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Wear resistance of vanadium-niobium carbide layers grown via TRD

Fabio Enrique Castillejo-Nieto [a], Jhon Jairo Olaya-Florez [b] & José Edgar Alfonso [c]

[a] *Departamento de Ciencias Básicas, Universidad Santo Tomas de Aquino, Bogotá, Colombia. fabiocastillejo@usantotomas.edu.co*
[b] *Departamento de Ingeniería Mecánica y Mecatrónica, Universidad Nacional de Colombia, Bogotá, Colombia. jjolayaf@unal.edu.co*
[c] *Grupo de Ciencia de Materiales y Superficies, Departamento de Física, Universidad Nacional de Colombia, Bogotá, Colombia. jealfonsoo@unal.edu.co*

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Abstract
Nb-V complex carbide coatings were produced on AISI D2 steel substrates using the thermo-reactive diffusion (TRD) process in order to improve the surface hardness and wear resistance of this tool steel. The carbide coating treatment was performed using molten borax with added ferroniobium, ferrovanadium, and aluminum at temperatures of 1223, 1293, and 1363 K for 2, 3, 4, and 5 h. The coating layers were characterized with scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDS). The growth rates of the coatings were studied, and a kinetic model of the layer thickness was constructed as a function of the time and temperature treatment. The hardness and friction coefficient (COF) of the coatings was measured through nanoindentation and pin on disk test respectively. The carbide layers had a homogeneous thickness and a hardness of 37.63 GPa, which is close to values obtained in super-hard materials, and the COF was in the range of 0,3 for the coated steels.

Keywords: Thermo Reactive Diffusion, Niobium-Vanadium Carbide, Hardness, wear

Resistencia al desgaste de capas de carburo de vanadio-niobio crecidas por medio de TRD

Resumen
Recubrimientos mixtos de carburos de niobio - vanadio se fabricaron utilizando el proceso de difusión termo-reactiva (TRD) a fin de mejorar la resistencia al desgaste en aceros para herramientas. Los recubrimientos fueron depositados en un baño de bórax con ferroniobio, ferrovanadio y aluminio, con temperaturas de depósito de 1223, 1293 y 1363 K para 2, 3, 4 y 5 h. Los recubrimientos producidos se caracterizaron con microscopía electrónica de barrido (SEM), difracción de rayos X (DRX) y espectroscopia de dispersión de energía (EDS). Se estudiaron las tasas de crecimiento de los recubrimientos, y se construyó un modelo de la cinética del espesor de la capa como una función del tiempo y la temperatura del proceso. La dureza y el coeficiente de fricción (COF) de los recubrimientos se midieron a través de nanoindentación y bola sobre disco, respectivamente. Las capas de carburo presentaron un espesor homogéneo alcanzando durezas de 37,63 GPa que está cerca de valores obtenidos en materiales súper-duros, y el COF presento valores cercanos de 0,3 para los aceros recubiertos.

Palabras clave: Difusión Termo-Reactiva, Carburo de Niobio-Vanadio, dureza, desgaste.

1. Introduction

Various surface treatments are used to produce coatings, mainly of carbides and nitrides of transition metals, in order to improve the tribological performance of materials that are subjected to conditions of high wear, such as forming dies and cutting tools such as drills and chisels in the metalworking sector. These coatings are generally produced using techniques such as physical vapor phase deposition (PVD) and chemical deposition in the vapor phase (CVD) [1,2]. These procedures require the use of complex equipment and high-vacuum conditions, which makes their industrial implementation costly. An affordable and competitive alternative has been the application of hard

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coatings with excellent wear resistance through deposition via the thermo reactive diffusion (TRD) process [3] on substrates containing a carbon percentage higher than 0.3% w [4]. The coatings deposited through this process exhibit good adhesion to the substrate, good thickness uniformity, low friction coefficients, and a high degree of hardness. [5]. In the TRD process, a bath of molten salts formed by borax, aluminum as a reducing element, and carbide forming elements (CFE's) such as titanium, niobium, chromium, and vanadium can be used [4]. The carbide is formed when the metal element dissolved in the salt bath has a relatively low energy of formation of carbide and an energy of formation of the oxide higher than that of boron oxide (B2O3) [6,7]. If this condition is not fulfilled, the boron atoms are not oxidized and are free to diffuse in the steel, where they combine with the iron to form layers of iron boride (FeB or Fe2B) [8,9].

