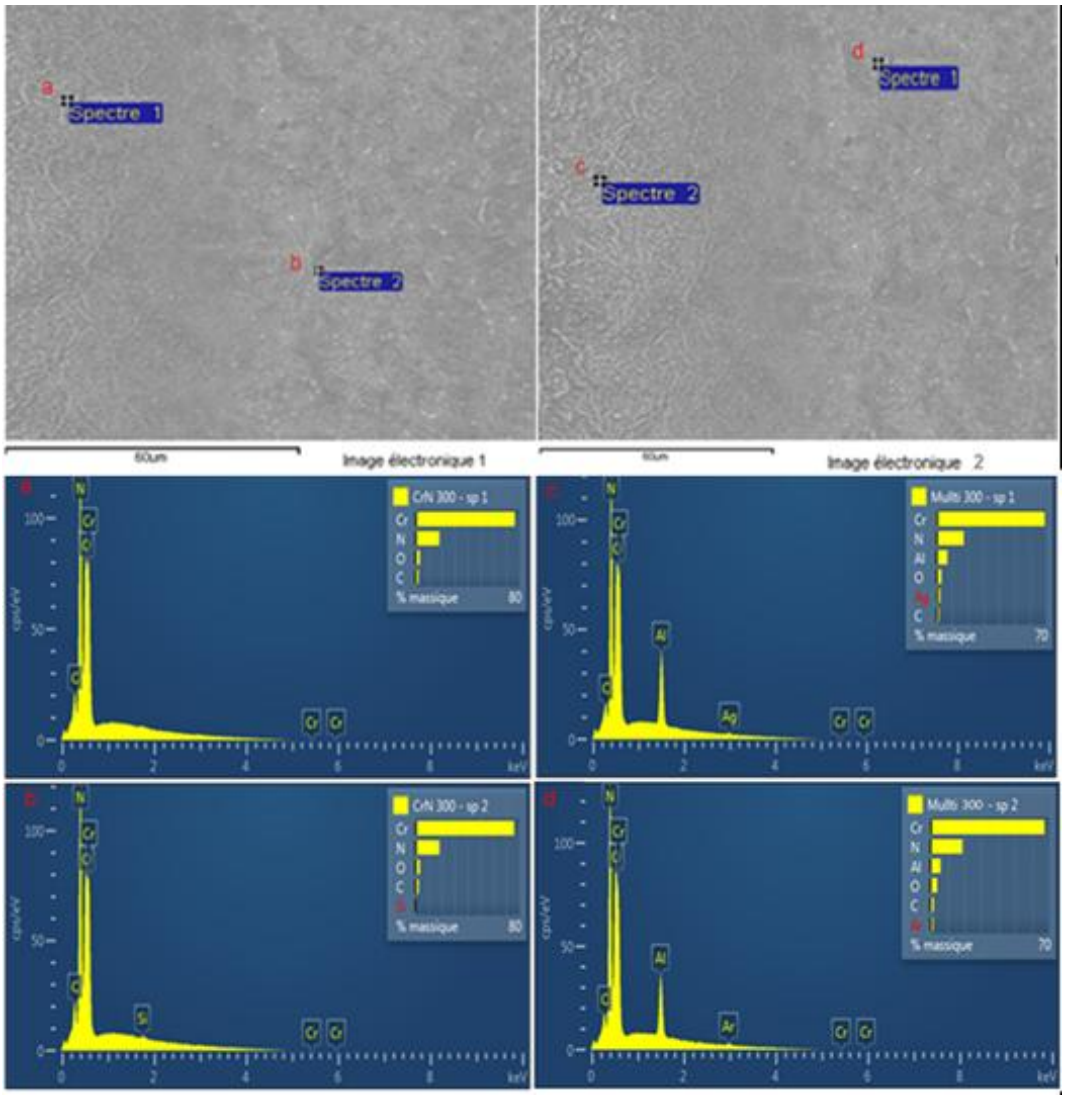

Figure 6 – EDS spectrum and elemental analysis of: a,b) CrN monolayer an c,d)Cr/CrN/CrCrN/CrAlN multilayer film Chemical composition

Table 1 – Chemical composition of the layers and multilayers obtained by EDS

Coating	N(%at)	O	Al	Cr
CrN	43,1	6,5	---	50,4
CrAlN	44,0	5,3	18,8	32
Cr/CrN	43,1	6,5	---	50,4
Cr/CrN/CrAl/N	42,71	6,01	8,08	43,2

3.4 Wear tests

Several experiments with ball-on-disc wear tests were performed to understand the tribological behavior of the studied multilayers and thus to observe the interaction of aluminum alloy with the surface of the coatings. We specifically studied the beginning of the tribological test because it that can be assimilated to the highest force required to start the movement of the spindle during the demolding test.

