



Construction and implementation of a 4-probe measuring System to determinate the temperature dependent sheet Resistance of thin films

Thesis submitted for the degree of master in mechatronics

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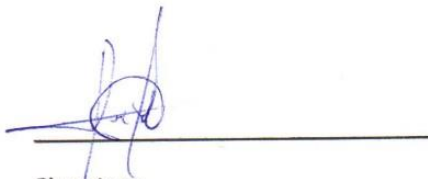
Without their help this would not have been possible.



II. Declaration

Hereby, I declare that I have elaborated the present work without any non-specified assistance. The people involved in the research, literature, as well as any another resource used in this thesis, have been completely specified throughout at the end of the text.

19st April 2016, Ilmenau



Signature

III. Resumen

Con el fin de construir máquinas, dispositivos electrónicos, es necesario conocer todas las propiedades de los materiales. Las máquinas y los dispositivos electrónicos utilizan piezas que están interconectados, las propiedades mecánicas son importantes, pero para algunas tareas específicas son más importantes las propiedades eléctricas. En este sentido es necesario predecir el comportamiento de estas piezas a diferentes temperaturas. La presente tesis se centra en la implementación de un sistema de medición 4-puntas para determinar la resistencia laminar de las muestras de las láminas delgadas, que demuestran la dependencia de la resistividad respecto al espesor de la lámina, así como de la temperatura de deposición. El método utilizado para determinar la resistividad es el van der Pauw modificado. Por lo tanto, es importante la medición de la corriente y la caída de tensión en la muestra. También es importante la medición de la distancia entre las puntas, para calcular la resistividad. Sin embargo, también es importante encontrar la correcta transformación que mapea cuatro puntos de un plano a un nuevo plano con cuatro puntos en lineados. Para la dependencia de la resistividad de la temperatura se utilizará el método der Pauw van modificado. Las mediciones se controlan a través de LabVIEW y los datos medidos se muestran en una interfaz de usuario.



IV. Abstract

In order to build machines, electronic devices, it is necessary to know all properties of the materials. The machines and electronic devices use parts that are interconnected, the mechanical properties are important, but for some specific tasks the electrical properties are more important. In this sense it is necessary to predict the behavior of this parts in different temperatures to the environment. The present thesis focus on implementation of a 4-probe measuring system to determinate the sheet resistance of thin film samples showing the dependency of the resistivity on the film thickness as well as on the deposition temperature. The method used to determine the resistivity is the modified van der Pauw Method. Therefore, it is important the measurement of the current and the voltage drop in the sample. It is also important to measure the distance between tips, in order to calculate the resistivity. Furthermore, it is also important to find the correct transformation that maps any four point of a plane to a new plane with four collinear points. The measurements are controlled via LabVIEW and the measured data is displayed in the user interface.



V. Kurzfassung

Um Maschinen und elektronische Geräte zu bauen ist es notwendig alle Eigenschaften der Materialien zu kennen. In Maschinen und elektronischen Geräte werden Teile verwendet, welche miteinander verbunden werden, wobei deren mechanische Eigenschaften wichtig sind, aber für manche spezifische Aufgaben sind die elektrischen Eigenschaften wichtiger. In diesem Zusammenhang ist es notwendig vorauszusagen, wie sich das Verhalten dieser Teile bei verschiedenen Temperaturen zur Umgebung ändert. Die vorliegende Arbeit konzentriert sich auf die Implementierung eines 4-Punkt-Messsystems zur Bestimmung des Plattenwiderstandes von dünnen Filmproben sowie der Abhängigkeit des spezifischen Widerstands von der Filmdicke als auch der Fertigungstemperatur der Probe. Das Verfahren, welches verwendet wird um den spezifischen Widerstand zu bestimmen, ist die modifizierte van der Pauw Methode. Deshalb ist das Maß des Stroms und der Spannungsabfall in der Probe wichtig. Es ist noetig, die Entfernung zwischen den Messpunkten zu bestimmen, um den spezifischen Widerstand zu berechnen. Desweiteren ist es auch wichtig, die richtige Transformation zu finden, um jeden der vier Punkte einer Ebene in einer neuen Ebene mit vier Collinearen-Punkten kartografisch darzustellen. Die Messwerte werden mit Hilfe von LabVIEW ueberwacht, und die Messdaten werden in einer Benutzeroberflaeche angezeigt.

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1 Introduction

Nowadays, almost everybody is interacting using electronic devices in order to make life easier.

Every piece of these devices has different properties (electrical, mechanical, etc.). The knowledge of each component’s properties can lead to its proper function. The common components used in the electronic or electric circuits are resistors, inductors, capacitors, diodes, transistors, conductors and insulators. These consist of different types of materials. For example diodes and transistors are made of semiconductors with different type of doping. Capacitors are made of two conductors and in between there is an insulator (dielectric material). Inductors are made of a coiled conductor wire with an insulating core (e.g. air). The conductors connect the other components, and the insulators avoid the electrical contact between conducting components. Therefore, in order to apply a material properly, it is very important to know the properties.

One of the properties that concerns the most to the developers of electric circuits is the conductivity (σ) of the used material. This property indicates whether a material allows the flow of current or not. This leads to the classification of the materials in conductors, semiconductors and insulators. Additionally the electrical resistivity (ρ) is important as this property indicates the resistance of a material to the flow of current.

$$\sigma = \frac{1}{\rho}$$

Equation 1: Relationship between conductivity and resistivity

Where:

σ : Conductivity [S/m]

ρ : Resistivity [Ω m]

The development of new materials for micro electromechanical systems (MEMS) as well as integrated circuits based on thin solid films is a challenging task especially when it comes to the thickness dependency of the electrical resistivity [1]. More challenging is this task if the investigated samples have an arbitrary shape and should be measured at elevated temperatures [2].

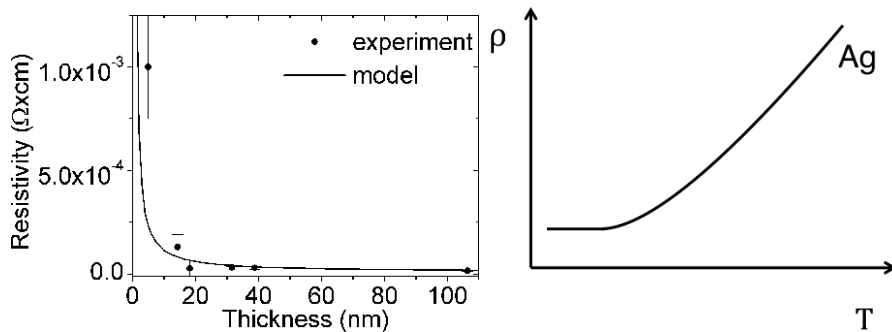


Figure 1: Dependence of the resistivity from the thickness (a) and dependence of the resistivity from the temperature (b) [1] [2]

Various models and methods have been suggested to measure the electrical resistance, nevertheless there is correction factors in order to be more precisely (shape of the sample, contact resistance, temperature, etc.)

Among others, the methods to measure the resistivity can be carried out using two probes, four probes, in van der Pauw setup, etc.

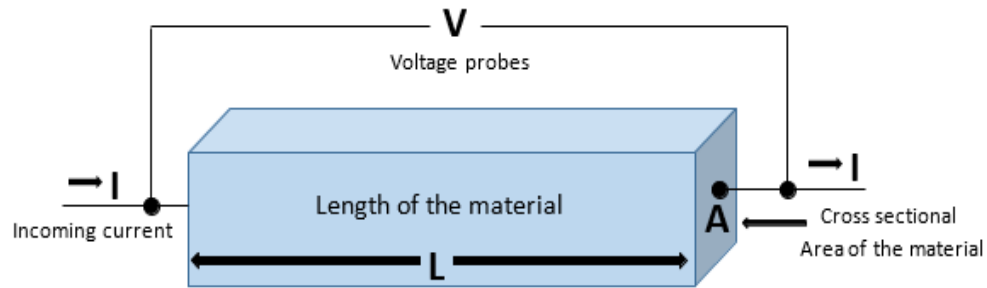


Figure 2: Electrical resistivity measured by the two probes method. [3]

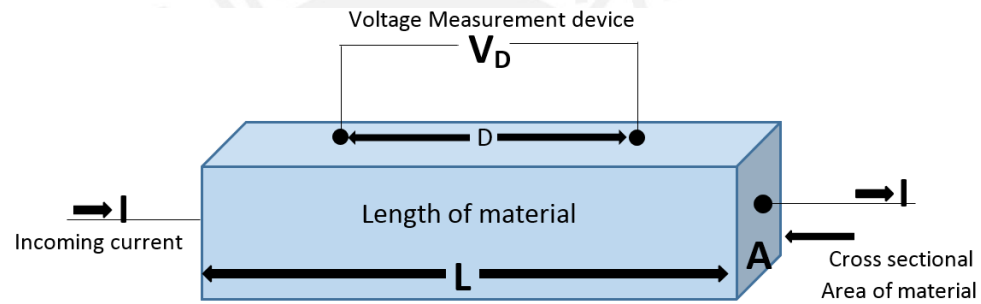


Figure 3: Electrical resistivity measured by the four probes method. [3]

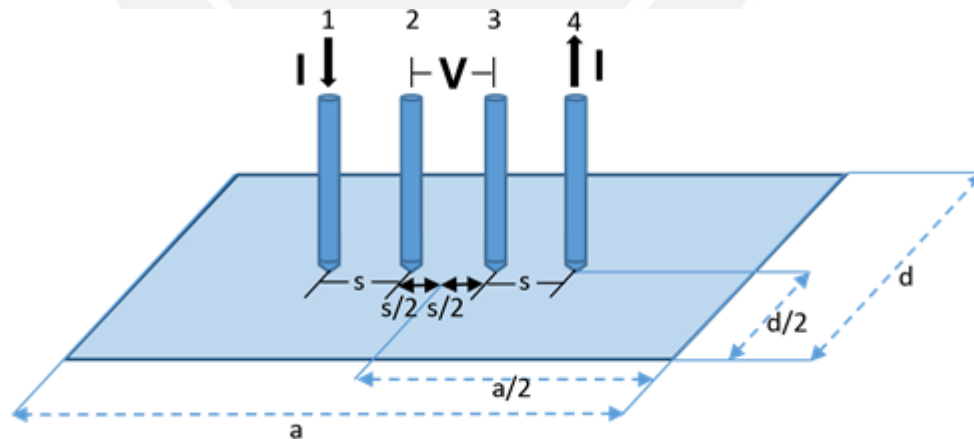


Figure 4: Electrical resistivity measured by four point probe method. [3]

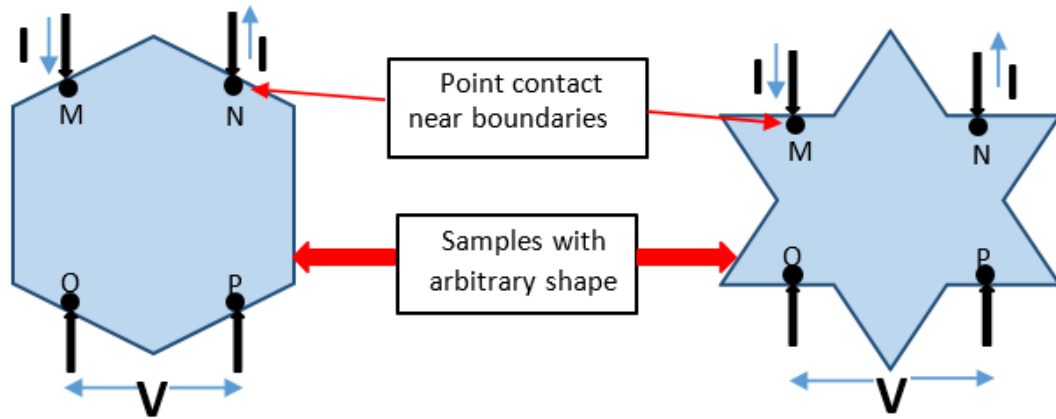


Figure 5: Van der Pauw schematic measurement. [3]

The atoms in a solid is in a constant movement due to the vibrating, these are bonded with each other the temperature is an important factor in the measurement of the resistivity,

$$\rho(T) = \rho_{20}(1 + \alpha * \Delta T)$$

Equation 2: Calculation for resistivity a different temperature of 20

Where:

$\rho(T)$: Resistivity due to the phonons [Ω m]

ρ_{20} : Resistivity at 20 °C [Ω m]

α : Temperature coefficient

ΔT : Gradient of temperature [$^{\circ}$ C]

The aim of this Master's thesis is the implementation of a 4-probe measuring system to determine the sheet resistance of thin film samples showing the dependency of the resistivity on the film thickness as well as on the deposition temperature. As the sample might have an arbitrary shape four freely moving probes should be used. Therefore, an optical tracking to determine the distance between the tips should be implemented. A third task in this thesis is the implementation of a heat source based on a Peltier element. Therefore, a profound understanding of the working principle of such an element should be demonstrated

2 State of the Art

2.1 Resistivity

The electrical resistance is the degree of resistance a material presents when electrical charges (electrons, ions and holes) flows through it [4]The resistance will vary according to the material itself, as well as to the length, width and thickness of the material.

$$R = \frac{L}{A} \rho$$

Equation 3: Relationship between resistance and resistivity

Where:

R: resistance [Ω]

L: length [m]

A: area [m^2]

ρ : resistivity [$\Omega \text{ m}$]

So, the resistance depends on both, the geometry of the system being measured and the nature of the material itself [4]. Figure 6: Schematic of electrical resistance and how it is measured shows the schematics of typical electrical resistance measurement.

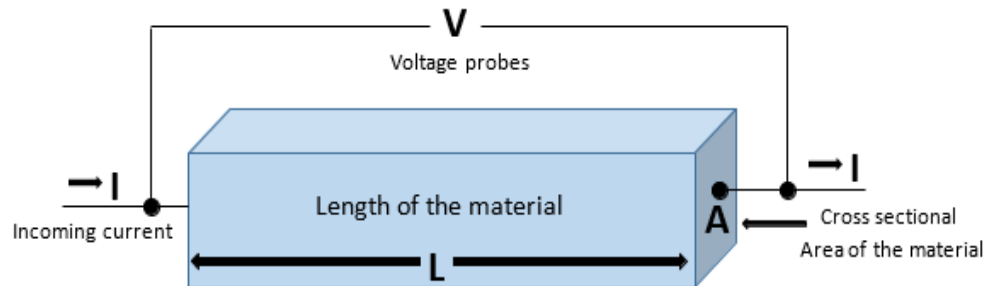


Figure 6: Schematic of electrical resistance and how it is measured [3]

The specific resistivity (ρ) is an intrinsic property of the material. This means, that it depends only on the material and not on its geometry [4]. This property defines how well a specific material conducts the flow of electric current. Due to this property, materials are classified as metals, semiconductors and insulators (Table 1).

