Comparative Study of Four-Point Measurements of Copper Thin Films Resistivity in Aligned and Square Configuration at Low Frequencies

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Authors’ contributions
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ABSTRACT
The main objective of this research work is the comparison of the two Van Der Pauw four-point measurement configurations (aligned and square) commonly used to measure the resistivity of metal thin films which are widely used in the manufacture of planar components such as inductors.
transformers, insulators, circulators, etc. Electrical characterization is very important because it ensures the quality of the metal supposed to conduct electrical current in the components mentioned above. The resistivity measured by using the square or aligned configuration sometimes has significant differences. Making a comparative study therefore allow to know which gap between points, which frequencies to choose when measurements must be made by using square or aligned configuration.

In this paper, the measured thin films are made of copper which is broadly used in the structures because of its physical, chemical and mechanical properties [1]. The copper is deposited on alumina substrates by magnetron radiofrequency sputtering. Rectangular alumina substrates of dimensions 50mm x 20mm x 635μm were used. Thin film resistivity measurements using a four-point measurement method were performed by using HP 4284A LCRmeter in the frequency range of a few Hz to a few kHz. The comparison was made between the four-point aligned method and the four-point square method based on the frequency, the gap between points and the thin film thickness was made.

The results obtained by the study show that both configurations measure the same resistivity up to 1 kHz and then the increase of resistivity in aligned configuration limits its use beyond this frequency. The square configuration can still measure the same resistivity up to 40 kHz. The study also shows that the gap between points is limited to 3 μm in aligned configuration while the square configuration allows a gap up to 5 μm.

Keywords: Thin films; characterization; LCRmeter; Van Der Pauw; resistivity; thickness; copper.

1. INTRODUCTION

In the manufacturing process of planar components, thin films represent the basic material from which all other operations must take place. The quality of these thin films is therefore of crucial importance. For this reason several characterizations are performed on thin films in order to know their intrinsic properties before their use. These characterizations can be electrical, mechanical, magnetic or material types. This paper deals with electrical characterization. In electrical characterization, the paper focuses on measuring metal thin films resistivity [2-8].

The principal objective of the work detailed in this paper is a comparative study of two four-point measurement configurations used to measure copper thin films resistivity at low frequencies: aligned configuration and square configuration of Van der Pauw. The research work must allow to highlight validity in the frequency of the configurations and measurements precision according to gaps between points and thin films thickness.

This characterization work is carried out on thin copper films deposited on alumina substrates. Characterizations are performed on copper thin films deposited on alumina substrates by magnetron RF sputtering technique. Substrates have rectangular dimensions of 20mm x 50mm x 635μm. Characterizations of copper thin films are performed using HP 4284A LCRmeter coupled to a four-point tester. Results are presented in the following sections.

2. MATERIALS AND METHODS

This part of the work presents the used deposition technique, four-point configurations and the measuring device principle of operation.

2.1 Copper Thin Film Production

The deposition technique used in copper thin films production is magnetron radiofrequency sputtering. Radiofrequency sputtering consists of bombarding a target with energetic ions and particles grabbed away the target are projected onto a substrate placed on their paths. Energetic ions are created from an inert gas argon and plasma is created by applying a high frequency electrical voltage between the target and substrate [2,4,8-11]. Fig. 1 presents the radiofrequency sputtering principle.

The experimental apparatus consists of a vacuum chamber with residual pressure in which two planar electrodes, cathode and anode, have been arranged, separated by a few centimeters. The cathode, on which the target is fixed, is connected to the negative pole of RF voltage generator. The substrate is fixed on the anode which is connected to ground. After vacuuming the chamber, the pressure is allowed to rise to
Fig. 1. Radiofrequency sputtering principle [2]

about $10^{-2}$ or $10^{-3}$ mbar by introducing a controlled flow of argon and keeping a dynamic pumping to maintain a circulation of pure gas. An electrical discharge occurs between the electrodes when a high voltage is applied between them.

The deposition is nourished by maintaining a flow of argon in the enclosure. The deposition is improved by posting magnets in the neighborhood of the target to increase electrons’ chance of ionizing argon atoms [02].

RF sputtering parameters are grouped in the following Table 1.

Thin film thicknesses are measured using a profilometer. Several measurements were performed. The retained thicknesses values are 3.3 and 5.2 μm.

2.2 Four-Point Measurement Configurations

Four-point measurements are used to correct two-point measurements insufficiencies in particular resistances of cables and contacts which are overlapped with measurements. In the process of four-point measurements, two points are used to inject current and two others are used to measure the potential difference as close as possible to the Device Under Test (DUT) terminals. In this way, cables and contact resistances with DUT are eliminated from measurement results. Knowing the injected and measured current, the potential difference and the thin film thickness, resistivity is then calculated using the Van der Pauw method according to the two configurations: square and aligned configurations [3,5-8].

