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Performance of chromium nitride based coatings under plastic processing conditions

L. Cunha^{a,*}, M. Andritschky^a, K. Pischow^b, Z. Wang^b, A. Zarychta^c, A.S. Miranda^d, A.M. Cunha^e

^aDepartamento de Física, Universidade do Minho, Campus de Gualtar, 4710-057 Braga, Portugal

^bSavcor Coatings Oy, Insinöörinkatu 7, FIN-50100 Mikkeli, Finland

^cInstitute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

^dDepartamento de Engenharia Mecânica, Universidade do Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

^eDepartamento de Engenharia de Polímeros, Universidade do Minho, Campus de Azurém, 4800-058 Guimarães, Portugal

Abstract

Chromium nitride based coatings were produced in the form of monolithic and multilayer coatings, by DC and RF reactive magnetron sputtering. These coatings were deposited onto stainless steel and tool steel substrates. Chromium nitride coatings have proved to be wear and corrosion resistant. The combination of these characteristics was necessary to protect surfaces during plastic processing. In order to select the best coatings, some mechanical and tribological tests were performed. Hardness and Young's modulus of the produced coatings were measured by nano-indentation, adhesion was assessed by scratch test experiments and wear was evaluated by performing pin-on-disc experiments. The behaviour of these coatings in chemically aggressive environments was studied by different corrosion experiments. The behaviour of these coatings in thermoplastic processing equipment was also studied. The abrasive and corrosive wear of chromium nitride based coatings was assessed under relatively high pressures and temperatures and was compared with the surface wear of hardened tool steel substrate by heat treatment, nitrided by chemical-heat treatment, or protected by hard chromium coating deposited in a galvanic way. Monolithic coatings were harder, and had higher Young modulus and adhesion when compared with multilayered coatings. Their wear resistance, measured by pin-on-disc test, was between two and three orders of magnitude higher, but multilayer coatings showed significantly better corrosion resistance. In general, monolithic and multilayer coatings have significantly better behaviour when compared with traditional processes of protecting functional surfaces for plastic transformation equipment. The measured wear of the best performance obtained by chromium nitride based coatings can be more than two orders of magnitude lower than nitrided surfaces or hard chromium coated samples. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Chromium nitride; Monolithic coatings; Multilayer coatings

^{*}Corresponding author. Tel.: +351-253-604-330; fax: +351-253-678-981. E-mail address: lcunha@fisica.uminho.pt (L. Cunha).

1. Introduction

The hardness of transition metal nitride coatings in combination with their corrosion resistance have been used to protect surfaces of various functional components like tools and machine parts. Titanium nitride is the best known of these materials, but its relatively low oxidation resistance increased the interest of studying other transition metal nitride, such as chromium nitride based coatings. However, defects like pores and pinholes, typical of PVD deposition processes can strongly affect the potential of these coatings to protect surfaces in chemically aggressive environments [1-6]. Some solutions have been tried, such as the deposition of a metallic layer between the substrate and the nitride coating [7,8], or producing multilayer coatings with nitride layers altering with metallic layers [9,10]. The latter case was very interesting because there was the possibility of maintaining a hard nitride component while reducing the residual stress by relaxation with the metal layers. Some authors have produced double layered coatings [11]. References on improving the mechanical properties have been found by producing graded layered coatings [12-16].

In this work, results concerning mechanical and tribological properties and also the corrosion behaviour of chromium nitride based coatings deposited in the form of monolithic and multilayer coatings were presented. The behaviour of these coatings under the flow of glass reinforced thermoplastic melt was also tested.