Table 1: Values of specific resistivity of common materials [4] and graphite [5]

Material	Resistivity ρ ($\Omega \cdot m$)
Conductors	
Copper	1.68×10^{-8}
Gold	2.44×10^{-8}
Iron	1.00×10^{-7}

Semiconductors	
Carbon(Graphite)	$3-60 \times 10^{-5}$
Germanium	4.60×10^{-1}
Silicon	6.40×10^2
Insulators	
Mica	$10^{11} - 10^{15}$
Rubber (Hard)	$10^{13} - 10^{16}$
Teflon	10^{23}

One way to understand this classification is by using the electronic band structure. In this case, to understand the differences, it is necessary to understand the energies available for the electrons in the material. Instead of having discrete energy levels, such as in the case of free atoms, the energy states available for an infinite group of atoms all together, form bands (Figure 1). The valence band is representing the energy of the valence electrons. There is a gap that appears between the valence and conduction bands (forbidden region) and it has a width that varies from zero to ten electron volts [6]. In order to move from the valence band to the conduction band, the valence electrons must gain energy. In the case of conductor materials, the valence band and the conduction band are overlapping. So the valence electrons can cross to the conduction band easily. In the case of semiconductor materials, these bands are separated by a 2 eV. This gap represents the energy that the electron must gain in order to move to the conduction band. This gap is small enough so that the electrons can move to the conduction band by a small source of excitation such as thermal excitation. If the source of excitation is constant, like an electric field, these free electrons can move in this band even if they lose energy and fall back to the valence band. This constant electric field gives the valence electrons enough energy to return to the conduction band. In the case of insulator materials, the gap between the valence band and the conduction band is 5 eV big enough that thermal excitation or an electric field cannot give the valence electrons 5 eV energy to move to the conduction band [6].

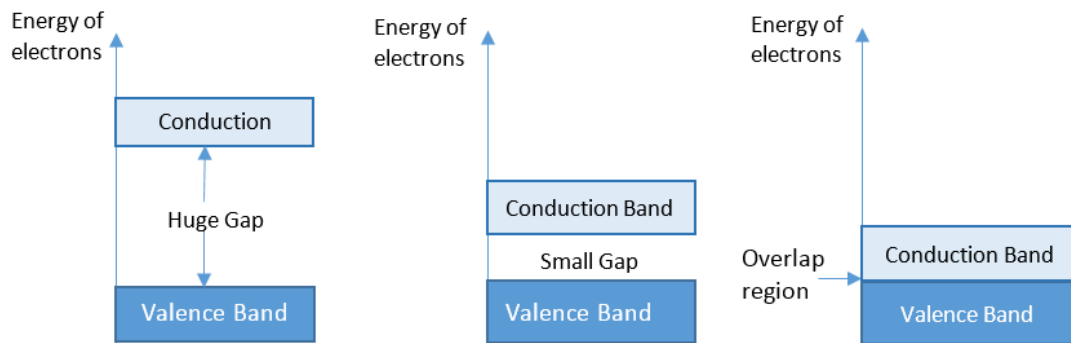


Figure 7: Energy bands in solids

In the case that impurities (phosphorus, boron, etc.) are added to a semiconductor material (silicon), the conductivity is increased. This process is known as doping. The impurities are added in order to increase the energy of the valence band and reduce the energy needed for a valence electron to

move to the conduction band. These impurities (phosphorus, boron) add electrons or holes to the materials depending on the doping process. An n-type doping, consists in adding a pentavalent element like Phosphorus to an intrinsic semiconductor such as silicon. Phosphorus has five valence electrons while silicon has only four. When the phosphorus is added to the silicon, it forms four covalent bonds. As a result, there is an extra electron “free” in the material and with a higher level of energy. A p-type doping consists in adding a trivalent element like boron, to an intrinsic semiconductor like silicon. As boron has three valence electrons, there will be three covalent bonds and one “hole” when it is added to the silicon.

The resistance of a conductor changes with changing temperature. This gradient of temperature caused a changing activity of the atoms that change the material resistance. The effect of heat on the atomic structure of a material induces a vibration of atoms. The higher the temperature gets the more the atoms vibrate (phonons).

$$\rho(T) = \rho_{20}(1 + \alpha * \Delta T)$$

Equation 4: Resistivity calculation for a temperature different to 20 °C

Where:

$\rho(T)$: Resistivity due to the phonons [$\Omega \cdot m$]

ρ_{20} : Resistivity at 20 °C [$\Omega \cdot m$]

α : Temperature coefficient

ΔT : Gradient of temperature [$^{\circ}C$]

In a material, which has a large number of free electrons flowing through it, the vibration of the atoms causes many collisions between the free electrons and the captive electrons. Each collision consumes some energy from the free electron and is a cause of resistance. The more the atoms vibrate in the material, the more collisions are caused and hence the greater the resistance to current flow.

$$\rho = \rho_{th} + \rho_{imp} + \rho_{def}$$

Equation 5: Resistivity due to Matthiessen's Rule [2]

Where:

ρ_{th} : Resistivity due to temperature

ρ_{imp} : Resistivity due to impurities

ρ_{def} : Resistivity due to defects

Materials have a positive temperature coefficient if the resistance increases with an increase in temperature. In general conductors have a positive temperature coefficient (α), while (at high temperatures) insulators have a negative temperature coefficient (α) (Figure 8 and Figure 9). [4]

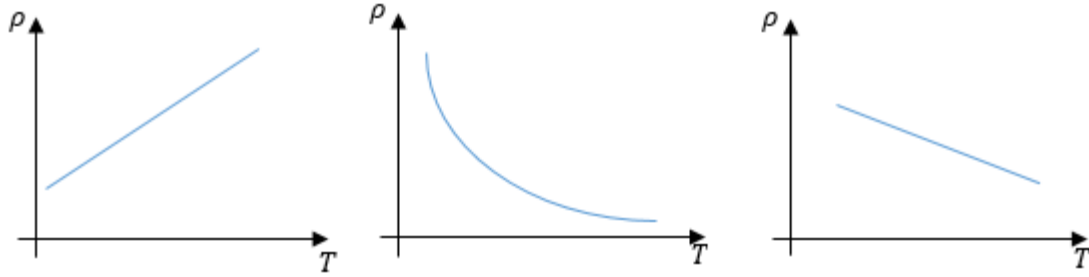


Figure 8: Resistivity VS Temperature (conductors, intrinsic semiconductors and insulators) [2]

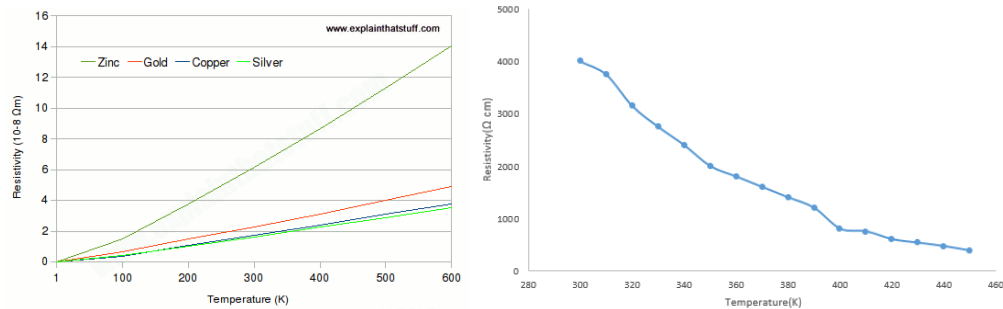


Figure 9: Resistivity VS Temperature (conductors, Semiconductor SnSe) [7] [8]

2.2 Electrical effects caused by thermal change

Thermoelectric effects involve an interaction between electronic and thermal properties of a system. These can be observed by the measurement of voltage and current obtained due to thermal gradients. The voltage and current can create a heat flow. In this case the more important thermoelectric effects are the Seebeck effect and Peltier effect. [9]

2.2.1 Seebeck effect

When a thermal gradient is applied to a conductive material, the charge carries (electrons, ions and holes) move along the thermal gradient from hot to cold, as a result an electric potential difference is created. In closed loop formed by two metals joined in two places with temperature difference between the joints create a flow loop (thermo electric current) and a magnetic field. At the junction of the two different metals free electrons can migrate in one direction across the junction, setting up an electric field and a fixed potential (thermo electromotive force (e.m.f)). [9]

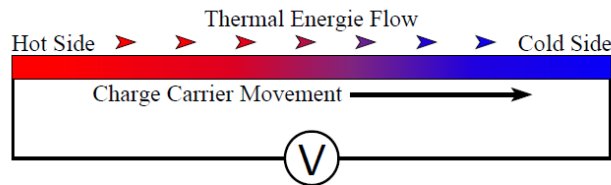


Figure 10: Charges carries move along the thermal gradient [9]

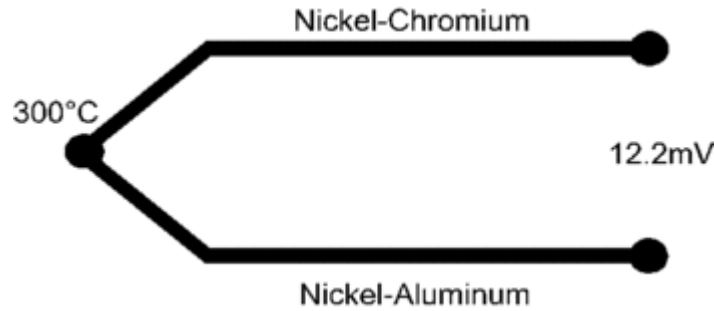


Figure 11: Voltage generate by the temperature between to different conductors [10]

It was shown that the electron diffusion current produced the magnetic field in the circuit a changing voltage of e.m.f. (Lenz’s Law). The magnitude of the voltage produced between the two junctions depends on the material and on the difference of temperature of the element 1 and element 2 ΔT_{12} through the linear relationship defined by the Seebeck coefficient S for the material (Figure 11). Values of Seebeck coefficient are shown in Table 2.

$$\Delta V = S \Delta T_{12}$$

Equation 6: Relationship between voltage and temperature

Where:

ΔV : Voltage difference [V]

S : Seebeck coefficient [V/°K]

ΔT_{12} : Temperature difference between element 1 and element 2 [°K]

Table 2: Values of coefficient of Seebeck of metals and semiconductors [11]

Material	Seebeck coefficient ($\mu\text{V}/\text{K}$)
Conductors	
Copper	6.5
Gold	6.5
Iron	6.5
Aluminum	3.5
Lead	4.0
Semiconductors	
Selenium	900
Tellurium	500
Germanium	300
Silicon	440

The Seebeck effect can be used to identify the type of dominating charge carriers in the material, if the electrons are the dominating charge carriers, the cold side will be charged negatively, whereas for holes dominating materials the cold side is charged positively [12]

2.2.2 Peltier Effect

The flow of electricity introduced into a thermocouple circuit causes an exchange of heat between a junction and its surroundings that can be reversed by reversing the direction of the current (Figure 12). The effect at one junction is independent of the temperature of the other junction or the size of the wire, and it is directly proportional to the current [13]. In Peltier effect devices, a temperature difference is created: one junction becomes cooler and one junction becomes warmer.

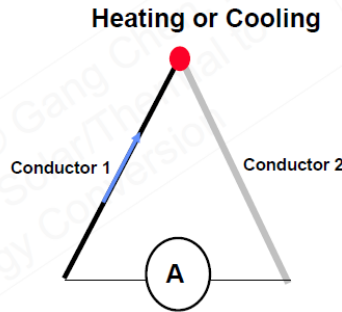


Figure 12: The flow of the current will determine the temperature of the joint. [14]

A thermo material can be a semiconductor. The electrical behavior is determined by the majority charge carriers (electrons and holes). When n- and p-type semiconductors are biased in the same direction, their charge carriers flow in opposite directions.

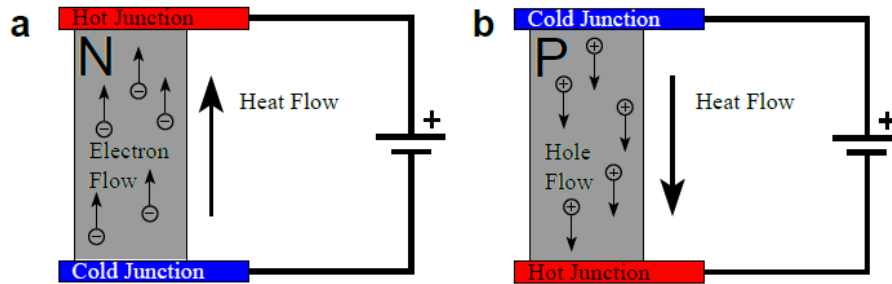


Figure 13: N-type versus P-type Peltier elements [9]

The power of a single Peltier element is not enough for a realistic situations. In order to increase their power a Peliter device is made of many n-type and p-type semiconductors (see Figure 13). They are connected in series using a metallic junctions individually. In consequence charge carries flow freely in both directions. In a Peltier device, the individual elements are arranged in an array that the n-and p-type heat flow in the same direction (see Figure 14)

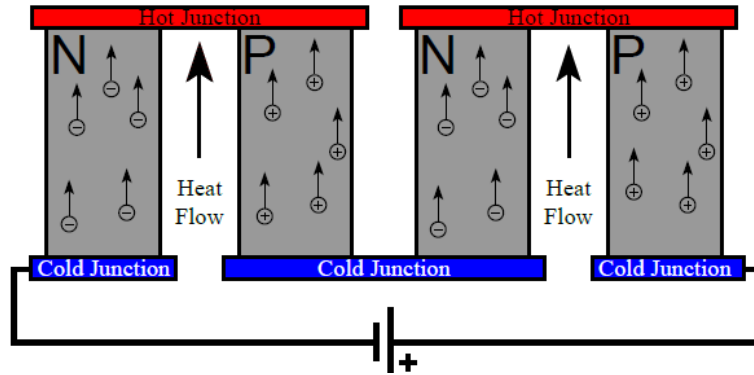


Figure 14: Array of a Peltier Device [9]

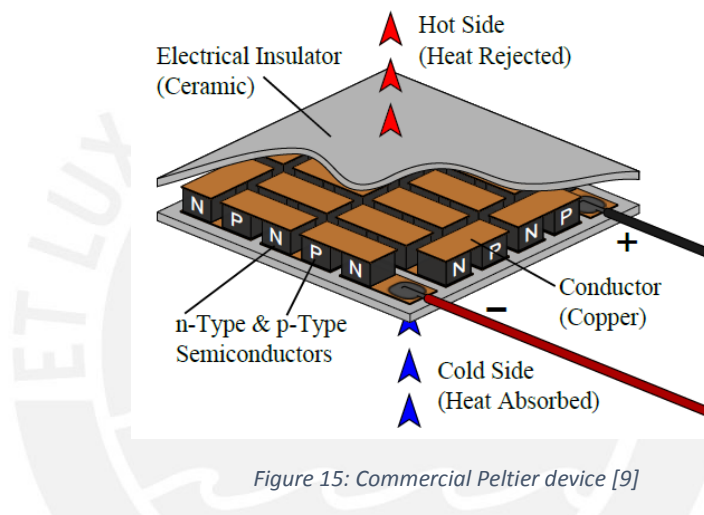


Figure 15: Commercial Peltier device [9]

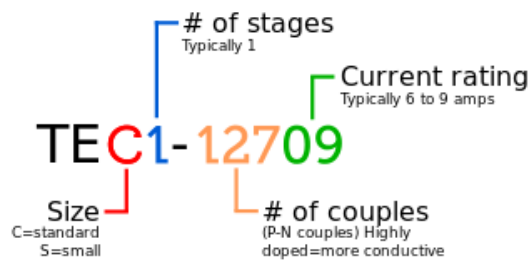


Figure 16: Universal identification specification of Peltier devices [15]

