

Tribological Study of Molybdenum Nitrides Under the Effect of Vanadium

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Our study of the MoVN ternary thin films has the objective of improving certain tribological and mechanical characteristics and of finding the appropriate stoichiometry to have the microstructure that corresponds to the desired properties using the characterization techniques: SEM -XRD-EDS-XPS- WDS. Nano-indentation and the scratch-test. Note that wear and friction resistance does not depend on the internal characteristics of the materials in contact as is usually the case for mechanical properties, but rather depends mainly on the structure and morphology and experimental conditions. Hypotheses will be presented in order to explain the tribological behavior of deposits and to match them to their physicochemical and mechanical properties. The EDS-XPS microanalyses revealed that the atomic ratio (N/V) ~ 1 for VN, that (N/Mo) = 1.22 for MoN and that (N/(Mo + V)) = 1 for the MoVN ternary films.

From this figure we find that the MoN film has an average coefficient of friction of 0.65. This film then has good wear resistance. Comparing this value with that obtained on MoN coatings deposited by the Alcatel PVD sputtering system on AISI substrate, (which is of the order of 0.45) therefore better than that deposited on XC100 and Si. This variation can be explained by the nature that differs between the coating/substrate interface and the other links that are generated. By comparing the Mo-V-N and MoN films, we can then say: increasing the V content in the Mo-V-N film is more favorable for the production of films with a low average value of the coefficient of friction. Which remains lower than that of MoN film (0.65)? So this is regular with the mechanical properties.

Keywords: Microstructure, Covers, PVD, Morphology, Coefficient of friction, Tribology.

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1. INTRODUCTION

The needs for the industry in cutting devices endowed with a hardness and an excellent wear resistance, led, for several years in the development of thin layers of nitrides of metals of transition (TiN, CrN, MY) put down by physical processes in phase vapor (PVD) These techniques allow to put down a superficial layer which brings remarkable improvements in the holding in service of the mechanical parts, particularly the cutting devices, These technologies are connected to the space, we also call them the nanotechnologies. The latter constitute surface treatments by application of covers in thin layers $\ll 10 \mu\text{m}$ of thickness [1]. A considerable attention in several laboratories was just concentrated to study these covers with the vision to prepare a harder material than the diamond, Consequently, the physical limit for the maximum of the increase of the hardness in covers nano composite stays an open question, which is extensively studied in many laboratories [2]. The texture is strongly influenced by the conditions of deposits (preceded to elaboration, temperature, pressure of gases, tension of polarization, the hardness and the tribological properties optimized are most of the time the first ones to be considered because of the potential application of these layers in the field of the manufacturing. Parts dressed in materials with molybdenum are not enough studied during these last decades. In spite of their good mechanical properties, these covers did not find applications in the industry. Indeed, compared with the nitride of chromi-

um, they present a coefficient of rather weak friction. The formation of oxides of high-temperature Mo allows to reduce the friction. Furthermore; Cr, Mo and N are important elements of addition allowing the hardening of the metallic alloys and the formation of passive films to fight against the chemical corrosion [3]. And a good adhesion to the steel substrata because of the solubility of Mo in the ferrous alloys [4] consequently. Ti-C-N is wear resistant abrasive and in the oxidation until a temperature about 800 °C. Shan and al. [5] indeed demonstrated that the brush resistance of these films against a steel ball was better than that of the film of TiN, because of its higher hardness and in the presence of the Carbon. Nitrides of metals of transitions (TiN, CrN, ZrN) their covers possess one High melting point, a high value of hardness and a big wear resistance, were widely studied During these last years and found of numerous Industrial applications. As well as TiN, the addition of Si in the ZrN resulted in a structure of cover nano composite in this study the moderate pure hardness of the cover ZrN (38GPa) is extremely raised compared with the values reported usually in the literature [6]. The moderate sizes of grains exactly are not reported, but are said varied between 10 and 20 nm according to the concentration of silicon. Pilloud and Al [7] found that the addition of Si in the ZrN makes decrease only the moderate hardness, slightly for low concentrations Si ($< 3.5\%$), then largely with the addition of more Si. We show that if the internal constraints are too important, it is them who control the crystalline organization of the growing deposit, and

