

Characterization of Microstructure and Mechanical Properties of Chromium Carbide Coatings Formed on 100Cr6 and X200Cr12 Steels through Conversion Process

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ABSTRACT

A number of deposition processes can be utilized to produce transition metal carbide and nitride coatings. The choice of the deposition process is determined by various parameters, such as coating thickness, use of the deposit, adhesion of the deposit to the substrate, and production cost. In this study, hard chromium carbide coatings are prepared through a conversion treatment on 100Cr6 and X200Cr12 steels. This process consists of electrode position of chromium on the surface of 100Cr6 and X200Cr12 steels followed by a diffusion annealing treatment at 1000 °C and 1100 °C for 1 and 2 hours. All the conversion treatments conducted result in a chromium carbide layer formation on the steel surface. The constituting phases of the formed layer depend on the temperature of the diffusion annealing treatment and the duration at this temperature. In addition, the X-Ray Diffraction (XRD) analysis confirms the presence of chromium oxide in addition to chromium carbides. The micro-hardness profile indicates that both samples attain high hardness values, which are more than four times higher than the base substrate hardness. A well-defined transition zone is also observed, offering the highest adhesion between the deposited layers and the substrate. Scratch tests, in addition to micro-hardness values, determine the improved performance of the X200Cr12 steel sample.

Keywords-100Cr6 steel; X200Cr12 steel; coating; diffusion; hardness; scratch test

I. INTRODUCTION

In recent decades, the use of thin film deposition techniques has experienced a rapid growth, particularly in the processing of materials or mechanical components including the essential tools employed in machining operations. The physical and chemical properties of thin films are often substantially different from solid materials [1, 2]. The chemical composition, morphology, and crystallographic structure of the deposited films are linked to the interaction between the source material and the underlying substrate during the film fabrication production process [3].

Carbides and nitrides of transition metals are frequently used due to their interesting properties, such as high melting point, high hardness, good resistance to abrasive and adhesive wear, good resistance to corrosion, and hot oxidation [4, 5].

Several methods and techniques have been developed to obtain hard films of chromium carbides on steels. Among these techniques are Physical Vapor Deposition (PVD) and the Thermo-Reactive Diffusion (TRD) deposition technique [6-9]. In the case of PVD films the thickness is generally much less than 10 μm , which in turn reduces their resistance to high stress. Moreover, TRD coatings require very long processing times at high temperatures. Common industrial methods exist that can be combined to produce chromium carbide coatings.

Authors in [10] optimized the conditions to produce chromium carbide layers on alloy steels. The results showed that the best chromium carbide layers were obtained through diffusion annealing carried out at temperatures between 1000 $^{\circ}\text{C}$ and 1100 $^{\circ}\text{C}$ and holding times between 1 and 2 hours. The deposited layer of chromium on case hardened steel is completely transformed into chromium carbide after a holding time of 50 min at 1100 $^{\circ}\text{C}$ [11]. The best properties are usually obtained by expensive methods, such as PVD and Chemical Vapor Deposition (CVD). These properties can be obtained in a simple and inexpensive manner in comparison to PVD or CVD techniques [12].

In this work, chromium carbide coatings with high hardness, good adhesive properties, and sufficient thickness were produced on steel with significant carbon content, based on a two-step process. The comparison of the obtained results allowed identifying the optimal conditions for the production of chromium carbide thin films.

II. MATERIALS AND METHODS

In the two step process, the first step is to deposit a layer of chromium electrochemically on the surface of the treated steel. The second step is to perform heat treatment at temperatures that allow the carbon to diffuse to the surface of the steel. At that point it will react with the chromium to form chromium carbides in the entire outer layer. The quality of the chromium carbide layers obtained on the surface of 100Cr6 and X200Cr12 steels will then be evaluated.

A. Materials

For the conversion treatment, 100Cr6 and X200Cr12 were chosen, which are widely utilized in the mechanical industry. 100Cr6 is a low alloy steel containing 1% carbon and 1.5%

chromium. X200Cr12 consists of 2% carbon and 12% chromium. The detailed chemical composition of the two steel types is given in Table I.

