On the thickness measurement of metallic thin films

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ABSTRACT

Thickness is a key factor on the physical properties of the new miniaturized technology of thin film devices. Thin film physical properties depend on the thickness value and its control. This paper highlights the importance of the accurate determination of the thickness in thin films. Many efforts have been made through the years to develop reliable thickness measurement techniques. In this work, some of these techniques implemented to measure the thickness are briefly discussed. A theoretical model for film thickness determination was discussed. We proposed a simple method based on the variations of electrical resistance of a probe film to measure and control *in situ* the metallic film thickness with good resolution. Gold films were deposited by free evaporation and sputtering in order to apply the proposed method to measure and control the film thickness.

Keywords: *film thickness, electrical resistance, metallic thin films.*

Sobre la medición del espesor en películas delgadas metálicas

RESUMEN

El espesor es un factor clave sobre las propiedades físicas en la nueva tecnología miniaturizada de los dispositivos de película delgada. Las propiedades físicas de las películas delgadas dependen del espesor y de su control. Este artículo resalta la importancia de conocer con gran aproximación el valor del espesor de películas delgadas. Se han realizado muchos esfuerzos a lo largo del tiempo para desarrollar técnicas de medición del espesor para obtener un valor confiable. En este trabajo se discuten en forma breve, algunas técnicas implementadas para medir el espesor y se propone una técnica simple basada en las variaciones de resistencia eléctrica para medir y controlar *in situ* el espesor de películas delgadas metálicas con buena resolución. Se depositaron capas delgadas de oro con las técnicas de evaporación libre y sputtering, con el objetivo de aplicar el método propuesto para medir y controlar el espesor de las películas depositadas.

Palabras clave: *espesor de películas, resistencia eléctrica, películas delgadas metálicas*

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INTRODUCTION

Due to the relation of film thickness with the physical properties, film thickness is a very important parameter in several applications of thin films, such as the production of very large scale integrated (VLSI) devices. During the last two decades, device feature size has decreased from 1 µm to 90 nm, while the microprocessors velocity increased the working frequency from 66 MHz to 4 GHz [Dan, 2004; Shamiryan, 2004]. However, the metal interconnections between elements on the integrated circuits can reduce this capacity causing delay on the response. The electrical resistivity of metallic materials is one of the limiting parameters causing the mentioned delay. Aluminium and aluminium alloys are typical materials used for interconnections, but recently copper has gained use due to its lower resistivity (36% less than Al, and 6% higher than Ag), and its cheaper cost [Shamiryan, 2004].

Knowing the film thickness and its associated properties is useful to control and evaluate its technological application. However, film thickness depends not only on the preparation technique, but also on the technique used for measurement.

There exist two general methods to measure the film thickness value [Echertová, 1986]: during films preparation (*in situ* method) and after films preparation (*ex-situ* method). The first method is the most appropriate method for controlling film properties but usually it is more expensive than the second one due to the equipment used and its installation into the deposition chamber. Complex techniques have been developed for *in-situ* thickness determination such as interferometry, quartz balance, and vibrating quartz crystal. The latter is the most used technique given the high sensitivity and reliability obtained for thickness determination.

Ex-situ methods have the advantages of simplicity and lower cost, but they have several inconveniences related to the control of the thickness. Therefore, depending on the range of film thicknesses, researchers and industrials use profilemeters (such as talysteps), atomic force microscopy, x-ray photoelectron spectroscopy, and micrometers, among others, for *ex-situ* thickness determination.

But, how can film thickness be defined? Normally, film thickness is defined depending on its property or quality to be measured. Thus, if we measure the step formed between a film and its substrate, the measured thickness will depend on the deposition rate, the surface roughness, and the deposition technique; however, if we measure the mass deposited on a surface by a change of frequency on a quartz crystal; then, we will measure the mean thickness value assuming that the material is symmetrically distributed on the substrate. Nowadays, the real film thickness definition is not clear yet and depends strongly on the technique used for measurement.

On microelectronic applications, metallic film thickness plays a crucial role on their physical properties. The improvement of electronic conduction is one of the most important goals of the integrated circuits, together with the atmospheric and chemical stability.

In this work, the implementation of an *in situ* method to control and measure the metallic film thickness by means of variations of the electrical resistance in a probe film is described. The feasibility to use this method with different metals and their combinations by means of a theoretical analysis developed for pure metals is demonstrated. Gold/glass films were deposited by thermal evaporation and sputtering techniques for experimental purposes, and their thicknesses estimated by this method were compared with the thicknesses measured with a profilemeter.