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this one will turn in the direction $\langle 111 \rangle$ [8]. The Layers VN won a lot of attention recently in various sectors, such as in microelectronics, as barriers of broadcasting in integrated circuits thanks to their weak electric resistivity in comparison with TiN and for applications of the hard covers thanks to a big corrosion resistance a big hardness [9-10], a weak coefficient of friction, a good adhesion with the support and a very important electric and thermal conductivity [11]. The reasons of a privileged orientation and a type of texture remained unexplained which have explained the crystallographic orientation of the covers PVD on the basis of a relation between the energy of surface and the residual constraints Hibbs and al [12] has underlined that the hardness of the layers of TiN decreases with the concentration of chinks at the level of the joints of grains and thus are easily deformable. the hardness of the layers of TiN is between 2000 and 3000 Kg/ mm², however the porosity and micro cracks decrease the hardness. Les consistes binary form of CrN have a low coefficient of friction and resist effectively has the oxidation [13]. the speed of wear of CrN compare has TiN, ZrN, TiCN and AlTiN remains lower with essays of a steel ball 100Cr6 according to Rodriguez and al [14] deposits in ZrN are use for the cutting of materials non-ferrous previous studies have watch as well as the CrVN presents a coefficient of lower friction with A low volume of wear compared to that of the CrN and CrAlN. The compounds such as CrWN, MoCrN; TiCrN, CrN, bN, presents a good oxidation behavior and a big hardness compared with the other films of MoN or CrN. Abboudi.A et al found that the coefficient of friction of MoZrN is 0.42 for zirconium levels of 30% [15]. Chermime et al see that the MoVN compound in comparison with others shows a greater friction with the rate of vanadium which increases gradually [16]. Indeed, this type of covers presents not only a resistance increased in the wear but also a coefficient of friction enough weak which will allow to work without lubrication. On the basis of this concept, textures $\langle 111 \rangle$, often met in the literature, translate the presence of strong residual constraints within the deposits [17]. The research works which we began at the level of the Laboratory of Engineering and Advanced Materials Science (ISMA), Université Abbas Laghrour de Khenchela, first of all preceded by a bibliographical study summarizing the results of the researchers in the field of the nanotechnology and more exactly that of the thin layers (binary and ternary). The current work begins with a summary of the data and also the objectives follow-up of an introduction. As a consequence we have showed the material and the equipment which we have used to make our experimental essays followed by the results and the discussions. the study is divided into a structural study characterized by the AFM DRX micro written form. (Fig.1).

Another mechanical study which analyzes the hardness, the roughness and the tribology. And we draw at the end of work the adequate consequences expressed in the form of conclusion which regroups the results by comparing it with the objectives which leave the open door towards profound investigations towards new materials likely to be submitted to strong thermal, mechanical or chemical requests forced in search of new effective solutions.

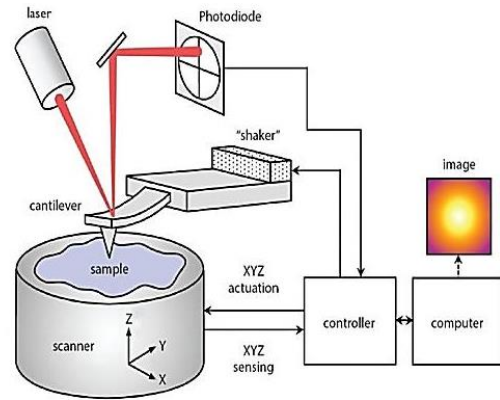


Fig 1 - Illustration of the Principe of the Atomic Force Microscope

2. EXPERIMENTAL DEVICES AND MATERIALS

The recording of the force between the end of the beam makes it possible to produce images (Figure 2) in order to measure the roughness of the surface studied up to a resolution close to the atomic scale (in contact mode). Determine the grain size or qualify the porosity of a layer. The three dimensions can be controlled by the tip; we can directly obtain an image of the surface, with a depth resolution of 0.01 nm and a lateral resolution of 0.1 nm [16-18].

