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Article

Behavior of M2 steel with CrN/TiN thin films by cathodic arc PVD method

Comportamiento del acero M2 con películas delgadas de CrN/TiN por método PVD por arco catódico

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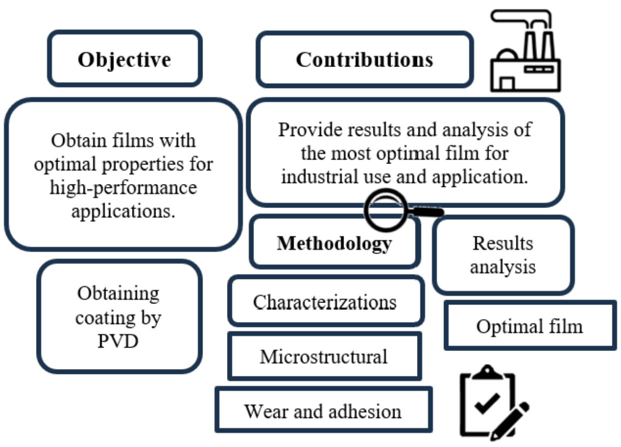
Abstract

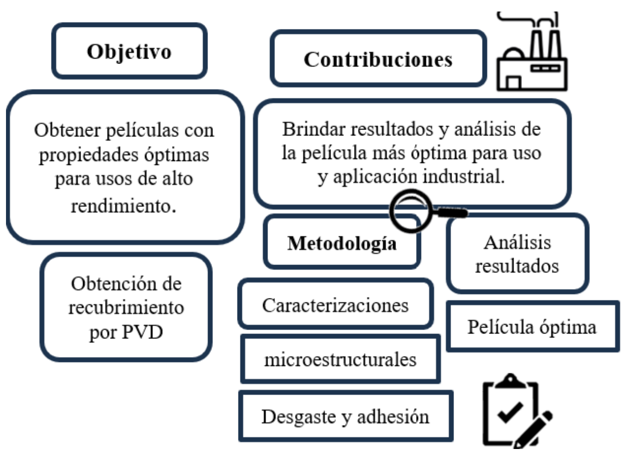
The objective of this project is to obtain results on the films generated on M2 high-speed steel substrates, used in cutting applications and wear resistance. These samples were coated with CrN as the base layer and TiN as the film, using the PVD cathodic arc method. The project was carried out in collaboration with SADOSA S.A. de C.V., a company specialized in industrial coatings for high-speed cutting tools.The methodology included microstructural characterization through scanning electron microscopy (SEM) and composition analysis (SEM-EDS), along with topographical analysis using optical profilometry. The film thickness was determined using the calotest method, and tribological analysis was conducted with the pin-on-disk technique, obtaining wear tracks, volume loss, and specific wear rate according to ASTM G99 standards. The adhesion of the films was also evaluated using VDI 3198 standards to assess the adhesion quality of the samples. Finally, the data were collected for the analysis of the obtained results.



Characterization, film, PVD, Tribology

Resumen

El objetivo de este proyecto es obtener resultados sobre las películas generadas en sustratos de acero M2 de alta velocidad, utilizados en aplicaciones de corte y resistencia al desgaste. Estas muestras fueron recubiertas con CrN como capa base y TiN como película, empleando el método PVD por arco catódico. Se realizó la colaboración con la empresa SADOSA S.A. de C.V., especializada en recubrimientos industriales para herramientas de corte de alta velocidad. La metodología incluyó la caracterización microestructural mediante micrografías (MEB) y análisis de composición (MEB-EDS), además de un análisis topográfico mediante perfilometría óptica. Se determinó el espesor de la película usando el método calotest y se realizó un análisis tribológico con la técnica de perno sobre disco, obteniendo huellas de desgaste, volumen perdido y desgaste específico según la norma ASTM G99. También se evaluó la adhesión de las películas utilizando la norma VDI 3198, para determinar la calidad de adhesión de las muestras. Finalmente, se recopilaron los datos para el análisis de los resultados obtenidos.



