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Choice of a Substrate for Pd_xNi_{1-x} Thin Films used as Hydrogen Sensors

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Abstract: In this work we studied the effect of the order at the interface of a $Pd_{20}Ni_{80}$ thin film deposited on different substrates on the electric resistance (R) thin film. We analyzed the fluctuations of the electric resistance (δR) of $Pd_{20}Ni_{80}$ thin film after 1000 measurements. The relative error of the electric resistance (δR/R) of a $Pd_{20}Ni_{80}$ thin film is minimal when the surface of the substrate is ordered, obtained with an α-alumina single crystal substrate, while this ratio presents a strong drift in the absence of order at the interface when the substrate used is an amorphous silica glass. After 1000 measurements at strong current (I = 50 mA), the mean relative error of the electric resistance of the $Ni_{80}Pd_{20}$ films deposited on an α-alumina single crystal is 33 times weaker than the relative error of the electric resistance obtained for the same film deposited on silica glass.

Keywords: Resistivity, interface-order, $Pd_{20}Ni_{80}$ thin film, specular-interface.

1 Introduction

In the context of an increase in production and general public use of hydrogen, the problem of safety is an essential parameter.

The present study aims at the development of thin film alloys Pd_xNi_{1-x} thin for hydrogen sensor.

The Pd_xNi_{1-x} alloys have the characteristic to be stainless and can function in a broad range of temperatures and hydrogen pressures [1, 2]. They have a low solubility for the hydrogen which one can control by the composition of the alloy. The sensors can be used in the form of thin films or multi-layer deposited on an insulating substrate (α-alumina, glass).

The functionality of these sensors depends on the physicochemical modifications in bulk. The reactivity of thin films is followed by the modification of the electric properties produced by the controlled bulk insertion of atomic hydrogen.

The electrical resistivity of multi-component metallic systems is extremely sensitive to the microscopic arrangement of the various atomic species on the crystal lattice [4, 5]. To vary the rate of order at the interface thin film/substrate we worked on three insulating substrates: an α-alumina single crystal, an α-alumina polycrystal and glass of silica. The objective being the choice of the substrate presenting the best electric response of the $Pd_{20}Ni_{80}$ thin film.

2 Preparation of the Samples

The $Pd_{20}Ni_{80}$ alloy was elaborated under vacuum in an induction furnace.

The $Pd_{20}Ni_{80}$ thin films are prepared by evaporation of the $Pd_{20}Ni_{80}$ alloy in vacuum under electron beam.

Thickness measurements are obtained using an interference microscope. The thickness of films is, w = 220 nm.

3 Measurements of the Electrical Resistivity of a Thin Film by Four Points Probes Technique (Fig.1)

In this method one imposes a sweeping of D.C. current (I) via both external probes (Fig-1) and one measures the potential drop between the two internal probes (ΔV), the electric resistance is deduced: R = ΔV/I. In this study we used a power source (KEITHLEY-6220) to supply a D.C. current to the two external probes and a nanovoltmeter

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(KEITHLEY-2182A) to measure the voltage between the two internal probes.

The four aligned probes being separated from the same distance s. The resistivity of film is given by the relation of Smit [3], ρ = R*w*C(s, d), w being the thin film thickness of and d the diameter of the film, C (s, d) the geometrical correction factor.

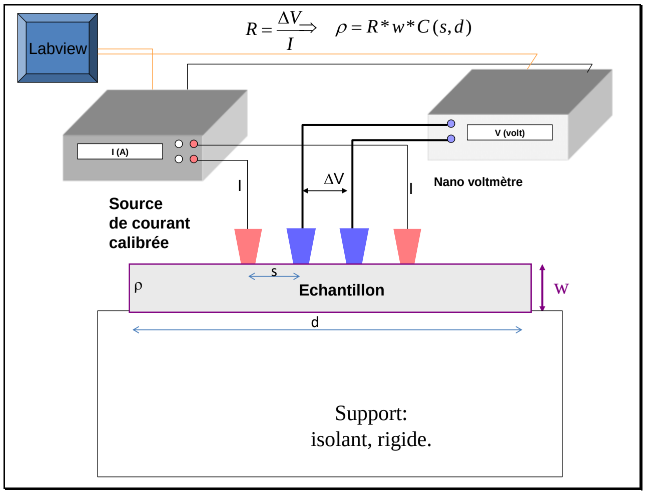

Fig-1: Principle of measurement of the resistivity (ρ) by the method of the four points probes.