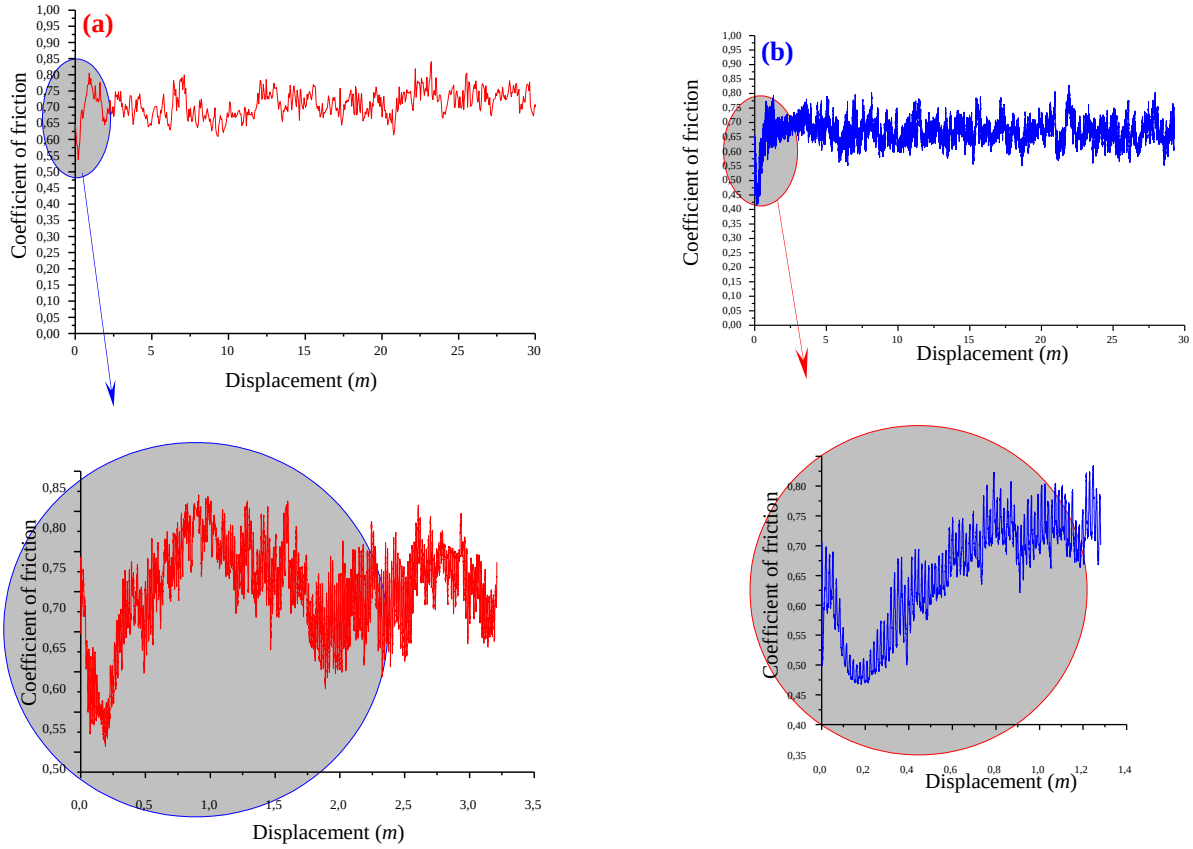

Figure 7: Evolution of the friction coefficient of: (a) Cr/CrN/CrAlN and (b) Cr/CrN multilayer against aluminum ball. The insets shows the evolution of the COF at the beginning of the wear test

Figure 7 shows the coefficient of friction (COF) of Cr/CrN/CrAlN and Cr/CrN multilayers against aluminum balls. For all tribological couples, the evolution of the COF shows the same tendency at the beginning of the test: a run-in step followed by increase of the COF down to a steady state. The Cr/CrN multilayer shows the lowest COF. (average of is 0.65) while the Cr/CrN/CrAlN multilayer one is in average 0.70.Indeed, at the beginning of the wear test, the COF of the Cr/CrN/CrAlN multilayer decreases from 0.75 to 0.55 and from 0.65 to 0.45 for the Cr/CrN one. Then it increases abruptly up to 0.80 for Cr/CrN/CrAlN and 0.75 for Cr/CrN to stabilize at the average values.