The impressions were carried out using a hardness testing machine, which consisted: support pin for Rockwell B (diameter bead) and optical microscope; clamp; approach vice with mam; the type selector; monitor and program selector 15.6-250 kg; maneuvering level; rule for the dimensions; validation button, Testwell-Testor HT 1a, equipped with a Brinell indenter A 2.5 mm diameter ball was used. For each sample of coated XC 100, two tests were performed using standard parameters (1860 N).

The coefficient of friction is a property widely used to qualify and quantify the behavior of the surfaces in tribology contact despite the fact that its measurement is delicate and that the values obtained must always be interpreted according to the test conditions, in particular the atmosphere (humidity, temperature, etc.) in which the test takes place. The coefficient of friction and the wear of a coating are strongly dependent on its physical, chemical and mechanical properties (for example in the case of the C-polycrystalline coating) [17-19] and the apparatus measurement used. The tracking of the coefficient of friction and wear is an essential aspect in understanding the damage mechanisms that occur on cutting tools. Tribology tests are designed to analyze the behavior of a material subjected to a friction cycle against an opposing part.

The tribometer (Fig. 2.) is an apparatus that allows friction testing between a sample and a ball (eg steel, ceramic, carbide, etc.), subjected to a given load, under chosen conditions, with or without lubrication. The ball (of given dimension) is mounted on a fixed support and the sample is reciprocating or rotating. This device is equipped with a digital acquisition chain which records the evolution of the friction force, the coefficient of

friction, the depth of penetration of the ball in the sample, the temperature, etc., depending on the distance of friction (duration of friction, number of cycles) and this, during all the test.

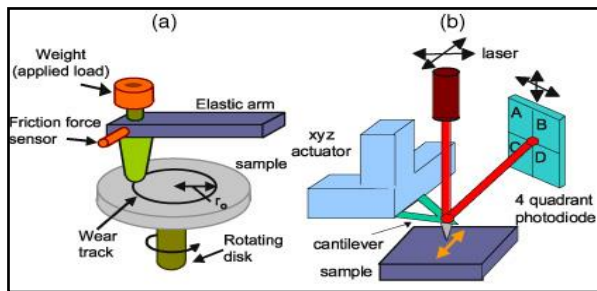


Fig. 2 – Principle of the (a) rotary and (b) alternating tribometer [18]

Table 1 – Chemical composition of XC100 steel wt. %

XC100	C	Mn	S. P	Si	Cr	Ni	Cu
wt. %	0.95	0.25	S < 0.025	0.15	0.15	0.2	0.2

During our study a tribometer (rotataif and alternating) was used to perform friction tests on our coated samples without lubrication under ambient laboratory conditions. The characteristics of the tribometers used are the following: Stable contact point and no parasitic friction, Possibility of testing samples of variable dimensions and geometries, tests compatible with DIN 50324, AS TM G99 and ASTM G133, possibility of performing high temperature tests (up to 800 OC) for the rotating tribometer only. To determine the coefficients of friction and to study the wear of our films, ball-type friction tests on discs were carried out using tribometers in linear reciprocating mode (Table 1) and a rotating tribometer [18].

Preparation of Si substrates. Clean square or non-square substrates in 1 wafer of Si (100) polished 1 side (cut on the polished side) using the diamond stylus and a ruler, make samples of 1cmx1cm. The Si thickness is 380 μm.

Preparation of XC100 steel substrates. Check the R_a (average) and R_t (peak-to-peak) roughness of the current samples: have an R_a of the order of 200 to 500 nm maxi, an R_t of 1 μm maxi see a little less, but have the same Roughness for all samples. Define the number of square Si for constraints, other (non-square) for composition,

DRX, MEB, etc. (But not too small either!) To put at each deposit to get enough for all the characterizations to be done thereafter. Similarly, calculate the nb of steel samples to be put for each deposit, thus the total nb of samples to be prepared, so that it is necessary to optimize the placement of the samples on the substrate holder (steels and Si) , And to always put them in the same place at each deposit for reproducibility NB: Consider keeping a sample of each (Si and steel) for future analyzes (1 Si and 1 steel)