4 Measurements of electric resistance according to the time of application of the current, Fig.-2.

Using the delta mode (Fig-2), we could study the evolution of electric resistance R(t) of a thin film according to the current on the one hand and time of application of the current on the other hand. For this delta mode one sends a periodic current and one measures tension at the points A, B, C, D…, one thus carries out with this procedure 1000 measurements. The measurement of tension over the first period is:

In order to establish a criterion associating at the same time the average value of electric resistance ($\overline{R}$) and the drift compared to average resistance during the time of application of the current we defined a parameter of experimental test, $K_i(t)$, representing the absolute relative variation of the electric resistance of films according to time. Fig-3 and defined by:

$$1st \quad Delta \quad reading := \left(\frac{A-2B+C}{4}\right).(-1)^0$$

$$K_i(t) = \sqrt{\left(\frac{R(t)-\overline{R}}{\overline{R}}\right)^2} = \sqrt{\left(\frac{\delta R}{\overline{R}}\right)^2}$$

$\overline{R}$ is the average resistance on the interval of time of measurement and R(t) the instantaneous resistance.

The chart of $K_i(t)$, (Fig-3), enabled us to determine the average threshold of electric output from which thermal agitation at strong current (2 mA < I ≤ 50 mA) in $Pd_{20}Ni_{80}$ thin film by comparison with the electronic statistical noise with weak current becomes the prevalent factor of the growth of electric resistance. On the figure-3 one can see that the transition between the two fields from variations from $K_i(t)$ is produced for a current in the interval: ± 200 µA < I < ± 2 mA.

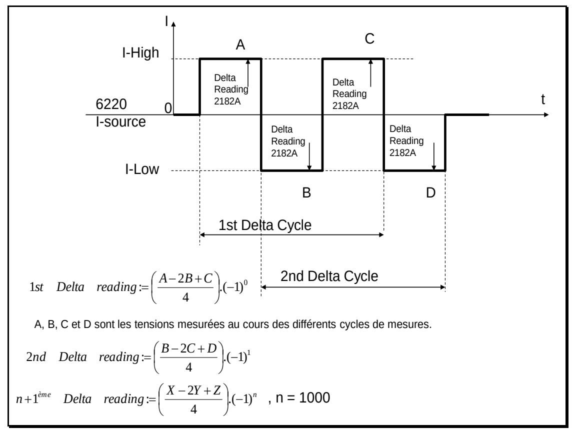

Fig. - 2: Principle of a cycle of 1000 measurements in delta mode.

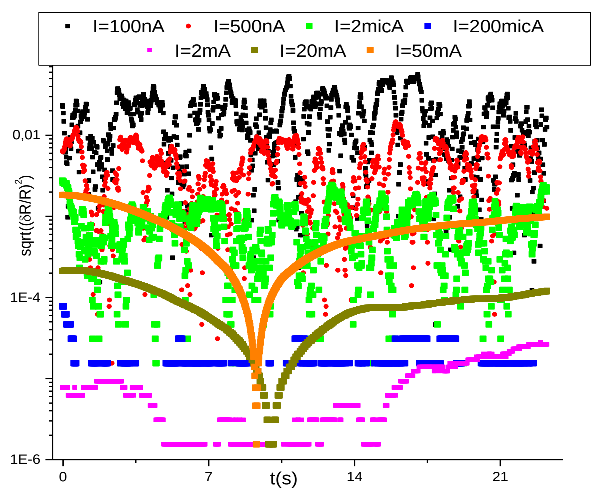

Fig-3: Chart of the parameter $K_i(t)$ according to the time and the current for thin film $Ni_{80}Pd_{20}$ deposited on glass substrate.

