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FABRICATION AND CHARACTERISATION OF THIN FILM COAXIAL AC/DC RESISTORS FOR THE DETREMINATION OF R_K

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Abstract: This paper describes the fabrication and the characterization of ultra thin films of NiCr deposited on cylindrical substrates of ceramic aiming to get coaxial ac-dc resistance standards. The layers are obtained by magnetron sputtering technique and their structural characterization are carried out by using Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and Atomic Force Microscopy (AFM). A first set of resistive sticks has been obtained with a good homogeneity of thickness. The layer thickness varies from 8 nm to 70 nm and correspond to a wide range of resistance values, from 1 kΩ to 100 kΩ.

Keywords: Impedance, AC measurements, calculable resistance, thin films.

1. INTRODUCTION

To contribute to the future International System of units (SI) based on fundamental constants, LNE is involved in the development of experiments leading to a best experimental knowledge of two quantum phenomena which are in use in electrical metrology: the first one is the Josephson effect, which is observed in superconducting tunnel junctions and relates voltage and microwave frequency through a constant K_J (Josephson constant); theoretically, $K_J = 2e/h$, where e is the electron charge and h is the Planck's constant. The other quantum standard is the quantum Hall effect (QHE), which is observed in semiconducting heterostructures at low temperatures and high magnetic fields. Quantum Hall effect gives rise to very flat resistance plateaus as a function of magnetic field, with universal values $R = R_K/i$, where i is an integer and R_K is the von Klitzing constant, whose theoretical value is $R_K = h/e^2$. Note that the latter value is linked to the fine-structure constant α by exactly known constants c (speed of light in vacuum) and μ_0 (permeability of vacuum) as $h/e^2 = \mu_0 c/2\alpha$. Experiments have shown that the values of K_J and R_K are universal and independent of material or experimental conditions [1]. However, the exactness of their relation with fundamental constants, e and h, has not yet been demonstrated with metrological accuracy. LNE and LERMPS laboratories have started a project whose goal is to give a contribution to the experiment in which the relations $R_K=h/e^2$ will be tested with relative uncertainty approaching one part in 10^8. The confidence on this equality will be established through the calculable capacitor of Thompson-Lampard [2,3], which allows a direct determination of R_K, by comparing the obtained values with α determinations from measurements of h/M ratio (M is the mass of an atom of Cs or Rb, or neutron mass). R_K is determined through a long experimental chain with different steps, starting from the calculable standards of capacitance and resistance and the associated measurement bridges in ac current, up to measurements in dc current of the quantum Hall resistance. Therefore, to get R_K with an uncertainty of one part in 10^8 or less becomes a very big challenge, since that requires to minimize the self-contribution of every component of this chain. Calculable ac-dc resistance standards with a coaxial design have been developed and are based on cylindrical substrates of ceramic coated with metallic thin films of NiCr [4,5]. Analytical and numerical calculations showed that the change with frequency of these standards is lower than some parts in 10^9 from dc to some kHz. In addition to these best metrological characteristics that this design could offer, it particularly allows to obtain high resistance values while keeping the coaxial structure of the standard, which makes it possible to use the more precise 1:1 ratio in the frequency calibration of the quadrature bridge resistors of 10 kΩ, 20 kΩ and 40 kΩ.

To reach these high resistance values by magnetron sputtering technique, the NiCr films have to be as thin as possible while covering well the ceramic substrate. That implies deposit conditions leading to a layer by layer growth to ensure homogeneity in thickness and in chemical structure for a best distribution of the physical effects (inductive and capacitive) behind the frequency behavior in the ac-dc resistance standards. However, for metrological purpose it remains pretty obvious that these resistive films have to be characterized by a stability of resistance and thermal coefficients good enough to be consistent with the lowest uncertainty.