THEORY

The electrical resistance of a metallic material that obeys the Ohm' s law is given by the relation:

$$R=\rho L/tw \qquad (1)$$

Here, L is the sample length, w the width and t the thickness. The electrical resistivity, ρ, of a material is an intrinsic property that usually does not depend on its geometrical characteristics. However, for film thicknesses below certain critical value, the resistivity increases and presents a strong dependence on thickness [Weller, 2001]. In this work, we will assume that the value of ρ remains constant at the bulk value for the range of thicknesses studied. Therefore, the expression for the thickness can be written from equation (1) as $t=\rho L/Rw$. Defining $M=L/w$, as the slender relation, then, the equation for thickness can be re-written as

$$t=\rho M/R \qquad (2)$$

Our proposed method for thickness measurement requires two components: a very thin probe-film with four electrodes which is used as reference, and a clean substrate where the new film will be deposited. These components are placed together in a twin sample-

holder into the vacuum chamber such that they receive the evaporated material in similar conditions. Thus, the thickness of the new film can be estimated from the changes of resistance measured in the probe-film. Two cases will be discussed: a) when the evaporated material is the same material as that of the probe-film and, b) when the evaporated material is quite different to that of the probe-film.

a) Same metallic films

Initially, we deposited a very thin metallic film (30 nm, for example) to be used as a film of reference (probe film). A first approximation of the proposed method assumed that the material to be deposited is the same material as that of the probe film. By measuring the electrical resistance of the probe-film before deposition (R_i) and after deposition (R_f), the new thickness of the deposited film can be associated with the change of resistance of the probe-film. From equation (2), the probe-film thickness before deposition can be calculated by $t_i=\rho M/R_i$; and after deposition, the total probe-film thickness is given by $t_f=\rho M/R_f$. Thus, the change of thickness as a function of the electrical resistance is given by:

$$\Delta t = t_f - t_i = \frac{\rho M}{R_f} - \frac{\rho M}{R_i} = \rho M\left[\frac{1}{R_f} - \frac{1}{R_i}\right] = \rho M\left[\frac{R_i - R_f}{R_i R_f}\right] \quad (3)$$

As equation (3) shows, the electrical resistance diminishes when the film thickness increases. Assuming that the slender relation M and ρ are constant (for technical implementation), the simultaneous deposition of two films under same conditions is needed: one on the probe-film and the other on the clean substrate. Then, a twin-sample holder for films deposition is required. Thus, changes on the electrical resistance of the probe-film permit us to determine the thickness of the twin film deposited on the clean substrate, under same conditions.

By plotting equation (3), a limitation of the probe-film geometry can be observed. Figure 1 shows the theoretical behavior between the electrical resistance and the gold film thickness, calculated for three values of the slender relation M. If very thin films thicknesses are to be deposited, the use of probe-films with small M values is suggested; contrarily, if we need to deposit thick films, the recommendation is to use probe-films with higher M values to appreciate well the changes of electrical resistance. However, better results can be achieved for very small thicknesses of the probe films, since they will provide larger changes on the electrical resistance. A recent study [Camacho y Oliva, 2004] has demonstrated that after 15 nm-thicknesses of Au films the electrical resistivity reaches the bulk value. Then, 15 nm is suggested as the minimum thickness for gold probe film.

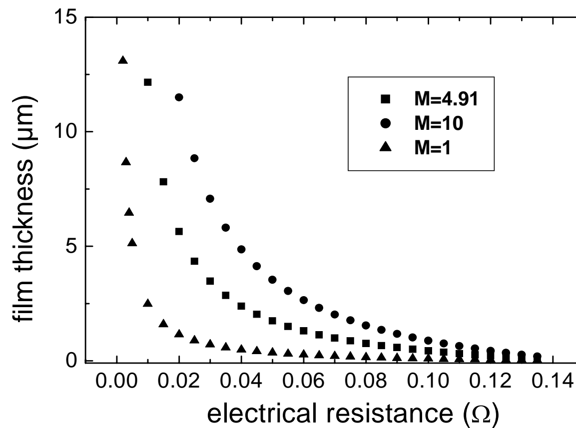


Figure 1. Gold films resistance calculated as a function of different film thicknesses for three different slender relations M (obtained from equation 3).

b) Different metallic films

By using the proposed method it is also possible to estimate the thickness when two different metallic films are deposited, forming a bilayer. This can be useful to develop thin films alloys for different applications [Mesa-Laguna, et al., 2005]. Following a treatment similar to the previous one, and assuming that a bilayer is formed, the equivalent shunt

resistance R_e, in terms of the slender relation M and the electrical resistivity of the pure metallic films, is given by:

$$R_e = \frac{M\rho_1\rho_2}{t_2\rho_1 + t_1\rho_2} \quad (4)$$

where subscripts 1 and 2 represent the probe-film and the new deposited film, respectively.