2. Experimental

The coatings were produced by reactive magnetron sputtering (RF and DC) and deposited on polished stainless steel (AISI 316) and heat-treated tool steel (AISI O1) substrates. In order to improve adhesion and corrosion resistance, a Cr layer with a thickness of approximately 100 nm was deposited prior to the deposition of monolithic coatings. These coatings were deposited using a pure Cr target in an argon and nitrogen atmosphere under a working pressure of 0.45 Pa. The multilaver coatings, with chromium nitride layers alternating with Ti ductile metal layers, were deposited using pure Ti and Cr targets. Each monolayer was deposited when the substrates were static in front of the Ti target with an Ar atmosphere, for Ti layers, and in front of Cr target with mixed Ar and N₂ atmosphere, for CrN layers. The most important deposition parameters and some characteristics of the coatings are summarised in Table 1. The composition of the coatings was obtained by Rutherford backscattering spectroscopy (RBS) and the composition of the top layer was

Table 1 Some deposition conditions and characteristics of coatings:^a

Samples	Туре	V _s [V]	T _s [°C]	% N ₂	P [W]	t _C [μm]	t _m [nm]	n	ρ [10 ³ kg/m ³]
CrN-2	M	0	300	40	500	3.6			3.5
CrN-8	M	-50	300	40	500	3.1	_		3.6
CrN-12	M	-50	300	40	600	3.5	-	_	4.9
CrN-13	M	-50	350	40	500	3.2	-	_	3.6
CrN-14	M	-50	350	40	600	3.9	_	-	4.3
Cr N -15	M	- 25	300	40	500	3.0	=	-	4.4
CtN-16	M	- 75	300	40	500	3.1		-	4.2
CrN-23	M	0	300	40	600	4.5	-	-	3.2
G/CrN-2	M	-50	~ 180	40	796	6.0	-	-	4.7
G/CrN-6	M	-80	~ 140	40	760	3.0	_	_	5.1
riCrN-13	ML	-50	~ 180	40	Cr-786 Ti-612	3.5	10-12	300	4.1
ΓiCrN-14	ML	- 50	~ 180	40	Cr-763 Ti-643	3.0	100	30	4.9
CiCrN-16	ML	-80	~ 140	40	Cr-735 Ti-726	4.1	14	300	5.1
ΓiCτN-17	ML	-80	~ 140	40	Cr-770 Ti-970	5.1	8	600	4.3
ľiCrN-18	ML	-50	~ 200	40	Cr-840 Ti-690	2.5	83-100	30	5.1
iCrN-19	ML	-80	~ 200 `	40	Cr-840 Ti-690	2.4	80-100	30	5.0
TiCrN-20	ML	-50	~ 200	40	Cr-840 Ti-690	2.2	7-10	300	3.5

^aM-monolithic coating; ML-multilayer coating; V_s -substrate bias; T_s -substrate deposition temperature; N_2 -percentage of nitrogen in the working atmosphere; P-source power; t_c -thickness; t_m -thickness of monolayers in multilayer coatings; n-number of monolayers; ρ -density of the coatings.

obtained by X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES).

Coatings residual stresses were calculated using Stoney's equation [17] after measuring the parabolic deflection of the substrate by laser triangulation. Hardness and Young's modulus perpendicular to the surface were determined using a Berkovich indenter, with instrumented indentation depths of 100, 200 and 300 nm. Adhesion was determined by scratch test employing a Rockwell diamond tip with a radius of 200 µm, a loading rate of 100 N/min and a scratching speed of 10 mm/min. For each sample five tests were performed. The damage was analysed by optical microscopy and quantified by: (i) first critical load (L_{C1}) corresponding to the occurrence of coating damage by conformal cracking, in and at the edge of the scratch track; (ii) second critical load (L_{C2}) corresponding the removal from the substrate of more than 50% of the coating.

To characterise the tribological behaviour of the coatings a pin-on-disc tribometer was used with a $\mathrm{Si}_3\mathrm{N}_4$ ceramic pin, with a flat circular end with a diameter of 1 mm. This test was performed at room temperature and at 300°C, in air with relative humidity between 50 and 60%, sliding speed of 0.5 m/s under a normal load of 5 N. The volume of removed coating was calculated as the product of the wear track length multiplied by the average of the measured area of the transversal wear profile obtained by a profilometer. The profile cross-section was measured in five different positions. The test was finished when the sliding distance was sufficiently large to define the wear evolution correctly

or when the coating was completely worn. A load cell incorporated into the pin-on-disc tester enables the continuous measurement of friction. The wear rate (K) was determined as the volume of removed coating divided by normal load and sliding distance.