2. THIN FILMS DEPOSITION

The magnetic field in the magnetron sputtering deposition in which pressure is very low, allows to increase the ionization rate of the plasma gas around the target and consequently the growth speed. In addition, the emitted vapor flow is concentrated in a very directional way, which could present, on wide substrates, structural or morphological inhomogeneities. To obtain ultra thin layers on ceramic substrates of 45 mm in length and 8 mm in diameter, both last aspects have been carefully taken into account, particularly by finding a compromise between a growth speed which must be small enough to get very thin thickness and an adequate interaction rate of the sputtered chemical elements with plasma (depending on the distance target-substrate) which is of great influence on the growth mode.

The $Ni_{80}Cr_{20}$ samples are made in a limit vacuum of 10^{-6} mbar during evaporation with plasma of argon. The cylindrical substrates are placed in a rotating holder (100 turns per minute) at a distance of 13 cm from the target (Fig. 1).

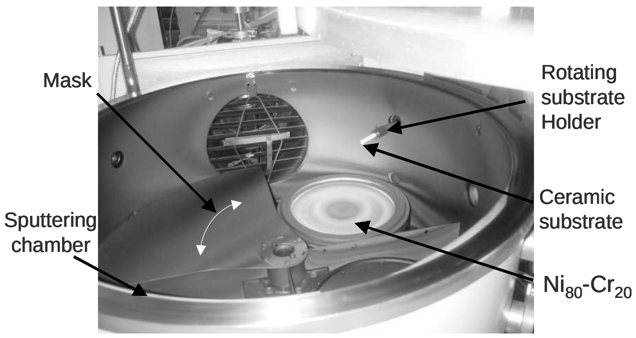

Figure 1: Magnetron Sputtering System

However, first deposits have been done on plane glass and ceramic substrates to calibrate the thickness value and the resistance per square of the samples. Before the sputtering process, the NiCr target is classically cleaned in a dc current (0.2 A, 400 V) to avoid any contamination of the films by surface oxides and impurities. Moreover, an ionic cleaning phase of the substrate is carried out over 5 minutes by applying a pulsed dc current (0.3 A, -300V, 240 kHz/1.4 μs) due to the insulation character of ceramic. Because of the growth speed is directly linked to the applied current to the NiCr target, low current values ranging from 0.08 A to 0.5 A were used during evaporation (with a voltage of –300 V between the target and the enclosure) to obtain the lowest deposit speeds. Finally, the elaborated films of NiCr are characterized by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

3. NiCr FILMS CHARACTERIZATION

Sputtered layers have been realized on square samples of ceramic of 1×1cm^2 of dimensions. These substrates have been stuck along a ceramic rod, as it is shown in figure 2a, to check the thickness homogeneity of the NiCr films over the length and the diameter when they would be deposited on cylindrical substrates. A part of the square samples was fitted with masking tapes (black strips), creating after deposition a step of NiCr film (Fig.2b). The NiCr step allows to measure the film thickness by using an Atomic Force Microscope as a profilemeter, simply by measuring the step height. This measurement procedure is applied to each NiCr step of the different square samples to determine the variation of the film thickness according to their position on the ceramic rode.

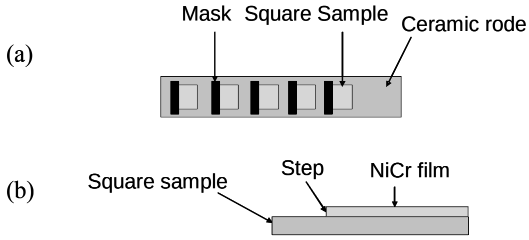

Figure 2: (a) Square samples layout on ceramic rode and (b) NiCr film step

AFM observations of NiCr layers clearly show a good covering rate of the deposited layers without any porosity of the films even for very thin thicknesses. AFM microscopy is also used to measure the NiCr step thickness (Fig.3) on various plane substrates laid out along the ceramic stick.