Cleaning of substrates. Before loading a substrate (Si or steel) into the enclosure, it must be cleaned ex situ. Put alcohol at 95° in the ultrasonic bottom and then put samples in a beaker with: trichloroethylene (99.5 %) for 5 min , pure acetone (99.5 %) for 5 min, ethanol (99.5 %) for 5 min. At the end of each stage, the

substrates are rinsed with deionizer water and stored in pill bottles containing absolute ethanol (for Si) or in a desiccators for steels (to avoid oxidation) up to their usage. NB: clean the Si assemblies (all of them clean all at once) and the steels in several steps, do not put steels with Si at the risk of damaging them. Check that you have cleaned the chamber and that you have the correct target (especially that of Mo) mounted on a cathode and mark it (top or bottom to know the RF generator and the tuning box to use)

To check the cleanliness of the enclosure, iron a sopalin paper cleaning substrates in situ and target to be done before each deposit when residual vacuum reaches around 10^{-6} or 8.10^{-7} mbar on Alcatel gauge procedure but apply 12 kV with the HT generator for 5 min on substrates and target under Ar. NB: for the first deposit, after the machine has been stopped for more than 3 months, strike the target at least 15 mn under Ar.

The pre-pulverizing operation consists in placing the target under the deposition conditions for a time of 5 to 10 minutes. The target, after ionic cleaning, is not nitride. There is therefore a transient state under unstable conditions. Since some of the deposits have durations of the order of 5 to 10 minutes, this transient state introduces an error which can be not negligible in the calculation of the deposition rates. It is therefore necessary to nitridate the target before starting the deposition.

3. RESULTS AND CONCLUSIONS

3.1 The Atomic Force Microscope (AFM)

The Atomic Force Microscope (AFM) is à widely used technique for providing images of our sample surface. It reaches very high resolution levels up to the atomic level. So we can make images of surfaces with a very high resolution. When the tip approaches the surface of the sample (At a distance of a few tenths of a nanometer), Forces of repulsion van der Waals between the atoms of the tip and those of the test surface cause deflection of the beam, depend on the distance between them. The main mode of use of an AFM microscope is based on the use of a feedback loop to maintain a constant distance between the tip and the surface analyzed by means of a piezoelectric actuator. Images of the surface of some of our films were obtained using a constant force of 0.035 N/m and determined their respective roughness. It is observed in the AFM images Fig. 4a, b; the surface density of the columns decreases with the thickness, so the columns are bigger. The layers must show a hardness decreases as their thickness increases. Note that the roughness decreases with the width of the column. The roughness varies from 44 nm to 3.7 nm for the Mo-V-N coating and from 73.2 nm to 2 nm for the Mo-N coating. Fig.3 indeed, at the beginning of the growth of the layer is first creating islands that will coalesce and hinder the movement of dislocations. Impede the movement of dislocations. For Mo-N and Mo-V-N it is obtained that the particle size of about 80 nm randomly dispersed.

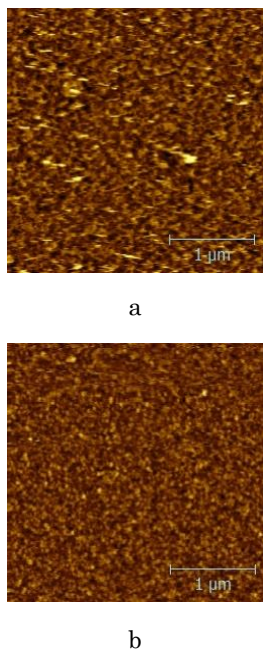


Fig. 3 – The surface orphologies: a – Mo-N and b – Mo-V-N

3.2 XRD data

The X-ray diffraction spectra were recorded using a X'PERT PRO MRD type diaphragm meter from Panalytical, equipped with a copper anode RX tube. An acquisition time of 5s per angular step of 0.04 ° was used over the interval between 30 ° and 55 ° (2θ). The identification of the crystalline phases presented was made by comparing the observed lines with those of the appropriate phases contained in the PDF2 database. The spectra of DRX Fig.6 Deposits of the vanadium nitride with different concentrations of V on the XC100 steel are mentioned in the figure. The observed peaks reveal the presence of VN, V2N, MoN, Mo2N, and MoVN of cubic structure (JCPDS not 004-0850). Presence of the phases VN, V2N, orientation (111) and (200) between the angles (2θ). (40° and 50°) this is explained by the strong presence of residual stresses.