2.3 Methods for measuring resistivity

2.3.1 Four point probe measurement

One of the methods used to measure the resistivity on materials (conductors and semiconductors) is the four-point probe method. This method is widely used in device design and manufacturing [16]. This method uses four probe tips equally spaced (linear) which are in contact with the surface of the sample. The exterior tips (1 and 4) shown in Figure 17 are used to inject current into the sample, while the inner tips (2 and 3) are used to measure the voltage through the surface of the sample [17]

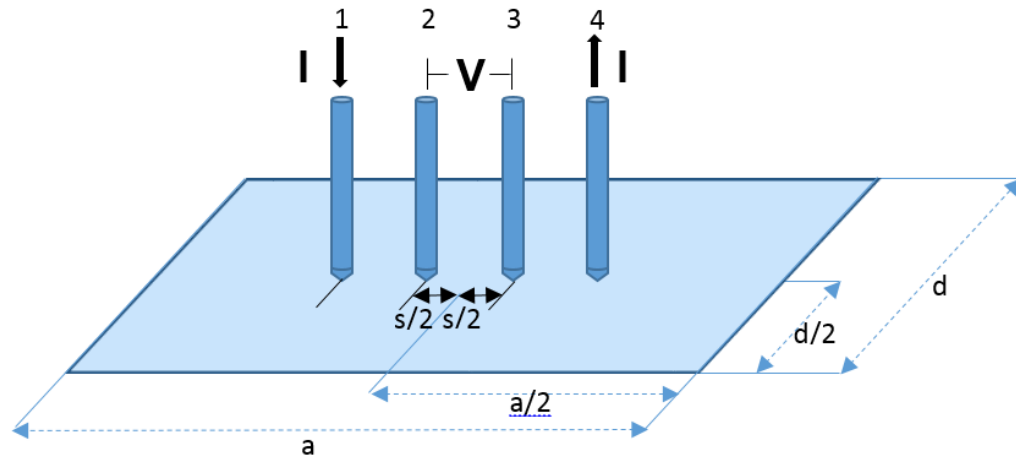


Figure 17: Four point probe schematic [18]

The relationship is:

$$\rho = \left(\frac{\pi}{\ln 2}\right) * \left(\frac{V}{I}\right) * d * k$$

Equation 7: Resistivity due to the four probe measurement

Where:

ρ : resistivity

V : voltage measured between 2 and 3

I : current injected between 1 and 4

d : thickness of the wafer sample

k : correction factor, based on the relationship between the diameter of the tips and the diameter of the wafer; and on the relationship of the wafer thickness and the separation between the tips [19].

$$k = 4\pi \left[\ln \frac{\alpha_1 \alpha_2}{\alpha_3 \alpha_4} + \ln \frac{\beta_1 \beta_2}{\beta_3 \beta_4} \right]^{-1}$$

Equation 8: Perloff correction factor [19]

Where:

$$\alpha_1 = (v_2 - v_4)^2 + (u_2 - u_4)^2$$

$$\alpha_1 = (v_2 + v_4)^2 + (u_2 - u_4)^2$$

$$\alpha_1 = (v_2 - v_1)^2 + (u_2 - u_1)^2$$

$$\alpha_1 = (v_2 + v_1)^2 + (u_2 - u_1)^2$$

And

$$\beta_1 = (v_3 - v_1)^2 + (u_3 - u_1)^2$$

$$\beta_1 = (v_3 + v_1)^2 + (u_3 - u_1)^2$$

$$\beta_1 = (v_3 - v_4)^2 + (u_3 - u_4)^2$$

$$\beta_1 = (v_3 + v_4)^2 + (u_3 - u_4)^2$$

Where v_n and u_n coordinate points where the tips are located on the surface of the wafer sample [19].

The use of four tips eliminates the errors due to the tips, the conductor's resistance, and the contact resistance between the metal tip and the semiconductor sample.

2.3.2 Van der Pauw method

Another technique to measure the resistivity in semiconductors is the van der Pauw method [20]. This method like the previous one, uses four tips; but the difference is that this method locates the tips at the boundary of the sample, injects current by two adjacent tips and measures the voltage with the other pair of tips.

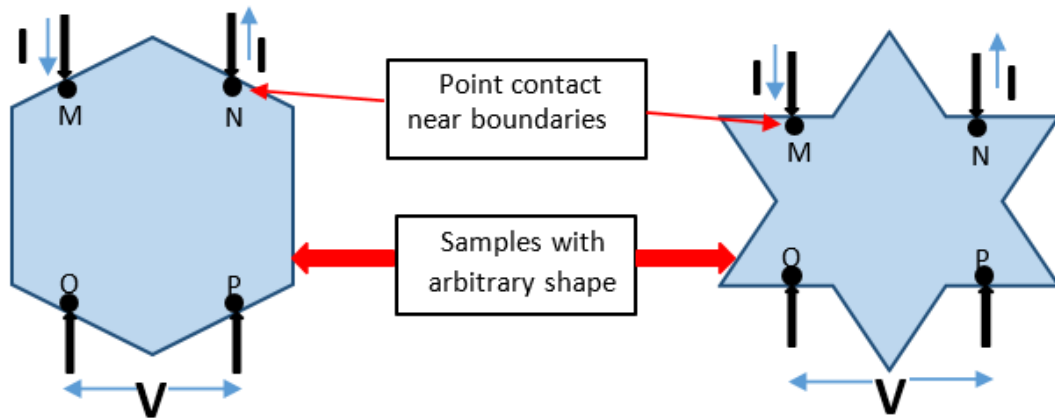


Figure 18: Van der Pauw schematic [3]

With those measurements, it is possible to calculate the sheet resistance. Subsequently, the van der Pauw relationship gives the possibility to calculate the resistivity of the material.

The van der Pauw method is one of the most utilized measurements methods for the evaluation of electrical properties such as resistivity, Hall coefficient, carrier density and mobility. It also allows to

measure samples of arbitrary shape with high precision when the following conditions are satisfied [21]:

- The point contacts are placed at the edges of the sample
- The sample quality has to be homogenous and it's thickness must be constant
- The sample must not have any isolated holes
- The area of contact between the tips and the sample is sufficiently small

The van der Pauw relationship expresses the connection between the resistances measured when the voltage and current contacts are interchanged as shown in Equation 9

$$\exp\left(\frac{-\pi d}{\rho} R_{MN,OP}\right) + \exp\left(\frac{-\pi d}{\rho} R_{NO,PM}\right) = 1$$

Equation 9: Van der Pauw Relationship [20]

Where:

$R_{MN,OP}$: The resistance equal to the potential differences between contact M and N divide by the current passing through contacts O and P

$R_{NO,PM}$: The resistance equal to the potential differences between contact N and O divide by the current passing through contacts P and M

ρ : Resistivity

This method is quite useful for samples whose thickness is too small compared to its length and width.

2.3.3 Including the distance of the tips into the calculations

Although the van der Pauw method is used to calculate the resistivity of materials following some conditions. One of them states that the tips must be on the boundaries of the sample [22]. There is no solution for the van der Pauw method when the tips are at arbitrary positions on the sample. The challenge is that locating the tips of a very small area at the very edge of the sample while ensuring mechanical stability of the contact [23]

Lim et al. [23], developed a modified form of the van der Pauw relationship (Equation 10) when the contacts are placed at arbitrary locations on the surface of the sample. In this case the boundaries of sample are at infinite distance from the contacts. Which means that the location of the probes are far away from the boundaries.

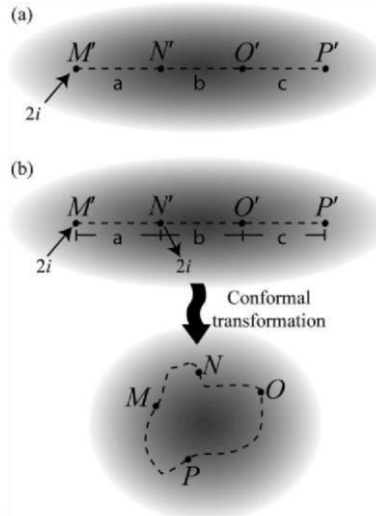


Figure 19: Modified Van der Pauw [23]

The modified van der Pauw relationship is formed by mapping the contacts from an infinite plane to a semi-infinite plane. In this relationship, the transformation maps the collinear contacts on the infinite plane (Figure 19a) onto an infinite plane with arbitrary contacts on the surface of the sample (Figure 19b).

The new relationship obtained is:

$$\exp\left(\frac{-2\pi d}{\rho} R_{M'N',O'P'}\right) + \exp\left(\frac{-2\pi d}{\rho} R_{N'O',P'M'}\right) = 1$$

Equation 10: Modified Van der Pauw relationship [23]

This new expression differs from the previous (Equation 10) only in the factor of two in the exponential terms. This expression becomes accurate when the contacts are placed at infinity distant from the boundaries [23].

This technique is closely related to the van der Pauw method but has the advantage that the tips can be placed at any position at an infinity distance of the boundaries where the results becomes accurate.

Another expression given in this paper [23] is the relationship between the resistance in terms of the resistivity, the thickness and the distances between the tips (Equation 11).

$$R_{M'N',O'P'} = \frac{\rho}{2\pi d} \ln \left[\frac{(a+b)(b+c)}{b(a+b+c)} \right]$$

Equation 11: Resistance in terms of resistivity and distance between tips [23]

Where:

$R_{M'N',O'P'}$: The resistance equal to the potential differences between contact M' and N' divide by the current passing through contacts O' and P.

ρ : Resistivity

a, b, c : Results of mapping the distance between the tips of an infinity plane onto infinite plane such that M', N', O' and P' are collinear points (Figure 19b). The distance between M' and N' is " a ", " b " is the distance between N' and O' , " c " is the distance between O' and P' .

For the present work, it has been working with the modified van der Pauw method to determinate the resistivity of the samples.

The conformal transformation that maps any four points in a plane to new general locations in the plane is done with the Moebius transformation (Equation 12) from the complex v -plane to the complex w -plane, $w = f(v)$ where $f(v)$ is the function:

$$w = \frac{p_0v + p_1}{p_2v + p_3}$$

Equation 12: Moebius transformation from v -plane to w -plane [23]

Where:

p_0, p_1, p_2, p_3 : are four (complex) parameters obtained by specifying the destinations in the w -plane of four points in the v -plane.

3 Connections and Program

3.1 Connection setup and devices

For the present work a program has been developed in LabVIEW and the modified van der Pauw method was implemented in it. The Keithley libraries has been used, in order to interact with the different Keithley devices. In order to perform the task required, the system implemented has the following devices: one Keithley 2400 Source Measure, one Keithley 2001 Multimeter, one Keithley 2002 Multimeter, one Keithley 2000 Multimeter and one Keithley 7001 switch/control, all connected through the IEEE-488 or commonly named GPIB. The libraries from the Vision and Motion of the LabVIEW allow the interaction with the camera through USB port. The interconnection diagram for the modified van der Pauw measurement is shown in Figure 20, the interconnection between the Keithley devices and the channel selector is shown in the Figure 21 and the station for the measurement is shown in the Figure 22.

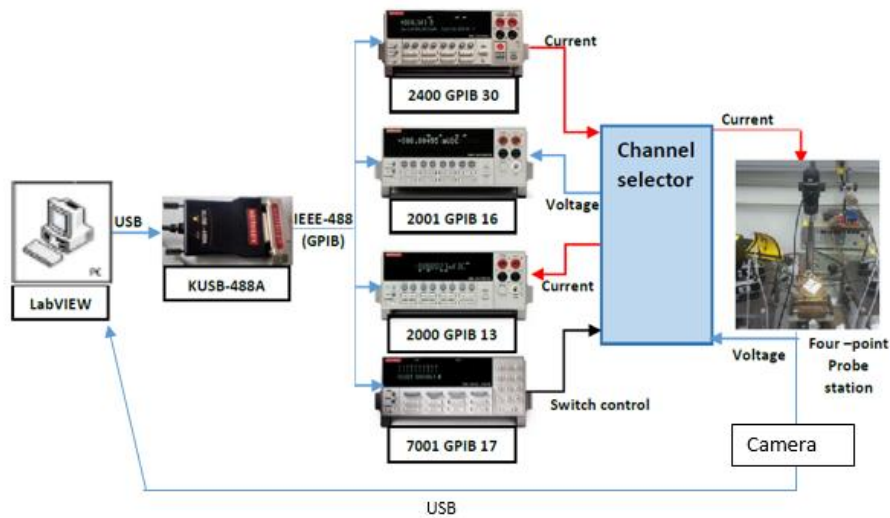


Figure 20: Interconnection diagram for the modified van der Pauw measurement



Figure 21: Interconnection between Keithley devices and channel selector

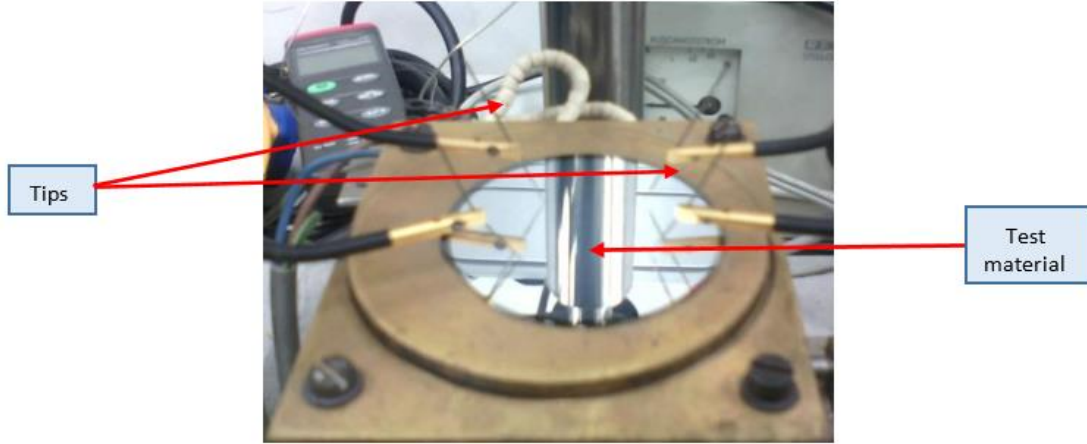








Figure 22: Four probe station

The description of the Keithley devices are shown in the Table 3.

Table 3: Keithley devices description

	<p>Keithley 2400 Source Measure Unit (GPIB 30) This source can provide with precision voltage and current sourcing with a 6 ½ resolution.</p>
	<p>Keithley 2001 7 ½ - Digit High Performance Multimeter (GPIB 16) This device configured as a voltmeter, is perfect for users whose applications demand exceptional resolution, accuracy, and sensitivity combined with high throughput. With a 7½ resolution.</p>
	<p>Keithley 2002 8 ½ - Digit High Performance Multimeter (GPIB 15) This device configured as a voltmeter, is perfect for users whose applications demand exceptional resolution, accuracy, and sensitivity combined with high throughput. With an 8½ resolution.</p>
	<p>Keithley 2000 6 ½ - Digit Multimeter (GPIB 13) Configured as an ammeter is a fast, accurate, and highly stable instrument that's as easy to operate as it is to afford.</p>

	<p>Keithley 7001 switch/control Mainframe 80-channel (GPIB 17)</p> <p>This device is used as general switching and test configuration for applications involving high and low current and voltage, temperature measurement, High frequency testing and RF.</p>
	<p>Keithley KUSB – 488A IEEE – 488 GPIB Interface</p> <p>This device turns any computer with a USB port into a functional GPIB controller. The product is USB 2.0 compliant and has an IEEE data transfer rate upwards of 1.5 MB/s through the USB port.</p>

3.2 LabVIEW program structure

The developed LabVIEW interface interacts with the devices previously described. The necessary data such as voltage and current is collected by Keithley devices using the Keithley block. Afterwards, the resistance of the material is obtained as well the resistivity. Furthermore, the V-I diagram is plotted on the interface. It also offers the possibility to show the data in an Excel sheet.