The behaviour of the samples in aggressive environ--ments was studied by performing three different corrosion experiments [21]. Aqueous corrosion was studied by open circuit potential (OCP) and potentiodynamic polarisation experiments in HCl (1 M) solution. A potentiostat, a standard calomel electrode (SCE) as a reference, and platinum as the auxiliary electrode were used. OCP was measured during one h. Potentiodynamic polarisation measurements were done with an applied voltage from -500 mV to +800 mV and a sweep rate of 15 mV/min, with an upper current limit of 5 mA. The corrodkote experiment consists of a test where the samples were covered by a chemically aggressive paste in an environment with a temperature of $38 \pm 2^{\circ}$ C and 80% < RH < 90%, during 16 h [22]. This test was developed and standardised for electroplated coatings and was applied for reasons of comparison; however, it reveals coating defects and may cause fracture in case of excessive internal stresses. Hot corrosion experiments were performed by placing the samples in a furnace, inside a glass tube, at a temperature of 350°C during 1 h, with a gas flow of approximately 100 sccm containing HCl-H₂O. The HCl-H₂O flow originated from an HCl 36% aqueous solution. A test cycle consists of heating the samples in a laboratory atmosphere to 350°C during one h, exposing them for

Table 2
Some mechanical and tribological properties of monolithic and multilayer coatings^a

Samples	σ _R [GPa]	Hardness [GPa]	E [GPa]	$L_{ m C1}$ [N]	L_{C2} [N]	K (RT) [m ² /N]	K (300°C) [m ² /N]
AISI 316 subs	-	4.3 ± 0.7	_		-		
AISI O1 subs	= .	7.2 ± 1.3	-	_	_	_	_
CrN-2	-0.7 ± 0.05	22.5 ± 3.8	283.4 ± 28.4	(1)	52.3	-	_
CrN-8	-4.9 ± 0.5	28.6 ± 7.7	297.5 ± 64.6	14.0	34.9	7.7×10^{-16}	3.2×10^{-14}
CrN-12	-4.8 ± 0.4	25.4 ± 4.2	303.9 ± 39.5	22.1	52.1	_	_
CrN-13	-3.8 ± 0.3	25.4 ± 5.7	290.3 ± 39.5	14.7	45.8	_	_
CrN-14	-3.7 ± 0.3	25.2 ± 6.1	300.6 ± 25.5	14.7	39.1	1.5×10^{-15}	3.7×10^{-14}
CrN-15	-5.2 ± 0.4	19.2 ± 4.8	260.6 ± 45.5	(1)	74.9	9.9×10^{-16}	6.6×10^{-14}
CrN-16	-6.2 ± 0.5	24.8 ± 4.0	302.2 ± 38.1	24.1	71.2	1.2×10^{-15}	5.5×10^{-14}
CrN-23 (matt)	-2.0 ± 0.1	4.0 ± 1.0	126.3 ± 29.1	20.4	62.8	1.6×10^{-15}	2.7×10^{-14}
CrN-23 (met.)		27.8 ± 5.8	367.1 ± 66.0				
G/CrN-2	-	19.3 ± 2.1	261.3 ± 19.2	_	_	_	_
G/CrN-6	-2.6 ± 0.3	21.2 ± 1.6	315.4 ± 23.3	(1)	54.0	1.5×10^{-15}	1.2×10^{-14}
TiCrN-13	< -1	10.5 ± 1.4	176.5 ± 15.7	(1)	46.3	_	_
TiCrN-14	< -1	10.1 ± 1.3	176.9 ± 29.0	(1)	45.3	_	_
TiCrN-16	< -1	13.5 ± 0.9	234.0 ± 15.3	_		3.5×10^{-12}	1.2×10^{-11}
TiCrN-17	< -1	14.1 ± 0.5	233.1 ± 16.5	_	_	2.8×10^{-12}	5.8×10^{-11}
TiCrN-18	< -1	12.0 ± 1.0	212.3 ± 16.7	(1)	39.1	1.7×10^{-12}	2.3×10^{-12}
TiCrN-19	< -1	12.4 ± 1.7	215.4 ± 21.4	(1)	46.9	3.9×10^{-12}	4.0×10^{-12}
TiCrN-20	< -1	15.7 ± 1.8	250.1 ± 16.3	(1)	37.2	6.5×10^{-12}	2.6×10^{-12}

 $^{^{}a}\sigma_{R}$ -Residual stress; E-Young's modulus measured; L_{C1} -First critical load; L_{C2} -Second critical load; K (RT)-Wear rate (pin-on-disc) at room temperature; K (300°C)-Wear rate (pin-on-disc) at 300°C; (1) not measurable; not measured.

one h to the aggressive atmosphere and cooling to room temperature (1 h). The samples were analysed by XPS and RBS before and after this corrosion test.