Table 2 – Parameter and crystallite size of MoN, Mo-V-N and VN coatings; thickness atomic percent

deposit		MoN	Mo _{0.39} V _{0.15} N _{0.45}	Mo _{0.20} V _{0.30} N _{0.47}	Mo _{0.06} V _{0.45} N _{0.49}	VN
gaz	Ar/N ₂	75/25				
Mo	P _{Mo} (W)	650	640	650	350	-
	U _{Mo} (- V)	800	850	850	500	-
V	P _V (W)	-	400	550	650	650
	U _V (- V)	-	550	850	800	900
N		48.6	45.0	47.0	49.0	48.0
Mo		48.0	39.0	20.0	05.0	-
O		3.4	1.0	3.0	1.0	3.0
V		-	15	30.0	45.0	49.0
N/(Mo+V)		0.99	0.83	0.94	0.98	0.97
e (μm)		1.36	1.42	1.64	2.2	1.3

3.4 Parameter and Crystallite Size

Below is a summary of the data with the corresponding results. The potential of Mo and V is

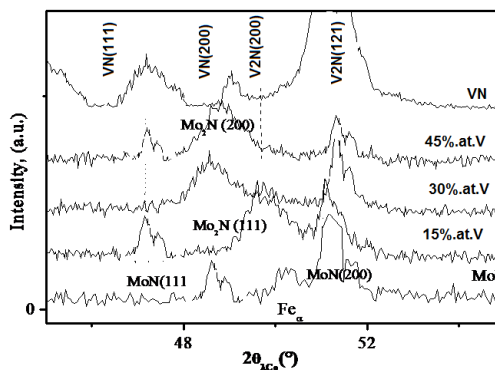


Fig. 4 – XRD of MoN, Mo-V (10%at.V)-N, Mo-V (30%at.V)-N, Mo-V (45%at.V)-N, VN.

3.3 Hardness

It is clearly seen from Figure 5 that the hardness is a function of the vanadium content however it increases and takes a value and remains constant this is explained by the saturation of the MoVN compound vanadium atoms then the hardness reaches a peak which is the order of 25Gpa.

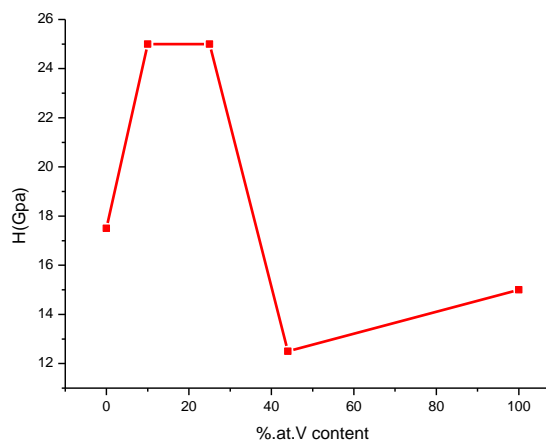


Fig. 5 – Hardness of MoVN as a function of % at.V

no more Mo. Then the Thickness decreases considerably.

See the corresponding mesh parameters in Table 2.

The ternary MoVN is taken and the vanadium level is gradually varied (0 %, 15 %, 30 %, 45 %, 100 %) and the results are recorded which increase to a peak (30 % 25 GPa) for MoN (18.5GPa) and VN (15GPa) Fig7.