Caracterización, película, PVD, tribología

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Introduction

As a result of significant advances in the field of design, the emergence of new alloys and improvements in heat treatment processes, tool and high speed steels (HSS) have achieved, since the 20th century, a remarkable participation in the industrial field, especially as cutting tools.

The optimisation of materials to achieve better performance has led to the emergence of new alloys, such as high speed steels (HSS) focusing on M2 steel, which have a great application in the industrial area, such as the manufacture of cutting tools, drills and others.

Currently, these types of steels have a great relevance and multiple applications, as they are produced in large volumes worldwide. This is because they have ideal mechanical properties that allow them to perform their cutting function at high speed, standing out for their exceptional toughness and strength.

The inherent properties of high speed steels (HSS) enable them to offer excellent performance for the specific purposes for which they were designed. However, the continuous advancement of the industry and the significant increase in demand for these steels has led to the need for materials that not only fulfil their original function, but also exceed performance expectations.

When a steel does not meet expectations in relation to the function for which it is required, failure begins on its surface, resulting from a lack of suitable properties to perform that function. This process gives rise to a phenomenon known as wear. Wear is defined as the gradual removal of material from the solid surface.

Causes that can lead to this effect include mechanical, chemical and corrosive factors. In the case of HSS steels we will focus on mechanical wear, this phenomenon occurs due to the plastic displacement of material on and near the surface, resulting in the detachment of particles that contribute to the formation of wear debris.

To address this need, effective solutions have been explored, most notably surface coatings within the field of surface engineering. These coatings not only improve the original characteristics of the steel, but also increase certain properties such as improved hardness, wear resistance and corrosion resistance.

As a result of this quest for improvement, methods have been developed to deposit thin layers of materials on the surface of steel, which provide these additional properties and optimise its overall performance.

Physical Vapor Deposition (PVD) Cathodic Arc.

A method used to transfer material on an atomic scale by evaporating the material to be deposited. Once the material has evaporated, it condenses on the steel to be coated to form a thin film.

This is a good coating method that works in a vacuum medium which provides improved wear and corrosion resistance, durability and monetary value to the products. It is a functional and applicable method for tooling, blades and dies.

To improve the properties of steels, hard coatings such as TiAlN, AlCrN or TiN are applied for the specific purpose of significantly increasing the properties of the steel. These coatings have the ability to improve service life and reduce damage caused by extreme use.

This vapour deposition technique has the quality of being able to deposit elements, compounds, alloys, which provide a good finish and better adhesion, it should be noted that adhesion is a vital part of the performance of this film.

Box 1

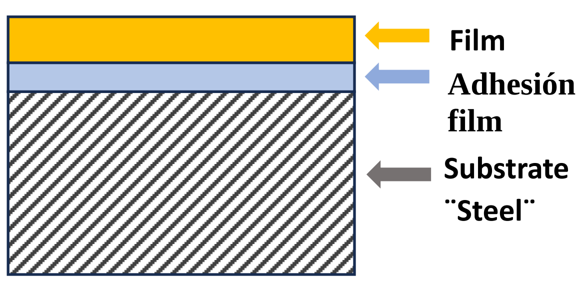

Figure 1
Films on M2 CrN/TiN substrate
Source: *Own elaboration*

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Methodology

M2 steel samples were used, which were metallographically prepared to achieve an optimum finish. The deposition of CrN/TiN thin films was carried out using the cathodic arc physical vapour deposition (PVD) method, where a CrN adhesion layer was initially deposited, followed by the application of a TiN film. Five samples were prepared, varying the deposition times of the TiN layer, while keeping the same deposition time for the adhesion layer.

Box 2

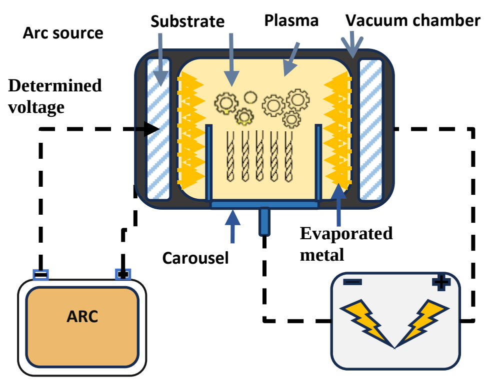

Figure 2
Cathodic arc PVD diagram

Results

Scanning electron microscopy (SEM).

