

RESEARCHES ON OBTAINING THIN FILMS OF STAINLESS STEEL FROM INOXIDABLE STEEL BY PHYSICAL VAPOR DEPOSITION MAGNETRON ASSISTED

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ABSTRACT

The experimental researches carried out in this paper aimed at obtaining thin films of stainless steel and their characterization in terms of structural, optical and electrical properties. The films were made using a PVD spraying system consisting of a vacuum chamber with a capacity of 2 liters, a plane magnetron with ferrite magnets ($\varnothing 40 \times 22 \times 9$) neodymium ($\varnothing 15 \times 8$), a vacuum pump with sliding blades, a DC source of 100 to 600 volts. The atmosphere used for the maintenance of plasma during deposition was rarefied argon in a pressure range of $3 \cdot 10^{-2}$ - $8 \cdot 10^{-3}$ mbar. The argon flow rate used was $100 \text{ cm}^3/\text{min}$. The support material consisted of glass plates of $76 \times 25 \times 1$ mm dimensions and copper plates of $80 \times 20 \times 1$ mm. It has been noticed that the properties of the deposited films are influenced by the parameters used. It has been found that when increasing the deposition time, the surface resistance and transparency decrease.

KEYWORDS: d.c. magnetron, electrical and optical properties, stainless steel deposition

1. Introduction

Thin films have become a major research area with applications in electronic, optical, magnetic, biochemical, protective, sensing or catalyzing devices. Nanometric dimensional control allows very good flexibility in adjusting thin film properties and adapting both the properties and behavior of the devices containing them.

There are several methods of thin films deposition, the PVD method being the most widely used. Choosing one of them depends on: the requirements for the thin layer properties, the maximum temperature the substrate can withstand, the compatibility of the process with the processes applied to the substrate before and after deposition, and finally the production costs, the efficiency and the large-scale manufacture of the products.

The control of the deposition of thin films from the vapor phase involves a thermodynamic and kinetic control of the phase transition (evaporation - condensation), nucleation of the crystalline phase to condensation of molecules on the surface of the substrate, growth of nuclei or film grains, and activation of processes such as diffusion and desorption of molecules.

The production of thin films from austenitic stainless steel is the basis of applications in the microelectronic, micromechanical, food, chemical, petrochemical, energy and medical industries requiring materials resistant to corrosion, abrasion, friction, oxidation at high temperatures as well as in making decorative pieces.

The terrestrial, naval, aerial, space transportation means also includes components made of stainless-steel coated materials of good mechanical strength as well as resistance to aggressive environments and atmospheres.

The unlimited resistance to erosion of austenitic stainless steels has been noted, compared to the poor corrosion resistance of copper where the corrosion products are fragile and easy to shear [1, 2].

Resistance to high temperature oxidation and corrosion in different environments, correlated with mechanical and physical properties, are primary elements, stimulating the creation of new films of stainless steel.

The research carried out in this paper has as general objective the setting up of a technology for obtaining thin films of austenitic stainless steel X10CrNi 18-8 and their characterization.

2. Experimental Conditions

A circular austenitic stainless-steel plate with a diameter of 46.5 mm and a thickness of 1 mm was used as the target for the films.

The support material consisted of glass plates of 76x25x1 mm and copper plates of 80x20x1 mm.

The films were made using a PVD sputtering system consisting of a vacuum chamber with a capacity of 2 liters, a planar magnetron with ferrite magnets ($\phi 40 \times 22 \times 9$) neodymium ($\phi 15 \times 8$), a vacuum

pump with slides blades, a DC source of source of 100 to 600 volts.

The installation allows varying the magnetron substrate deposition distance between 25 and 90 mm, and the substrate temperature can be monitored with a chromel - aluminum thermocouple.

The atmosphere used for plasma maintenance during deposition was thinned argon in a pressure range of between $3 \cdot 10^{-2}$ - $8 \cdot 10^{-3}$ mbar. The argon flow rate used was $100 \text{ cm}^3/\text{min}$.

Fig. 1 illustrates the device used for the experimental researches.