For investigation of the wear resistance in plastic processing a nozzle was used, adapted from the 'DKI platelet method' [18-20]. The coated samples were arranged in the die which-was fitted to a reciprocating scan injection moulding machine, Krauss-Maffei KM 60-120A. The plastic used for tests was polyamide 6.6 with 35% of glass fibre (Ultramid A3 G7). The molten plastic was pressed through a rectangular aperture $(0.3 \times 7.0 \text{ mm})$ at a temperature between 290 and 320°C and a pressure between 10 and 12 MPa. This plastic was selected because it was extremely abrasive and corrosive [19,20]. The same profilometer was used to measure the wear track cross-section caused by the abrasive polymeric melt. The volume of removed material was calculated as the product of the length of the wear track multiplied by the average of the measured transversal area at five different positions. Wear was determined as the volume of removed coating, divided by the mass of polyamide passed throw.

3. Results and discussion

3.1. Mechanical and tribological properties

The composition of the coatings was stoichiometric (Cr:N 1:1) or slightly sub-stoichiometric regarding nitrogen (Cr:N 1:0.9). All the coatings have a metallic grey appearance and reflectivity, except CrN-23 which mixes matte and metallic grey areas. In general this heterogeneous appearance was found to occur with unbiased samples [23].

Monolithic coatings can be highly stressed. In general the compressive residual stresses increase with ion bombardment during deposition (Fig. 1). Multilayer coatings have significantly lower residual stresses what was related with the relaxation due to metallic layers (Table 2).

The average hardness of monolithic coatings was approximately 23 GPa, but the hardness of multilayered coatings was much lower. In fact the hardness of these coatings was approximately the average of the measured hardness of monolithic chromium nitride and of metallic titanium coatings (~ 5 GPa). Values between 10.1 and 15.7 GPa have been found in this work. This hardness difference has a strong influence on the wear resistance of these coatings as shown in Fig. 2. The multilayer coatings were softer and additionally can present some adhesion problems as a consequence of the several interfaces within the coating. The failure mode observed during scratch tests is related to hardness [23]. Harder coatings show conformal crack-

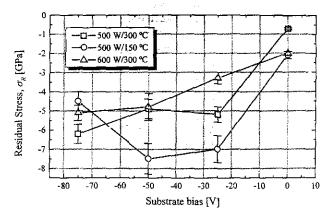


Fig. 1. The residual stress of three series of monolithic chromium nitride coatings, under the variation of the substrate bias.

ing for low loads ($L_{\rm C1}$). The softer monolithic coatings fail by small cracks within the scratch track. The quantity of these small cracks increases as the indenter load increases, until the coating was scratched off. This kind of failure also happens on multilayer coatings. In comparison to titanium/chromium nitride multilayer coatings, chromium nitride monolithic coatings are harder and have much higher wear resistance, but they can be highly stressed.

3.2. Corrosion behaviour

The corrosion tests show two corrosion mechanisms: coating corrosion and interfacial corrosion [21]. During aqueous corrosion tests, the corrosion products enter into the aqueous solution but the surface coating composition observed by XPS was not changed. The current measured in monolithic coatings was caused by the coating decomposition and corrosion through pinholes. In multilayer coatings, the corrosion through pinholes

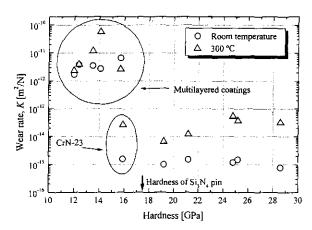


Fig. 2. Relation between wear rates, measured after pin-on-disc experiments, and hardness for chromium nitride based coatings. The hardness of the coating CrN-23 was an average of the measured hardness on matte and metallic grey areas.