If both films are deposited with the same geometry, equation (3) can be re-written as:

$$\frac{t_T}{\rho_T} = \frac{t_1}{\rho_1} + \frac{t_2}{\rho_2} \quad (5)$$

where, ρ_T is given by:

$$\rho_T = \frac{t_T\rho_1\rho_2}{t_1\rho_2 + t_2\rho_1} \quad (6)$$

Although the expression of the equivalent resistivity ρ_T may not have a real significance, its definition results useful for our model.

Thus, it is possible to obtain a film thickness measurement when different metallic films are deposited. However, the measured electrical resistance needs to be within a useful range. Because of this, it is necessary to determine the adequate range to assure high resolution during the electrical resistance measurements.

Now, similar to the previous case, we will use equation (3) to calculate the thickness value when different metallic films are deposited, this is,

$$t_2 = t_T - t_1 = \frac{M\rho_T}{R_T} - \frac{M\rho_1}{R_1} = \frac{M}{R_T}\left[\frac{t_T\rho_1\rho_2}{t_1\rho_2 + t_2\rho_1}\right] - \frac{M\rho_1}{R_1} \quad (7)$$

where

$$t_2 = \rho_1 M\left[\frac{t_T\rho_2}{(t_1\rho_2 + t_2\rho_1)R_T} - \frac{1}{R_1}\right] \quad (8)$$

The product of the final electrical resistance (R_T) and the deposited film thickness in equation (7), can be obtained as:

$$R_T t_2 = \frac{M\rho_2\rho_1(t_1 + t_2)R_1}{(t_1\rho_2 + t_2\rho_1)(R_1 t_2 + M\rho_1)} \quad (9)$$

Rewriting equation (9) as a function of film thickness t_2, we obtained the following quadratic equation:

$$t_2^2 + t_2\left(t_1\frac{\rho_2}{\rho_1} - M\frac{\rho_2}{R_T} + M\frac{\rho_1}{R_1}\right) + \frac{M}{R_T}t_1\rho_2\left(\frac{R_T}{R_1} - 1\right) = 0 \quad (10)$$

by solving equation (10) for t_2, we obtain two possible solutions [López-Garduza, 2004]:

$$t_{a,b} = \frac{-\left[t_1\frac{\rho_2}{\rho_1} - M\frac{\rho_2}{R_T} + M\frac{\rho_1}{R_1}\right] \pm \sqrt{\left(t_1\frac{\rho_2}{\rho_1} - M\frac{\rho_2}{R_T} + M\frac{\rho_1}{R_1}\right)^2 - 4\frac{M}{R_T}t_1\rho_2\left(\frac{R_T}{R_1} - 1\right)}}{2} \quad (11)$$

The predicted behaviour of films thickness for different combinations of metallic films can be observed by plotting equation (11) as a function of the electrical resistance. Figure 2 shows these results for different Al, Au and Cu film bilayers with M=0.78. Note the differences amongst the film thicknesses and related electrical resistance depending on the type of metals used as probe-film. In this figure, Al-Cu means that Al (new film) is deposited onto Cu (probe-film). Individual resistivity of each metallic layer was taken at room temperature (RT). No experimental results for bilayers will be

presented in this work. We will only discuss experiments with films deposited on probe films of the same material.

