MODERN METHODS AND PROCEDURES FOR SUBMISSION OF THIN LAYERS IN VACUUM

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Abstract: This paper tackles bringing to the attention of the specialists in the field, of some modern methods of increasing wear resistance of tools, for working wood. Thus, the results are processed and analyzed by laborious experimental testing procedures for submission of thin layers in vacuum, using the cathode pulverization, type magnetron. The experimental tests were performed on modern installations, using the performing equipment of measurement and control, both the working parameters, and the thickness of the deposited layers; they were conducted in the laboratories of University Cluny of France.

Keywords: procedure, layer, magnetron, vacuum

1. INTRODUCTION

1.1. Advanced technologies regarding the cathodic pulverization, type magnetron. Procedures for submission of the thin layerss in vacuum

The cathodic pulverization, type magnetron, starts from the classical pulverization, which consists of a plan cathode, provided with a screen to limit the plasma and an anode; behind the cathode is located a magnetic system, and at a certain distance from the front of the cathode (the pulverization aim), optionally, an additional anode [1].
The magnets or the electromagnets which create the magnetic field from the front of the cathode are arranged to be at least one region in the front of the cathode, where the locus of the magnetic field lines, parallel to the aim surface, to generate a closed curve. By adding at one pulverization cathode of a magnetic system, a pulverization apparatus, called Penning cathode, is obtained; by adding to it an anode to ensure a certain configuration to the electric field, a special pulverization apparatus is made, with the electric and magnetic fields cross, called pulverization apparatus, type magnetron [2].
Sometimes, the anode, providing a certain configuration to the electric field between it and the cathode, is not integrated in the construction of the pulverization apparatus, but belongs to the pulverization system, called PVD magnetron pulverization system. This is the working principle adopted in the experimental attempts, made by the author of this work, in the laboratories of the University Cluny, France.
This crossed electric and magnetic fields leads to substantial modification of the spatial distribution of the electric charges that occur in the luminescent discharge, by the combination of these at the surface of the pulverization aim.
The charged particles from the cathode pulverization plasma, influenced significantly by the existence of crossed electric and magnetic fields [3, 4], are:
- the positive ions of argon and nitrogen eventually, generated in large numbers and which bombard the cathode, respectively the anode;
-the secondary electrons released from the pulverization aim under ion bombardment;
-the secondary electrons, resulting at the anode;
- the electrons resulting from ionization work gas (argon).

After much research, we can say that due to inhomogeneity of the electric and magnetic fields, and the deviation from the perpendicularity between them, the trajectories of the electrons emitted from the cathode, which are crucial in increasing plasma to the aim surface, are not perfectly cycloidal [5 , 6].

1.2 Basic Parameters Of The Magnetron Cathode Pulverization

The basic parameters of the magnetron cathode pulverization are:
- the tension between electrodes; these magnetron pulverization systems operate at low voltage, so that does not exceed 1000 [V]; the working practice voltage is 300 ... 700 [V]. The aim working is negative polarized and the anode, rule, is null.
-the current (current density) in the discharge, respectively the average power dissipated in discharge, depends on many factors, such as: the working voltage, the working gas pressure, the magnetic field induction, the magnetic system configuration, the nature of the material of the pulverization aim, and limit by the power supply; so the current density reaches average values of 80 [mA / cm ^{2}] for cylindrical magnetron, 160 [mA / cm ^{2}] for magnetron with conical aim and 200 [mA / cm ^{2}] for magnetron with the plane aim . Correspondingly, the average power obtained in the magnetron pulverization system is 40 [W / cm ^{2}] for cylindrical magnetron, 80 [W / cm ^{2}] for

magnetron with conical aim and 100 [W / cm ²] for magnetron with the plane aim;
- the geometry and the size of the magnetic field induction is the most important parameter for many features of the magnetron pulverization; so, the horizontal component of the magnetic induction at the pulverization aim surface is in the range (0.03 .... 0.1) T;
- the working pressure; it can work at working pressures of 0.01 to 1 [Pa].

