

Study of the Influence of Cementation Layer Thickness on Properties of Chromium Carbide Obtained by Conversion Treatment

Rabah BOUBAAYA^{1,2*}, Omar ALLAOUI¹, Mokhtar DJENDEL^{1,2}, Younes BENARIOUA³,
Zied DRISS⁴

¹Laboratoire de Génie des procédés, Université Amar Telidji - Laghouat, Algeria

²Département des Sciences et Techniques, Faculté des Sciences et de la Technologie, Université de Bordj Bou Arreridj, 34000, Bordj Bou Arreridj, Algeria

³Département de Génie Mécanique, Faculté de la Technologie, Université de M'sila, Algeria

⁴Laboratory of Electro-Mechanic Systems (LASEM), National School of Engineers of Sfax (ENIS), Tunisia

* Corresponding Author : raboubaaya@yahoo.fr

ORCID:0000-0002-3672-3185

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Abstract:

Steel substrates low carbons were face-hardened by cementing in case, and then thin layers of chromium were deposited by electrolytic way on these substrates. After deposition, the samples were exposed to isothermal annealing in the temperature of 950°C. The characterization of the thin layers was made by means of optical microscopy and interferometry Vickers micro-hardness. From the obtained results, we have established the kinetics of phase shift (under effect the layer of cementing) in the thin layers of chromium which are transformed into chromium carbide while passing by metastable phases of transition. These transformations occurred by diffusion of the carbon atoms coming from layer of cementing, germination and growth in solid phase. This fact has examined according to the temperature of annealing, the evolution of the lattice parameter and the morphology of the deposited chromium layer. As regards the mechanical properties, it was established that the micro-hardness believes with the evolution of the phase shift.

1. Introduction

Over the past two decades, the transition metal carbides of the coatings were found to be one of the most effective materials for increasing the life time of the machine components, cutting tools and forming tools, in particular to improve wear and corrosion resistance [1,2]. Among these metal compounds, chromium carbide has the best mechanical and chemical resistance in harsh conditions. Recent research on chromium carbide stressed their resistance to oxidation and corrosion and suggested that they have great potential to replace hard chrome electroplating as protective coatings [3,4]. The chromium carbide layers can be obtained by physical vapor deposition (PVD) [5,6] or thermo-reactive deposition/diffusion (TRD) technology [7,8]. In the case of PVD thickness is

much less than 10 μm making those unfit also withstood the high contact pressure and further adhesion to the substrate is less than that obtained with other types of coating techniques. TRD coatings must very long process times at high temperatures. For example, a thickness 8 μm chromium carbide layer was obtained on a steel AISI D2 with a treatment at 1030 °C for 4 h [9] and a thickness of 13 μm was obtained with treatment at 1000 °C for 3 h [10]. Indeed, there are very common industrial methods that can be combined in any manner to produce chromium carbide coatings.

In order to produce a high-hardness coating, a good adhesive properties and a sufficient thickness with stand severe loading conditions, we propose an alternative technique based on a three-step process using only standard industrial methods. The first step intends to increase the carbon content at the

surface of the substrate using a standard carburizing process. The second step consists to coat the carburized material with a layer of chromium. The final step considers the heat treatment at temperatures which allow the diffusion of carbon into the surface of the material. In this work, we propose to determine the experimental conditions which allow complete conversion of the chromium layer in a uniform layer of high hardness of chromium carbide and a good adhesion to the substrate.

2. Experimental methods

2.1. Materials and coating deposition

Cylindrical steel samples were selected of 20 mm in diameter and 15 mm in length. The chemical composition of the substrate reported in Table 1 was determined by spectrometric analysis using a spectrometer HILGER.

Table 1. Chemical composition of steel to be coated (%).

Fe	C	Si	Mn	P	S	Cr
95.99	0.19	0.22	0.94	0.003	0.056	1.12
Mo	Ni	Al	Co	Cu	Ti	Sn
0.07	1.18	0.0116	0.01	0.20	0.0012	0.010

The preparation of the substrate consists on a mechanical polishing on each sample using abrasive papers. The finishing operation provides a good polishing with felt washed down with a suspension of alumina in water. The polished surface must be cleaned thoroughly with water and dried with compressed air and hot filtered. To conduct a comparative study, we divided the samples into 4 groups as is reported in Table 2. Cementing is performed using a cement coke powder. To accelerate cementing process, barium carbonates $BaCO_3$ is added to coal activators.