The research reported to date has studied binary metal carbides deposited via the TRD process, and has characterized its wear resistance, microstructure, and mechanical behavior. For example, several studies have focused on producing VC and NbC coatings on substrates of AISI H13, AISI M2, and AISI D2 steel, reporting hardness values of up to 2300 HV [3,5]. Other investigations have studied the growth kinetics of niobium carbides on AISI 1040 [10], iron boride on AISI 4140 steel [11], and chromium carbide on AISI D2 steel [12| These papers reported the growth kinetics for binary systems such as NbC or VC, obtaining a layer thickness that depended on the time and the temperature of the treatment. Regarding electrochemical behavior, there are studies of iron boride and aluminum formed using borax on steels AISI S1, S2, and S3, varying the manganese content [13] and there are also studies about tribological and electrochemical performance of niobium-chromium carbides [14]. However, there are few micro-structural and electrochemical studies of possible ternary systems produced using two EFC's. Therefore, the aim of this work is to deposit ternary niobium-vanadium carbide coatings on a D2 steel substrate using the thermo reactive deposition technique and to study their structure, hardness, and wear resistance.

2. Experimental Methods

Coatings of ternary carbides were deposited on AISI D2 tool steel. The dimensions of the substrate were 15 mm in diameter and 4 mm in thickness. The preparation of the samples was performed through metallographic polishing with 220, 360, 400, 600, 1000, and 1200 sandpaper, ending with an ultrasonic cleaning in acetone. The chemical composition of the steel, as stated by the manufacturer, was 1.5% wt C, 11.5-12.5% wt Cr, 0.15-0.45% wt Mn, 0.8% wt Mo, 0.8 wt% V and Fe balance. The steel samples were supplied in the annealed condition with a maximum hardness of 2.6 GPa.

The coatings were produced using a salt bath formed by molten borax (Na2B4O7), 8% wt ferro-niobium (Fe-Nb), 8% wt ferro-vanadium (Fe-V) and 3% wt aluminum (Al). The aluminum that is added in the salt bath had the role of a reducing agent, i.e., it reduced boron oxide and oxidized it in order to prevent the added metal (niobium or vanadium) from oxidizing so that these metals are released and can bind with the carbon of the steel to form carbides. The growth rates of the coatings were studied through surface treatment with TRD at three different temperatures of 1223, 1293, and 1363 K for 2, 3, 4, and 5 hours, with preheating at 873 K.

The crystallographic structure of the coatings was analyzed using an X-PertPro Panalytical difractometer operating at 45 kV and 40 mA, with a configuration of θ - 2θ at an interval of 30° to 90° and steps of 0.02°, with the Kα line of copper (1.594 Å). The average thickness of the coatings was measured with a Philips scanning electron microscope, working at 40 KV and the EDX was used to determine the chemical composition of the coatings surfaces. The wear cracks were observed using the confocal microscopy. The hardness and the elastic modulus of the coatings were determined from measurements of nano-indentation using CSM Instruments equipment, with a Berkovich indenter at a speed of approach to the sample of 2000 nm/min with a load application rate of 10 mN/min and a maximum load of 30 mN every 15 s. The results obtained were results from an average of 6 measurements. The tribological properties of the coatings were measured at room temperature, via a CETR-UMC-2 tribometer, using the ball-on-disk technique with Al2O3 balls of 6 mm diameter and applying a load of 4 N at a speed of 50mm/s for 10 minutes with a humidity of 60%.