3.2.1 Keithley VI modules:

The VI modules from the Keithley in LabVIEW which were used in the present work as well as a brief description are the following ones:

3.2.1.1 Keithley 2400 Source Meter Unit

The VI modules from the Keithley 2400 Source Meter Unit used in the present work are shown in Table 4 :

Table 4: Keithley 2400 VI Modules used in the program

VI Icon	Module	Description
	Keithley 24XX.lvlib:Initialize.vi	Establishes communications with the instrument and optionally performs an instrument identification query and/or an instrument reset.
	Keithley 24XX.lvlib:Enable Output.vi	Enables the source to the output the assigned voltage or current to the device under test.
	Keithley 24XX.lvlib:Configure Output.vi	Configures whether the output is in terms of current or voltage, the amplitude of the output and compliance settings.
	Keithley 24XX.lvlib:Close.vi	Performs an instrument error query before terminating the software connection to the instrument.

Some important VI modules are the “Configure Output” VI-module and the “Enable Output” VI-module. The parameters for the “Configuration Output” VI-module are shown in the Figure 23, a brief description and set values of the parameters in the Table 5. For the “Enable Output” VI-module are shown in the Figure 24

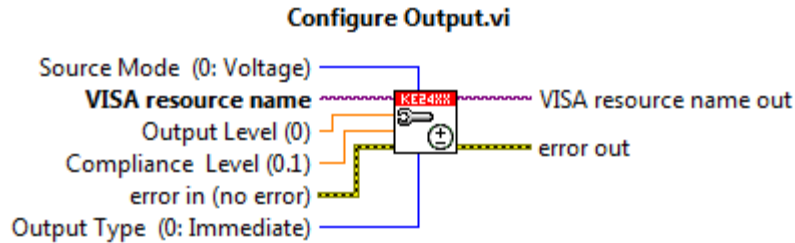


Figure 23: Configure Output VI-module

Table 5: Parameters and set values of the “Configuration Output” VI-module [24]

Parameter	Description
Source Mode	Selects the type of source mode to be configured, voltage or current. The possible values are: 0: Voltage(Default); 1: Current Set value: 1(Current Source)
VISA resource name	The reference of the instrument used, this was initially created by “Initialize” VI-module
Output Level	The desired value of current or voltage for the device, this depends on the type of source mode. The valid range is: Voltage: 5 μ V to 210 V Current: 50 pA to 1.05 A Maximum power source: 22 W Set value: Current 50 pA to 1.05 A
Compliance Level	Restricts the power that can be delivered to the device, this limits the not regulated value of the source. In this sense, for a current source the compliance is the voltage. Conversely, for a voltage source the compliance is the current. The valid range is: Voltage: \pm 210 mV to \pm 210 V Current: \pm 1.05 μ A to \pm 1.05 A Set value: Voltage was 2 V, 20 V and 200 V,
Output Type	Determinate if the source will be triggered or will immediately reach the “Output Level”. The possible values are: 0: Immediately(default) 1: Triggered Set value: 0-Immediately

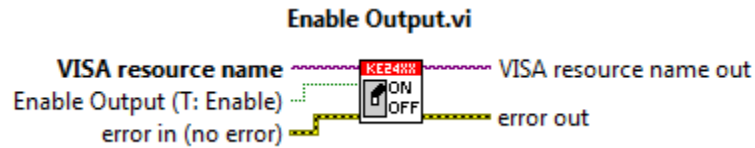


Figure 24: Enable Output VI-module




Table 6: Parameters of the “Enable Output” VI-module

Parameter	Description
VISA resource name	The reference of the instrument used, this was initially created by “Initialize” VI-module Set value: 30
Enable Output	Enable the source to output the assigned current value to the device. The possible values are: T: Enable(default) F: Disable Set value: T

3.2.1.2 Keithley 2001 Multimeter Unit

The VI modules from the Keithley 2001 Multimeter Unit used in the present work are shown in Table 7

Table 7: Keithley 2001 VI Modules

VI Icon	Module	Description
	Keithley 2001 DCV Config.vi	This VI is used to configure the settings for the dc voltage measurements.
	Keithley 2001 Single Read.vi	This VI is used to configure the measure layer (Trigger) and take a single measurement
	Keith 2001 Reset / Preset.vi	This VI for witch System allows the operator to send a “reset” or “preset” command to the unit

An important VI module is the “Keithley 2001 DCV Config” VI-module. The parameters of this VI-module are shown in Table 8 and the set values in Table 9.

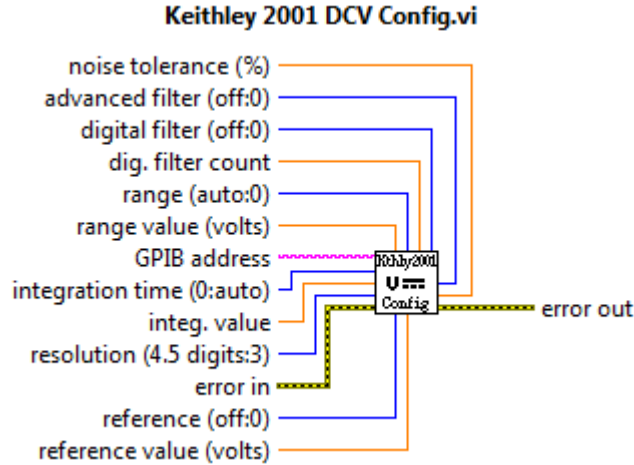


Figure 25: Keithley 2001 DCV Config VI-module

Table 8: Parameter of the “Keithley 2001 Config” VI-module

Parameter	Description
noise tolerance	The noise tolerance level is expressed as a percent of the last averaged reading. 1 (default)
advance filter	This selection is for an averaging filter with a noise window
digital filter	The digital filter is based on reading conversions. The displayed, stored or transmitted reading is simply an average of a number of reading conversions
dig. filter count	The filter count is the number of readings that are acquired and stored in the filter buffer for the averaging calculation
range	This command is used to manually select the measurement range
range value	This is the value of the range
GPBI address	The reference of the instrument used, physical address
integration time	This is the speed of the measurement.
intg. value	The value for the integration time.
resolution	Number of digits for the measurement
reference	This is used to establish a reference value for the specified function. The result will be the algebraic difference between the input signal and the reference value. The possible values are: 0: off 1: on
reference value	Is the value for the reference

Table 9: Set values for the “Keithley 2001 Config” VI-module





Parameter	Value	Description
noise tolerance	1	Noise tolerance 1%
advance filter	Off	Default value
digital filter	Off	Default value
dig. filter count	10	Default value
range	autorange	Default value
range value	1000	Default value
GPBI address	16	Address of the physical device.
integration time	Set to minimum	Measurement speed 0.1 PLC (medium speed)
intg. value	0.01667	1 PLC
resolution	4.5 digits	Default value
reference	Off	Default value
reference value	0	Default value

*1 PLC: power line cycle 16.67 msec for 60 Hz, 20 msec for 50 Hz and 400Hz

3.2.1.3 Keithley 2000 Multimeter Unit

The VI modules from the Keithley 2001 Multimeter Unit used in the present work are shown in Table 10.

Table 10: Keithley 2000 VI Modules

VI Icon	Module	Description
	Keithley 2000.lvlib:Initialize.vi	This VI passes the addressing information in the instrument descriptor to the instrument Open VI and returns the Instrument ID
	Keithley 2000.lvlib:Configure DC Current.vi	Configures the instrument to measure DC current
	Keithley 2000.lvlib:Data Read Single.vi	Reads a measurement or calculation from the instrument
	Keithley 2000.lvlib:Close.vi	Closes the I/O interface with the instrument

Some important VI modules are the “Configure DC Current” VI-module and the “Data Read Single” VI-module. The main parameters for the “Configure DC Current” VI-module are shown in the Figure 26. A brief description and set values of the parameters are shown in Table 11. The “Data Read Single” VI-module is shown in Figure 27. A brief description and set values of the parameters are shown in Table 13 and **Error! Reference source not found.**

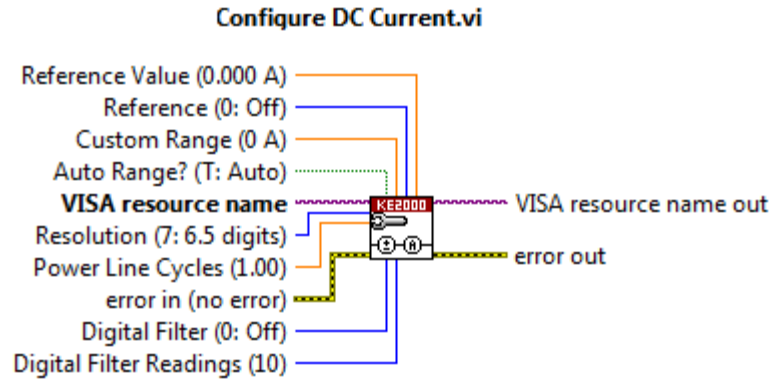


Figure 26: Configure DC Current VI-module

Table 11: Parameters of the “Configure DC Current” VI-module

Parameter	Description
Reference Value	Value of the reference, units in amps.
Reference	Enable the possibility to use the reference
Custom Range	Sets this to the absolute value of the expected reading and the instrument will adjust to the most sensitive setting that can measure that value
Autorange	Allows the instrument to adjust the range according to the inputs. The possible values are: T: Enable F:Disable
Visa resource name	The reference of the instrument used, this was initially created by “Initialize” VI-module
Resolution	Sets the number of digits to display
Power Line Cycles	The measurement speed
Digital Filter	Sets the mode of the digital filter function of the instrument. The possible values are: 0: Disable 1: Moving filter 2:Repeting filter
Readings	Sets the number of readings used in the instrument. The value default is 10 readings

For the thesis work, the values used for “Configure DC Current” VI-module are shown in the

Table 12: Set values for the “Configure DC Current” VI-module

Parameter	Value	Description
Reference Value	0	No reference value
Reference	0	Reference disable
Custom Range	0	No custom range
Autorange	T	Enable
Visa resource name	13	Address of the physical device.
Resolution	7	Seven number of digits
Power Line Cycles	1	Measurement speed 16.67 msec
Digital Filter	Off	No digital filter ,only one measurement
Readings	10	Not enable as a result of “Off” in the Digital filte

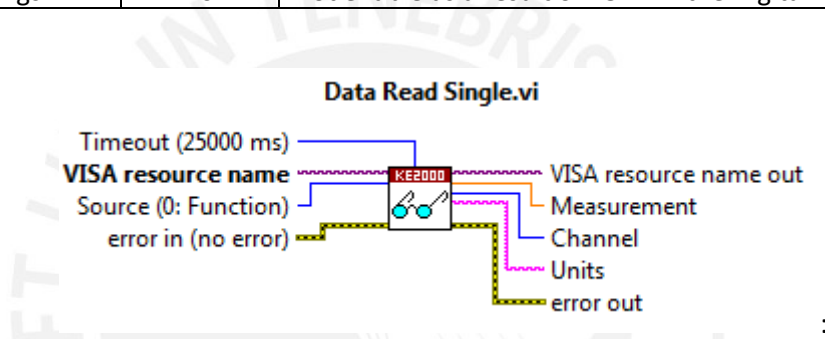


Figure 27: Data Read Single VI-module







Table 13: Parameters of “Data Read Single” VI-module

Parameter	Value	Description
Timeout	250	Sets the amount of time to wait for a measurement. Return error if the measurement is not returned within the amount of time. Chosen value time in milliseconds to make quickly
Visa resource name	13	The reference of the instrument used, this was initially created by “Initialize” VI-module. Address of the physical device.
Source	0	Sets the source measurement to read. Read the last instrument reading

3.2.2 State chart

The developed program uses state chart. The VI modules used in the work are shown in Table 14

Table 14: Statechat Modules

VI Icon	Module	Description
	Run Statechart	This VI executes the state chart to which this function is linked.
	State	Define a state, which is a unique in which the statechart can be.
	Region	Defines the area in which the user can place states and pseudostates.
	Initial	Specifies the first state that the enclosing region enters when the statechart enters that region.
	Terminal	Specifies a state that can terminate the execution of the enclosing region.
	Transition Node	Transitions define the conditions under which the statechart can move between states. By creating transitions between states, you specify how the statechart reacts to certain triggers or guards, which triggers cause the statechart to move to certain states, and in what order the statechart can move between states.

The main modules from the Statechart are “Run Statechart”, “State”, “Region” and the “transition node”. The parameter for the “Run statetchart” VI-module are shown in Figure 28, a brief description and set values of the parameters in Table 15.

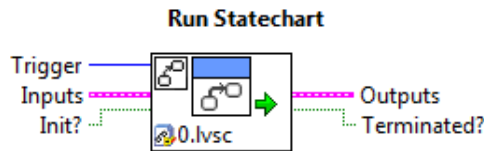


Figure 28: Run Statechart VI-module

Table 15: Parameters of “Run Statechart” VI-module

Parameter	Description
Trigger	Specifies the trigger to send to the statechart, the user can send only triggers that has been created by using the Edit Triggers and Groups dialog box
Inputs	Inputs specifies the input data to the statechart.
Init?	Restart the statechart from any initial conditions you provide
Output	Returns the output data from the statechart
Terminated?	Returns TRUE after the entire statechart finishes executing.

Figure 29 shows the parameters of the Statechart. The user can configure the Triggers, the inputs, outputs and the diagram.

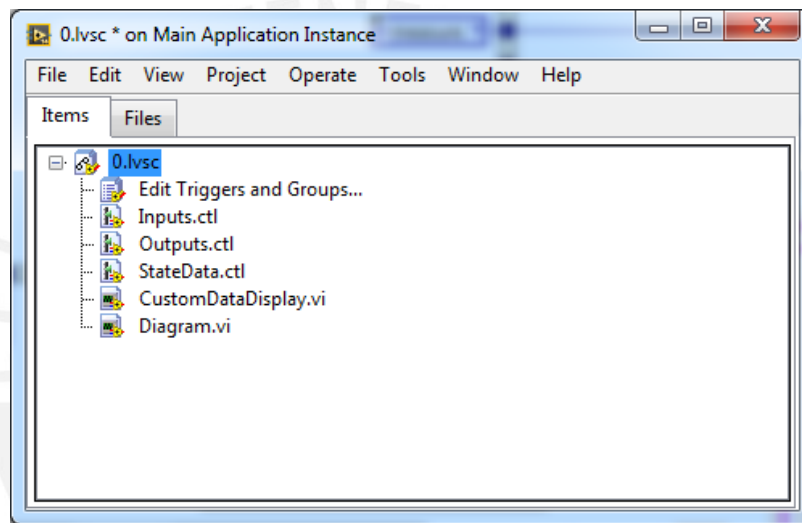


Figure 29: Statechart

The “State” module performs an action that will be configured inside of the “State” module itself. The user can access to the configuration by a double click on the “State” (Figure 30). The “Transition Node” also can perform an action, but its configuration is needed. The user can access to the configuration by a double click on the “Transition Node”, the user can configure the trigger (Figure 31) and the “Guard” (condition to execute the action).