Table 2. Carburizing test parameters.

Group	I	II	III	IV
Hold time	00	03 hours	06 hours	12 hours
Cementing thickness	E1= 00 mm	E2= 0.6 mm	E3= 1.3 mm	E4= 2.5 mm
Temperature		900 °C	900 °C	900 °C

Samples were chrome electroplating, the standard composition of plating bath: 250 g/l CrO_3 , 2.5 g/l SO_4H_2 , bath temperature (40 - 44 °C), antimony lead anode, retention time 0.5 hour and deposition rate of about 40 $\mu m/h$.

In order to obtain a layer of chromium carbide, chromium coated samples were a thermal annealing treatment in the temperature of 950 °C. The hold time is taken 1 hour.

2.2. Characterization and analysis techniques

Microscopic observation requires fine polishing of the surface or the cutting of the deposit to be observed. Cleaning with acetone and drying by hot air flow end the mechanical polishing. The second phase consists of a chemical attack using the product nital, the composition and proportions by volume are nitric acid (5 ml) and ethanol (95 ml). Chemical etching can reveal the different phases presenting the optical microscope. The equipment used is a universal optical metallographic microscope ZEISS type.

The micro Vickers hardness test allows us to measure the size of the indentation made on the deposit. Measurements of the micro hardness of the deposits have been made on surfaces of polished sections. The indentations under a load of 50 grams were made through a microdurometer type Buehler MICROMET (HARDNESS TESTER) which is equipped with a Vickers diamond indenter. The samples have been tested in the Vickers micro-hardness with five measurements on each sample. Considering the average value obtained over five fingerprints to reduce measurement errors.

3. Results and discussion

3.1. Morphology

Figure 1 shows the observation of metallographic faces layer/substrate after annealing at 950 °C. From these results, it has been observed that no chromium carbide trace could be revealed in the pictures corresponding the sample without cementation ($e_1 = 0$ mm). The sample of group II ($e_2 = 0.6$ mm), a chromium carbide edge occurs in the vicinity of the interface. This fact is reflected by the low diffusion of carbon cementation zone in the deposited chromium layer. In the picture corresponding to the sample of group III ($e_3 = 1.3$ mm), it has been noted that the microstructure is predominantly in chips arranged on the substrate surface. This kind of morphology is found in several cases of phase change in the solid state. The structure of the incipient phase is usually in the form of needles certain plane oriented parallel to the surrounding parent phase. Increasing the amount of carbon diffusion from the substrate causes a progressive propagation of chromium carbide in the deposition of chromium. For the sample of group IV ($e_4 = 2.5$ mm), chromium matrix is transformed completely to chromium carbides. The formed layer has a granular structure. The morphology of the deposit of chromium carbides mainly depends on the treatment temperature, which promotes the mobility of atoms in the layer and the substrate. Mobility supports