2. EXPERIMENTAL TESTS

2.1. Presentation Of The Installation, Type Magnetron, For Vacuum Submissions - PVD

The installation used for submissions, type Nordiko, was modified to allow the independent working, or simultaneously, with two aims (fig. 1), and consists of:
1-vacuum chamber, 2 - RF generator (radio frequency) for Zr aim, 3 - RF generator for Cr aim, 4 - central panel, 5 - generator for substrate cleaning, 6 - cryogenic pump for the secondary vacuum, 7 - mass flow meter, 8 - switches to access in the vacuum chamber of the working gas (Ar and N_2), 9 - programming block of the submission time and the used aims (Zr and Cr), 10 - voltage control block on the Zr aim, 11 - voltage control block on Cr aim, 12 - gas cylinders working (Ar and N2).

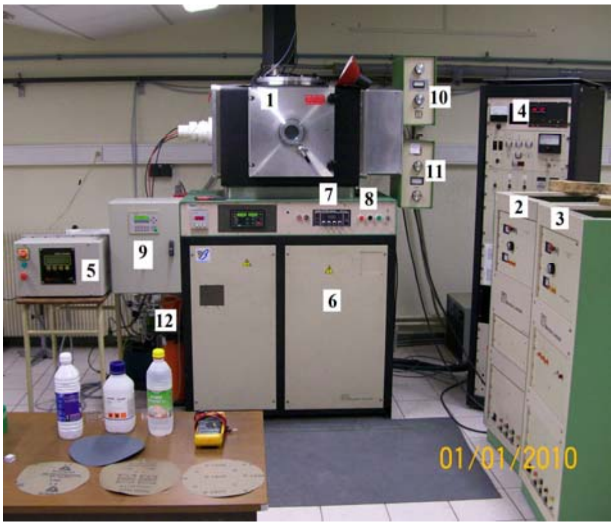


Figure 1. Installation of vacuum submissions by

On the substrate holder several samples (substrates) were fixed in order to obtain a more complete characterization of the made submissions. The characterizations were performed by determining the following elements: the submitted layer thickness, the growth speed of the deposed layer thickness, the chemical composition of the submission, the determining of the internal stresses in the submitted layer.

1 - Zr aim, 2 – Zr aim shutter, 3 - Cr aim, 4 - Cr aim shutter, 5 - substrate holder, 6 - fixed substrates on the holder, in order to initiate the submission process.
The performed characterizations analysis, yielded some conclusions about the Zr submission process, which have allowed the optimization of this process, for several types of tested materials (fig. 2), as: Zr, ZrN, CrZrN. For a complete analysis of the process, the parameters for each submission (which were set for the laboratory installation), were recorded in the file of the submission process and were presented for each type of material tested.
The submission installation was of laboratory that allowed the process development, with the operator intervention at each stage with values of the parameters, time and settings, so that they can perform various regimes and identify the influence on the submitted materials, by the characterizations effectuated.