Microscopy was used to obtain micrographs of the surface of the films. Figure # shows a micrograph of the sample M1 CrN/TiN 610S where white spots can be observed on the surface of the coating, this is due to the cathodic arc PVD method technique because the technique generates agglomerated material due to the large amounts of CrN and TiN condensed on the surface of the steel M2.

Box 3

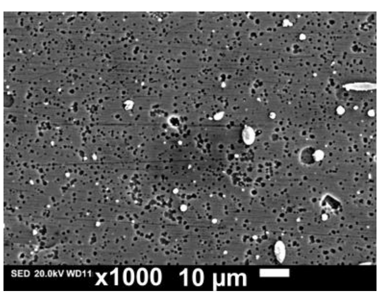

Figure 3
Micrograph of sample M1 CrN/TiN 610s on M2 at 1000x magnification

Figure 4 shows the micrograph of the M2 CrN/TiN 750s test where white dots can be observed, the white dots as in the previous sample refer to the accumulation of material by the film deposition process, it is observed quite uniformity in general of the surface, no detachment or microcracks can be perceived.

Box 5

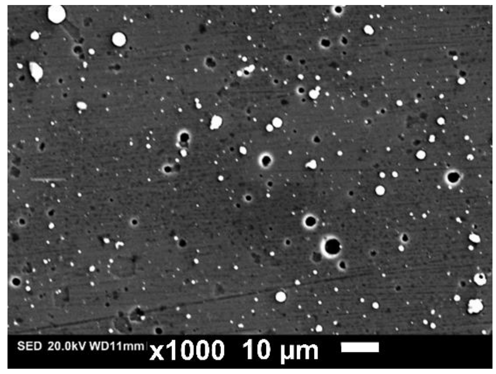

Figure 4
Micrograph of M2 CrN/TiN 750s sample on M2 at 1000x magnification

Energy dispersive spectroscopy (EDS)

Graph 1 shows the elements present in the deposited film, with which the following percentage was obtained in the film.

Box 6

Table 1
Percentage of elements present in M1

%Ti	%N	%Cr
80.49	19.49	0.02

Box 7

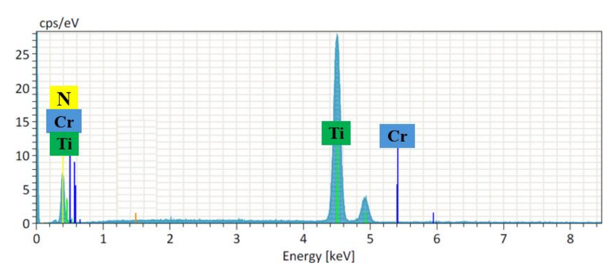

Figure 5
Percentage of elements present in deposited film M1 CrN/TiN 610s

The elements present in the film were identified, which correspond to the compounds CrN/TiN.

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Box 8

Table 2

Percentage of elements present in M1

%Ti	%N	%Cr
75.90	20.52	0.39

Box 9

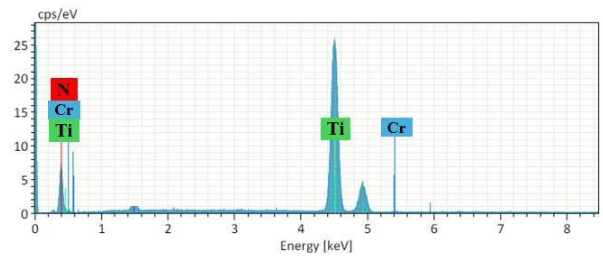

Figure 6
Percentage of elements present in deposited film M2 CrN/TiN 750s

X-ray diffraction

The method of ray diffraction was carried out with a Bruker d8 discover equipment, it allowed the obtaining of the present phases of the compounds deposited by the cathodic arc PVD method, in the sample M1 CrN/TiN 610s in the graph 3 the identified phases are observed, revealing in the case of CrN the crystallographic plane (002) has greater intensity of CrN and TiN with the plane (111) with greater intensity, in both cases.