TABLE I. CHEMICAL COMPOSITION OF 100Cr6 AND X200Cr12 (WT-%)

	Steel	
	X200Cr12	100Cr6
C	1.98	1.00
Si	0.19	0.35
Mn	0.25	0.26
Cr	11.78	1.43
Mo	0.08	0.08
Ni	0.241	0.12
N	0.03	0.13
Fe	85.96	96.56

Figure 1 illustrates the initial microstructures of 100Cr6 and X200Cr12 in which carbides with perlite are present. The initial hardness of these steels is 240 HV and 270 HV, respectively.

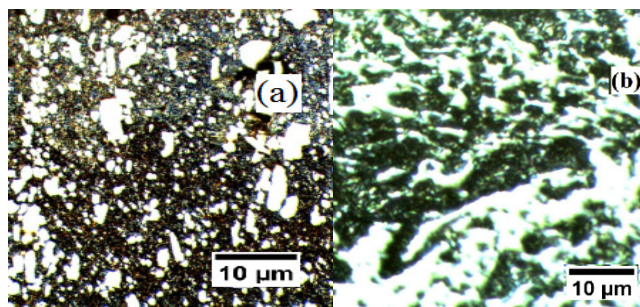


Fig. 1. Initial microstructures at a cross section of (a) 100Cr6 and (b) X200Cr12.

Prior to any treatment, all samples are polished with sandpaper to reduce the roughness to a maximum R_a value of 0.50 μm . In addition, prior to the hard chrome plating process, degreasing and rinsing operations are carried out on the substrates to ensure the high quality of the deposit.

B. Conversion Treatment

In the first step of the conversion treatment, electrolytic chromium plating is performed to cover the substrates with 10 μm thick layer. The chemical composition of chrome plating bath and the conditions under which it is performed are: 250 g/l of chromic acid, 1.5 g/l sulfuric acid, antimony lead anode, bath temperature of 40 $^{\circ}\text{C}$, and the holding time in the bath is 30 min.

In the second step, the diffusion annealing treatment, the chromium layer deposited on the surface is transformed into a layer of chromium carbide. The process is performed for 1 and 2 hours at temperatures of 1000 $^{\circ}\text{C}$ and 1100 $^{\circ}\text{C}$ with a heating rate of ~ 200 $^{\circ}\text{C}/\text{h}$. After the annealing, the samples are cooled in the furnace for 24 hours, corresponding to a cooling rate of approximately ~ 50 $^{\circ}\text{C}/\text{h}$. This process is accomplished in an electric muffle furnace with a high-precision PID temperature controller. The above process is depicted in Figure 2.

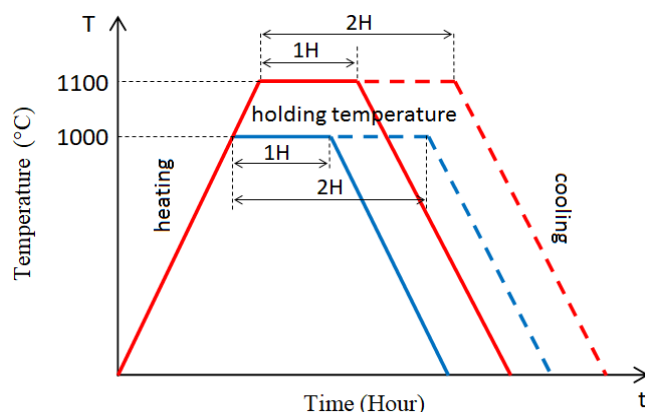


Fig. 2. Schematic representation of the annealing treatment.

C. Phase and Microstructure Characterization

The study employed optical microscopy (LEICADMLM), scanning electron microscopy (TESCAN VEGA3) micrographs, as well as XRD (BRUKER-AXS type D8) to observe and identify the chromium carbide layers obtained on the surface of the treated steels. The BRUKER-AXS type D8 utilized a Copper K- α radiation source with a wavelength λ value of 1.5418 Å.