This phenomenon of the coefficient of friction increase is less pronounced for the Cr/CrN multilayer (Fig 6) and can be explained by the grain size that is greater for the Cr/CrN/CrAlN multilayer (Fig.5). The COF decrease can be explained by the sliding movement before the contact of the asperities. Then, the contact pressure causes a plastic deformation, adhesion and the consequent formation of local junctions which explains the sudden increase of the COF.

The figure 8 shows the EDS analysis of the residues obtained on the traces of wear carried out on the coatings of Cr / CrN and and of / CrN / CrAlN.

In order to study the wear mechanism, two small areas in Figure 8 of the Cr / CrN and Cr / CrN / CrAlN wear trace are chosen for SEM observation and EDS analyzes. In general, after the wear tests, a non-negligible oxide level is obtained, which indicates the existence of a local temperature in the contact zone between the ball and the surface. We observe a significant amount of iron, which shows that the ball rubs on the substrate and this proves that the two multilayers are completely torn off. The significant presence of Aluminum is due to wear by the adhesion of the aluminum of the ball to the substrate.

A 3D optical profilometer (VEECO, Wyko NT-1100) was used to observe the wear profiles.

We observed in figure 9, by optical microscope, the two balls used for the wear test. We notice that the wear on the ball used for the Cr / CrN / CrAlN multilayer is more pronounced than that used for the Cr / CrN multilayer, which probably due to the roughness of the coatings, the grains are larger for the Cr film. / CrN / CrAlN.

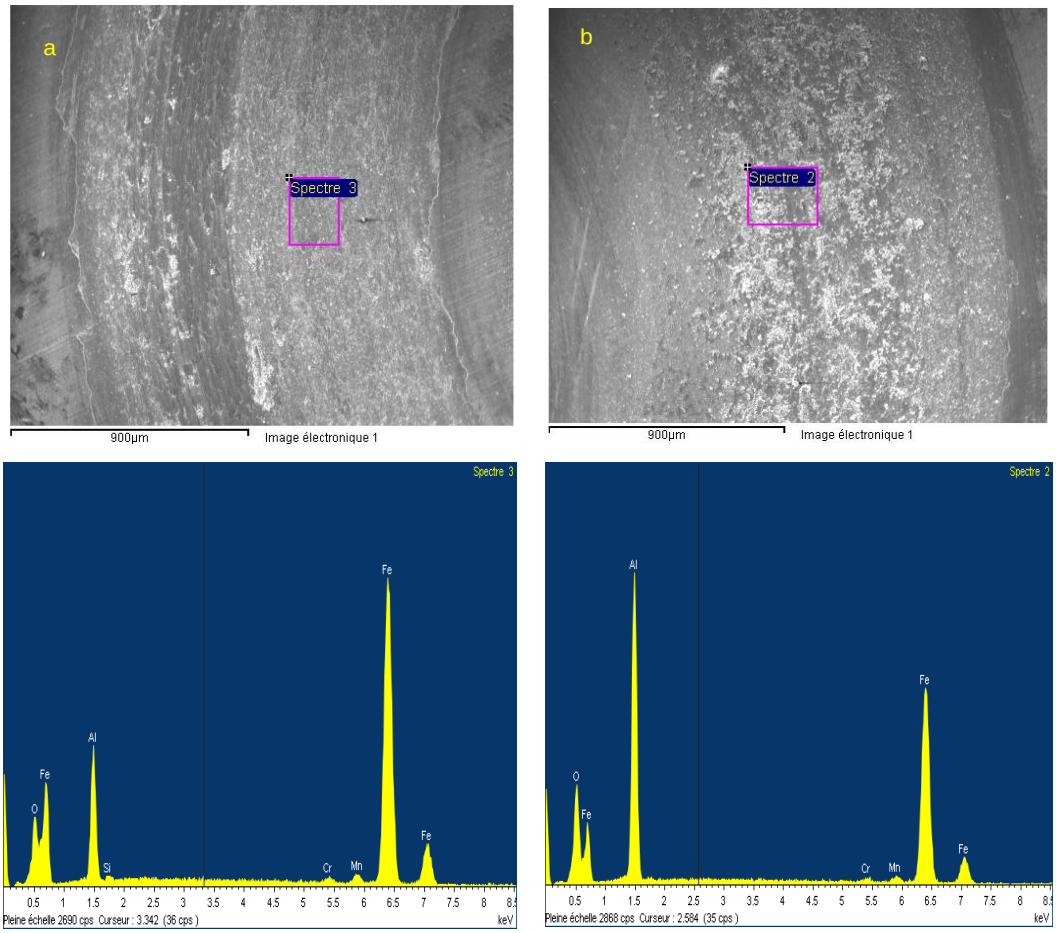

Figure 8 – EDS analysis of the residues obtained on the traces of wear track of the coatings (a) Cr / CrN and (b) Cr / CrN / CrAlN