2.2.1 Aligned four-point configuration

In this study, the aligned configuration is used with a rectangular structure of length $a$ and width $d$. The four points are aligned at equal distance $s$ on a thin film surface as regards to $I_{Hcu} V_{Hpot} V_{Lpot} I_{Lcu}$ arrangement as shown in the following Fig. 2 where $I_{Hcu}$ and $I_{Lcu}$ are dedicated to current injection and $V_{Hpot}$ and $V_{Lpot}$ are dedicated to measuring the potential difference. The four points are connected respectively to four-terminal of LCRmeter by coaxial cables. The measuring current is injected by the LCRmeter and the potential difference is measured by an incorporated device.

The resistivity is calculated by using the following formula:

$$\rho = \frac{V}{I} * C \left( \frac{d}{s^2} \right) * e \quad (1)$$

In this formula $\rho$ is resistivity, $V$ measured voltage, $I$ injected courant $C$, the correction coefficient which depends on the ratio between the width $d$ of the thin films and the distance between the tips $s$ on the one hand and the ratio between the length $a$ and the width $d$ on the other hand and $e$ the thickness of the copper thin films [3,8,12-13].

2.2.2 Square four-point configuration

In the square configuration, the four points are arranged on thin films edges and numbered from 1 to 4 to form a square as shown in Fig. 3.

The current is injected between terminals 1 and 2, the voltage is measured between terminals 3 and 4 and the resistance $R_A$ is calculated.
The current is then injected between terminals 2 and 3, the voltage is measured between terminals 1 and 4 and the resistance $R_B$ is calculated.

If currents are injected and voltages measured as shown in the figures, the resistances $R_{12,43} = R_A$ and $R_{23,14} = R_B$. Resistivity $\rho$ is a solution to Van der Pauw equation:

$$\exp\left(-\frac{\pi \rho}{\rho} R_{12,43}\right) + \exp\left(-\frac{\pi \rho}{\rho} R_{23,14}\right) = 1$$  \hspace{1cm} (2)

With the symmetry of the square, the Van der Pauw equation can be simplified and the resistivity is given by the following equation [2-3,8,12-16]:

$$\rho = \frac{\pi \rho}{ln2} R = \frac{\pi \rho}{ln2} \frac{V}{ln2}$$  \hspace{1cm} (3)

### 2.3 Measurement Methods

Four-point measurements are performed using a LCR meter type HP 4284A associated with a tester under tips. This LCR meter is an impedance bridge based on measurement of current-voltage vector across Devices Under Test (DUT). It is from this vector that complex impedance $[Z]$, complex admittance $[Y]$ as well as inductance, resistance, capacitance and other parameters of the device are calculated. To measure this current-voltage vector, the LCR meter uses a self-balanced bridge method shown in Fig. 4 [2].

The bridge consists of two oscillators, an R-range resistor and a zero detector. Measurement principle is to cancel potential at point P by controlling, in amplitude and phase, delivered signal by the oscillator 2. For this, the zero detector measures the potential of P point and imbalance current (difference of currents $I_x$ and $I_R$ in DUT and resistance $R$) and controls the oscillator 2 in order to obtain a zero potential at point called virtual mass. The bridge balance gives a zero-imbalance current so that $I_x = I_R$ and then:

$$I_x = \frac{V_x}{Z_x} = -\frac{V_2}{R}$$  \hspace{1cm} (4)

### Table 1. RF sputtering parameters [3]

| Code | Substrate polarization | Interval target - Substrate (cm) | Plim (mbar) | Pdép (mbar) | Power FWDP (W) | Ar (sccm) | Duration (min) |
|------|------------------------|---------------------------------|-------------|-------------|---------------|-----------|---------------|
| 1    | Connected to the enclosure | 6.7                              | 5.4.10⁻⁷    | 2.10⁻²      | 300           | 20        | 20            |
| 2    | Connected to the enclosure | 6.7                              | 2.10⁻⁶      | 1.8.10⁻²    | 300           | 20        | 30            |

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**Fig. 2.** Aligned Van der Pauw configuration [3,8,12-13]

**Fig. 3.** (A-B). square Van der Pauw configuration [3,8,12-13]
The DUT complex impedance $Z_x$ is therefore given by the equation:

$$Z_x = -\frac{V_1}{V_2} R$$  \hspace{1cm} (5)

Voltages $V_1$ and $V_2$ are transmitted to a digital acquisition chain that performs processing to determine real and imaginary parts of DST impedance and admittance.

The tester under tips allows to measure voltage drop at DUT terminals using configurations presented above.

Measurements were performed in serial RL mode with a SHORT correction to eliminate residual impedances. The results will be presented in the results and discussion section [2-8].

3. RESULTS AND DISCUSSION

The results presented in this section are obtained by measuring samples manufactured according to techniques, equipment and methods presented in previous section.