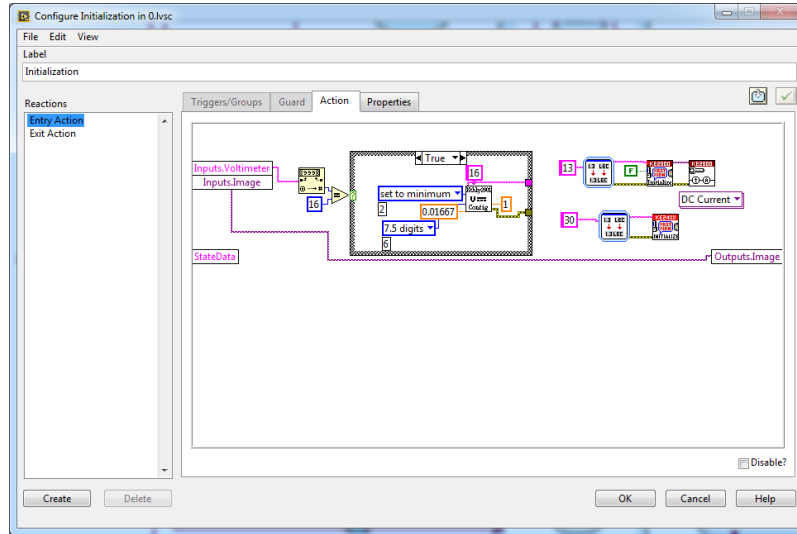


Figure 30: Action performed inside of the "Initialization" state

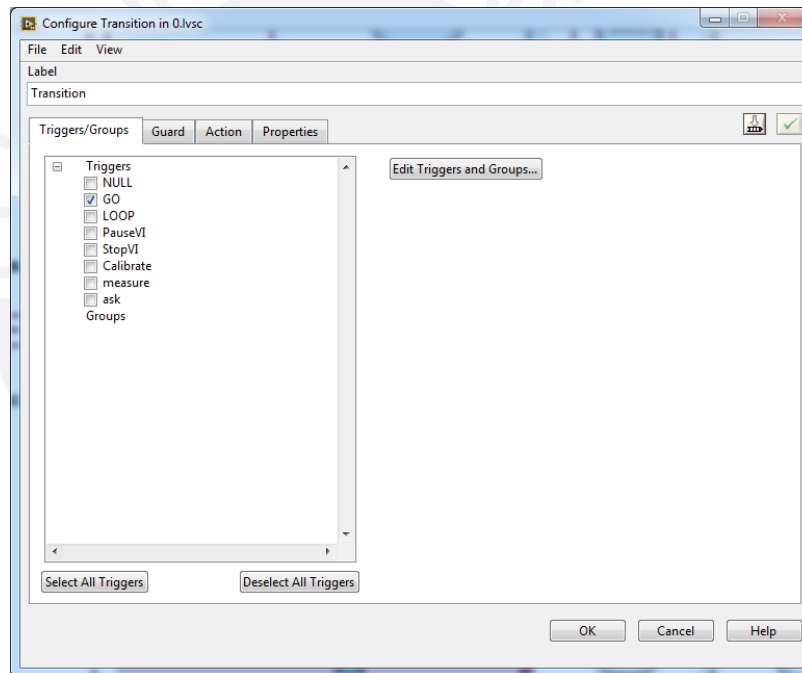


Figure 31: Configuration of the trigger of the "Transition Node"

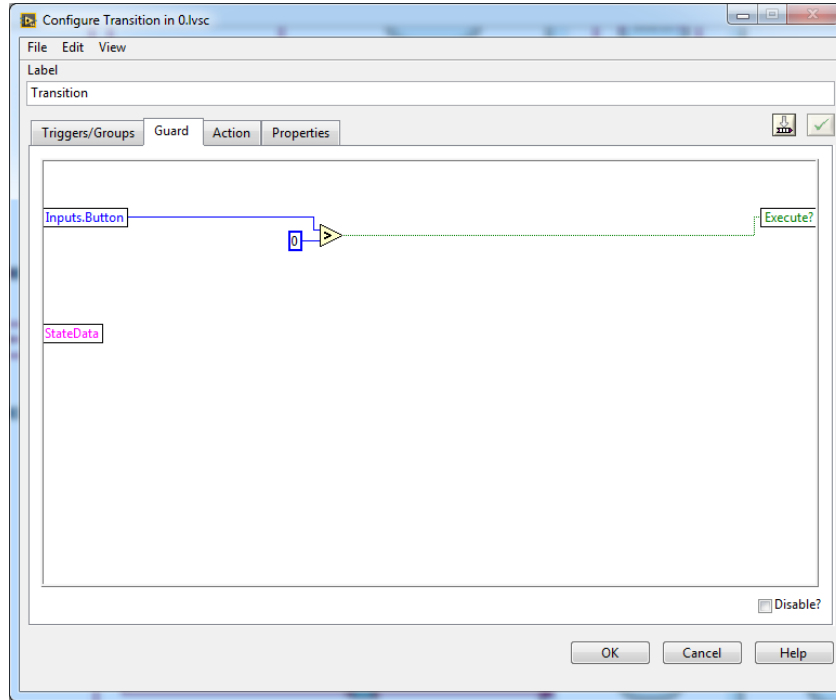


Figure 32: Configuration of the Guard of the “Transition Node”

Figure 32 shows the configuration of the guard of the “Transition Node”, the condition to execute the action is that the value of the Input.Button should be greater than “0”. Only when the condition to execute is true and both, the trigger of the “Transition Node” and the trigger created by the event case are the same, the action will be performed (Figure 33)

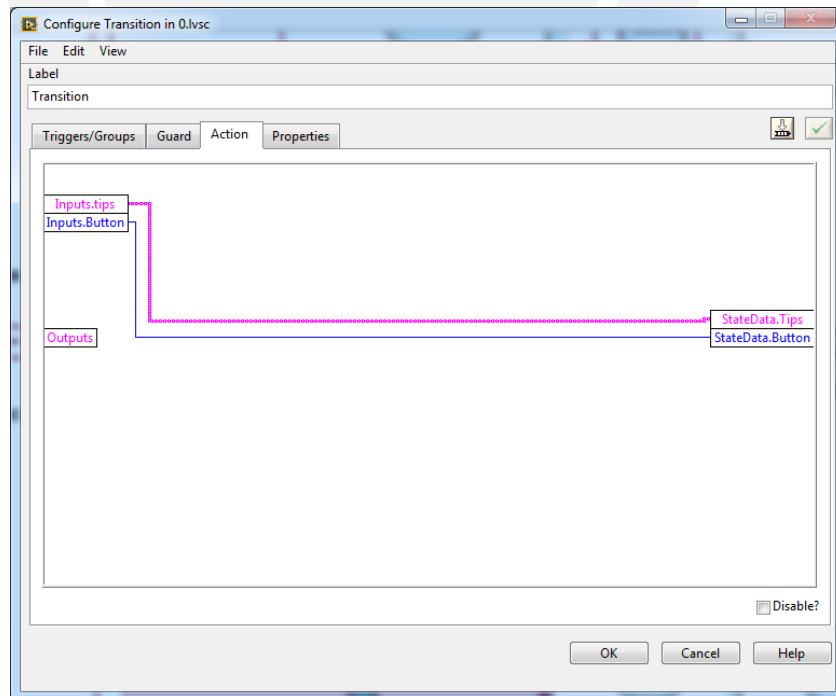


Figure 33: Performed action by the “Transition Node”

3.3 LabVIEW Program

The developed program fulfills the requirements of fast processing and easy programming. For the fast processing the program was developed in state chart.

3.3.1 State Chart

The developed state chart is shown in the Figure 34.

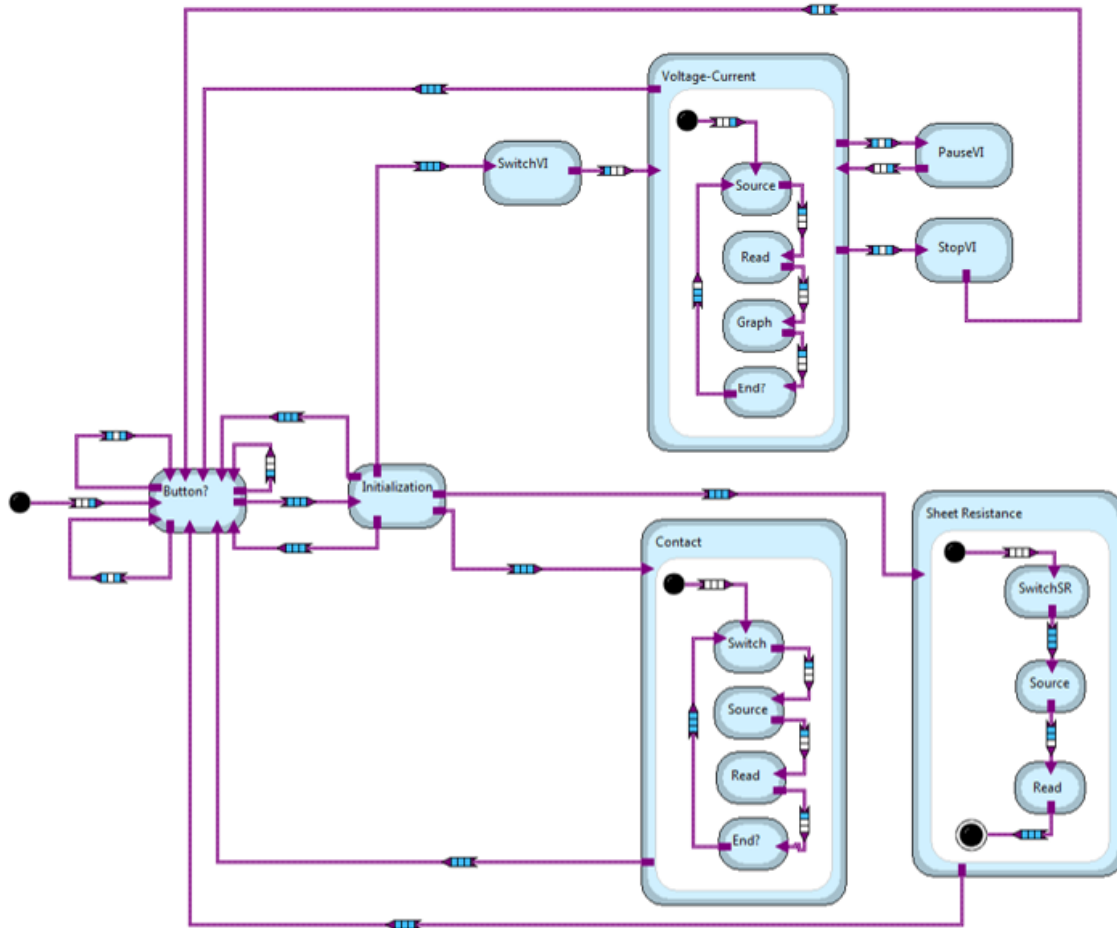


Figure 34: State Chart developed for the program

The program has three main sequence, which correspond to each tab of the graphical user interface (Contact, Voltage-Current, and Sheet Resistance) which will be described below.

3.3.2 Graphical user interface

The developed program has a user interface with three tabs, each tab performs a different task.

3.3.2.1 Contact and Info tab

The first tab (Figure 35) is the Contact and Info tab. Here, the user can evaluate whether the tips are in contact with the sample surface or not. This tab has six light indicators. Each light indicator changes to a bright color when the contacts between the two tips evaluated are right (flow of

current). In order to do not damage any sample, the evaluation is done with a current of one milliamp.

On the left bottom corner of the tab, there are two buttons. The “OK” button begins the evaluation of the contact between tips. The “Reset” button changes all the lights to a dark color, in order to perform again the evaluation of the contact between tips.

Next to the light indicators is the “Recommendations” part, which helps the user to understand the performance of the program, giving advices in order to make the interaction between user and interface easier. Next to the “Recommendations” are two images. The first image shows the Equation 10 used to obtain the resistivity (modified van de Pauw) with a reference image. The second image shows the tip distribution seen by the camera.

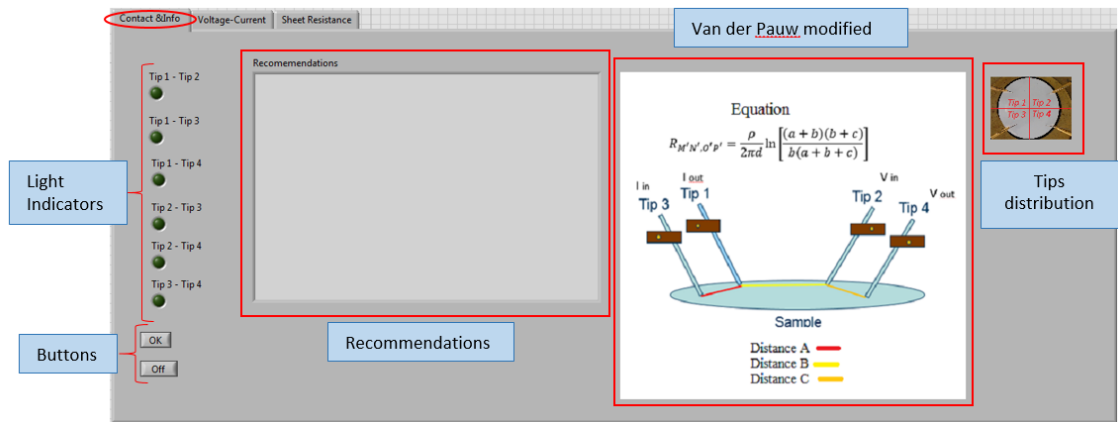


Figure 35: Contact&Info tab of the developed program

3.3.2.2 Voltage-Current tab

The second tab (Figure 36) is the voltage-current tab. First, in this tab the user might select from the menu voltmeter the one that will be used for the experiment. Additionally, the user can select tips where the current will be injected, as well as the tips to measure the voltage. At the upper part the values of the current that will be used in the experiment should be entered. The entered current data is in milliamps. If the value entered into the “Start” input is higher than the “Stop” input, the program will change automatically the values of the “Start” input with “Stop” input.

In this tab the measured data will be shown in a data table in real time. This table has three columns. The first one is the column of the current measured by the amp meter in milliamps.

The second column is showing the measured voltage in millivolts. The last column is for the resistance calculated by dividing the voltage and the currents measured in ohms. Finally there is an XY-Graph were all the data collected before will be plotted. The axis shown are the voltage on the X axis in millivolts and current on the Y axis in milliamps.