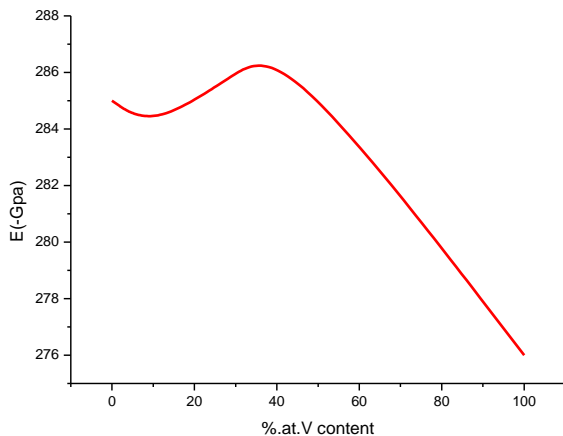


Fig 6 – MoVN young module as a function of % .at. V

3.5 Tribological Behavior

Note that wear and friction resistance does not depend on the internal characteristics of the materials in contact as is usually the case for mechanical properties, but rather depends mainly on the structure and morphology and experimental conditions.

Hypotheses will be presented in order to explain the tribological behavior of deposits and to match them to their physicochemical and mechanical properties.

The microanalyses by EDS-XPS revealed that the atomic ratio (N/V) ~ 1 for VN, that (N/Mo) = 1.22 for MoN and that (N/(Mo + V)) = 1 for the ternary films MoVN.

The chemical composition of these films strongly depends on the voltage applied to the targets. The molybdenum content increases when the tension applied to it increase while the N content is unchanged. XPS analyzes revealed that ternary Mo-V-N films show a solid solution of molybdenum of zirconium and nitrogen in the form of a ternary Mo-V-N compound. the actual indication of the nitrogen content is much greater because of the preferential spraying of N during argon bombardment of the layers. The films are homogeneous and isotropic. The results by XPS were confirmed by the DRX.

Surface and cross-sectional microstructural analyzes by AFM, MEB-FEG showed that all Mo-V-N films are homogeneous on surfaces where a deposit is applied (silicon, steel,) thanks to a thickness of 2.2 μm and the high mobility of the

atoms arriving on the substrates and to highlight the influence of the percentage of V on the evolution of the coefficient of friction of the Mo-V-N layers deposited on XC100 substrate, we presented on the IV.11) the variation of the coefficient of friction as a function of the V content.

From this figure we find that the MoN film has an average coefficient of friction of 0.65. This film then has good wear resistance. Comparing this value with that obtained on MoN coatings deposited by the Alcatel PVD sputtering system on AISI substrate, (which is of the order of 0.45) therefore better than that deposited on XC100 and Si. This variation can be explained by the nature that differs between the coating / substrate interface and the other links that are generated. By comparing the Mo-V-N and MoN films, we can then say: increasing the V content in the Mo-V-N film is more favorable for the production of films with a low average value of the coefficient of friction. Which remains lower than that of MoN film (0.65)? So this is regular with the mechanical properties.

In fact, the Mo-V film (30 % at.) - N has the lowest average value of (0.42), which explains the concordance with the dynamic movement of dislocations during the zirconium substitution in the MoN network. As molybdenum nitride has a lower coefficient of friction than zirconium nitride, then with the increase in V content we also increase the VN content relative to MoN in Mo-V-N films and consequently we increase the coefficient of friction.

The VN layer has a coefficient of friction of the order of (0.68) which is larger than that of MoN coatings.

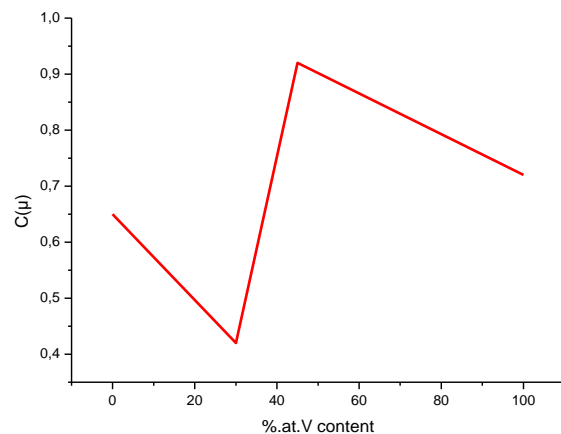


Fig. 7 – Coefficient of friction \sim Mo-V-N films obtained on steel substrates XC as a function of the V content

Figure 8 represents the coefficient of friction of the film MoVN as a function of the thickness, the figure shows for a fixed value of the vanadium rate 25% the way in which the coefficient of friction varies if an enlargement (zoom) is made around the value of the coefficient of friction of 0.4 to 25% V at 25% Vanadium the coefficient of friction varies substantially according to the thickness Fig. 8 (from 0.38 to 0.46).