D. Microhardness and Scratch Tests

A Vickers micro-hardness tester (MVK-H2) with 25 and 200 g loads is utilized to perform the micro-hardness profile measurements. The test includes measurements from the surface to the depth of chromium carbide layers formed on 100Cr6 and X200Cr12 steels surfaces. Three micro-hardness measurements were averaged to ensure a consistent reproducibility of the results.

For the scratch test, a REVETEST device with two sensors is utilized. The first sensor is an acoustic emission sensor, which measures the load at which damage occurs in situ. The second sensor allows the direct recording of tangential force, enabling the determination of the instantaneous coefficient of friction. During the test the surface of a sample is scratched using a diamond indenter with a hemispherical diameter of 200 μm moving at a constant speed of 5 mm/min. Moreover, the test is performed in a single pass under an increasing normal load at a rate of 1 N/mm of travelled distance, varying from 0 N to 200 N. This type of test makes it possible to determine the Critical Load (LC), which corresponds to the appearance of damage on the tested layer.

III. RESULTS AND DISCUSSION

A. Microstructure and Phase Analysis

In Figure 3, after the first step of the conversion treatment, the evolution of the microstructure of the formed layers on the surfaces of 100Cr6 and X200Cr12 steels is presented. In Figures 3a and 3b a distinct formation of a chromium layer on the surfaces of the investigated steel samples is observed. Additionally, the formed layers on the substrates are uniform, homogeneous, compact, and adherent along the surfaces.

After chromium plating, the initial microstructures of 100Cr6 and X200Cr12 substrates remain unchanged. On

100Cr6 a microstructure containing very fine grains randomly distributed is observed. In contrast, X200Cr12 exhibits a net microstructure. The thickness of the deposited layer of chromium on the surface of the both steels is of the order of 10 μm and practically equivalent. Figure 4 illustrates the spectra of XRD analysis.

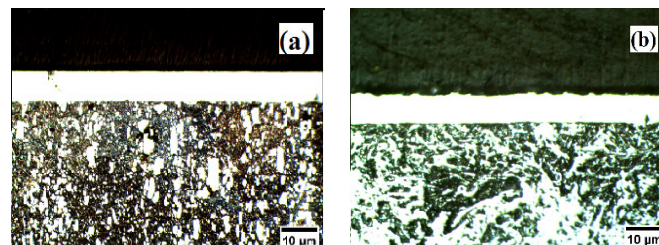


Fig. 3. Cross section of: (a) 100Cr6 and (b) X200Cr12 after the chromium electroplating.

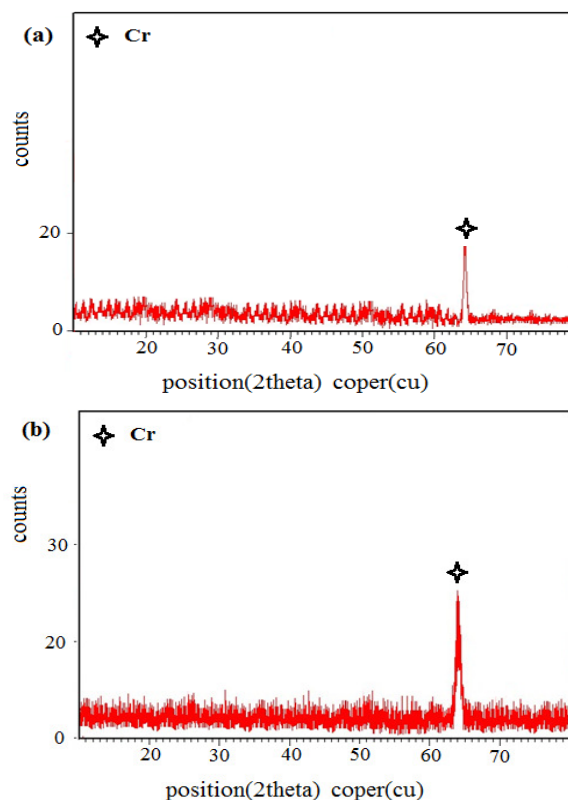


Fig. 4. XRD spectra of: (a) 100Cr6 and (b) X200Cr12 after the chromium electroplating.