Box 10

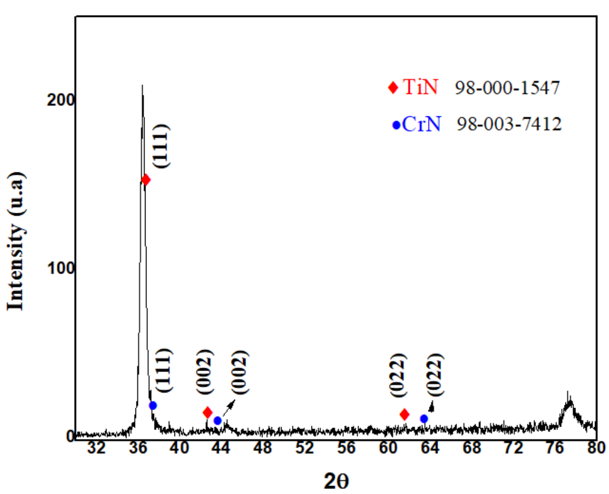

Figure 7
Diffractogram M1CrN/TiN 610s

Box 11

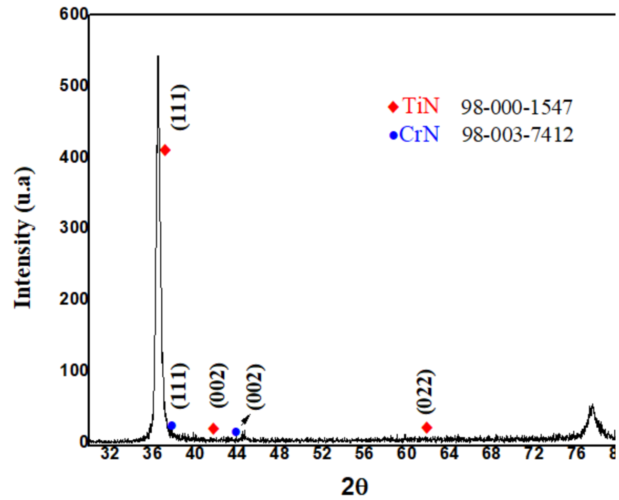

Figure 8
Diffractogram M2CrN/TiN 750s

Optical profilometry

The growth of valleys and ridges is observed in all the surface taken from the sample M1 in figure 9, in general uniformity is perceived, with the growth of ridges of some higher ridges, but not marking significant distinction with respect to the wole measured area, an arithmetic roughness of 59.79 nm was obtained.

Box 12

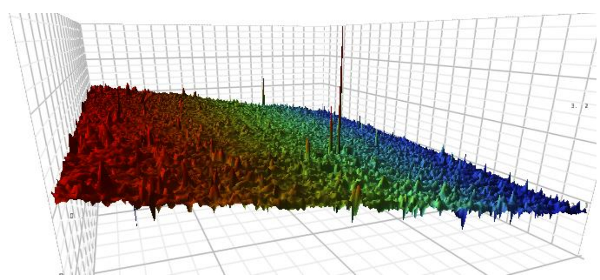

Figure 9
3D topography view of sample M1 CrN/TiN 610s

In figure 10, sample M2 has an arithmetic roughness of 64.98 nm, it was determined as a homogeneous and uniform surface.

Box 13

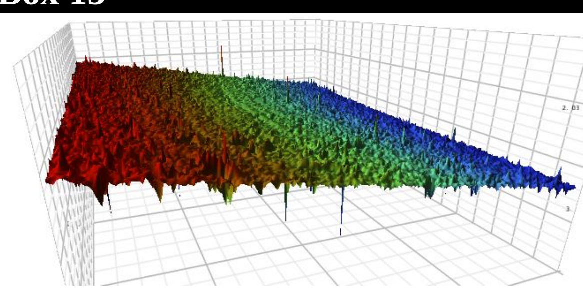

Figure 10
3d topography view of M2 CrN/TiN 750s sample

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Calotest

The layer thickness was determined by means of the calotest method, CrN adhesion film thickness and TiN film thickness were obtained for both samples.