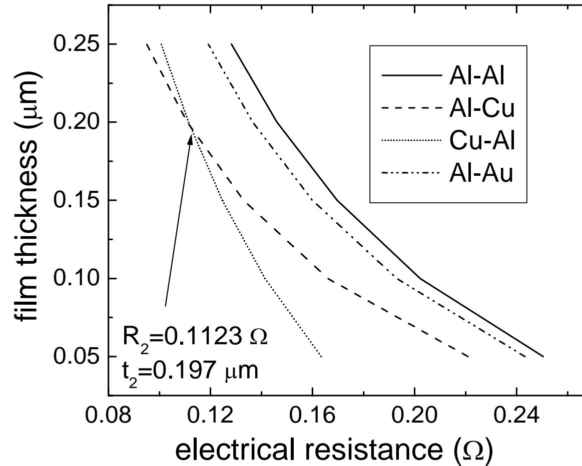


Figure 2. Behavior of the electrical resistance with film thickness for different combinations of metallic films obtained from equation (11).

Equation (11) can be used to calculate the new thickness of deposited films when probe films of different materials are used. By taking the + sign, equation (11) reduces to equation (3) if we assume that the two deposited metals are identical. The negative sign yields a negative thickness which does not have physical significance.

EXPERIMENTAL SETUP

Usage of thin films as probe films is required by the process. The probe film is placed along with the new substrate on a twin-sample-holder. Gold thin films (5x20 mm^2) with different thicknesses were deposited on Corning glass 7059 substrate by free evaporation and sputtering techniques into a vacuum chamber (~10^{-6} Torr). High purity gold (Cerac, 99.999%) was used as source material for thermal evaporation. Ar gas for plasma production and a high purity gold target (5 cm, diameter) were used for the sputtering deposition. The target-substrate distance was 5 cm and Ar+ pressure was maintained at 10^{-4} Torr after gas flow. Figure 3 shows the twin sample-holder used to deposit the gold sputtered films. Target is placed above sample-holder such that plasma was produced into their distance gap. Substrates were cleaned according to a standard process [López-Garduza, 2004]. Film thicknesses were verified after deposition with a Dektak 3030 profilemeter. The deposition rate varied between 0.01 nm/s and 0.4 nm/s and film thickness ranged from 29 to 1200 nm. In order to apply the collinear four-probe technique through the film for electrical resistance measurement, four electrodes of AWG 36 copper-wire were glued to the film with conductive silver paint. A programmable HP-6643A power supply provided electrical current through the external electrodes and a programmable voltmeter HP-3458A measured the voltage through the internal electrodes. Initially, a small current is applied by the external electrodes to obtain thermal stabilization and determine the initial resistance R_i value. Under this condition, the deposition initiates and the changes of resistance are measured over time. The temperature on the substrate surface was simultaneously measured with high resolution by means of a small-mass Omega K-type thermocouple and captured in real time through a GPIB interface. Temperature is a key parameter in the resistance determination, in order to correct data for reliable information. Home-made software developed in HP-VEE language was used to control and acquire data in real-time to the PC. Knowledge of the material type, the changes of electrical resistance, and the evolution of the substrate temperature, allow us to estimate the film thickness according to Eqs. (3) and (11).

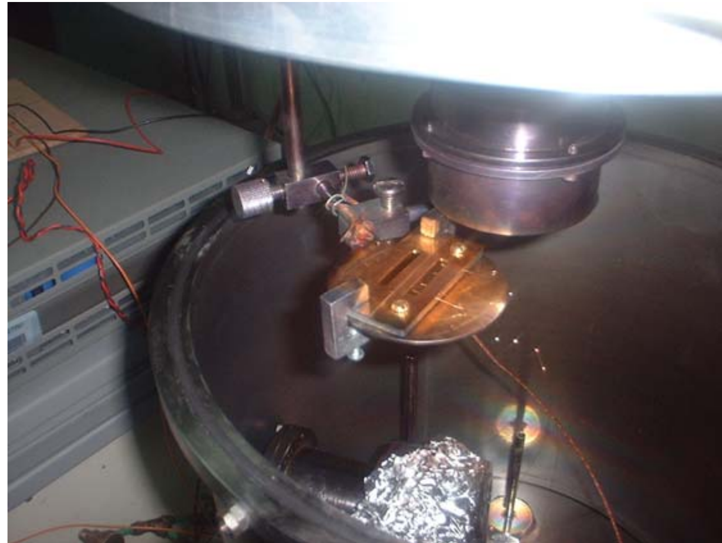
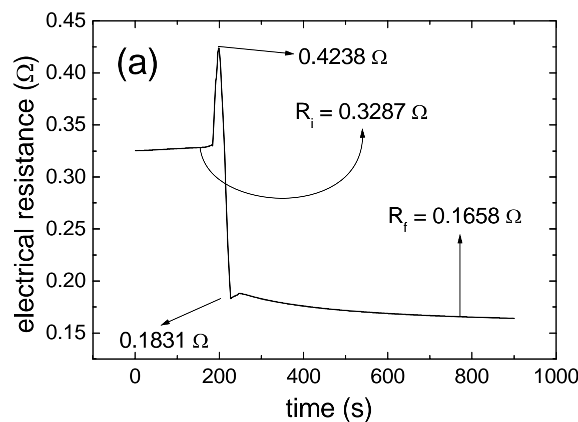