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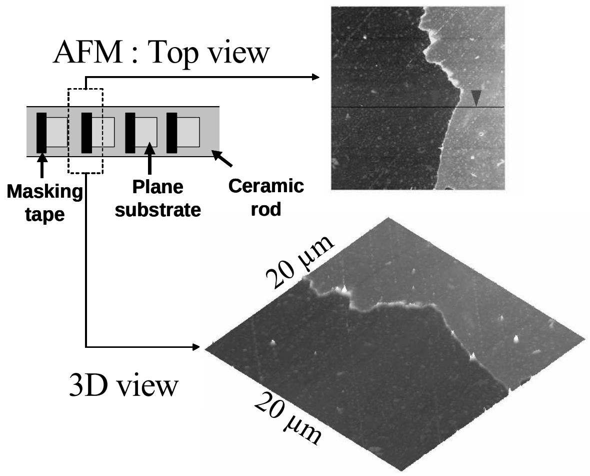

Figure 3: Layout of the square ceramic samples and AFM photos of NiCr layer steps

Figure 4 shows AFM profiles of such steps at the ends (Fig.4a, Fig.4c) and at the center (Fig.4b) of the ceramic stick. No thickness variation is observed, that indicates a very good homogeneity of the NiCr films. The mean value of the measured thickness is in this case of about 10 nm.

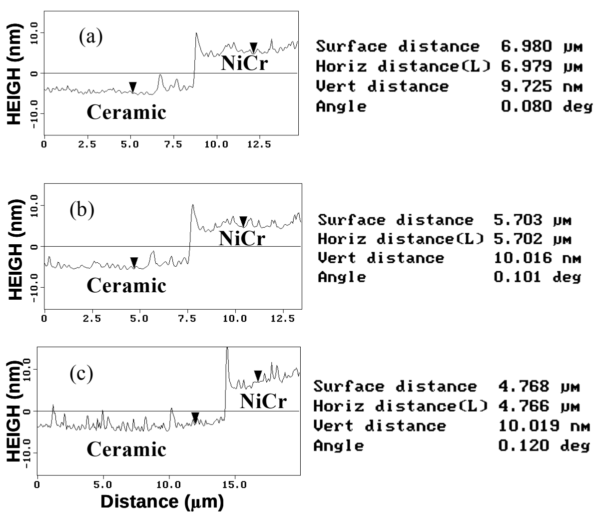

Figure 4: AFM step profiles of NiCr layers on ceramic plane substrate

4. RESULTS

Keeping all the deposit parameters, which give an optimal quality of the layers, NiCr films have been deposited on cylindrical sticks with extremely low growth speeds. In this case, only the thickness ***t*** was adjusted in order to obtain different resistance values, as showed in figure 5.

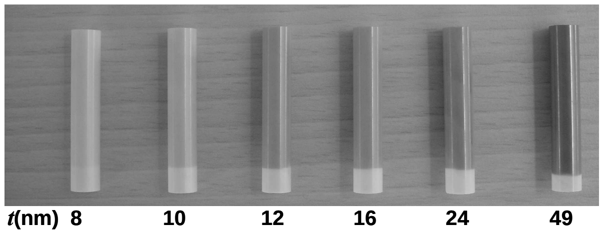

Figure 5: Photos of NiCr films on cylindrical ceramic sticks of various thickness t in nm.

Figure 6 presents the measured resistance values of this first set of NiCr layers with thickness varying from about 8 nm to 73 nm corresponding to deposit times from 30 s to 280 s. We can see clearly a drastic change in resistance around 10 nm of thickness, which occurs when the ceramic substrate starts to be well covered by the NiCr layer. Above t=10 nm, resistance values ranging from 1 kΩ up to 100 kΩ are obtained, which is a very promising result for coaxial ac-dc resistor applications.

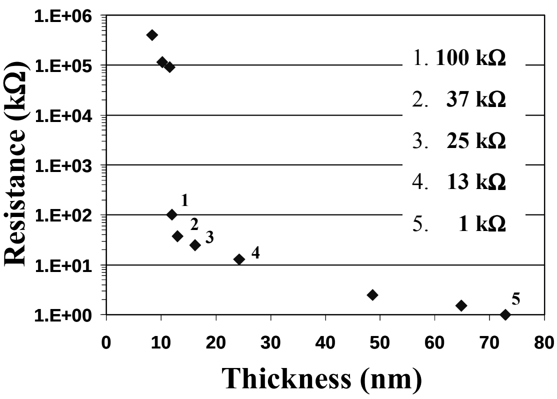

Figure 6: Resistance of NiCr films vs the thickness t .