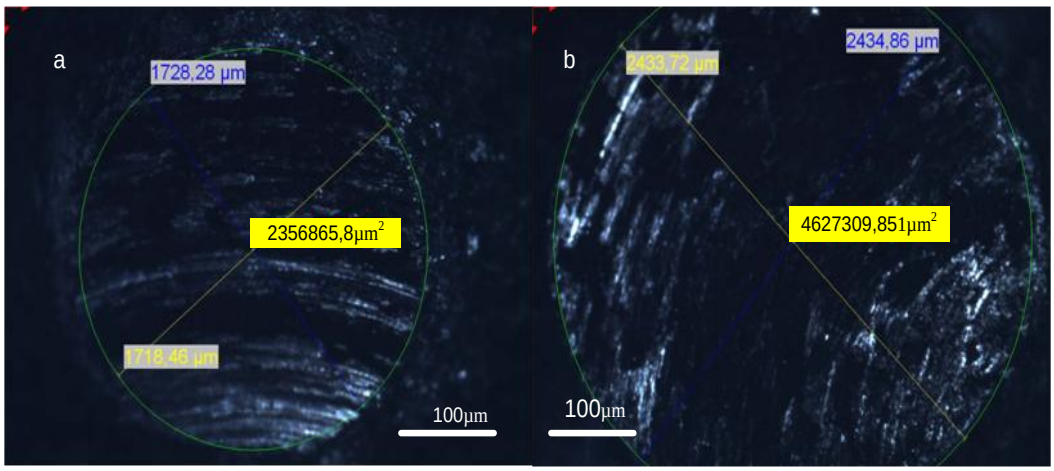

Fig. 9: optical images of the two balls after the wear test (a) Cr/CrN et (b) Cr/CrN/CrAlN

3.5 Demolding test

The curves obtained with the average values of the maximum demolding forces measured during the various tests show that the movement of the samples starts when the force is the highest. Then, from the maximum, the force decreases constantly until the complete demolding of the pin (Figure.10). The curves obtained for the Cr/CrN coated samples are different from the Cr/CrN/CrAlN ones.The maximum force required to start the movement of the pins is lower in the case of the

Cr/CrN/CrAlN coated samples. The start of the movement is followed by an oscillation of the force (saw tooth shape curve) and by almost linear decrease of the demolding force. Similar results were obtained by Terek et al. [42] is also obvious that the oscillation of the demolding forces is much pronounced for Cr/CrN/CrAlN multilayer than for the Cr/CrN one.

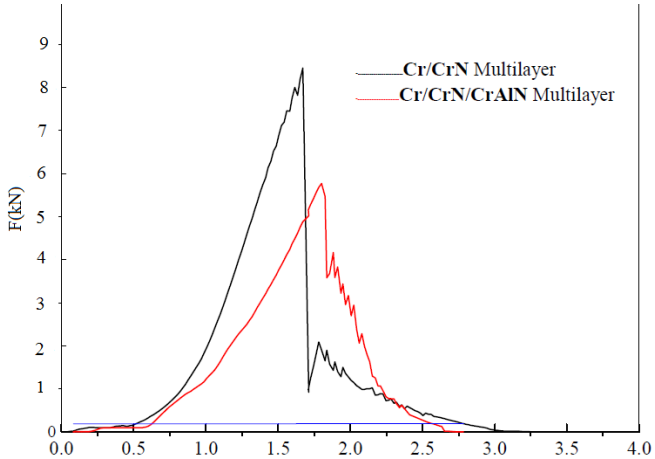

Fig. 10 – Demolding curves obtained for Cr/CrN and Cr/CrN/CrAlN coated pins.

Optical observations of pins after demolding are presented in Figure 11. Qualitative information about the soldering tendency of cast alloy towards pin material and galling processes that develop during the pin ejection are obvious.