3. Results and Discussion

3.1. *Microstructure and growth kinetics of the coatings*

Fig. 1 shows the cross section (SEM) of two coatings of vanadium-niobium obtained at temperatures of 1363 and 1223 K for a deposit time of 5h for both coatings. The images allow it to be established that the coatings have thicknesses of 19.3±3.2 μm and 9.6±0.8 μm, respectively. Higher values of thickness were obtained at the higher temperature. Furthermore, the micrograph allows it to be seen that coatings were formed by splats with dimensions on the order of a micron.

Fig. 2 shows the coating thickness as a function of time, with the temperature as a study parameter. Fig. 2a shows that the thickness does not exhibit linear behavior with respect to the time of thermal treatment. Therefore, it can be assumed that the rate of diffusion of carbon in the layer determines the growth rate of the coatings [15], and if the layer growth is perpendicular to the substrate surface, we can assume that the film growth rate obeys an Arrhenius function (see eq. 1) [10]. From Fig. 2b, the coefficient of diffusion of carbon into the substrate was determined.

$$x^2 = Dt \tag{1}$$

Where x is the thickness (cm), D is the diffusion coefficient (cm^2/s) and t is the processing time (s). Given that the carbon that diffuses also reacts to form carbides, the diffusion coefficient calculated using Eq. (1) is an effective diffusion coefficient and, therefore, it does not correspond to pure diffusion of carbon in the substrate.

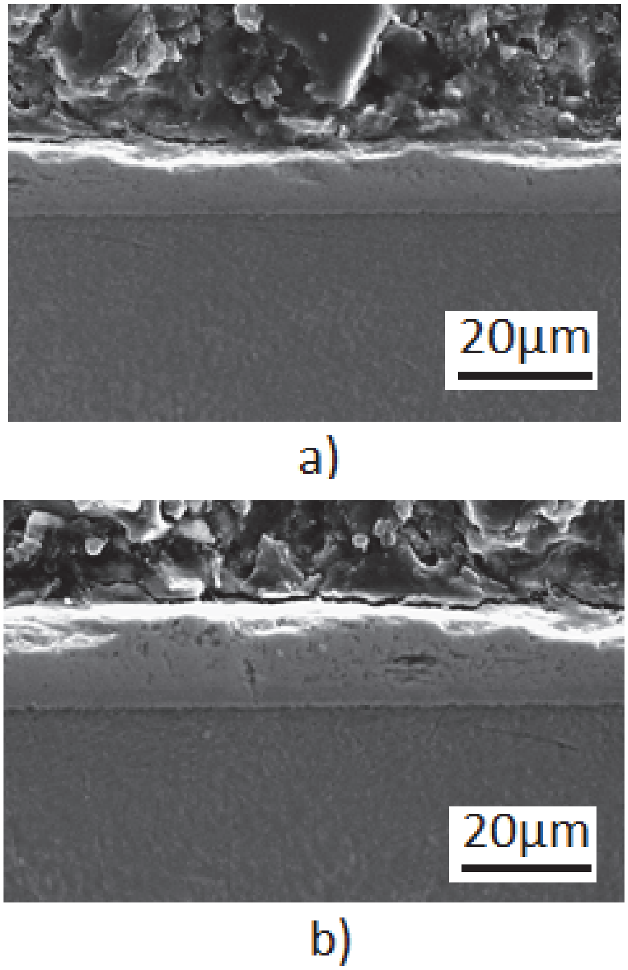

Figure 1. SEM images of a) Niobium-Vanadium carbide at 1223 K and 5 h. y b) Niobium-Vanadium carbide at 1363 K and 5 h.
Source: The authors

The relationship between diffusion coefficient and temperatures is given by [10]:

$$D = D_0 \exp\left(\frac{-Q}{RT}\right) \qquad (2)$$

Where D_0 is the frequency factor, Q is the activation energy, R the gas constant and T is the absolute temperature. Linearizing this function, one obtains:

$$\ln D = \frac{-Q}{RT} + \ln D_0 \qquad (3)$$

Fig. 3 shows the behavior of *ln D* as a function of the reciprocal of the temperature. In the inset, we show the values of the slope and the intercept, which allow determining that the activation energy Q = 137.688 kJ/mol (Ec. 3) and the value of D_0 is 3.88×10^{-5}. Substituting these numerical values into Equation 1, Equation 4 is obtained.

$$D = 3736\sqrt{t \exp\left(16651/T\right)} \qquad (4)$$

This expression allows calculating the thickness (t) of the coating in microns. In Table 1, the measured and calculated thickness values are shown.