3.1 Resistivity Study as a Function of Frequency and Tip Spacing

In this part of this study, spacing between tips is fixed. Resistivity is observed as a function of frequency, thickness and configuration. Five spacing between tips values ranging from 1 to 5 mm were achieved; frequency varies from 50 kHz to 40 kHz; thicknesses are 3.3 and 5.2 μm and aligned and square configurations have been performed. Figs. 5 to 9 show measurements results.

These figures show that whatever thickness and spacing between tips values, resistivity measured with square configuration is constant in the aforementioned frequency range (50 kHz to 40 kHz) while it is constant only from 50 Hz to 1 kHz in the case of aligned configuration. After 1 kHz, resistivity measured with an aligned configuration increases rapidly. Noting that at 1 kHz, skin and proximity effects that are the main causes of conductor resistance increase and therefore its resistivity are not yet very visible, this abnormal resistivity increase shows that aligned configuration is not used above this frequency.

However, figures show that for spacings between tips less than 3 mm, resistivity is the same for aligned and square configurations in the frequency range from 50 Hz to 1 kHz and for a given thickness.

For spacings greater than 3 mm, there is an inversion of the resistivity value as a function of thicknesses in aligned configuration, i.e., the resistivity of the thickest film becomes lower and that of the weaker film becomes higher. The resistivity in a square configuration is the same as in the case of spacings less than 3 mm. These results clearly show that in an aligned configuration, resistivity is a function of the spacing between tips. This restricts the use of aligned configuration. Indeed, whereas the two configurations measure the same parameter according to thickness, this inversion can only be justified by the limit of the aligned method. It is possible to cancel this reversal by adjusting injected current in order to control the measured voltage to obtain the same resistance. This can be the subject of another paper.

The various figures plotted as a function of frequency, thickness and spacing between tips show that to have a good measurement agreement between aligned and square configurations, it is necessary to limit the measurements to 1 kHz and a spacing of less than 3 mm. So what is the real impact of points spacing on resistivity value? In the next section, we will study resistivity evolution as a function of spacing.

3.2 Study of Resistivity as a Function of Tips Disposition

To go deeply into resistivity study according to configurations, resistivity evolution as function of points spacing and thickness was performed. Figs. 10 to 12 show this evolution for three frequencies: 50, 400 Hz and 1 kHz.

The figures show that for the square configuration, resistivity is constant for all thicknesses and spacings between tips (from 1 to 5 mm). The difference between curves of the configuration comes only from thickness according to the proportionality of the equation giving resistivity (eq. 3).

In the case of aligned configuration, resistivity change with spacing between tips and thickness. For a film with 3.3 μm thickness whose resistivity is less than the resistivity of solid copper, resistivity increases as soon as spacing between points is greater than 3 mm. It is just the opposite in the case of film of 5.2 μm thickness and whose resistivity is greater than the resistivity of solid copper which decreases when the spacing is
greater than 3 mm. These variations, it can easily be noted, do not depend on frequency. It is therefore an intrinsic phenomenon to aligned configuration. This square configuration behavior can be studied as a function of courant intensity and voltage in another work.

To have a comparable resistivity for both two configurations, spacing between points must be limited to 3 mm.

For a spacing of 1 mm between tips, there is a perfect concordance between measurements for the two configurations. The square configuration gives the same resistivity value for all the spacings studied values. Contrariwise, in aligned configuration, the resistivity depends on spacing of tips. So, what are the measurement errors compared to concordant measurement at 1 mm?

![Diagram](image.png)

**Fig. 4. HP 4284A Self-Balanced Bridge [2]**

![Graph](image.png)

**Fig. 5. Frequency-dependent resistivity for 1 mm between tips**

![Graph](image.png)

**Fig. 6. Frequency-dependent resistivity for 2 mm between tips**
3.3 Measurement Variances

Preceding figures have shown that measured resistivity in aligned configuration varies with spacing between tips and it is equal to resistivity in the square configuration when spacing is 1 mm. It is therefore important to estimate as a percentage of this reference value, variations in values as a function of spacing and frequency. Table 2 shows the percentage of the variance for four spacings and three frequencies. All variances are calculated from values obtained with a spacing of 1 mm.

