



Annealing study of thin chromium layers on cemented steel substrates



Younes Benarioua ^{a,*}, Rabeh Boubaaya ^b, Jacky Lesage ^c, Didier Chicot ^c

^a Département de Génie Mécanique, Faculté de Technologies, Université de M'sila, B.P 166 Ichbilila, 28000 M'sila, Algeria

^b Département de Génie Mécanique, Faculté de Technologies, Université de Jijel, B.P 98, Jijel 18000, Algeria

^c Laboratoire de Mécanique de Lille, LML UMR 8107, U.S.T Lille, IUTA GMP, BP 179, 59 653 Villeneuve d'Ascq, France

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ABSTRACT

The present study has been conducted in the objective of obtaining chromium carbide layers using a conversion treatment involving three main steps: 1) carburizing treatment in order to increase the amount of carbon atoms at the steel surface, 2) deposition of a pure chromium thin layer using electrolytic method and finally, 3) annealing treatment used for carbon diffusion and carbide formation until complete transformation of chromium layer. Depending on the treatment temperature, partial or complete conversion is obtained as a result of the diffusion process. The role of the annealing temperature on transformation rates of chromium into chromium carbide films was investigated. It is shown that for 1 h of treatment the layer is totally transformed at 1100 °C. Hardness and adhesion of layers produced were found to increase whatever the annealing treatment even for partial transformation at the lower annealing temperatures.

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1. Introduction

In the past two decades, carbides of transition metals coatings have proven to be one of the most effective materials to increase the service life of machine components, cutting tools and forming tools, especially to improve wear and corrosion resistance [1–4]. Among these metal compounds, chromium carbide presents the best chemical and mechanical behavior under severe conditions of use. Recent research on chromium carbide films emphasized their oxidation and corrosion resistance and suggested that they have great potential for replacing electroplated hard chromium as protective coatings [5–8]. Hard films of chromium carbides can be obtained by physical vapor deposition (PVD) [9,10] or thermo-reactive deposition/diffusion (TRD) technique [3,11]. In the case of PVD films the thickness is generally much less than 10 μm that renders them improper too resist high contact pressure and moreover their adhesion to the substrate is lower than that obtained with other types of coating techniques. The TRD coatings need very long treatment times at high temperatures. As an example, an 8 μm thick chromium carbide layer was obtained on an AISI D2 steel with a treatment at 1030 °C during 4 h [3] and a 13 μm layer was obtained with a treatment at 1000 °C during 3 h [11]. There are very common industrial methods that can be combined anyway to produce chromium carbide coatings.

In order to produce a coating of high hardness, good adhesive properties and of sufficient thickness to resist severe conditions of loading, we suggest here an alternative technique based on a three-step process using only standard industrial methods. The first step

has the objective of increasing the carbon content at the surface of the substrate using a standard carburizing process. The second step is to coat the carburized material with a layer of chromium. The last step is a thermal treatment at temperatures which would allow the diffusion of carbon toward the surface of the material where it is likely to combine with chromium to form chromium carbides throughout the coating. In these work we want to determine the experimental conditions that will allow the complete conversion of the chromium coating into a uniform carbide layer of high hardness and good adhesion to the substrate due to the carbon diffusion zone underneath the coating.

2. Experimental methods

2.1. Materials and coating deposition

The steel (AFNOR 20CN4) was used as the substrate material. The chemical composition in weight % is: 0.19% C, 1.12% Cr, 0.12% Si, 0.14% Mn, 0.38% Ni, 0.20% Cu, and 0.07% Mo. The samples to be coated were machined as cylinders of dimensions 20 mm in diameter and 10 mm in length. All specimens were mechanically polished in order to reduce the roughness to $R_a = 0.25 \mu\text{m}$ using sandpaper. The steel substrates received a pack cementation treatment at 900 °C for 3 h. The carbon was provided by a cement coke powder added with barium carbonate as activator. With this method a carburizing depth of 0.6 mm was obtained as pointed out in Fig. 1. Pre-treatment is necessarily used for the cemented steel substrate before being coated with a layer of chromium. After cooling at room temperature the samples were degreased during 10 min in a solution of sulfuric acid heated at 60 °C. Chromium coating was then performed using a standard

* Corresponding author. Tel.: +213 773 853 855; fax: +213 3555 1836.
E-mail address: benariouayounes@yahoo.fr (Y. Benarioua).

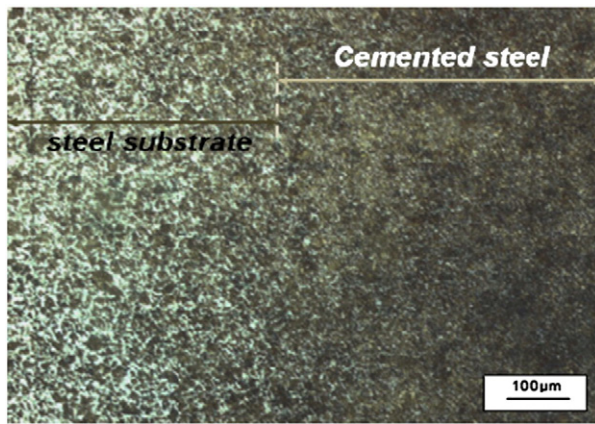


Fig. 1. Micrographic analysis of cemented steel substrate.

electrolytic procedure and the following setups: Current density of 10 A/dm^2 applied to the electrolytic solution at 40°C composed of chromium oxide (250 g/l) and sulfuric acid (2.5 g/l). The duration of the treatment was 30 min at 40°C . In these conditions the produced coatings are composed of pure chromium and are about $20 \mu\text{m}$ thick. The last treatment used to transform the chromium coating into a carbide coating was just a thermal treatment of one hour at test temperatures ranging from 500 to 1100°C according to the thermal cycles presented in Fig. 2. All the annealing treatments were carried out in a KO14 type furnace.