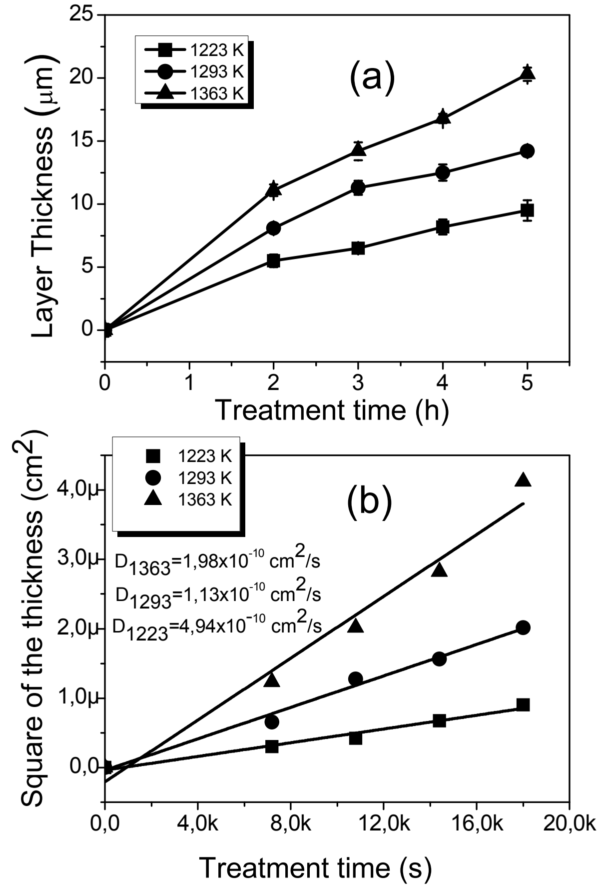

Figure 2. (Up) Thickness of the coatings and, (down) square of the thickness, as a function of treatment time.
Source: The authors

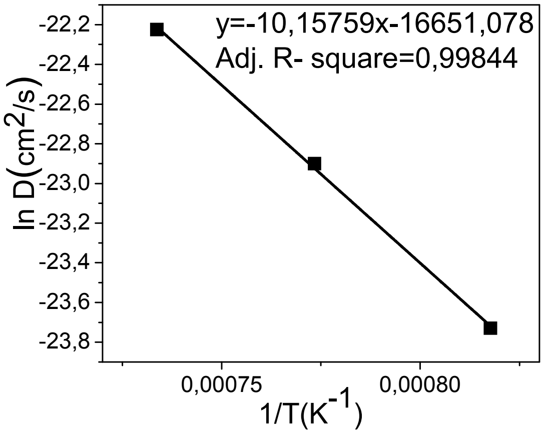

Figure 3. Natural logarithm of *D* as a function of the inverse of the temperature.
Source: The authors

Table 1.
Measured and computed thickness for $NbVC_2$, obtained through TRD at different times and temperatures.

	1223K			1293K			1363K		
NbV on D2	Measured	Calculated	Error%	Measured	Calculated	Error%	Measured	Calculated	Error%
Time(h)	Thickness (µm)	Thickness (µm)		Thickness (µm)	Thickness (µm)		Thickness (µm)	Thickness (µm)	
0	0	0	0	0	0	0	0	0	0
2	5.5	5.8	5.1	8.1	8.4	3.5	11.1	11.8	5.9
3	6.5	7.1	8.4	11.3	10.3	9.7	14.2	14.4	1.3
4	8.2	8.3	1.2	12.5	11.9	5	16.8	16.6	1.2
5	9.5	9.2	3.2	14.2	13.3	6.7	19.7	18.6	5.9