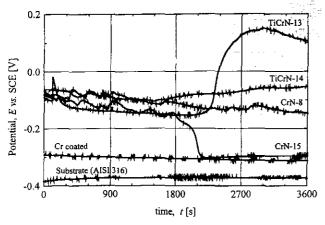


Fig. 3. Open circuit potential curves in a solution of HCl (1 M) for the uncoated substrate (stainless steel AISI 316) and five PVD coated samples: a chromium coating, two monolithic chromium nitride coatings (CrN-8, CrN-15) and two multilayered titanium/chromium nitride coatings (TiCrN-13, TiCrN-14). The deposition conditions of the chromium coating were identical of the interlayer of the monolithic coatings.

happens to a significantly lower extent. In general OCP tests do not show solution penetration through the coating as was observed for most monolithic coatings (Fig. 3). In fact, while the free corrosion potential of monolithic coatings can achieve the potential of the interlayer, for multilayer coatings a passive layer that causes an increase of the open circuit potential seems to occur. This was clearly shown for the TiCrN-13 sample. The current densities of multilayer coatings can be one order of magnitude lower than for monolithic coatings. The transpassive zone was achieved at higher potentials and, as can be seen in Fig. 4, the best multilayer coatings show significantly lower anodic current densities when compared with monolithic chromium nitride coatings.

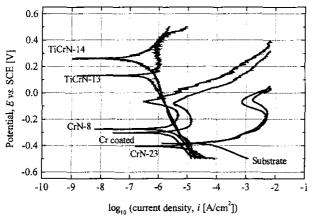


Fig. 4. Polarisation curves for the substrate, and PVD five coated samples: a chromium coating, two monolithic chromium nitride coatings (CrN-8, CrN-23) and two multilayered titanium/chromium nitride coatings (TiCrN-13, TiCrN-14). The deposition conditions of the chromium coating were identical of the interlayer of the monolithic coatings.

The hot corrosion experiments do not remove the corrosion products of the surface and they can be identified. During the experiment the coatings were attacked by the aggressive environment and XPS spectra show that there was a strong loss of nitrogen at the surface, and a strong rise of oxygen and a little increase of chlorine. A detailed study of chemical mechanisms occurring during the corrosion reactions on the coatings surface was published previously by the authors [21].

The interfacial corrosion was mainly caused by defects which allow the chemically aggressive agents reach the substrate or the interlayer. OCP tests show that this penetration was more probable on monolithic coatings. More important information was obtained from corrodkote and hot corrosion experiments. Cycles of these experiments were performed until the coatings were damaged. The coatings after corrodkote tests were identified as damaged when defects were detected by visual inspection. In hot corrosion experiments the coating detaches itself in the form of a fine grey powder. SEM images of this powder demonstrated that the coating fragmented and detached from the surface, along with fragments of substrate. However, it seems that the coating was not destroyed by the corrosive attack [21]. This suggests that corrosion attacks primarily the metallic substrate through pores or pinholes. In general the multilayer coatings could resist more cycles than monolithic coatings (Fig. 5). Multilayer titanium/chromium nitride coatings have better corrosion resistance than monolithic coatings, mainly because the quantity of defects like pores and pinholes was lower and there was no open porosity in this type of films.

3.3. Wear tests in plastic processing

The wear results of monolithic and multilayered coatings in plastic processing conditions, have been

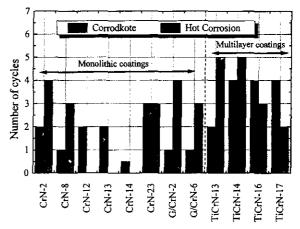


Fig. 5. Number of corrodkote and hot corrosion cycles up to failure performed by chromium nitride based coatings.