At the bottom left side of the Voltage-Current interface, there is a group of buttons which perform different actions. The “New” button starts a new measurement with new conditions (values of the current, selection of the tips and selection of the voltmeter) or not. If there is already data shown in the table or graph it will be erased and the new data will be entered into both the table and the graph. The “Add” button starts a new measurements with new conditions such as values of the

current, selection of the tips and selection of the voltmeter or with no new conditions. In this case, the data of the table and the graph will not be erased. The new data will be added to the previous data in the table and XY-Graph. The “Excel” button exports the all data obtained into an Excel sheet. The “Reset” button deletes all the data in the table and XY-Graph. The “Pause” button stops the actual measurement. Thus, for safety reasons the current source will be turned off and the switching will be stopped. After the pause, the measurement continues with the previous tip configuration. The “Stop” button turns off the current, stops the switching and allows the user the possibility to start a new measurement with all new conditions in Voltmeter, tips selection and values for the current.

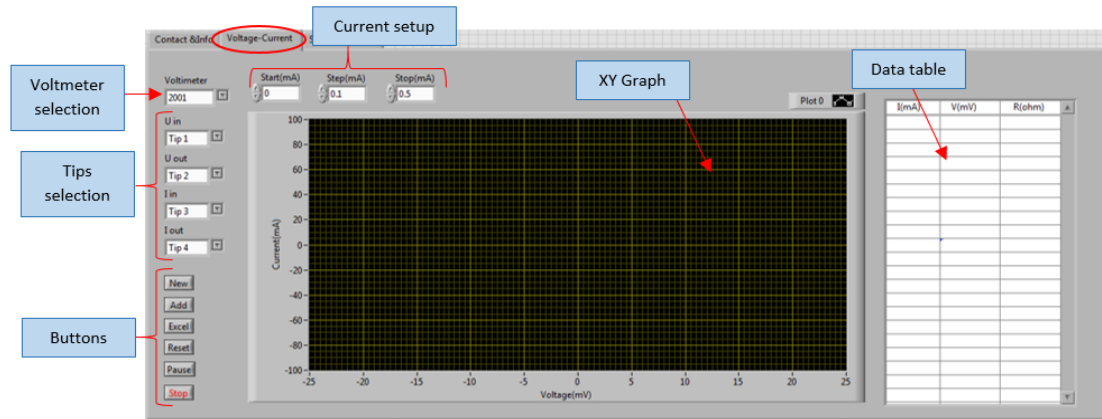


Figure 36: Voltage-Current tab from the developed program

3.3.2.3 Sheet resistance tab

The last tab (Figure 37) is the Sheet resistance tab. In this tab, the calculation of the resistivity of a sample is performed and the result is shown in a table. On the left upper side there is the tips selection. The user can choose the tips where the current will be injected, as well as the tips to measure the voltage. The tip configuration is located on the left side of the interface. At the upper part there are more input boxes. In the first one, the user should entered value of current that will be used during the experiment. The value of the current is in milliamps. In order to not spoil the sample the default value for the current is one milliamp. The second one is the “Compl(V)” box. In this box the user can choose from the menu one of the three pre-defined compliance voltage which are of 2 V, 20 V and 200 V or the fourth option “Free”, this “Free” option allow the user to enter any value for the compliance up to 210 V on the “Manual Compl Volt(V)”. This value is the limit of the Keithley device. The “Manual Compl Volt (V)” box is on right side of the “Compl(V)” box. The compliance limit acts as a power limiter for the device under test (DUT) and is set to protect it from damaging currents or voltages [24].

The next three boxes are for the distance between the tips. The data of these are in millimeters. The different distances belong to the separation between the tips according to the sequence shown in Figure 19b. The “Distance A (mm)” is the distance between the input current tip and the output current tip, the “Distance B (mm)” is the distance between output current tip and the positive voltage tip, the “Distance C (mm)” is the distance between the positive voltage tip and the negative voltage tip. These distances are used to calculate the measurement of the resistivity. The next box

is the “thickness nm)”, where the user introduces the thickness of the sample that is going to be analyzed. The thickness should be introduced in nanometers.

On the right side of the tab, there is the current image of the camera used to acquire the distances between the tips.

On the left side of this tab, there are buttons that perform different actions. In order to get a good measurement for the distances between the tips, is necessary to calibrate the image. The “Calibrate” button allows the user to adjust the image prior to the measurement of the sample. This button opens a menu that allows the user to use the values of the default calibration or make a new calibration (Figure 38). If the user selects the option “New”, the current image will be displayed on a new window (Figure 39). In order to calibrate the image, the user should put a calibration ruler, adjust the focus, draw a line of 5 cm and press enter. The user has the possibility to save this new calibration as default calibration. The “Measure” button help the user to verify if the calibration is correct or not. If the user select the “Measure” button, the current image will be displayed on a new window, the user can draw a line of a known distance by clicking on two points and press enter. The units of the measurement are in millimeters and it is shown in a small window. In order to get a correct measurement of the resistivity, it is only allowed to perform a measurement after the calibration of the image. The “New” button acquires the measure values for the voltage and current. It reads the “Current (mA)”, “Compl (V)”, “Thickness” and it displays a window with the current image, where the user can select the sequence of tips previously set on the tip selection. Then acquire the voltage and current data and calculate the resistivity of the sample. The “Add” button gives the user the possibility to add a measurement with two different options: “Distance”, where the user select again a new sequence points, or the “Current” option where the user can change the value of the current for the experiment and remain the values of the distance between the tips. The “Excel” button exports the data obtained to an excel sheet. There is a table in which all the data will be shown (Figure 40). The “Reset” button reset the obtained data shown on the table.

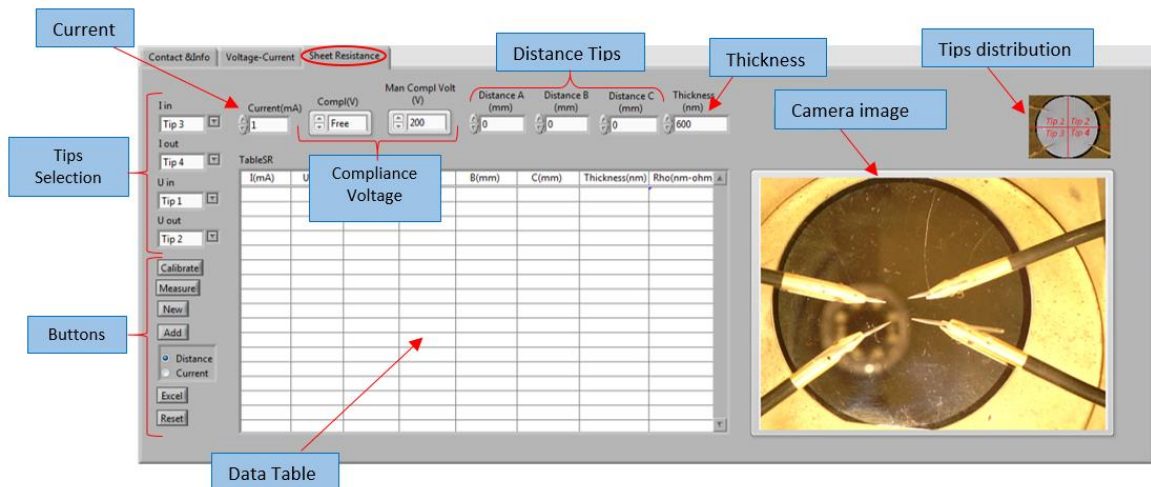


Figure 37: Sheet Resistance tab from the developed program

3.3.3 Description of the program

The program was developed using LabVIEW, the task requirements were:

- . Show a graphic with the current-voltage diagram
- . Show in a table the obtained data (Voltage, Current and the Resistance calculated)
- . Allow the user to introduce the current range and the step size of the current change
- . Allow the user to choose the tips it wants to use to perform the experiment
- . Obtain the resistivity of a sample
- Obtain the distance between the tips through a camera
- . Export all the data obtained to an excel sheet

When the program start, the state chart does the same and waits for an event that changes one of the inputs of the state chart (Figure 41). All the buttons from the tabs are matched with a determined event, the event will generate a number and trigger for the state chart (Table 16). After the user has pressed a button, the number and trigger generated by the event will remain for 100 ms.

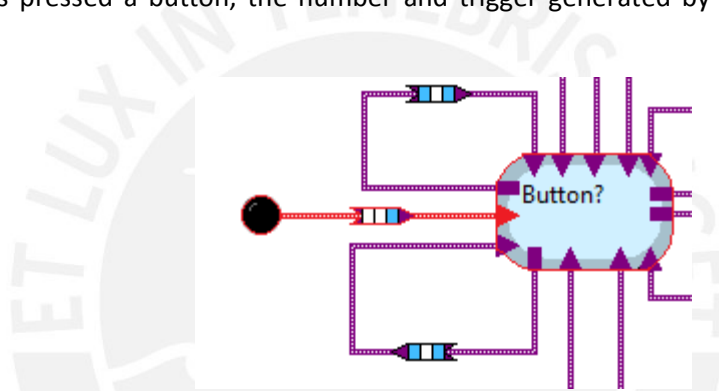


Figure 41: Sequence at the beginning of the program

Table 16: Number (first input of the state chart) and process to perform

Number	Trigger	Process to perform
1	GO	New voltage-current graph and table
2	GO	Add a voltage-current graph and table
3	GO	Create an excel with the voltage current data
4	GO	Reset the table and graph of the voltage current tab
5	GO	New row for the sheet resistance table
6	GO	Add a row for sheet resistance table
7	GO	Export the sheet resistance table to excel
8	GO	Reset the sheet resistance table
9	GO	Initialize the contact evaluation
10	GO	Change from bright to dark color the light indicators
-	PauseVI	Pause the measurement of the voltage-current
-	StopVI	Stop the measurement of the voltage-current
-	Calibrate	Help the user to calibrate the image
-	measure	Measure the distance between two points

3.3.3.1 Contact and Info tab

When the program starts, the “Contact&Info” tab is shown by default. This tab has six light indicators, two buttons, two images and recommendations. With the selection of the button “OK”, the user starts the evaluation of the contact between the tips. During this process, the program injects one milliamp of current in one tip and it measures the current in another tip following a predefined sequence. If the measured current is not in the range of 5 % tolerance the light indicator is not going to change to a bright color. The predefined sequence and the flow diagram are shown in Figure 42.

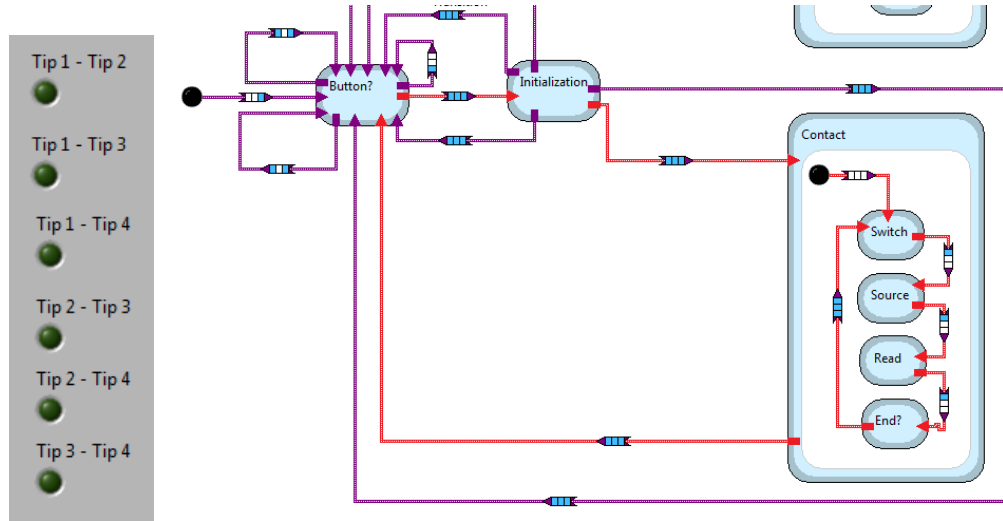


Figure 42: Predefined sequence and flow diagram of state chart for “Contact&Info”(Red lines)

The evaluation of the contact starts when the user press the button “OK”. This event will generate a number and trigger (Figure 43).

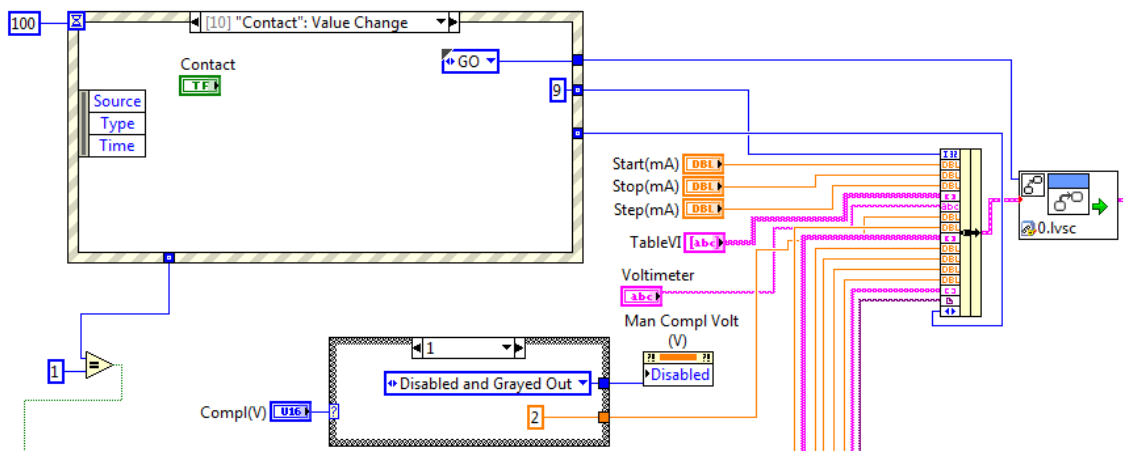


Figure 43: Event Case structure showing the new value for the input and trigger of the state chart

Now the state chart has an input and trigger, with number and trigger the state chart recognize which button was pressed, to pass the “Initialization” state the program performs a comparison, the first comparison is between the trigger generated by the event case and the trigger of the transition node (Figure 44), the second comparison is performed in “Guard” tab of the transition node between the number created by the event case and the value of 0 (Figure 45).