Box 14

Table 3

Calotest test parameters

Parameters	
Diamond suspension	1 micra
Steel ball	Steel 52100
Time	40s
Speed	500 rpm

Box 15

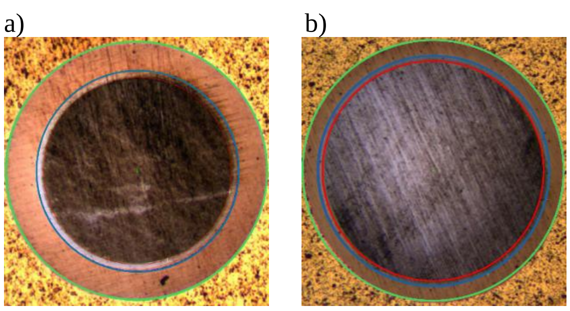

Figure 11

Calotest traces on steel, (a) M1 CrN/TiN 610s, (b) M12CrN/TiN 750s

Box 16

Table 4

Espesores M1 CrN/TiN 610s

Brand	Diameter (µm)	Thickness (µm)
Accession	469.8	0.70
Film	534.2	2.70

Box 17

Table 5

Thicknesses M2 CrN/TiN 750s

Brand	Diameter (µm)	Thickness (µm)
Accession	469.8	0.67
Film	534.2	3.52

Both samples have a CrN adhesion layer and a TiN film. The thickness of the films generated by the cathodic arc PVD method is determined by means of the wear of the counterpart with the sample placed, maintaining contact for a period of 40s causing wear until leaving at the end of the test a trace where the substrate is revealed, once this trace is obtained the thickness of each film is quantified.

Resistance to wear (Bolt on disc)

Box 18

Table 6

Bolt on disc test parameters

Counterpart	Steel ball 52100
Diameter	6 mm
Distance	250 m
Speed	0.15 m/s
Load	5 N

As shown in Figure 12, the friction coefficients of the samples M1 CrN/TiN 610s and M2 CrN/TiN 750s were obtained, the behaviour of the surface in the interaction of the 52100 steel counterpart can be seen, where in the first 10 m approximately, the settling period is shown, after that a stable period is reached throughout the test until the end of the 250 m of travel, this stability of the friction coefficient is presumed at 0.5 in both samples.

Box 19

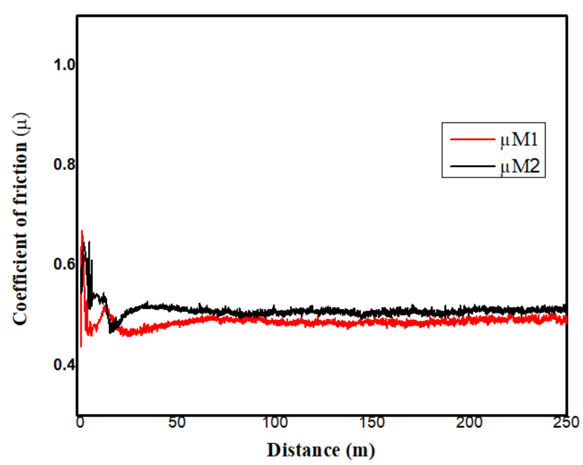

Figure 12

Coefficients of friction samples (M1, M2)

Derived from the wear test of the bolt on disc test, the wear traces obtained by an optical microscope at x40 were obtained.

It can be seen in figure 13, sample M1 CrN/TiN 610s the deterioration of the film, but not in its totality, at a parts of the film can be appreciated added on the surface, there are sections with adhesive wear due to the dispersed or smeared material observed on the surface in both images.

There are still no visual signs that the steel is already exposed.