Figure 3. Details of the sputtering vacuum chamber where the twin sample-holder and the mask are observed. The gold-target is above the sample holder. Electrical wires and a thermocouple can be observed at the right side of the probe-film.

RESULTS

Starting from a thin gold metallic film (170 nm as initial thickness, estimated from the initial value of resistance and the area) as a probe-film, we can deposit another metallic film over the previous one, such that the electrical resistance on the probe film shows variations with thickness. Homogeneity was observed on the deposited films given that the target size and the solid-angle of evaporation are higher than the size of the film. Figure 4 shows the temporal variations of the electrical resistance observed when a gold film is deposited by thermal evaporation for the first time over another gold film. In this case we used M=1.98 as the slender relation. In Figure 4a, four important changes in the probe-film resistance are observed: i) the steady state of the electrical resistance before deposition, ii) a sudden increase due to the received heat from the electrical crucible, iii) an abrupt decrease due to the gold film deposition, and, iv) the steady state reached after film deposition and cooling. The slope of the abrupt decrement of the resistance over time during film deposition is used to estimate the deposition rate. The film thickness and the deposition rate in Figure 4a were estimated as 157 nm and 2.21 nm/s, respectively.



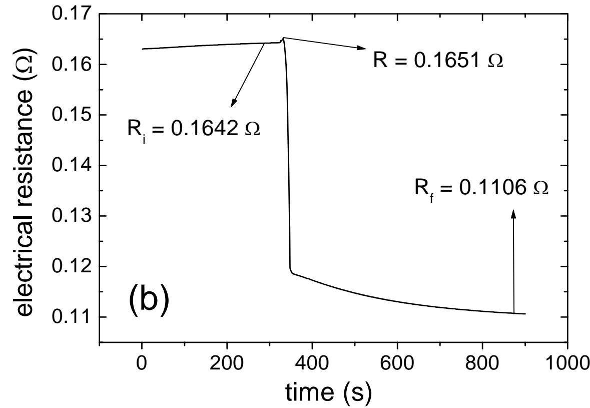


Figure 4. a) First and b) second deposition of gold film over a probe film. Variations of the electrical resistance were used for film thickness determination.

Figure 4b, shows the resistance behaviour when a second film is deposited on the same probe-film. Here, the probe-film did not suffer heating due to high deposition rate as compared with the first deposition. The initial and the final electrical resistance values are included in Figure 4. These values were used to estimate the film thickness deposited (155 nm) according to equations (3) and (4), and the deposition rate (5.34 nm/s).

Using this probe-film it was possible to deposit seven films of different thicknesses (forming a total thickness of 1007 nm). However, thermal evaporation technique results a quite fast deposition technique for controlling the rate of film deposition. Thus, a new deposition technique with slower deposition rates is required.

A second experiment was performed on a new probe-film by using the sputtering technique. This technique permits slower deposition rates and a better control of thickness. Figure 5 shows the sequential behavior of the resistance of six different gold films by using a new probe-film with M=2.02 and ρ_{bulk}=2.35x10^{-8} Ω-m (at RT). Initial thickness of the probe-film was 64.5 nm, estimated from the initial resistance.

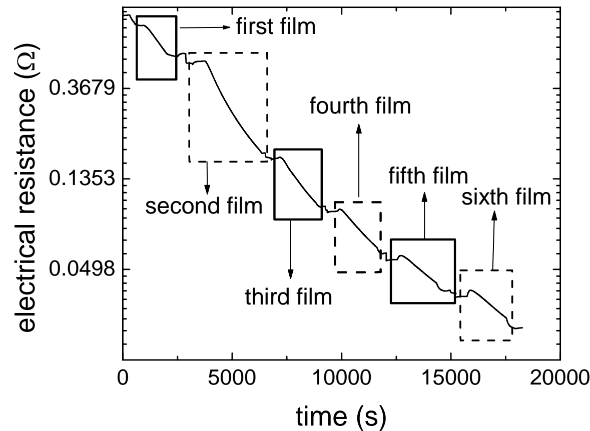


Figure 5. Variations of resistance over time of six gold films deposited with the same probe-film by the sputtering technique.