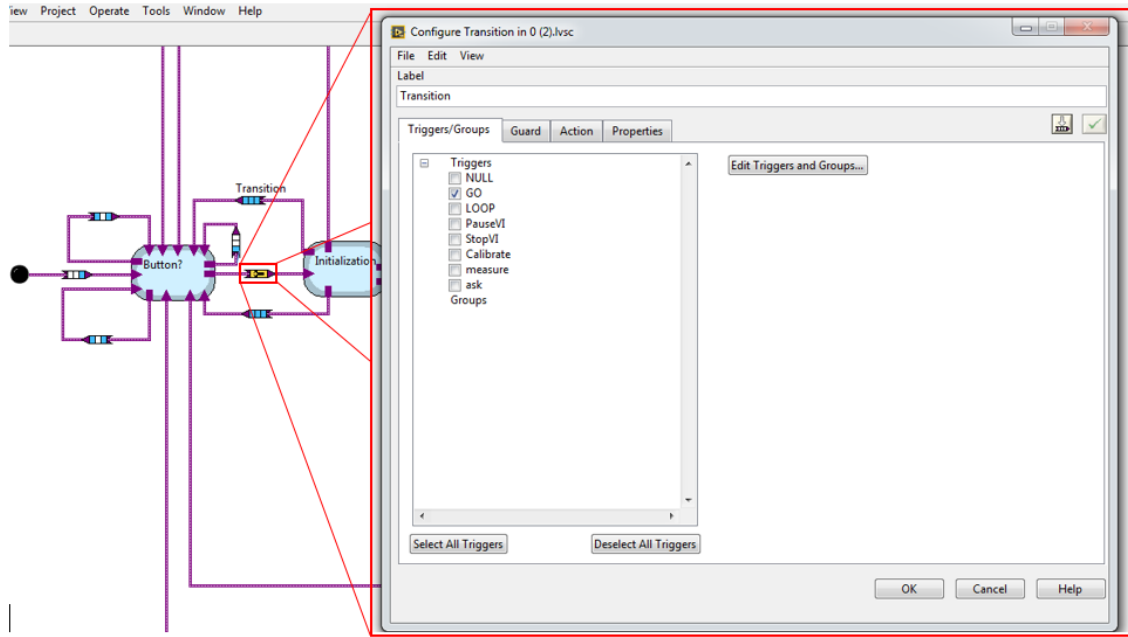


Figure 44: Trigger of the “Transition Node” located between “Button?” and “Initialization” states

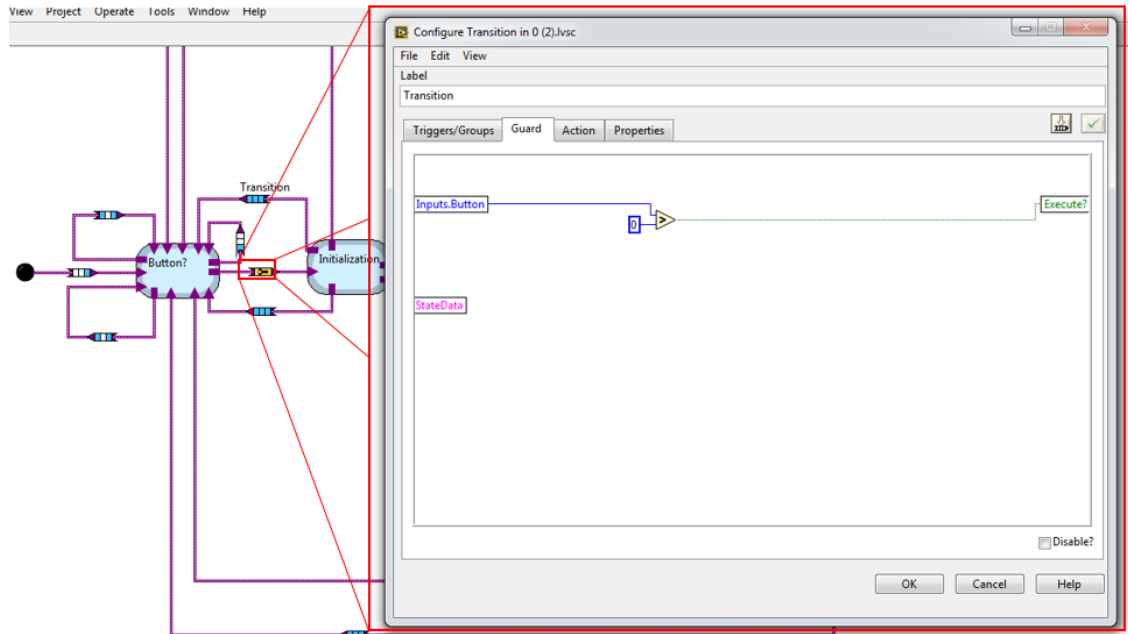


Figure 45: Guard of the “Transition Node” located between “Button?” and “Initialization” states

Inside of the state “Initialization”, the communication with the Keithley devices are initialized (Figure 46)

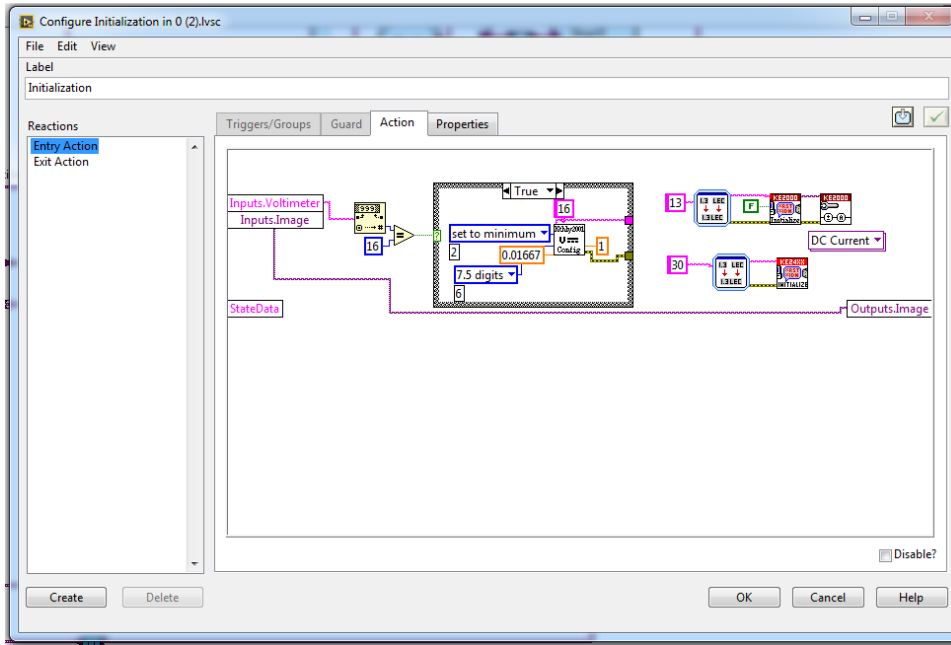


Figure 46: Initialization of the communication with the Keithley devices

With the communication initialized, the program evaluates which action should be performed. In order to pass to the next state, the “Guard” tab of the transition node uses the number previously created in the event structure to identify the process to perform (measure of resistivity, evaluate contacts, etc.). In Table 17 are shown all the buttons created by the event case (the tab and the number and trigger).

Table 17: Numbers and triggers matched to the buttons

Button	Tab	Number	Trigger
New	Voltage-Current	1	GO
Add	Voltage-Current	2	GO
Excel	Voltage-Current	3	GO
Reset	Voltage-Current	4	GO
New	Sheet Resistance	5	GO
Add	Sheet Resistance	6	GO
Excel	Sheet Resistance	7	GO
Reset	Sheet Resistance	8	GO
OK	Contact and Info	9	GO
Reset	Contact and Info	10	GO
Pause	Voltage-Current	-	PauseVI
Stop	Voltage-Current	-	StopVI
Calibrate	Sheet Resistance	-	Calibrate
Measure	Sheet Resistance	-	measure

The buttons with the numbers 3,4,7,8 and 10 do not perform any measurement. Therefore, after performing their actions (Table 16), they will return to the “Button?” state. The numbers “1” or “2”

are needed, in order to make a measurement in “Voltage-Current” tab. The numbers “5” or “6” are required, for the measurement of the “Sheet resistance” tab. To evaluate if the contact of the tips are correct, the number the “9” is needed. To perform the processes of “Pause”, “Stop”, “Calibrate” and “Measure” only their respective triggers are needed.

As previously written, the number “9” allows the evaluation of the contact between the tips. In the transition node a variable “SwitchC” is created with the value of “0”. This variable will change the tips combination to evaluate (Table 18). In the “Switch” pseudostate of the “Contact” state the performed action is the selection of the tips to evaluate (Figure 47).

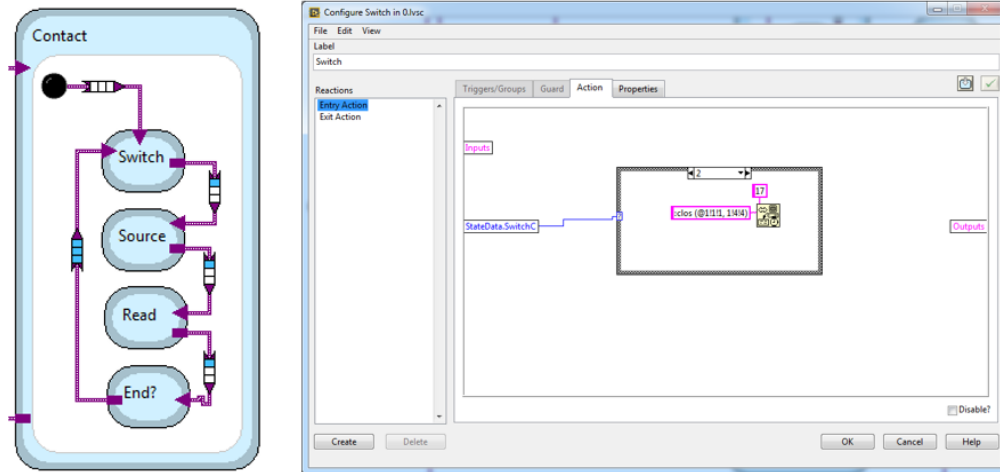


Figure 47: “Contact” region and the performed action in the “Switch” state

Table 18: Tips combination for the values of the variable “SwitchC”

Value of the “SwitchC” variable	Tips to evaluate
0	Tip 1 –Tip 2
1	Tip 1 –Tip 3
2	Tip 1 –Tip 4
3	Tip 2 –Tip 3
4	Tip 2 –Tip 4
5	Tip 3 –Tip 4

After the selection of the tips the current of 1 mA is injected. This process is performed by the “Source” pseudostate in the “Contact” state. The value is divided by 1000 due to the fact that the source is in amps and not in milliamps (Figure 48).

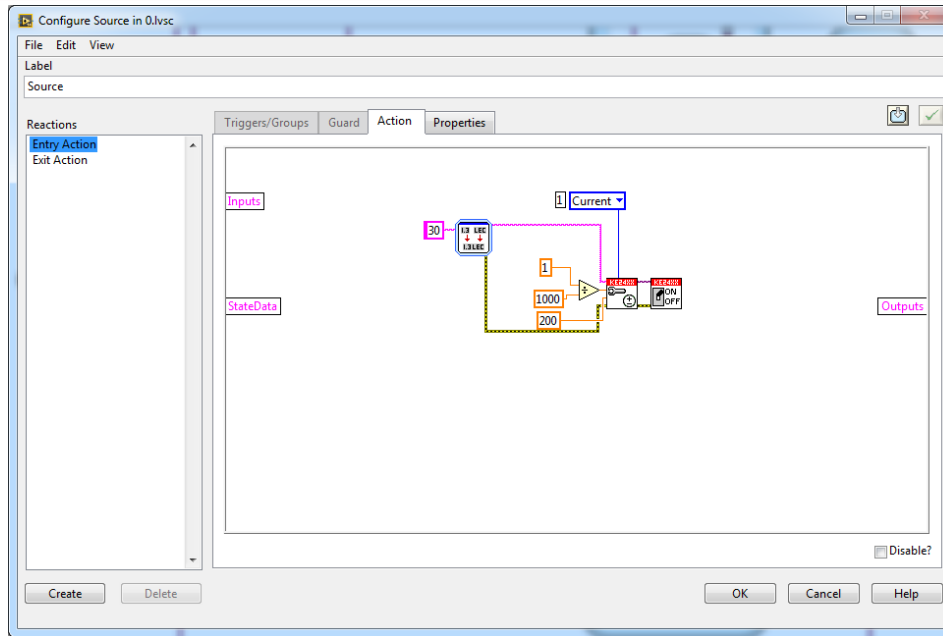


Figure 48: Action of the “Source” state of the “Contact” region

The next step is the reading process, it is a comparison between the current measured and one milliamp injected. The color of the light indicators will change, depending on the tolerance fulfillment (Figure 49).

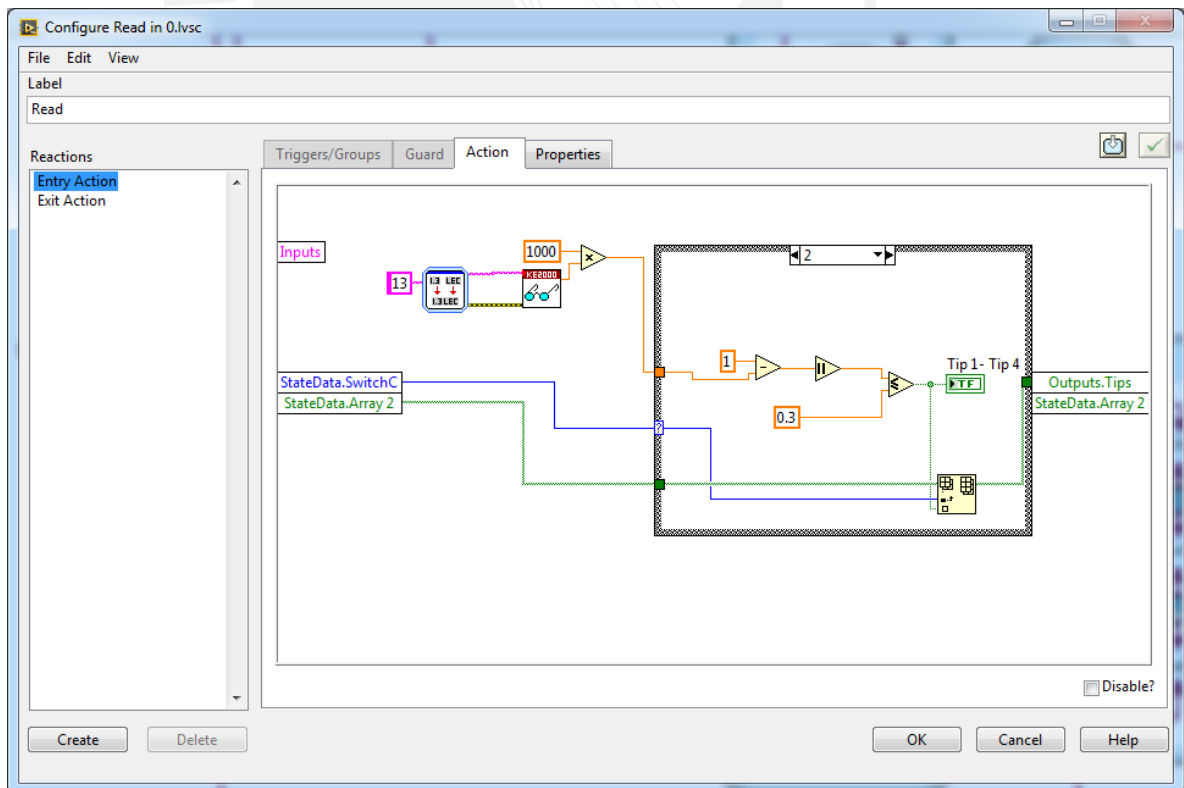


Figure 49: Action performed in the “Read” state of the “Contact” region

This process continues until the variable “SwitchC” is equal to the value of “6” (Figure 50). Whenever the “SwitchC” is equal to “6” the switching will be stopped and the source current will be turned off (Figure 51).