The minimum of FIG. 7 can physically be explained with a coefficient of friction equal to 0.4 (see the curve in Fig. 5). These two figures show extremums whose ternary compound is saturated with vanadium atoms (25% vanadium). The size of the vanadium atom is larger than that of the molybdenum nitrogen which causes a distortion of the crystal lattice and thus an increase in the coefficient of friction.

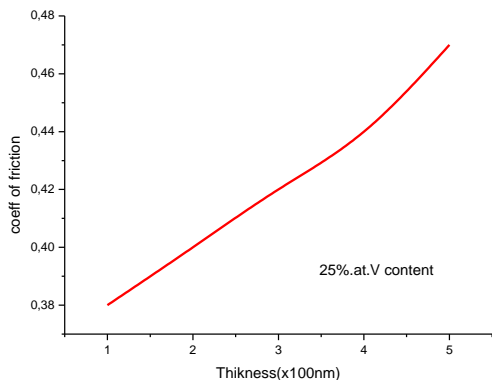


Fig. 8 – Coefficient of friction as a function of thickness

Figure 9 shows Micrograph of the scratch test of the damage zones (LCI) and tearing of these coatings (LC2) of the films of I) Mo-V (2% at.) – N II) Mo-V (5% at.) – N III)

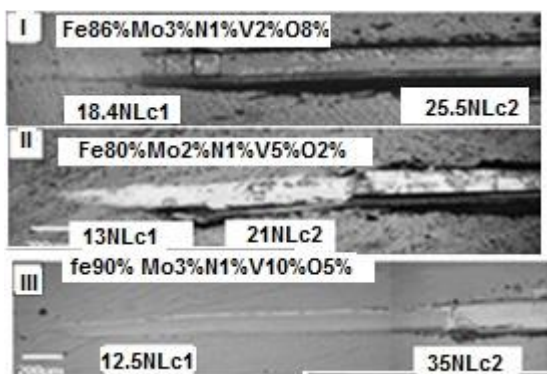


Fig. 9 – Micrograph of the scratch test of the damage zones (LCI) and tearing of these coatings (LC₂) of the films of I) Mo-V (2 %at.)-N II) Mo-V (5%at.)-N III)

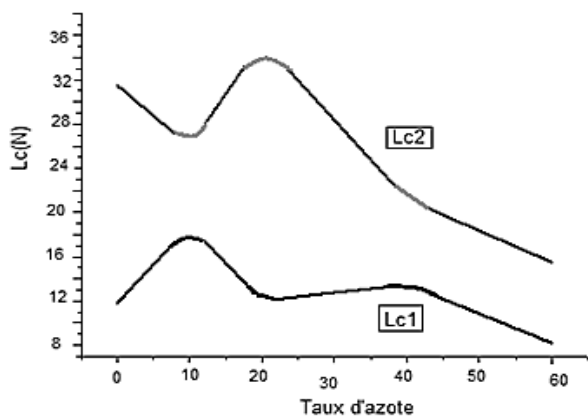


Fig. 10 – LCI and LC₂ critical loads for VN coatings, Mo-V-(15% at.)-N, Mo-V (30% at.)-N, Mo-V (45 at. %) -N and MoN obtained on XC100 Mo-V (10%at.)-N

In order to highlight the influence of the V content on the adhesion of Mo-V-N films, we performed Scratch tests (under the same conditions).

Figure 10 shows the evolution of LCI and LC₂ applied to the surface of Mo-V-N coatings as a function of V content. We defined LCI, as the normal force corresponds to the beginning of development of the

cracks in the deposited layer and LC₂ being the relative effort at the beginning of the tearing of this layer of its substrate.