Table I summarizes the thicknesses obtained for the six deposited films, the thicknesses measured by the Dektak 3030 profilemeter (for comparison), and the estimated deposition rates. Different sputtering powers were used in order to obtain different thicknesses. The uncertainties increase with thickness and deposition rate due to minor values of resistance measured at higher thicknesses. Minor differences

can be observed on the reported values by the two methods. Interpretation of the thickness significance will depend on the method used for measurement. Although in the electrical resistance method, thickness is the result of the global effect of all the material deposited between electrodes, in the profilemeter, thickness is the mean value obtained from various steps measured along the film borders. Normally, the thickness is not uniformly distributed in the substrate and, consequently, differences on thickness values may be reported.

In order to deposit only one film with a large thickness in only one step, we prepared a third experiment to obtain a film thickness near to 1 micrometer. Figure 6 shows the results of the changes of electrical resistance measured when 60 W power-sputtering was used. A nonlinear behaviour of the electrical resistance over time is clearly observed. Finally, we obtained a film thickness of 1.14 ± 0.04 μm after 2.2 hours of deposition at a mean deposition rate of 0.175 ± 0.006 nm/s. In this case, the probe film was used only once.

Table I. Electrical resistances, film thicknesses, and deposition rates calculated for the six gold films deposited with different sputtering- powers.

film number	sputtering power (W)	R_i [Ω]	R_f [Ω]	ΔR [Ω]	thickness t (this work) [nm]	deposition rate (nm/s)	thickness (profilemeter) (nm)
1	30	0.7360	0.5231	0.2129	29 ± 2	0.025 ± 0.002	30
2	40	0.4961	0.1797	0.3164	180 ± 10	0.010 ± 0.001	200
3	50	0.1700	0.0990	0.0710	220 ± 12	0.122 ± 0.007	300
4	60	0.0946	0.0631	0.0315	334 ± 15	0.185 ± 0.008	510
5	70	0.0554	0.0391	0.0163	400 ± 18	0.222 ± 0.010	580
6	80	0.0369	0.0259	0.0110	611 ± 25	0.339 ± 0.014	410-580

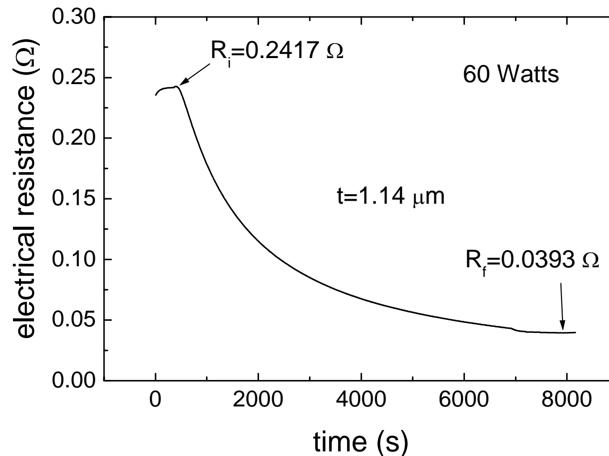


Figure 6. Behaviour of the electrical resistance for a thick gold film deposited by the sputtering technique.

CONCLUSIONS

An *in situ* method for metallic film thickness determination by measuring the variations of the electrical resistance was proposed. Experimental results were discussed for gold films deposited by thermal evaporation and by sputtering techniques under different conditions. The proposed method only requires an initial probe-film deposited with small thickness (some tenths of nanometres) which include four collinear electrodes to measure the variations of the electrical resistance. A theoretical model was proposed to determine the behaviour of the film thickness when same and different materials

are deposited on the probe film. Acceptable agreement between the estimated thickness by this method and the thickness measured with a profilemeter, was obtained. A range of thicknesses (from tenths of nanometres to microns) can be measured and controlled with this method. However, it is very important to take into account that electrical resistivities in metals present higher values for thicknesses of less than 10-20 nm. For such thin films, an adequate interpretation may need to consider quantum effects. A better control in the deposition rate with the sputtering technique was found, as compared with the evaporation technique, given the most controllable rate of film growth.

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