Source: The authors

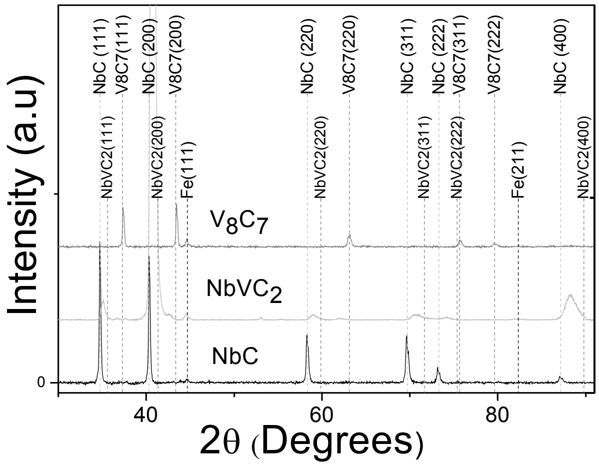

Figure 4. XRD patterns for niobium-vanadium carbides deposited on AISI D2 steel.
Source: The authors

Table 2.
Calculated and experimental angles of ternary carbides.

	$NbVC_2$	
Plane	Calculated	Experimental
Hkl	2θ	
200	41.335	40.96
220	59.886	59.91
311	71.647	71.88
222	75.369	74.98
400	89.802	89.02
331	100.568	100.12

Source: The authors

Table 3.
EDS results for the binary and ternary carbide coatings.

System	% at Nb	% at V	% at Fe	% at O	% at C
NbC	31,1	--	7.2	18,6	43,1
$NbVC_2$	37.2	18.7	5.3	6,2	32,6
V_8C_7	--	40,4	9.6	18,4	31,6

Source: The authors

Fig. 4 shows the XRD patterns of the coatings of the ternary vanadium-niobium carbides, obtained at a temperature of 1263 K and a time of 4 h of heat treatment. In order to establish the difference between the crystallographic phases in NbC and VC binary coatings [16] and in the ternary system, the three systems are included in the figure. Comparing XRD patterns allows establishing that the ternary vanadium-niobium carbides exhibit only the NbC crystallographic phase [17]. This result can be explained by considering that the free energy of the formation of NbC (-33.6 kcal) [18] is less than the free energy of formation of VC (-24 kJ / mol) [18].

Moreover, peaks with lower intensities present in the XRD pattern of the ternary system were assigned to the NbVC2 phase. The angular positions and the corresponding planes are shown in Table 2. The theoretical value was calculated with Powder Cell 2.4 software, in which values of the $NbVC_2$ crystallographic system were provided, such as the space group, the group number, and the Wyckoff parameters of this compound [19].

3.2. Chemical composition and mechanical properties

Table 3 shows the atomic percent measured through EDS analysis for every coating. The result shows that there is a greater percent of niobium than vanadium in the carbides; this can be explained based on the low formation energy of this carbide in comparison with the vanadium carbide.

Table 4 shows the values of hardness and elastic modulus of the coatings. Overall, the results show that coatings with ternary carbides increase their hardness value.

This increase in hardness may be associated with two factors: i) the crystal size reduction forming the coating, which is evidenced by the increased FWHM (Full Width at Half Maximum) of the peaks in the XRD, and ii) the macro-deformation, which is evidenced by the shift of the peaks with respect to their position in a binary carbide sample (see Fig. 4) [20-22]. The values of the hardness of the ternary coatings are close to those of the super-hard materials. [23].

Table 4.
Hardness and elastic modulus of the coatings of vanadium and niobium carbides deposited on AISI D2 steel.