We found that the effort required for the cracking of the MoN, LCI layers are 11.8N, and the stress on the LC₂ substrate detachment is 31.5N. We have noticed a clear improvement when V is added at this time. LCI and LC₂ change to 13 and 35.5 N. The adhesion of the Mo-V-N layers is improved when V is added. The LC₂ force takes a maximum value of 35.5 N for at V content of 20 % at., Then decreases respectively up to 26 N (for 15 % at. V) and finally at 21.2 N (for 42 % at. Zr).). But it always remains higher than that of MoN (which is of the order of 32N). These results can be explained by the fact that the residual stresses decrease LC₂ (with the addition of V) increases in the Mo-V-N system. LC1 changes in the same way as coefficient of friction that while with roughness it is inversely proportional.

Damage to the MoN film is observed (at 11.8 N) with tearing and delamination along the trace and appearance of conformal cracks at these edges at the beginning of the scratch test. The appearance of the residual stresses in the layer and responsible for this degradation which is due to the variation of the hardness between the two layers of the interface. We note that the effort of the beginning of delamination of the LC₂ layers of the MoN film is (31.5 N).

As for the film VN, it presents values of LCI and LC₂, respectively equal to 8.2 and 15.5 N at the beginning of the test the damage of this layer is of ductile type. The damage of the VN film is important and is done with tearing of the layer along the edges of the trace, this is probably due to their small thickness. Difference of mechanical properties of VN film and XCI 00 substrate.

On the other hand, by incorporating V in the MoN system, the LCI and LC₂ values for the Mo-V-N coatings increase to reach an optimal value of 13 and 35 N, respectively, for a V content of 10 at %. The Mo-V film (5 % at.) - N has the best adhesion compared to other films in this series. Indeed, from 10 % at. of V, the LCI and LC₂ efforts, although much higher than those of the VN or MoN binary deposits, respectively decrease to 12.5N and 21N, but still remain interesting for mechanical applications

This hypothesis can also explain the increase in Mo-V-N film stresses with the V content. In reality, the increase in the substitution of Mo atoms by atoms leads to the increase in the stress of Mo-V-N films

4. CONCLUSION

The results of researchers in the field of deposits of the nano-ternary and binary layers note that the compounds most used for their hardness such as CrN, TiCN; CrWN, AlTiN, ZrSiN, Ti-C-N resistant to abrasive wear and oxidation to a temperature of about 800° and that the resistance to friction is better than that of TiN.

CrN films tend to replace TiN films because they have very good corrosion resistance, good stability at high temperature as well as thick layers are required. The study on deposits about our work that remain

unexplored was approached by a bibliographic search which highlights the current state of research and the experimental chapter on compounds; MoN, VN, MoVN.

The morphological study of the ternary MoVN deposit showed that an orientation given by the dexterities of the texture (111, 222) is due to residual internal stresses.

MoN is less hard than CrN however more catchy and adheres more into the substrate; VN is hard, but does not have great grip. Now for the addition of vanadium in the molybdenum nitride, the hardness passes through a max to (35 %at. to V), for residual stresses max to (45 %at. V), the roughness is good for MoN with low vanadium concentrations.

MoVN has a max hardness with vanadium concentration and is less corrosion resistant than CrN. With regard to hardness, we gradually vary the vanadium content by 0 %, 15 %; 30 %; 45 %; Up to 100 % the results for the hardness fluctuate up to a max which has the value (30 % at. V; 25 Gpa) for MoN

(18.5 Gpa) and VN (15 Gpa) And the potential is also varied (550 v, 900 v, 800 v, - 900 v) and the power (300 w, 650 w, 650 w, 650 w).

The Young's modulus takes values almost very close, which is indicated for values below 100 % of V, so the crystal structure does not influence the resistance capacity.

Conversely, the most important residual stress is in 30 % V with a value of 1.9 Gpa therefore MoN and VN are less strained than MoVN, the introduction of the vanadium particles destabilizes the compound which is essentially intrinsic and Thermal deposits

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