The process reveals that the formed layer on the surface of both under-study steels is composed solely of chromium (Cr, ICSD PDF No: 151375), with a total absence of representative peaks of the substrate or chromium carbides. On the one hand, this can be attributed to the relatively large thickness of the deposited chromium layer (more than 10 μm) on the steels, which masks the appearance of the substrate phases. On the other hand, it is attributed to the absence of chromium carbides on the surface of steels before diffusion annealing.

Figure 5 displays the microstructures of 100Cr6 and X200Cr12 after the diffusion annealing treatment for 2 hours at 1100 °C, which corresponds to the second step of the conversion treatment. These structures exhibit changes with the appearance of chromium carbides and oxides. For 100Cr6 steel, the surface layer remains homogeneous and compact, and retains its initial thickness apart from the oxide layer. This behavior is also observed on samples treated at 1000 °C for 1 or 2 hours. The layers remain intact and show no signs of degradation. This is mainly attributed to their chemical composition and their ability to form protective oxides. On the contrary, X200Cr12 appears as two distinct layers as was already the case for the samples subjected to treatment at 1000 °C, for one hour or 2 hours.

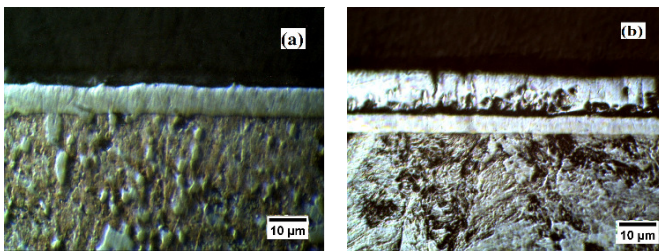


Fig. 5. Cross section of: (a) 100Cr6 and (b) X200Cr12 after the annealing diffusion treatment at 1100 °C for 2 hours.

Chromium carbides and chromium oxides, such as Cr_7C_3 , Cr_{23}C_6 , and Cr_2O_3 , were identified by XRD. The produced coatings exhibited no visible cracks or porosity, as observed in Figure 6. The micro-hardness profile confirms the inter diffusion of chromium in the substrate, which cannot be observed directly.

No detectable metallic chromium was identified through the XRD analysis of 100Cr6 and X200Cr12 samples subjected to 2 hour diffusion annealing at 1100 °C. This suggests that the precipitated chromium carbide phase fully extends to the outer layer of the deposit, aligning with the findings of [13]. According to the aforementioned changes on the surface of 100Cr6 and X200Cr12 steels, the following remarks were made:

- The chromium plating layers deposited on the surface of 100Cr6 and X200Cr12 samples are completely transformed into chromium carbides of Cr_{23}C_6 (ICSDPDFN°:43377), Cr_7C_3 (ICSDPDFNo:36-1482), and Cr_3C_2 (ICSD15086) types, with the presence of chromium oxide type Cr_2O_3 (ICSD 173470).
- Heating X200Cr12 steel at 1100 °C for 2 hours forms a chromium carbide layer on its surface. The final thickness of this layer is 7 µm, which is less than the initial thickness of the chromium deposit of 10 µm, excluding the chromium oxide layer. This decrease can be attributed to the diffusion of carbon from the substrate to the chromium layer and to the diffusion of chromium into the substrate. When the chromium atom is larger than the carbon atom, inter-diffusion causes a change in the size of the chromium layer. In addition, a large part of the chromium reacts with oxygen

to form a chromium oxide layer. Authors in [14] revealed that the diffusion of carbon from the substrate to the chromium layer reduces the decarburization phenomenon during the treatment.

- Under the same diffusion treatment conditions the chromium carbide layer of 100Cr6 steel is thicker than that of X200Cr12 steel. In addition, this layer is practically equivalent to the initial chromium layer. This explains the minimal reaction of 100Cr6 steel with oxygen and the lack of a chromium oxide layer, unlike the observation in X200Cr12 steel.
- According to XRD spectra, the formation of a chromium oxide (Cr_2O_3) in the chromium carbide layer during the diffusion annealing treatment is justified by the diffusion annealing treatment conditions. These conditions were carried out in an ambient atmosphere containing oxygen.