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Box 20

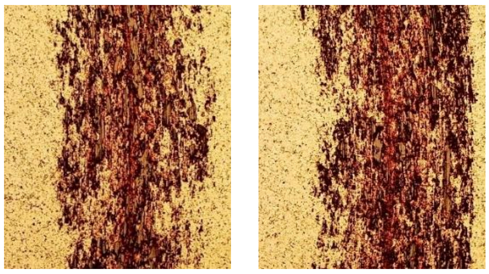

Figure 13
M1 CrN/TiN 610s wear track

Figure 14 shows the wear track of the M2 CrN/TiN 750s sample derived from the bolt on disc test, where sections of the deteriorated film are shown, adhesive and abrasive wear was generated as parts of the dispersed material can be observed on the surface and detachment of the film, in these images the trajectory of the detached material is visually perceived to such a degree that the steel is exposed, therefore the film suffered greater damage.

Box 21

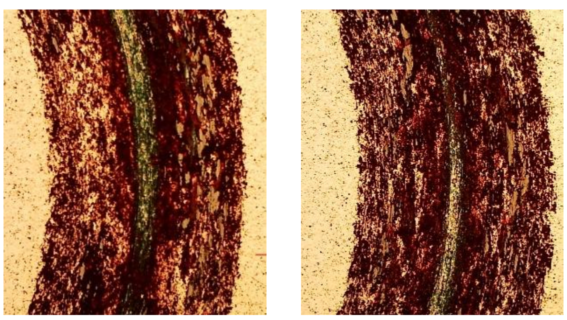

Figure 14
M2 CrN/TiN 750s wear pattern

With the data obtained from the bolt-on test, the calculation of the volume lost was generated, as well as the wear rate of both films. For this purpose, the ASTM G99 standard was used as a reference to obtain the calculations of these characteristics.

Equation Lost Volume

$$\frac{\pi(\text{track wear radius, mm})(\text{width of the footprint, mm}^3)}{6(\text{radius of the sphere, mm})}$$

Specific Wear Equation

$$K = \frac{\text{volume mm}^3}{(\text{Distance,m})(\text{Load N})}$$

With the data obtained, the following graph of lost volume was obtained.

As a result of the calculations obtained from the volume loss equation, it was observed that the sample M1 CrN/TiN 610s has a lower volume loss than the sample M2 CrN/TiN 750s.

Box 22

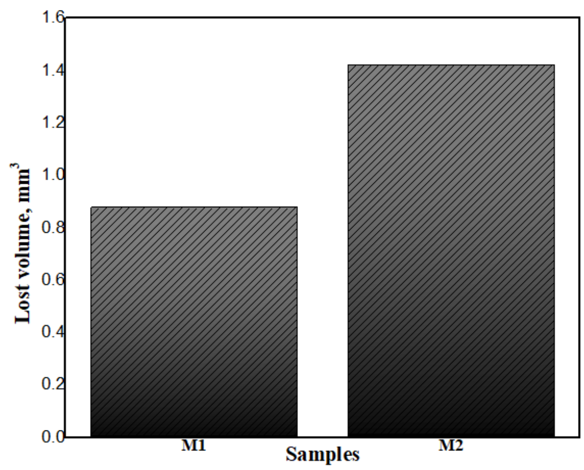

Figure 15
Volume loss of samples M1 and M2

With respect to the specific wear Figure 16, generating the calculations with the equation by the ASTM G99 standard, it was determined that the sample M1 CrN/TiN 610s has less wear, since it obtained a specific wear of 8.6×10^{-4} mm^3/N.m, while the sample M2 CrN/TiN 750s had a specific wear and tear of 1.4×10^{-3} mm^3/N.m.

Box 23

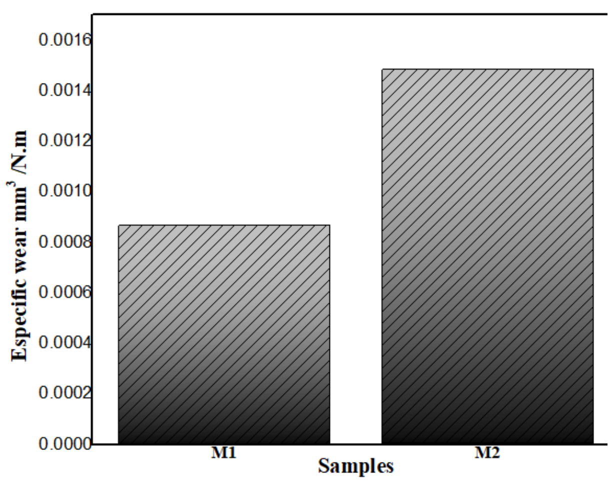

Figure 16
Specific wear of samples M1 and M2

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Accession

The VDI 3198 standard was used for the qualitative determination of the films with the best adhesion by applying an indentation of 150 kg with a Rockwell C machine.