System	Hardness (Gpa)	Elastic modulus (Gpa)
NbC	24.72 ± 1,4	420.6 ± 16,2
V_8C_7	25.46 ± 1,1	384.8 ± 14,8
$NbVC_2$	37.63 ± 1,7	435.1 ± 22,4

Source: The authors

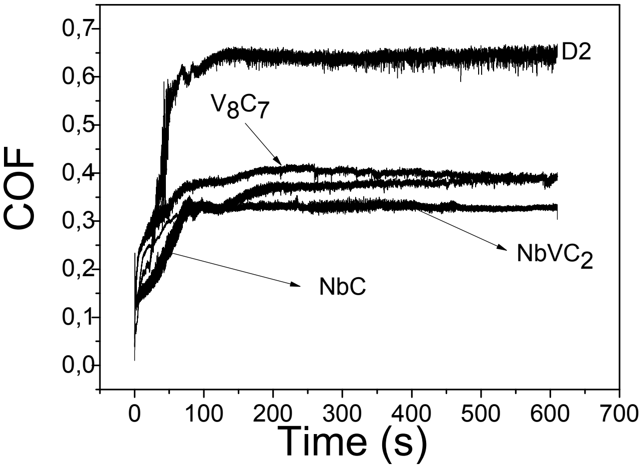

Figure 5. Friction coefficients in carbide coatings deposited on D2 steel obtained through the ball-on-disk test.
Source: The authors

3.3. *Coefficient of friction*

Fig. 5 shows the curve of the coefficient of friction as a function of the duration of wear testing for binary and ternary carbides systems deposited on AISI D2 steel. From the figure it can be established that the coefficient of friction (COF) of the binary carbides is less by about a factor two compared to the COF substrate. These results agree with those obtained in other studies [24-26]. In the ternary carbide systems, the COF values exhibit little change as a function of the percentage of vanadium, the coating with less vanadium having a lower COF value. The decrease of the COF in the coatings can be explained considering that on their surface there is an excess of aliphatic carbon atoms and graphite, which have Van der Waals type bonds. Calculation of the wear was not carried out because the depth of the wear scar was comparable to the roughness of the coating.

Figs. 6a to 6c show the EDS analysis, the morphology, and an image of confocal laser microscopy, respectively, of the NbC coatings. The EDS analysis shows signals of lines energy of Nb, Fe belonging to the substrate, and C. The SEM micrograph shows the plastic deformation of the coating, and the confocal image shows areas of maximum and minimum height of the coating and the line along which the roughness calculation was made. In Figs. 6d to 6f, we show the same information, but for vanadium carbide. The EDS analysis shows the presence of the K line of V, K line of Fe from the substrate, and K line of Al, possibly present in the salt bath. The confocal image allows the delamination of this carbide (dark green areas) to be observed, which appears mainly on the wear track.

Fig. 7 shows a SEM image for a wear track of $NbVC_2$ carbide deposited on AISI D2 steel. The cracking and delamination of the deposited carbide and the grooves or channels on the worn area is shown. The wear mechanism is based on the formation of cracks and parallel channels during the wear test. According to Adachi and Hutchings, this situation is known as a slotted wear, or an abrasion of two bodies [27]. These slots arise as a result of the cutting action of abrasive microparticles that remain essentially embedded in the ball during the wear test.

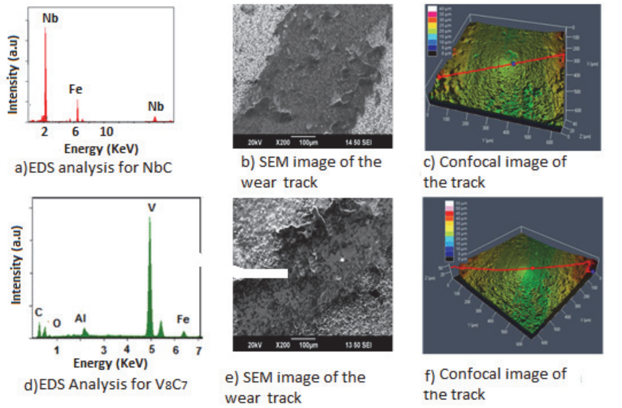

Figure 6. a) EDS analysis; b) SEM image and c) image of confocal laser microscopy, obtained for the tracks of binary carbides NbC, and the same information in figures d, e, and f for V_8C_7, both coatings deposited on AISI D2 steel.
Source: The authors