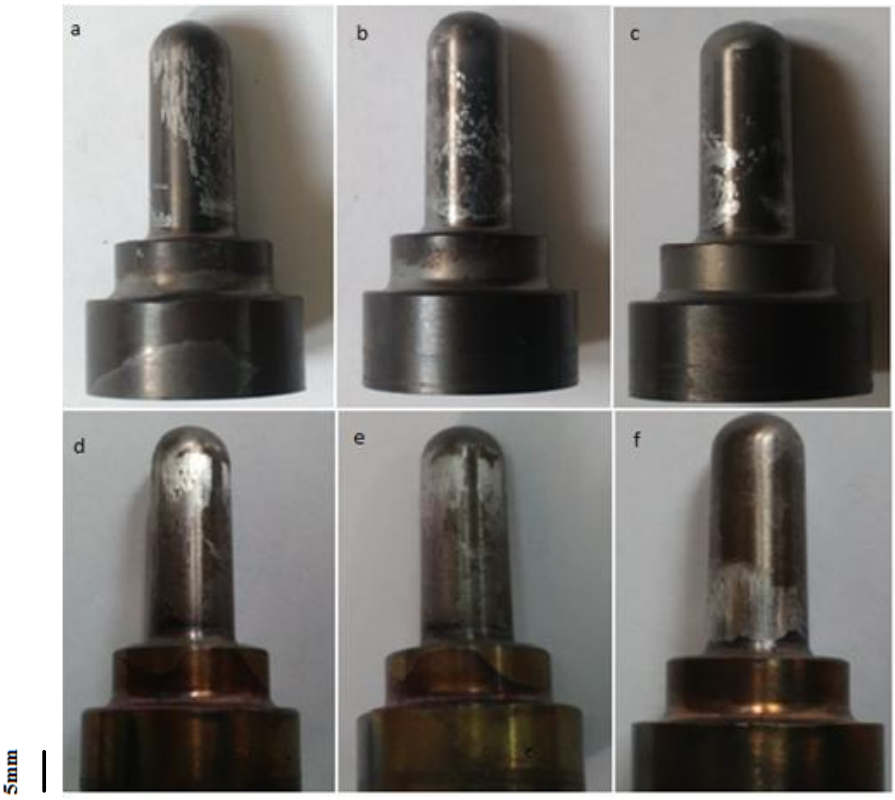

Figure 11 – Pictures of the Cr/CrN/CrAlN (a,b,c,) and Cr/CrN (d,e,f) coated pins after demolding test

These observations show that the alloy tracks are thicker and cover a smaller surface in the case of the Cr/CrN/CrAlN coated pins, (Fig.11 d, e, f). Besides, only fine spots on large surface are observed for Cr/CrN coated pins. The comparison is made on observations of three Cr/CrN/CrAlN multilayer coated pins and three others coated with Cr / CrN multilayer.

The result of the addition of the bonding surfaces observed on the pins shows that the bonding is more important on Cr/CrN coated pins.

4 Conclusions

This study deals with the application of commonly used surface treatments to decrease the adhesion phenomena and to increase the resistance to soldering of aluminum alloy casting tools. This work is focused on Cr/CrN and Cr/CrN/CrAlN multilayers, the pretreatment process consisting of quenching and tempering process leads to improve bonding between the coatings and the X38CrMoV8 steel substrate.

Home-made demolding tests showed that the demolding force is higher for the Cr/CrN multilayer coating, probably due to its low roughness and therefore to the adhesive strength.

The soldering is present on approximately on one third of the surface on the Cr/CrN/CrAlN multilayer coated pins and on more than a half on the Cr/CrN one. Nevertheless the spots are fine and cover a large surface for Cr/CrN multilayer coating while the layers were observed to be thicker and covered smaller surfaces in the case of Cr/CrN/CrAlN coated pins.

The growth mechanism for Cr/CrN/CrAlN multi-layer resulted in rough columnar structures which are clearly observed on the surface morphology SEM images, meaning a greater grain size. Different grain size and surface roughness influence the coefficient of friction, especially at the beginning of the friction test. It can be conclude that the performance of PVD coatings on hot working tools of casting aluminum alloys greatly rely on surface roughness and surface morphology of tool parts. The identified built-up layer of cast alloy is formed by the effects of mechanical soldering.

The casting sticking affects the production efficiency, the tool performance during operation and lowers the casting quality. This study showed that both Cr/CrN and Cr/CrN/CrAlN coatings are prone to Al–Si alloy sticking and galling.

Therefore, in order to avoid intense adhesive wear and high demolding force, the surface morphology of the coatings should be carefully considered in production of hot working tools intended of casting aluminum alloys.

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