The table analysis shows that resistivity variance is acceptable for spacings up to 3 mm because it remains less than 10% except for 3.3 μm thickness at 400 Hz and 1 kHz. This means that to correctly measure resistivity in aligned configuration, measurements must be performed with spacings below 3 mm and frequencies below 1 kHz. For thin thicknesses, it is necessary to remain well below 1 kHz.
Fig. 9. Frequency-dependent resistivity for 5 mm between tips

Fig. 10. Spacing-dependent resistivity at 50 Hz

Table 2. Resistivity variance in an aligned configuration

| Spacing (mm) | 50 Hz  | 400 Hz | 1 kHz  |
|--------------|--------|--------|--------|
|              | 3.3µm | 5.2µm | 3.3µm | 5.2µm | 3.3µm | 5.2µm |
| 2            | -5    | -5    | -5    | -5    | -5    | -5    |
| 3            | -9    | -9    | -20   | -9    | -20   | -9    |
| 4            | 61    | -20   | 61    | -20   | 61    | -19   |
| 5            | 46    | -27   | 46    | -27   | 46    | -27   |
4. CONCLUSION

The comparative study of copper thin films resistivity by using aligned and square configurations of Van der Pauw clearly shows that square configuration is more efficient because it allows good quality measurements up to more than 40 kHz in frequency and measurements do not depend on the spacing between tips. Indeed, various figures showed that the resistivity was the same (depending on thicknesses) in square and aligned configurations for frequencies ranging from 50 Hz to 1 kHz. The aligned configuration is limited to this frequency but the square configuration can measure the same resistivity up to 40 kHz. Considering the gap between points, the aligned configuration is limited to 3 μm and the square configuration to 5 μm. In the case of aligned configuration where increases in the resistivity value have been observed, the difference between measurements are acceptable when frequency and gap between points conditions are respected.

This study also shows that aligned configuration can be used under well-defined conditions. To obtain results comparable to square configuration, the frequency must be less than 1 kHz and spacing less than 3 mm. With these conditions, the difference between measures remains less than 10%.

This paper therefore contributes to knowing conditions of use of the two Van der Pauw measurement configurations. The choice of square or aligned configuration is guided by the frequency of use and available gap on the surface of the device under test. For frequencies below 1 kHz and small gaps, both configurations are valid. For high frequencies and the need of large gap, only the square configuration allows valid results.
COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Liliane Dedryver. The consumption of digital metals: A sector far from being dematerialized. Discussion paper, France Strategy, Paris. N° 2020-05. 2020;52.
2. Allassem Désiré. Contribution to the realization of a planar micro-inductor. PhD Thesis in Optics, Photonics, Microwave, Saint-Etienne: Jean Monnet University. 2010;117.
3. Yacoub Nassian Nimir. Measurement of metal thin films resistivity, comparison between the four-point method and the square points method. Research master's thesis in electronics, n'djamena: consortium of universities of chad and jean monnet university of saint-etienne, National Higher Institute of Science and Technology of Abéché. 2018;52.
4. Yaya Dagal Dari. Design, realization and characterization of planar inductors with magnetic film. PhD Thesis in Optics, Photonics, Microwave, Saint-Etienne: Jean Monnet University. 2013;167.
5. Matibeye Alain, Electrical characterization of thin films through its conductivity. Research master's thesis in electronics, n'djamena: consortium of universities of chad and jean monnet university of saint-etienne, university institute of science and technology of Abéché. 2013;50.
6. Abbas Mahamat Sied Haggar. Electrical characterization of copper thin films in low frequency using the LCRmeter. Research master's thesis in electronics, n'djamena: consortium of universities of chad and jean monnet university of saint-etienne, national higher institute of science and technology of Abéché. 2015;55.
7. Djerandouba Bayombaye. Measurement of metal thin films resistivity, study of the corrective factor of the Van der Pauw method. Research master's thesis in electronics, n'djamena: Consortium of universities of chad and jean monnet university of saint-etienne, national higher institute of science and technology of Abéché. 2017;56.
8. Jean-Pierre Chatelon. Magnetic thin films developed by RF sputtering. Micro-technology lecture notes, Saint-Etienne: Jean Monnet Université; 2008.
9. A. Mennad. Les techniques de dépôt des couches minces et leurs applications. Revue des Energies renouvelables. 2015; 18(4)713-719.
10. Noua Bouhssira. Development and characterization of zinc oxide thin films by evaporation. PhD Thesis in Physics, Constantine: University of Mentouri. 2005; 104.
11. Stéphane Andreiu. Introduction to thin films and Networks. Franco-Romanian School: Magnetism of nanoscopic systems and hybrid structures, Materials Physics Laboratory, Brasov. 2003;6.
12. F.M. Smits. Measurement of sheet resistivities with the four-point probe. The Bell System Technical Journal. 1957;171-178.
13. F.M. Smits. Measurement of sheet resistivities with the four-point probe. The Bell System Technical Journal. 1958;711-718.
14. Valdes LB, Member,IRE. Resistivity measurements on germanium for transistors. Proceedings of IRD. 1954;420-427.
15. Mike Bolt, Calvin College. Van der Pauw's Theorem on Sheet Resistance. Annual Meeting of the Michigan MAA Hope College. 2015;24.
16. Maria P, Gutierrez, Haiyong Li, Jeffrey Patton. Thin film surface resistivity. Male 210 Experimental Methods in Materials Engineering. 2002;25.

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