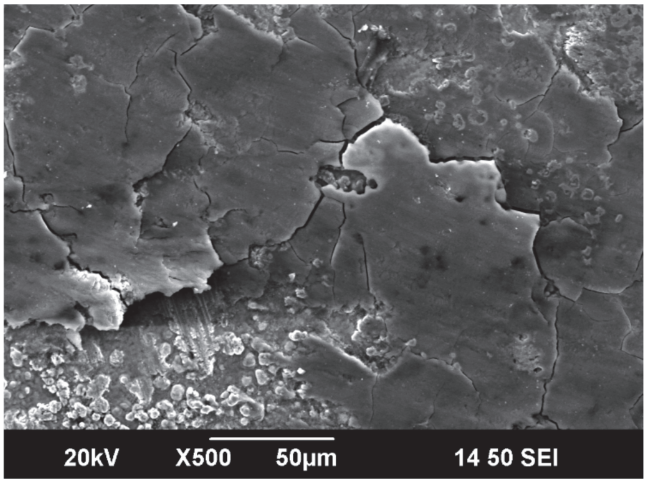

Figure 7. Detail of the wear track for the ball-on-disk test for a NbV coating deposited on AISI D2.
Source: The authors

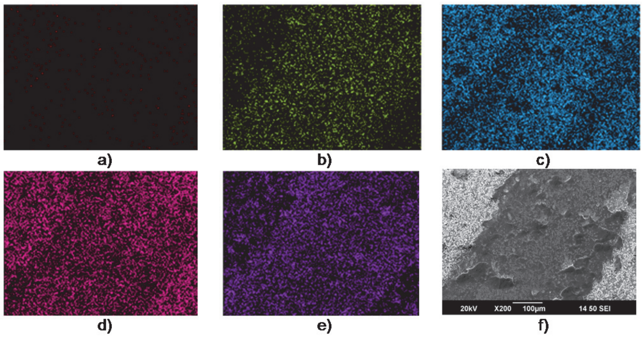

Figure 8. Elemental mapping of the coating after the wear test a) C, b) O, c) Nb, d) V, e) Fe, and f) normal SEM image.
Source: The authors

Fig. 8 shows the elemental mapping of the coating after the wear test. In the figure it is possible to see the reduction of niobium and vanadium in the wear area, and a large amount of iron is observed in the delaminated layer.

4. Conclusions

Ternary coatings of vanadium-niobium carbides were deposited via TRD; using XRD it was determined that the coatings were formed by binary compounds (NbC and VC) and a ternary one (NbVC2). The hardness value obtained was 38 GPa, which is close to the value obtained in super-hard materials. This increase in hardness can be explained by the formation of micro-deformations in the carbide and a decrease in crystal size, which is evidenced by a broadening of the XRD peaks that are exhibited in the ternary coatings. Additionally, the coating-substrate system was shown to have a lower coefficient of friction than that of the naked substrate. The binary systems show a good performance with hardness between 24 and 25 GPa and better wear performance than uncoated D2 steel.

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F.E. Castillejo-Nieto, received his BSc. Eng in Metallurgical Engineering in 1997, his MSc. in Physics in 2005, and his PhD degree in Engineering in 2014, all of them from the Universidad Nacional de Colombia. Medellin, Colombia. His research interests include: Coatings and thin films, wear and corrosion resistance of materials.

J.J. Olaya, is an associate professor at the Departamento de Ingeniería y Mecatrónica in the Universidad Nacional de Colombia, Bogotá, Colombia. He conducts research in the general area of development and applications of thin films deposited by plasma assisted techniques, corrosion and wear. He received his PhD in 2005 from the Universidad Nacional Autonoma de México, Mexico.

J.E. Alfonso, completed his BSc. degree in Physics in 1987 and his MSc. degree in Science - Physics in 1991, all of them being obtained from the Universidad Nacional de Colombia. In 1997 he completed his PhD in Science - Physics in the Universidad Autonoma de Madrid, Spain. He has taught at the Universidad Nacional de Colombia as a professor since 2000, where his research has been focused on material science, particularly on thin film processing as well as performance characterization, studying thin film optical, electrical and mechanical properties.