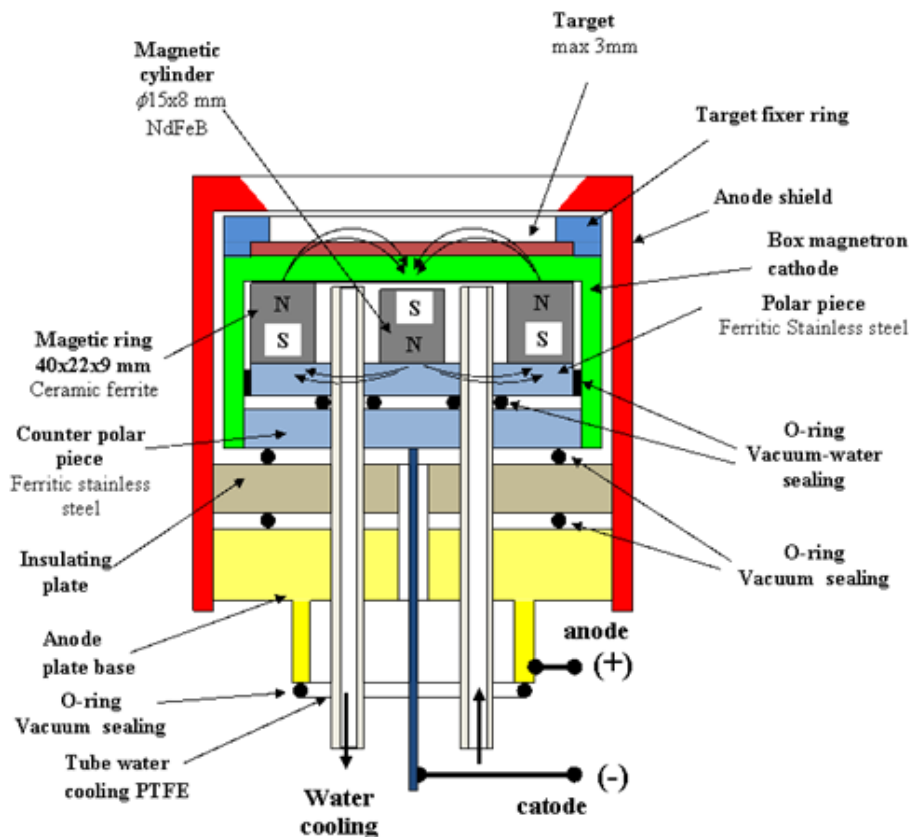


Fig. 1. Magnetron scheme

The microscopic analysis of the obtained films was performed using a Neophot 2 optical microscope with the acquisition of computerized data.

Transparency of films was determined using an electronic device that uses a light source and a photoreceptor. The light after passing through the film is measured with the photoreceptor and an amplifier and the result is displayed by means of an analogue device.

The electrical properties (resistivity) of the films were determined using the four-point collinear probe method using a laboratory device. As a principle, this is to inject the current through two external points and measure the voltage at two internal points.

With thin films, resistivity is calculated by the relation:

$$\rho = \frac{\pi \cdot t}{\ln 2} \left(\frac{U}{I} \right)$$

where: t – layer thickness, U – measured voltage, I – applied current.

$$\frac{\rho}{t} = \frac{\pi}{\ln 2} \left(\frac{U}{I} \right) - \text{surface resistance of the film.}$$

The stages of obtaining the deposits were:
 a. preparation of substrate surface
 b. film deposition by DC magnetron sputtering process

a. Preparation of substrate surface

This step consisted of washing the glass plates (size 76x25x1 mm) with a special detergent, washing them with water, then with distilled water, ultrasonic cleaning with ethanol and then drying with compressed air.

b. Film deposition by DC magnetron sputtering process.

To obtain the films, a circular austenitic stainless-steel plate was used as target, the chemical composition of which, revealed by spectral analysis, is shown in Table 1.

A series of regimes shown in Table 2 were used to obtain the films.

Table 1. Chemical composition of the austenitic stainless steel X10CrNi 18-8 used as target

Chemical composition [%]										
Mo	C	Si	Mn	S	P	Cr	Ni	Al	V	Ti
0.30	0.046	0.28	1.34	0.005	0.004	21.45	7.58	0.01	0.07	0.05

Table 2. Working regimes used in film deposition

Sample code	Voltage [V]	Current [mA]	Pressure [mbar]	Substrate temperature [°C]	Target – substrate distance [mm]	Deposition time [min]
P1	550	55	2.5×10^{-2}	35	66	10
P2	540	55	2.5×10^{-2}	35	66	20
P3	545	55	2.5×10^{-2}	34	66	40
P4	550	55	2.5×10^{-2}	34	66	40
P5	550	55	2.5×10^{-2}	34	66	20

3. Experimental results

The optic microscopy analysis revealed that the films obtained did not show cracks, were homogeneous and adherent as shown in Fig. 1.

The analysis of the electron microscope revealed the structure of the deposited film and its chemical composition by the EDS method, Fig. 2 and 3.