When the applied current varies in the interval [100 nA, 50 mA], the parameter Ki(t) decreases from the maximum value $K_{max} = 10^{-2}$ obtained with weak current: I = ± 100 nA to the minimum value $K_{min} = 10^{-6}$ obtained for a current 200 µA < I < 2 mA. The threshold of electric output for the activation of the thermal disorder in analyzed films is evaluated to 100 ± 50 μW. The value of this threshold depends on the resistance of film and resistance of the interface film/substrate.

5 Effects of the order distances at the interface film/substrate on the variation of the parameter Ki(t), Fi-4.

This part is devoted to the study of the effect of the substrate and more precisely of the contribution of the atomic order at the interface film/substrate on the evolution of the parameter Ki(t) determined at the strong electrical current (50 mA) in the $Ni_{80}Pd_{20}$ films. For that three types of substrates were considered: an α-alumina single crystal of orientation [001], an α-alumina polycrystal characterized by an average mean grain size equal to 0.7 μm and a null porosity on the one hand and amorphous silica glass on the other hand. The comparison of the parameters $K_1(t)_{Glass}$, $K_2(t)_{Single\ crystal}$ and $K_3(t)_{Polycrystal}$l, Fig.-4, given with strong electrical currents (50 mA) on the $Ni_{80}Pd_{20}$ films deposited respectively on glass on the α-alumina single crystal and the α-alumina polycrystal, show that the average value of the parameter $K_1(t)_{Glass}$ obtained on the $Ni_{80}Pd_{20}$ films deposited on a glass substrate is 33 times more important than the parameter $K_2(T)_{Single\ crystal}$ determined on the $Ni_{80}Pd_{20}$ films having as substrate an α-alumina single crystal. The parameter $K_3(t)_{Polycrystal}$ caracterizing the $Ni_{80}Pd_{20}$ films deposited on an α-alumina polycrystal is approximately 3 times the value of $K_2(t)_{Single\ crystal}$.

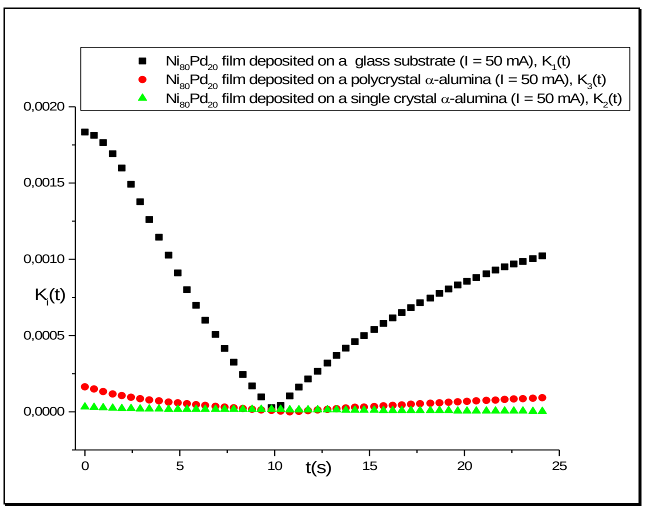

Fig- 4: Chart of the parameters $K_1(t)_{Glass}$, $K_2(t)_{Single\ crystal}$ and $K_3(t)_{Polycrystal}$ obtained on the $Ni_{80}Pd_{20}$ films deposited on the various substrates.

6 Conclusion

Because of the atomic order at short distance existing on the surface of the alumina single crystal, the interface of the film $Ni_{80}Pd_{20}$/alumina presents a more specular character by comparison with the $Ni_{80}Pd_{20}$/glass interface where the chemical disorder associated with the structural disorder of the surface of glass is at the origin of the diffuse character of this $Ni_{80}Pd_{20}$/glass interface. The order at short distances on the surface of the single-crystal substrate of alumina leads to a greater rate of probability of transmission of electrons at long distances in the $Ni_{80}Pd_{20}$ films and makes it possible to explain the weak variations of the parameter $K_2(t)$, at strong current if the films are deposited on a single crystal alumina.

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