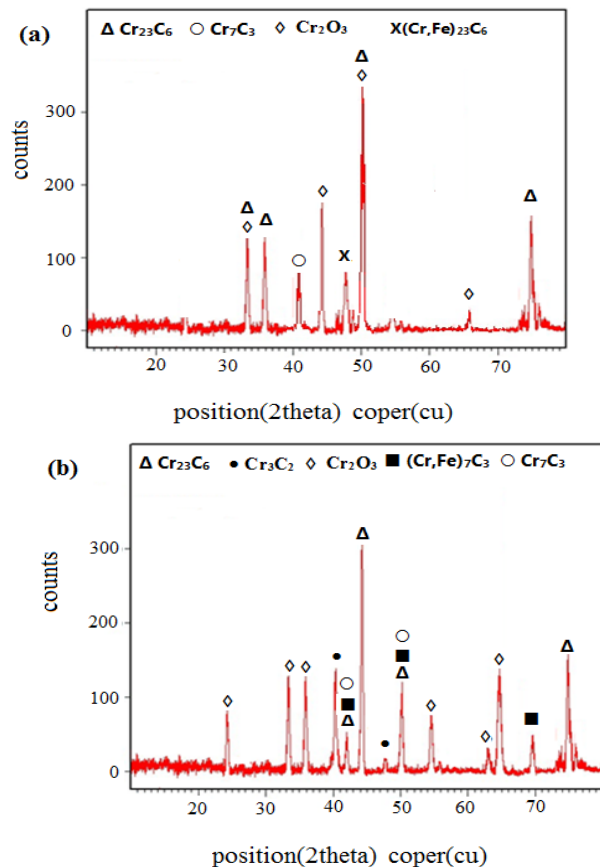


Fig. 6. XRD spectra of: (a) 100Cr6 and (b) X200Cr12 after annealing diffusion at 1100 °C for 2 hours.

- The presence of complex carbides $\text{X}(\text{Cr,Fe})_{23}\text{C}_6$ for 100Cr6 steel and $(\text{Cr, Fe})_7\text{C}_3$ for X200Cr12 steel can be attributed to the diffusion of iron and carbon atoms into the chromium layer during diffusion annealing. These phases do not exist for the annealing treatment of 1 hour. In [15], regarding 100Cr6, the ternary carbide $(\text{Cr,Fe})_7\text{C}_3$ only begins to appear at 1000 °C and for holding times higher than 1 hour.

Authors in [16] also disclosed the presence of the ternary carbide $(\text{Cr, Fe})_{23}\text{C}_6$ and the total transformation of the binary carbide Cr_7C_3 into a ternary carbide of type $(\text{Cr, Fe})_7\text{C}_3$ on an XC100 steel after an annealing treatment at 1000 °C for 2 hours.

B. Micro-Hardness

The micro-hardness values of the chromium carbide coating, the transition zone, and the substrate of 100Cr6 and X200Cr12 steels after chromium electroplating and diffusion annealing treatment at 1100 °C for 2 hours are presented in Figure 7.

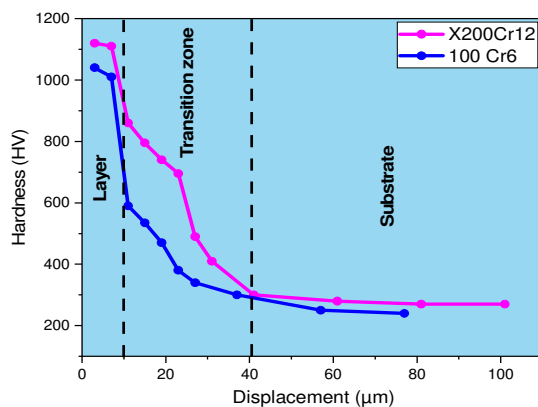


Fig.7. Micro-hardness profiles of the chromium carbide layer, the transition zone, and the substrate on 100Cr6 and X200Cr12 steels after the conversion treatment.