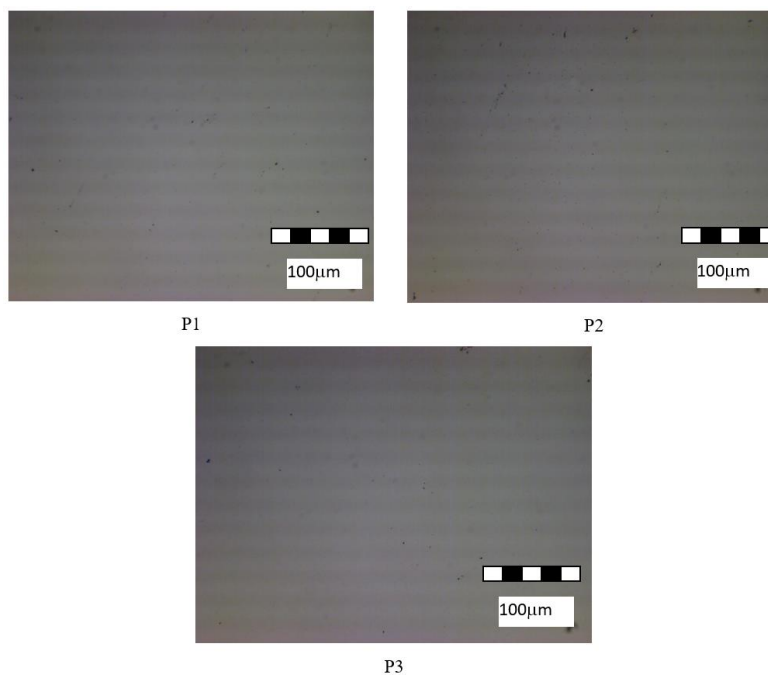


Fig. 1. Image of the films deposited under different regimes

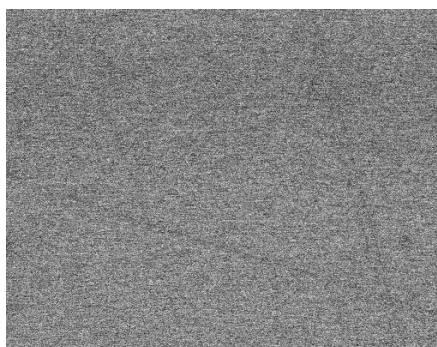


Fig. 2. SEM image of the austenitic stainless-steel film

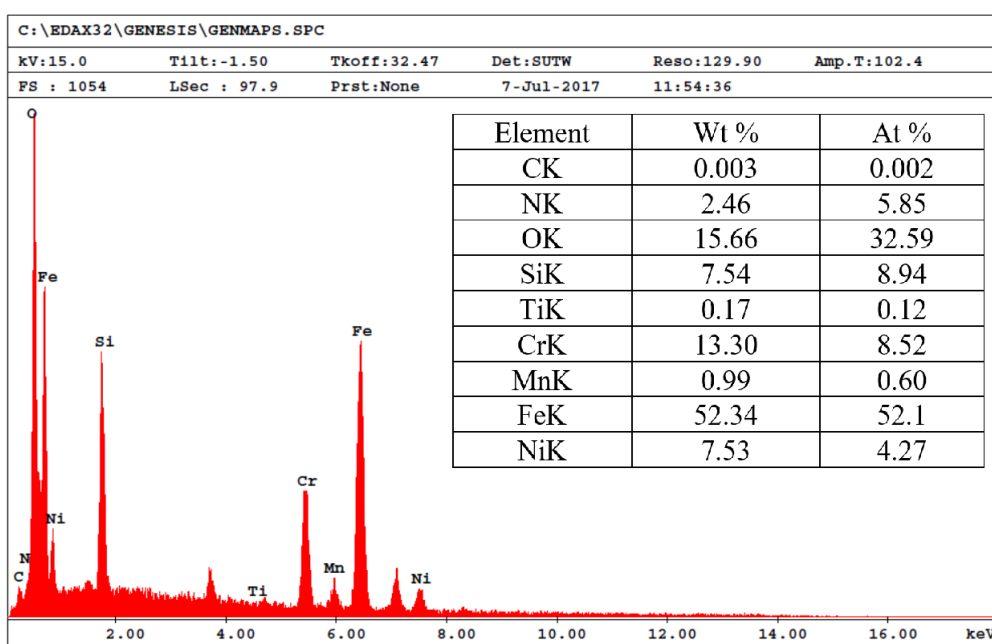


Fig. 3. EDS analysis of the austenitic stainless-steel film

The results of the transparency and electrical resistance measurements for samples P1, P2, P3 are shown in Table 3. The P4 and P5 samples were deposited on the support with copper base.