2.2. Characterization and analysis techniques

For reference samples examined at each step of the procedure and after thermal treatments carried out at 500 , 700 , 800 , 900 , 1000 and 1100°C , cross-sections were prepared and polished. The samples were then etched in a solution composed of 100 ml of water, 2 ml of HF at 40% and 5 ml of H_2O_2 at 30%. This type of etching is commonly used to enhance the boundaries. Using a B O71 Olympus microscope, this metallographic preparation allowed the observation of morphological details and the thickness of the chromium and/or chromium carbide layers. X-ray diffraction analysis was performed to determine the phases at the sample surface using a Phillips diffractometer. $\text{Cu K}\alpha$ characteristic radiation with a wavelength of 0.15418 nm was used over a 2θ range of 20 – 130° . The specimens were investigated using grazing incidence in order to limit the penetration of the X-rays. Cemented steel area, thickness of chromium

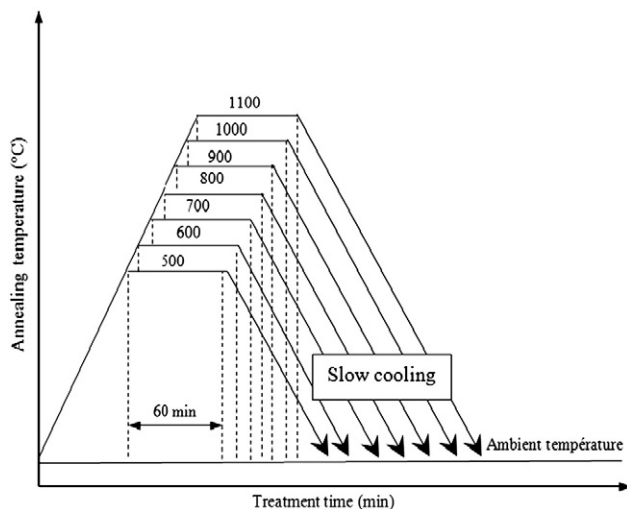


Fig. 2. Thermal cycles of annealing performed after carburizing and chromizing.

layers deposited and annealed was measured by an optical micrometer associated to the optical microscope.

The hardness profile of the samples was measured along the cross-sections using a Leco micro-indenter. Three Vickers indentations using a load of 10 g were performed at each depth in order to obtain a significant statistical value.

The adhesion of chromium and/or chromium carbide coatings to the carburized steel substrate was evaluated using a scratch-test technique with increasing loads applied to a spherical Rockwell indenter and further evaluation of the critical load from the acoustic emission signal. In these conditions the critical load that can be observed on the load displacement curve can be related to either cracking or chipping of the coating.

3. Results and discussion

3.1. Structure

Before chromizing, the hardened zone of the carburized steel is composed of ferrite and cementite phases with a maximum hardness of $320 \text{ HV}_{0.05}$ g at the surface. Fig. 3. shows the X-ray diffraction pattern of the carburized zone at the surface. In this figure, the two cementite and ferrite phases are observed. Toward the core of the material, the relative proportions of ferrite and cementite depend on the carbon content which varied from around 1% C at the surface to 0.2% C of carbon in the steel substrate.

Fig. 4 presents the X-ray spectra of chromium layers as deposited and treated at annealing temperature of 500°C , 800°C and 1100°C . For the untreated sample, the film is primarily composed of chromium.

The chromium layer treated at annealing temperature of 500°C reveals almost the same structural character than the as deposited chromium. For this temperature of treatment, no precipitation of chromium carbide phase was observed apart from the presence of chromium oxide characterized by a peak of low intensity. The presence of Cr_2O_3 at the surface of the samples is due to the reaction between the chromium layer and the oxygen present in the atmosphere of the furnace. The formation of chromium oxide phase can be explained by the low free energy of formation (-570 kJ/mol) [9]. Chromium carbide starts to be formed by carbon diffusion from the carburized zone of the steel substrate just before 800°C . The gradual formation of this new phase increases with the tested temperatures from 800°C to 1100°C . For these temperatures two separate layers are observed in the films. The outer region composed of pure chromium where the transformation has not started since the carbon has yet not reached this zone and the inner region where carbide precipitation has already started. Three different phases of chromium carbide: Cr_{23}C_6 , Cr_7C_3 and Cr_3C_2 can be identified in the

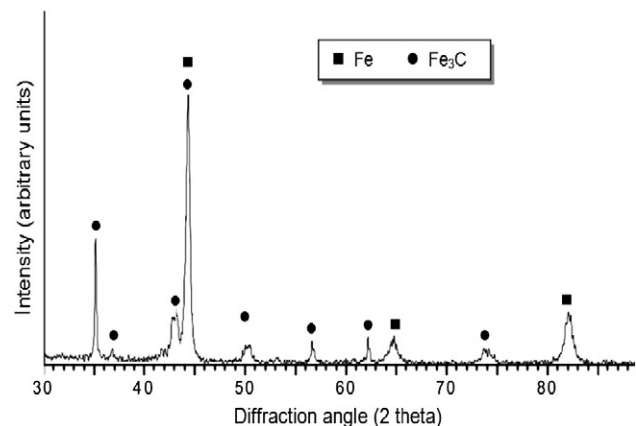


Fig. 3. X-ray diffraction pattern of the carburized layer at the surface of the steel.

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