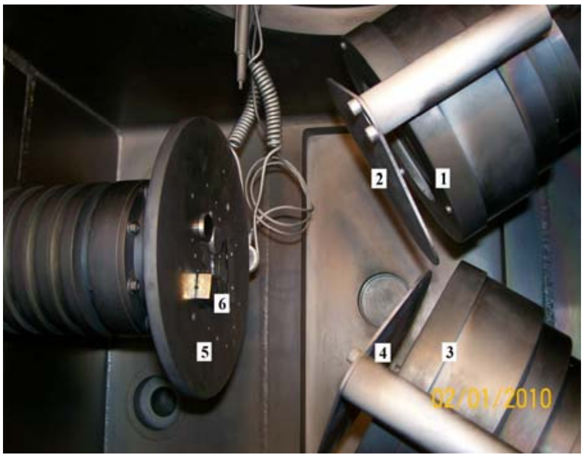


Figure 2. Interior of the vacuum chamber

Also, laboratory installation allowed, as for a number of parameters, the set values be more than the usual from industry, so the voltage on each aim can reach till 900 [V], while in industry, usually, are used values till 300 [V]. The use of such installation has the advantage that the submission process can be lead interfering in experimentation and improving the characteristics of the submitted materials. The disadvantage is to reduce the automation of the entire sequence of steps of the submission process, all the decisions on parameter values, durations, sequences (which are generally interdependent) are of the operator.
The manual management of the submission process, given the interdependence of the parameters and the need to experiment different values in order to determine the influence on the submitted layers, can lead to:
- the instability or the difficulty to achieve a balance between the values of the main parameters (pressure, voltage, power);

-the most difficult case, the loosing of the plasma in the different stages of the submission.
The plasma loosing, since before the submission stage, may attract repeat the experiment recurrence. This happens if working with two aims and the plasma is lost on one aim, if the plasma can not reacquire again soon. At the stage of submission, no matter how many aims work, the loss of plasma involves repeating the experiment. The situation is avoided because the preparation of a submission takes, for this installation (equipped with cryogenic pump for the second vacuum), between 8 and 10 hours. Also, the costs of the submission itself (materials, chemicals, electricity consumption) must be considered, too. It is mentioned that during the actual submission, the current consumption is 90 ~ 100 [A]. Given the above, it is important the presentation of the working steps for the submission installation.

2.1.1. Work Session For The Submission Installation In Vacuum, In Plasma

The working operations are multiple and done rigorously, starting with the cleaning of the substrate in vacuum, in plasma, by bombardment with argon before starting the submission process; after going the time proposed, the generator off and disconnect from the network of 380 [V]. The generator leads, automatically, the cleaning process of the substrate, according to the set values. The working operations are:
- cleaning of the aims in vacuum - in argon medium. Beginning with this stage, plasma must achieved and maintained, and the whole process management is manual. It also browses all the steps of this operation, and finally the parameter values are completed in the file of the submission process. They are: aim power: 200 [W], aim voltage: 500 [V], reflected power: 0 [W], working pressure: 10 [μBar]; cryogenic pump temperature: 12 [K]; time: 5 [min].
-before submission; then, the steps of this operation follow, that act to stabilize the process parameters;
-submission; the parameters values that must be kept under control during the submission process, for using a single aim and working gas Ar (Zr submission); the parameters values are completed in the file of the submission process;
-stop the pulverization process; steps taken in this operation led finally to disconnection from the network of 380 [V] of the pump, to cool the substrate;
-samples recuperation.

2.2. Submissions Made And The Parameters Of The Submission Process

2.2.1. Zr Submissions In Vacuum, In Plasma, In Argon Medium

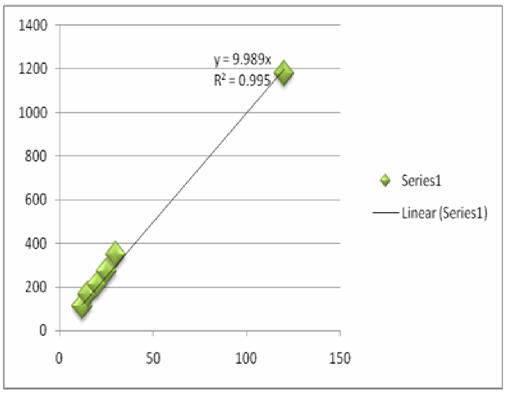
The Zr submissions aimed to obtain a knife with improved features, for effectuate the laboratory tests, needed to determine wear. The submission, having time 120 [min], was the first of the 29 submissions made, thus obtaining the first face of the knife - the settlement.
The second submission, of 120 minutes duration, was performed in order to cover with Zr the evolve face; for placing the knife on the substrate holder, the previous position was taken into account. The Zr aim features used are: purity: 99.95%, diameter: 4 [inches], thickness: 0.125 [inch].
The Zr aim was new, and the Cr was used before and kept mounted, from the previous submissions.
The mention has relevance with the notification that in the first submission processes for Zr aim plasma was very difficult obtained, but once obtained, it was stable during the submission process. For Cr aim, plasma was easily achieved, but was unstable (for the first submissions, when we worked with both aims, simultaneously).