Figure 1 shows the print generated by the test marked with the HF5 quadrant corresponding to the standard, with critical delamination in the diameter of the print, meaning a critical detachment of the film, determining it as unacceptable according to and in comparison with the HF5 quadrant of the standard.

Box 24

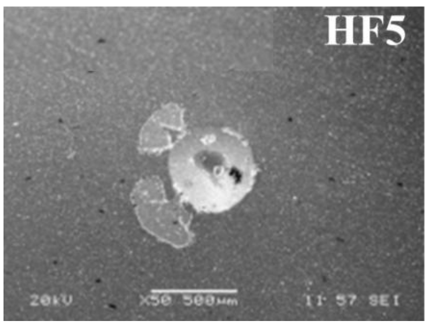

Figure 17
Indentation in sample M1 CrN/TiN 610s

With respect to figure 19, it could be observed that there is no presence of delamination on the surface of the film, there is no delamination in the circumference of the print, low amount of microcracks are perceived, according to the standard it is catalogued with the HF1 quadrant, with which it is determined as a film of acceptable failure.

Box 25

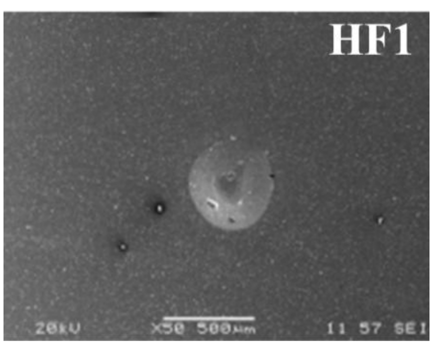

Figure 18
Indentation in sample M1 CrN/TiN 610s

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Conflicts of interest

The authors declare that they have no competing interests. They have no known financial interests or personal relationships that could have appeared to influence the reported article.

Contributions of the authors

Resendiz Albino, Juan David: characterization, results analysis, conceptualization, writing.

Melo Máximo, Lizbeth: contributed to advising in the development of this project, obtaining links with other institutions for the characterizations presented.

Melo Máximo, Dulce Viridiana: contributed to advising in the performance of the characterizations and reviewing results.

Vega Morón, Roberto Carlos: contributed to advising and reviewing the obtained results.

Financing

This research was conducted without funding.

Abbreviations.

1. AlCrN Chromium aluminum nitride
2. CrN Chromium nitride
3. EDS Energy-dispersive spectroscopy
4. HSS High-speed steel
5. m meter
6. mm milimeter
7. N Newtons
8. PVD Physical vapor deposition
9. SEM Scanning electron microscopy
10. TiAlN Titanium aluminum nitride
11. TiN Titanium nitride

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12. u.a Arbitrary units
13. XRD X-ray diffraction

Conclusions

According to the Energy Dispersive Spectroscopy (EDS) test, the presence of the elements Ti, Cr and N was verified in both samples M1 and M2, proving the effectiveness of the method for the deposition of these elements.

Both acceptable and unacceptable films were obtained according to the VDI 3198 standard, where the applied deposition factors for the M2 sample gave better adhesion in acceptable failures.

In the wear tests by the pin-on-disk method, it is concluded that a lower volume loss was obtained with the deposit factors in the M1 CrN/TiN 610s test.

Likewise, according to the specific wear according to the calculations generated with the ASTM G99 standard, a specific wear was obtained in the sample M1 CrN/TiN 610s of *m/N.m*, in comparison with our M2 CrN/TiN 750s reaching a value of *m/N.m,* with which a better wear resistance was determined in the M1 CrN/TiN 610s test, being the test with better wear resistance and lower volume loss.

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