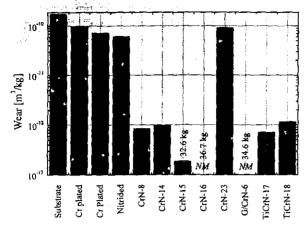


Fig. 6. Wear rates measured after the injection moulding experiments on heat treated steel substrate (AISI O1), two hard chromium coated samples (Cr plated), a nitrided sample, six monolithic chromium nitride coatings and two multilayered titanium/chromium nitride coatings. The quantities written in each column correspond to the amount of polymer used for each sample during the test. The wear in two of the samples (CrN-16, G/CrN-6) was not enough to be measured (NM).

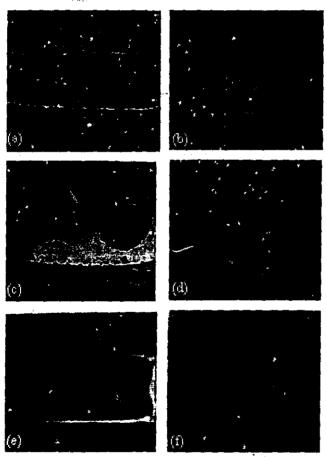


Fig. 7. Images of the surface of the samples after the polymer test. The track caused by the polymer flow was observed in all the samples: (A) heat-treated steel; (B) nitrided steel; (C) CrN-23; (D) CrN-16; (E) Cr plated coating; (F) TiCrN-18. Can be clearly seen corroded areas on A and B. On C the lighter area corresponds to detached coating. On D the wear track can be seen only because the difference in colour, but the profilometer could not measure any wear.

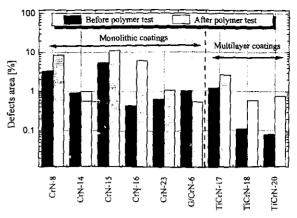


Fig. 8. Percentage of the observed area occupied by defects on the coatings wear track before and after the polymer injection-moulding test

performed in a special device attached to the plasticating barrel of the injection moulding machine (Fig. 6). The wear results of the heat-treated substrate, chromium-plated sample and plasma nitrided samples are also presented. Wear measured on the samples depends on the quantity of plastic used during the tests in the injection-moulding machine.

Nitriding and electrodepositing of hard chromium are two of the traditional ways of protecting the surfaces against wear in plastic processing. Using these surface treatments the wear was reduced between 1.7 and 2.8 times compared with a hardened steel substrate. However the PVD chromium nitride based coatings can reduce wear more than two orders of magnitude. In some of the samples (CrN-16, G/CrN-6) the profile of the wear track could not be detected even when using a higher quantity of polymer. One of the PVD coated samples (CrN-23) had a poor behaviour: after the test parts of the coating were completely worn (Fig. 7), apparently due to the inhomogenity of this coating. For the CrN-23 coating, hardnesses of 4 GPa (matte grey areas) and 27.8 GPa (metallic grey areas) were measured. The behaviour of the multilayered coatings was also good. The wear of these coatings was similar to the wear of some monolithic coatings (Fig. 6).

The surface of the coatings was observed using optical microscopy and some small corrosive spots being detected on the surface. The number of these spots was lower in the case of the multilayer coatings (Fig. 8). In the case of the uncoated substrate or the nitrided or Cr plated samples large corroded areas can be observed (Fig. 7).

4. Conclusions

Monolithic chromium nitride coatings showed a wear resistance, in pin-on-disc experiments, between two and three orders of magnitude better than the multilayer titanium/chromium nitride coatings. The reason of of this improved behaviour can be attributed to their higher hardness and adhesion.

Multilayer titanium/chromium nitride coatings showed better behaviour in chemically aggressive environments. This better performance was due to the low open porosity of these coatings. Monolithic...coatings can present pores that allow the aggressive agents to reach the substrate and cause interfacial corrosion.

In the plastic processing experiments the nitride coatings have significantly better wear resistance than the uncoated substrate or the substrates whose surfaces were protected by traditional processes—like nitriding or hard chromium coating deposition. However coatings with inhomogeneties, in terms of appearance and mechanical properties (CrN-23), can be produced with grounded substrate. In this situation a poor behaviour was observed and the coating can be rapidly worn. The best results have been achieved with the samples deposited under higher ion bombardement: CrN-16, C/CrN-6 produced with a substrate bias of -75 V and -80 V, respectively.

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