Table 1. Parameters of the submission process for Zr

Material	Time [min]	Press [μBar]	Ar [%]	N_2 [%]	Voltage Zr [V]	Voltage Cr [V]	Substrates used
Zr	120	4	100	0	900	-	Si, steel, carbide metallic
Zr	7	4	100	0	900	-	Si, otel
Zr	12	4	100	0	900	-	Si, otel
Zr	15	4	100	0	900	-	Si, otel
Zr	20	4	100	0	900	-	Si, otel
Zr	30	4	100	0	900	-	Si, otel
Zr	120	4	100	0	900	-	Si, steel, carbide metallic

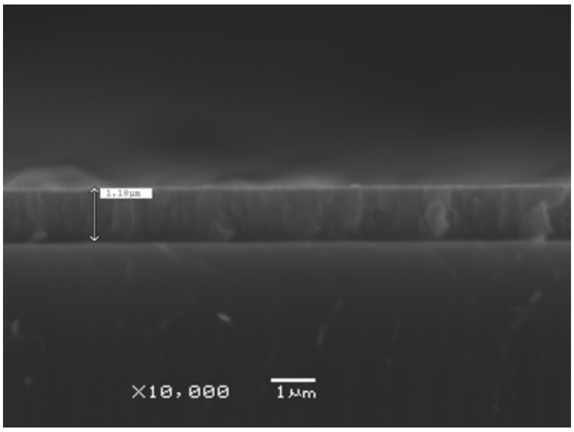
3. CONCLUSIONS

In conclusion, the movement of the electrons generated in the plasma, or ejected from the aim in electric and magnetic fields is strongly influenced by the magnetic field, their movement having cycloidal trajectories along the magnetic tunnel. Thus, the electrons way in plasma increases, and implicitly, the possibility to produce more ionization, by collision. The plasma, at the surface of the pulverization aim has a high degree of ionization, and a more intense bombardment of the cathode aim result, and thus relatively high efficiency pulverization.



Grosimea (nm)

Timpul (min)



Graph 1. Thickness increasing of Zr submission [nm] in function of time [min]

Submission	% mass O_2	% mass Zr	Total
Zr 120 min	2.3	97.7	100
Zr 30 min	15.7	84.3	100
Zr 25 min	2.2	97.8	100
Zr 20 min	2.2	97.8	100
Zr 15 min	2.1	97.9	100

Thus, the results of the experimental determinations are confirmed by above the charts.

To note is that this submission was made to the settlement face of the knife, for the submission on to the evolve face, the process parameters were identical. The aim of the submission was to determine the increasing rate of the layer thickness, leading thus to increase the wear resistance of the usage knife edge, and substantial, the working time on the processing of the wood industry.

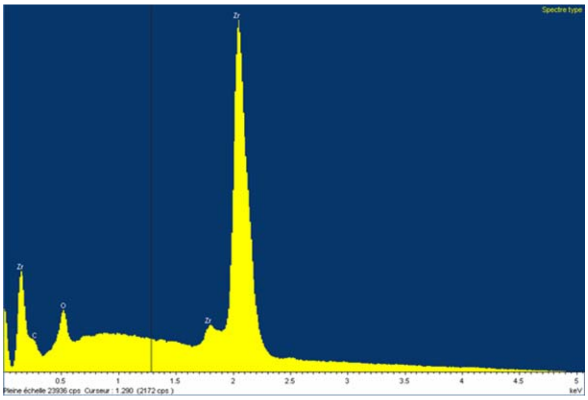


Figure 3. Chemical composition for the Zr submissions 120 [min]

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