The micro-hardness profiles in different zones confirm the reaction between the substrate and the chromium layer formed

in the first step. The diffusion of carbon atoms from the substrate to the chromium layer is favored by the small size of the carbon atoms and by the concentration gradient between the substrate and the chromium layer [14].

The micro-hardness values obtained for the different zones revealed the following:

- On the surface of X200Cr12 steel at a temperature of 1100 °C for 2 hours, the highest hardness obtained is 1120 HV. At a temperature of 1000 °C and for 1 hour, the hardness did not exceed 880 HV.
- On the surface of 100Cr6 steel at a temperature of 1100 °C for 2 hours, the highest hardness obtained is 1040 HV. At a temperature of 1000 °C and for 1 hour, the hardness did not exceed 810 HV.
- The micro-hardness of the chromium carbide layer and the transition zone in X200Cr12 steel is slightly higher in comparison to that of 100Cr6 steel, justified by the higher chromium and carbon contents in X200Cr12 steel.
- In both steels, the presence of a transition zone with an intermediate hardness between the hardness of the chromium carbide layer and the substrate provides good support for the layer, preventing its deterioration under severe stress.

The results of the comparison between the properties of the chromium carbide layers obtained in this study and those obtained by other researchers using processes and techniques other than conversion treatment are summarized in Table II [17-23].

TABLE II. PROPERTIES OF THE CHROMIUM CARBIDE LAYERS IN COMPARISON TO OTHER STUDIES

Process	Temperature (°C)	Time (h)	Substrate	Phases	Thickness (μm)	Hardness		Reference
						Coating	Substrate	
TRD	850	2 and 8	100Cr6	Cr_7C_3 , $(\text{Cr, Fe})_7\text{C}_3$	3-5	1730-1920 HV _{0.025}	723 HV _{0.025}	[17]
	800-1000	4, 5, and 6	AISI W1	Cr_7C_3 , Cr_{23}C_6 , Cr_3C_2	5-9	771-1700 HV _{0.025}	500 HV _{0.025}	[18]
PVD	900	4 and 10	X210Cr12	Cr , $(\text{Cr, Fe})_7\text{C}_3$	7 and 11	1846 HV _{0.025}	290 HV _{0.025}	[19]
Molten salt	1000	8, 10, and 12	AISI W1	$(\text{Cr, Fe})_7\text{C}_3$, Cr_7C_3 , Fe_5C_2	20-30	1782 HV _{0.025}	600 HV _{0.025}	[20]
Pack chromizing	1000	1	100Cr6	Cr_{23}C_6 , Cr_7C_3 , CrC , $(\text{Cr, Fe})_7\text{C}_3$, $(\text{Cr, Fe})_{23}\text{C}_6$	10	970 HV _{0.025}	720 HV _{0.025}	[21]
Magnetron sputtering	700-1000	1	XC100	Cr_7C_3 , Cr_{23}C_6 , $(\text{Cr, Fe})_7\text{C}_3$, $((\text{Cr, Fe})_{23}\text{C}_6)$, Cr_2O_3	2.64	2100 HV _{0.1}	520 HV _{0.025}	[22]
Conversion treatment	700-1100	1 and 2	16Mn2	Cr , Cr_7C_3 , Cr_{23}C_6 , Cr_2O_3	20	1580-2110 HV _{0.02}	320 HV _{0.02}	[23]
Conversion treatment	1100	2	X210Cr12	Cr_7C_3 , Cr_3C_2 , Cr_{23}C_6 , Cr_2O_3 , $(\text{Cr, Fe})_7\text{C}_3$	7	1120 HV _{0.025}	270 HV _{0.200}	Present work
			100Cr6	Cr_7C_3 , Cr_{23}C_6 , Cr_2O_3 , $(\text{Cr, Fe})_{23}\text{C}_6$	12	1040 HV _{0.025}	240 HV _{0.200}	

In Table II, it can be observed that the formed phases in this work, achieved through the conversion treatment, are practically very similar to the phases obtained by the other techniques. This phase consists of chromium carbides, chromium Ferro-carbides, or chromium oxides. In addition, the

micro-hardness obtained on the 100Cr6 and X210Cr12 steels exceeds 1100 HV. Therefore, it can be concluded that the conversion treatment enables the production of chromium carbide layers equivalent to those obtained by other processes and techniques. Table III depicts the critical loads for the

appearance of spalling and cracks at the bottom of traces of chromium carbide layers after the single-pass scratch test at increasing load. The load corresponding to the apparition of scaling is estimated from micrographs of the scratch mark.