Scanning Electron Microscopy and Energy Dispersive Spectroscopy were applied to analyze the quality of the obtained films. SEM photos of figure 7 give an overview of NiCr layers of different thicknesses (1 kΩ, 25 kΩ and 37 kΩ). These layers are of good structural quality and present good covering rates of the ceramic substrate. Moreover, EDS analysis show a homogenous distribution of Ni and Cr elements without any other contaminating ones. However, SEM and EDS observations will be carry on to see the effect of the natural oxidation of the films on the whole aspect of the layers and the evolution of the resistance values, which is very important for resistance standard application.

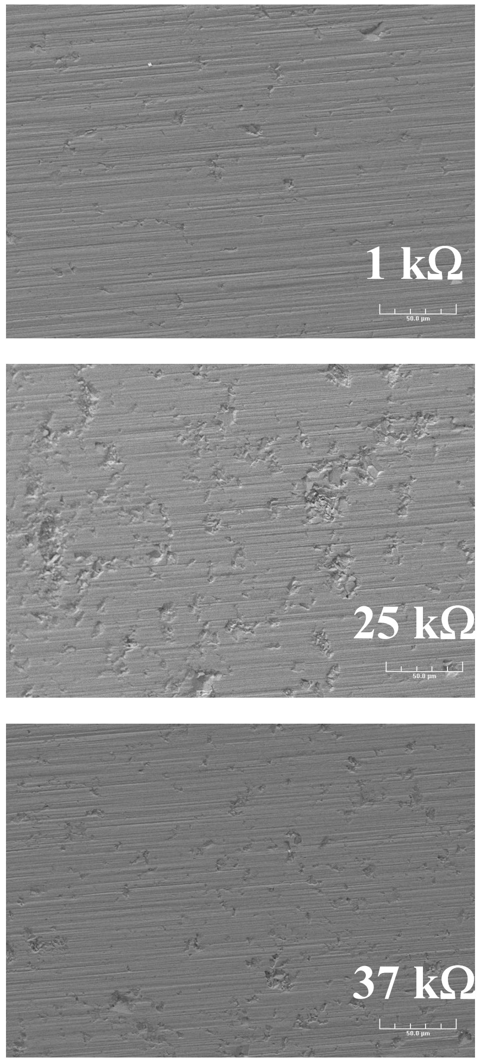

Figure 7: SEM photos of NiCr films vs thickness.

5. CONCLUSION

A new set of resistive sticks for ac resistors has been fabricated with resistance values up to 100 kΩ. Electrical measurements are being carried out on these samples to characterize their resistance stability and thermal coefficients and to determine their frequency performances. However, new deposits are also planed in which a best control of the growth speed should be achieved to get resistance values close enough to the nominal ones. The results of electrical measurements will be presented at the time of the conference.

REFERENCES

[1] J. Flowers, "The route to atomic and quantum standards", Science 306, 1324 (2004).
[2] A.M. Thompson and D.G. Lampard, "A new theorem in electrostatics and its application to calculable standards capacitance", *Nature*, vol 177, pp. 888 (1956).
[3] G. Trapon, O.Thévenot, J.C. Lacueille, W. Poirier, "Determination of the von Klitzing constant R_K in terms of

the BNM calculable capacitor – Fifteen years of investigations", Métrologia, n° 40, pp. 159 -171 (2003)

[4] A.Bounouh, "Numerical computations and measurements on calculable resistance standards based on NiCr thin film" CPEM04-Digest, London, UK, pp. 370-371, July 2004.

[5] A. Bounouh and S. Cadeau-belliard, "Calculations of ac resistance standard characteristics with Flux3D software" VII AMCTM, Lisbon-Portugal, June 2005.