Analyzing Table 3, it can be observed that when increasing the deposition time, surface resistance and

transparency decrease. This behavior is due partly to the increase in the thickness of the deposited film and, partly, to its oxidation. These aspects can also be seen on the graphs in Figures 4 and 5.

Table 3. Determining transparency and electrical properties of films

Sample cod	Film transparency	Film surface resistance [Ω]
P1	0.281	404.9
P2	$68 \cdot 10^{-4}$	214.5
P3	$12 \cdot 10^{-5}$	190.9

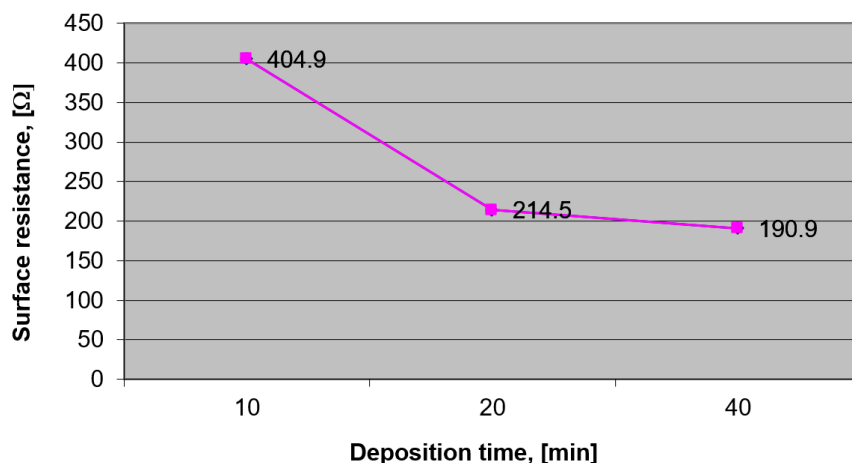


Fig. 4. The influence of the deposition time on the surface resistance of the film deposited

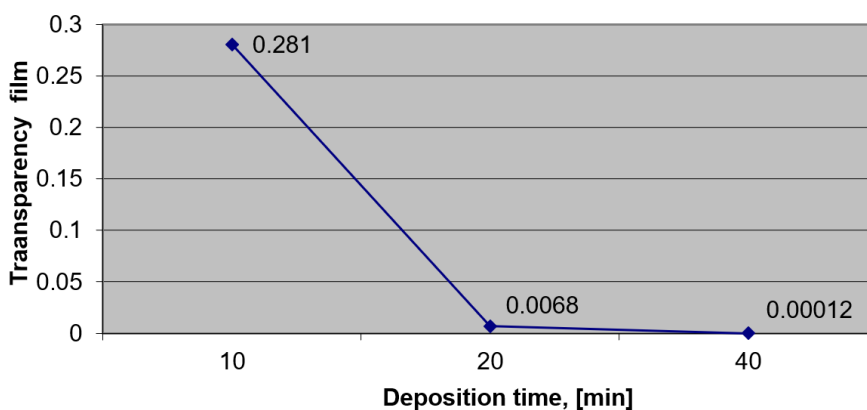


Fig. 5. The influence of the deposition time on the transparency of the film deposited

3. Conclusions

The experimental researches lead to the following conclusions:

- films deposited by DC magnetron sputtering process have highlighted an economical and simple way to synthesize several austenitic stainless-steel films.

- the films were obtained using as an atmosphere for the maintenance of plasma the rarefied argon in a pressure range between $3 \cdot 10^{-2}$ - $8 \cdot 10^{-3}$ mbar; the argon flow rate used was $100 \text{ cm}^3/\text{min}$.

- the structures and properties of the deposited films obtained depend on the parameters used.

- the microscopic analysis reveals that the films obtained do not show cracks, are uniform and adherent with mirror surface.

- when increasing the deposition time, the surface resistance and the transparency decrease the values obtained being in the range $404.9 - 190.9 \text{ } \Omega$ and $0.281 - 12 \cdot 10^{-5}$ respectively (this behaviour is

due on the one hand to an increased deposited film thickness, and on the other hand to oxidation their).

- the EDS analysis revealed the presence of alloying elements specific to austenitic stainless steel (Cr, Ni, Mn) which implies the achievement of the proposed objective.

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