TABLE III. CRITICAL LOADS AT THE BOTTOM OF TRACES OF CHROMIUM CARBIDE LAYERS

Steel type	Holding time at 1100 °C	Critical damage load LC (N)	Load corresponding to the apparition of scaling*(N)
100Cr6	1h	75	~140
	2h	85	~180
X200Cr12	1h	125	~180
	2h	135	>200

C. Scratch Resistance

Scratch tests with increasing load give rise to two different types of damage:

- Cracks at the bottom of the trace that propagates toward the back of the indenter. These kind of cracks are present in all samples. They appear in a curvilinear form perpendicular to the axis of the scratch, as portrayed in Figure 8a. According to [24], this type of crack is characteristic to stresses induced by a spherical indenter. The stress is concentrated just below the contact surface between the indenter and the tested surface. The actuate cracks oriented toward the front of the indenter suggest damage due to compressive stresses during the sliding of the indenter.
- Cohesive flaking that appears at the end of the scratch and at the edges of the traces. These cracks appear for high applied load. Theses palls are related to the intrinsic fragility of the coating and are generally observed in relatively thick layers, as presented in Figure 8b.

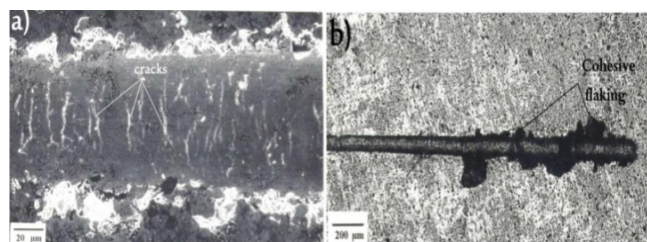


Fig. 8. Damage types after increasing load scratch test: (a) cracks at the bottom of the trace and (b) cohesive flaking at the end of the scratch.

The study revealed no cases of adhesive delamination at the coating/substrate interface. This behavior is confirmed by [24]. Specifically, the high temperature deposited coatings display high adhesion due to inter-diffusion at the interface (metallurgical continuity) and are, therefore, resistant to damage by interface decohesion.

The analysis of the samples subjected to the single-pass scratch test under increasing load reveals that the LC consistently coincides with the appearance of cracks at the base of the scratch paths. The results, as shown in Table III, indicate that the chromium carbide layer obtained on X200Cr12 steel showed no spalling, even at the maximum applied load.

IV. CONCLUSIONS

The present experimental work led to the following conclusions:

- Electrolytic coatings of chromium followed by various diffusion annealing treatments on 100Cr6 and X200Cr12 steels make it possible to obtain chromium carbide layers on the surface of treated steels.
- A transition zone with a depth of 30 μm is formed below the chromium carbide layer. The micro-hardness of the transition zone, intermediate between the layer and the substrate, provides significant support for the chromium carbide layer.
- The Metallographic analysis and X-Ray Diffraction (XRD) revealed that the chromium carbide layer formed on the surface of 100Cr6 and X200Cr12 consist of Cr_{23}C_6 , Cr_7C_3 and Cr_3C_2 chromium carbides, with the presence of Cr_2O_3 .
- The micro-hardness profile and the scratch test demonstrate that the X200Cr12 steel sample exhibited superior performance in comparison to 100Cr6.

The results of the present study, which include very high hardness and high friction resistance, enabled benefitting from this technology in many fields: aeronautics and space to protect components exposed to extreme conditions, automotive to enhance the durability of parts subjected to intense friction, oil and gas to safeguard equipment, such as pipelines, against erosion and corrosion, machine tools to extend the lifespan of cutting tools and molds.

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