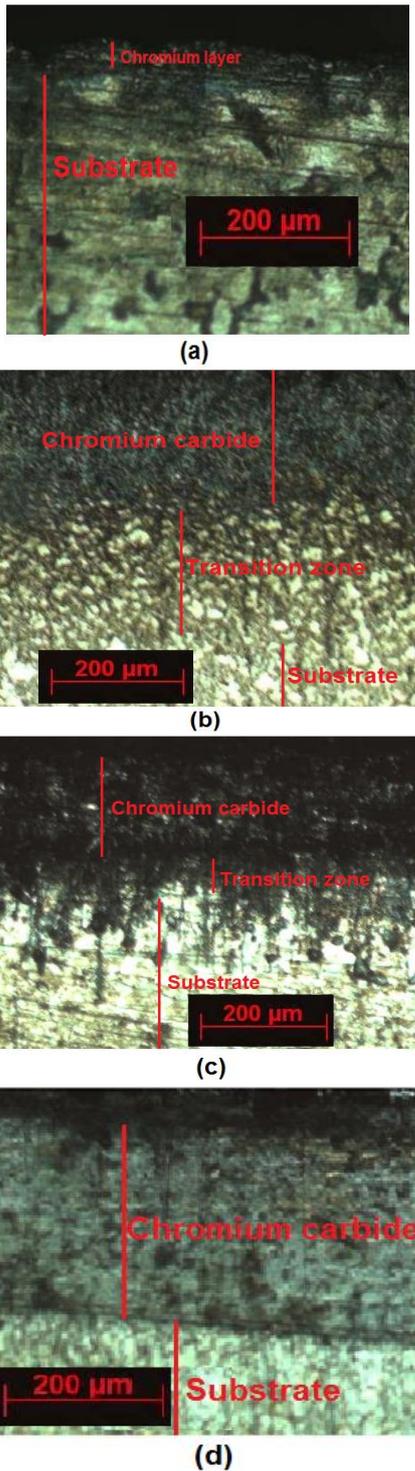


Figure 1. Observation of metallographic faces layer/substrate after annealing at 950 °C: (a) the sample of group I ($e_1 = 0$ mm), (b) the sample of group II ($e_2 = 0.6$ mm), (c) the sample of group III ($e_3 = 1.3$ mm), (d) the sample of group IV ($e_4 = 2.5$ mm).

traverses the diffusing carbon element concentration from the substrate to the layer. Indeed, the thickness of the carburized layer presents a great effect on the chromium conversion rate into chromium carbides.

3.2. Hardness

Fig.2. shows the effect of the thicknesses of the carburizing layer on the micro-hardness. According

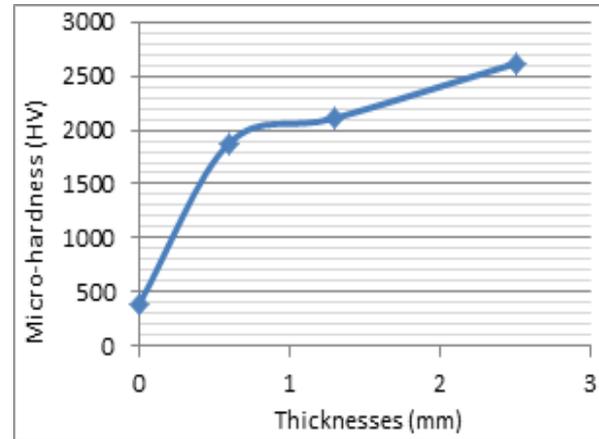


Figure 2. Effect of carburizing layer thicknesses on micro-hardness.

Table 3. Change in micro-hardness depending on the thicknesses of the carburizing layer.

Thicknesses of the carburizing layer	Micro-hardness (HV)
$e_1 = 0$ mm Without cementation	390
$e_2 = 0.6$ mm	1880
$e_3 = 1.3$ mm	2110
$e_4 = 2.5$ mm	2615

to these results, it is clear that the micro-hardness still is evolving in ascending order. Micro-hardness measurements made on samples e_3 and e_4 show that the micro-hardness is even greater than that of the samples e_1 and e_2 as it is shown in Fig.2 and reported in Table 3.

Curing would be linked to the complete formation of the chromium carbide that will cause a reinforcing of deposits. Diffusion of carbon atoms reaches the outer surface of the layer; the atoms occupy the chromium surface interstices; for thus, it will be a total loss of chromium phase.

In these conditions, the thicknesses of the carburizing layer e_2 , e_3 and e_4 are equal to: 0.6, 1.3 and 2.5 mm, respectively. From these results, we can confirm that the difference between the cementation thicknesses may cause difference in micro-hardness.

4. Conclusion

In this paper, we are interested on the increasing of the performance steels chromium carbide creation with high features. For thus and after carburizing

and deposition by electroplating, a chromium layer on the steel samples (layer/substrate) were subjected to annealing at 950°C. In this temperature, the deposited film is gradually converted to chromium carbide. Phase transformation which took place primarily by diffusion of the substrate carbon atoms in the layer was determined by metallographic analysis. The hardness of the produced layers evolved following the thickness of the carburized layer. The obtained value is in agreement with the changing properties determined by microscopic observation.

Indeed, it has been noted that for sample without cementation ($e_1 = 0$ mm), thin films retain their monophasic character, micro-hardness of the deposited raw layer is larger than that treated. It should be noted that for the sample of group II ($e_2 = 0.6$ mm) and the sample of group III ($e_3 = 1.3$ mm), the incipient phase is increased while the chromium mother phase undergoes a decrease. This causes a high hardening of the obtained deposit. For the sample of group IV ($e_4 = 2.5$ mm), the chromium layer is completely converted into chromium carbides that would be responsible for the increase of the hardness.

In this case, it is both the thicknesses of the layers of carburization temperature treatment which play an essential role in the transformation of the chromium layer to chromium carbides.

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