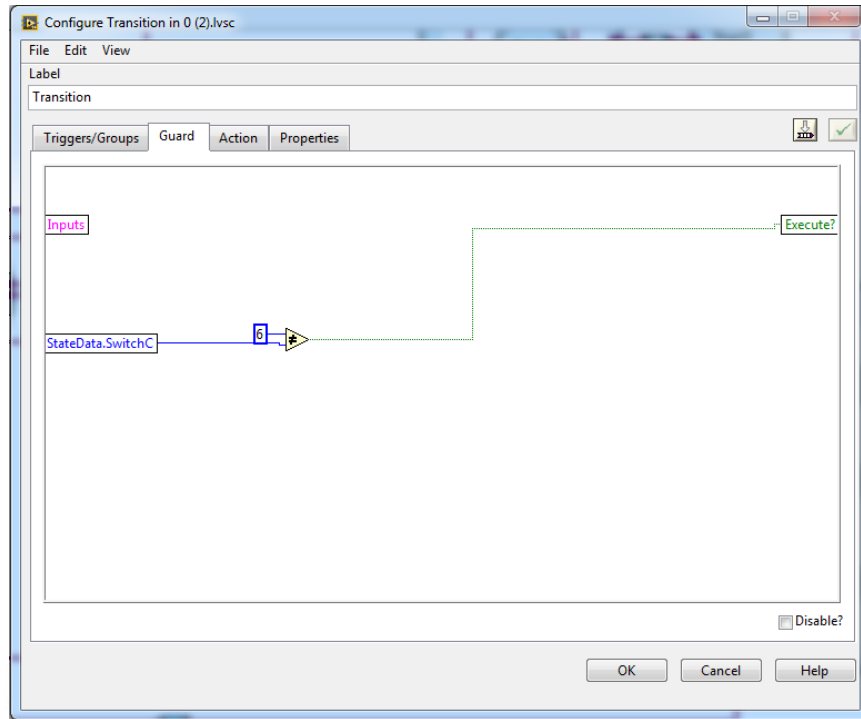


Figure 50: Comparison of the variable “SwitchC”

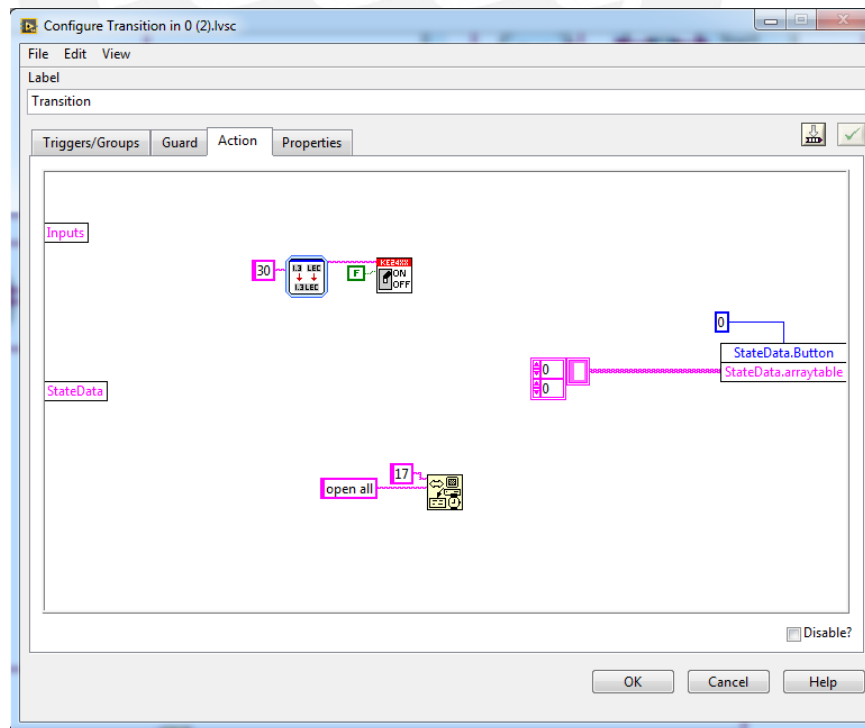


Figure 51: Transition between “Contact” region to “Button” state

3.3.3.2 Voltage-current

On the left side of the “Voltage Current” tab, there are five drop down menus (Figure 52a). In the first one the user can select the voltmeter. In the next ones, the user can select the tips, either for a new measurement or add another measurement, the user should be careful at the moment to choose the tips. The first two ones are for the selection of the tips for the voltmeter and the last two are for the selection of the tips of the source (Figure 52).

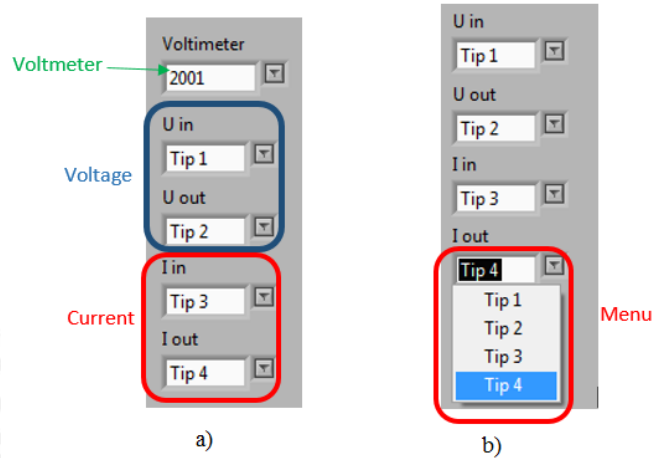


Figure 52: Voltmeter and tips selection (a) and drop down menu (b)

With the voltmeter and the tips selection the program can communicate with the Keithley devices and it performs the switching of the Keithley 7001. In order to make an easy inputs for the state chart, the tips selection were joined in an array (Figure 53), then it will be separated in the “Switch” state.

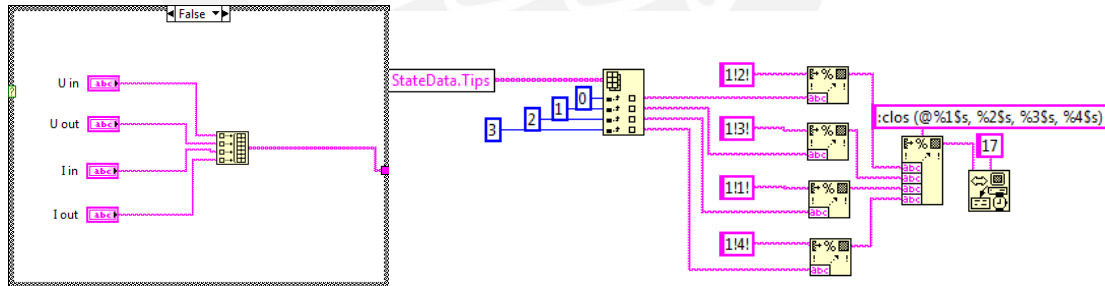


Figure 53: Tips selection into array and sending the tips selection to the Keithley 7001

The state chart reads the array and separates it in individual values, in order that the Keithley 7001 can understand the tips. These should have a determined format, this is done by the “Format into String” block and then joined together with “Format into String”, with the data ready, is send to the Keithley 7001 to perform the switching (Figure 53).

After setting all the values and selecting the tips, the user can start the program by pressing one of the buttons shown in the Figure 54. Each one of the menu buttons performs a different action.

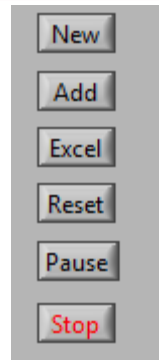


Figure 54: Menu buttons of the Voltage-Current tab

In the Figure 56 is shown the flow diagram of the state chart for the buttons. The actions of the “Excel” and “Reset” buttons are performed in the transition node from the “Initialization” state to “Button?” state, these action do not need to perform a measurement. A loop should be created, in order to get the measures for all the values of current created by the current configuration (Figure 55). This loop is built in the “Voltage-Current” region. To break the loop there are three possibilities: press the “Pause” button the “Stop” button or the condition in which the actual value of current is higher than the value of the “Stop” current. All the possibilities turn off the source and stop the switching in the Keithley 7001. Whenever the user presses the “Pause” button the loop will be broken. By pressing this button again, the user has the possibility to return to this loop and continue with the measurement. It is going to start with the last set values of current and switching. Pressing the button “Stop” will stop the measurement.

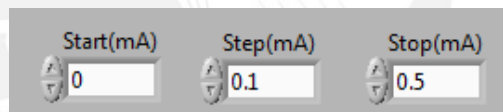


Figure 55: Default values for the current configuration

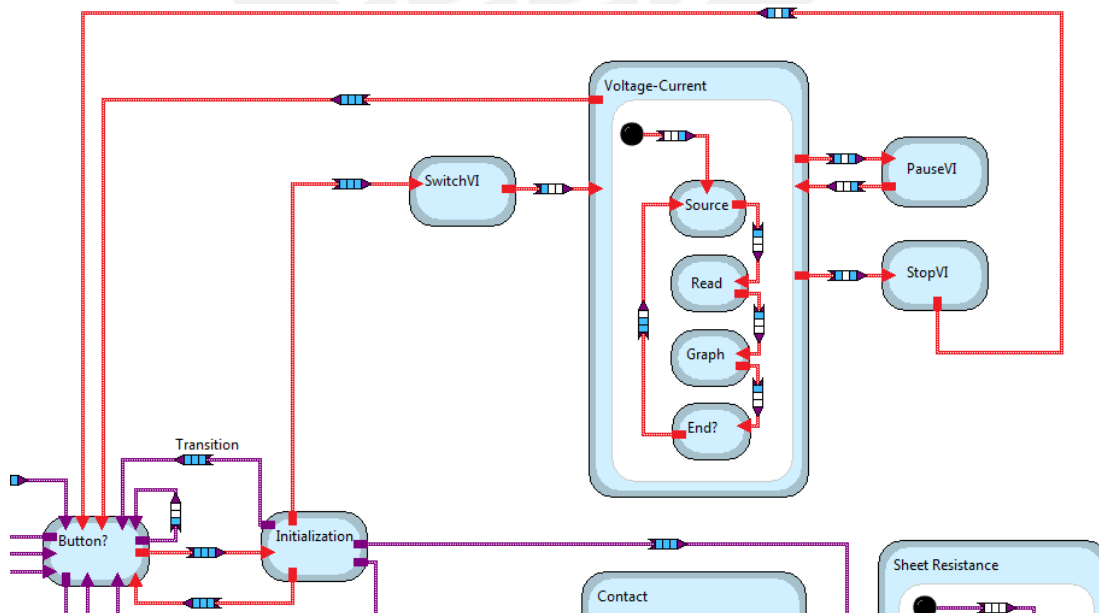


Figure 56: Possible action by pressing one buttons of the Voltage-Current tab

Four different states exist inside the loop, each one performs a different action. The “Source” state starts the current injection (Figure 57). Afterwards, the “Read” state reads the measurements of the current and the voltage. For the current measurement, the Keithley 2000 is used with the “Data Single Read” VI-module as ammeter. For the voltage measurement, the “Keithley 2001 single Read” is used as voltmeter. The measured value of current is multiplied by the factor of one thousand, with this factor the measurement can be shown in milliamps in the data table. The same is applied for the measurement of voltage. With the value of current and voltage, the resistance can be calculated by dividing the measured voltage by the measured current. The obtained result is in ohms (Ω). All the data collected (Figure 58) can be shown in the data table and plotted in the XY-Graph

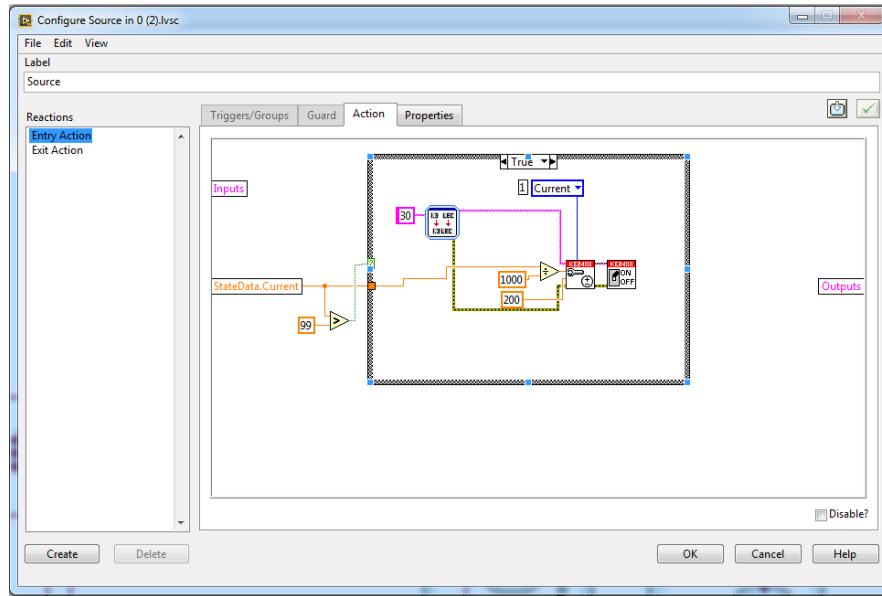


Figure 57: Current generation in the “Source” state of the “Voltage-Current” region

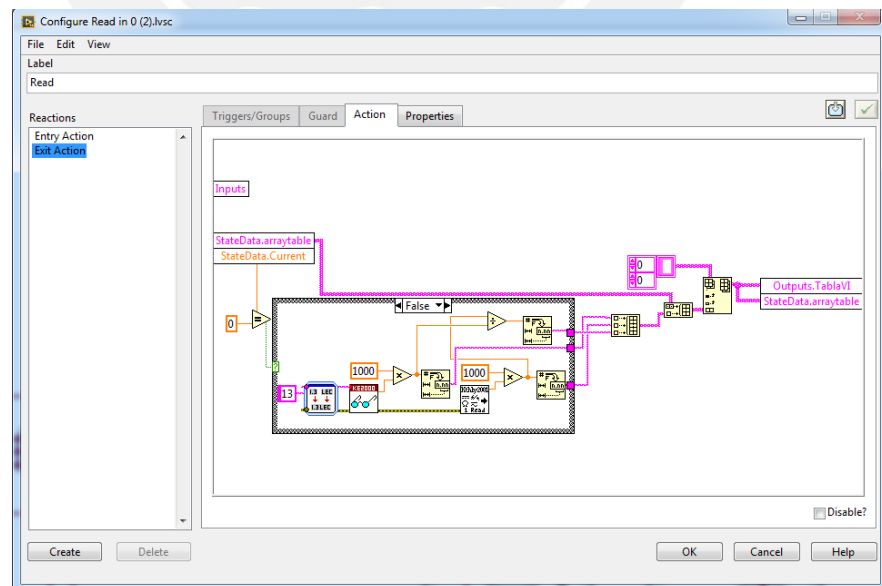


Figure 58: Data collected in the “Read” state of the “Voltage-Current” region

3.3.3.3 Sheet Resistance

Every time that the user selects the “Sheet Resistance” tab, a message will be shown (Figure 59), asking for the calibration of the camera. Only the buttons “Calibrate” and “Measure” are active. In order to activate the other buttons the user should calibrate the image of the camera. The user should press the “Calibrate” button, then a message will be display (Figure 60). If the user selects the “Use it” option the default calibration will be used. Else a new window will be displayed (Figure 61). The user should put the calibration ruler, adjust the focus and click on two points. The selection of a line should have the distance of 50 mm, the confirmation is done by pressing “OK”. A final message will be shown, giving the possibility to save this calibration as a default calibration. Else the calibration will be used only while the program is not finish.

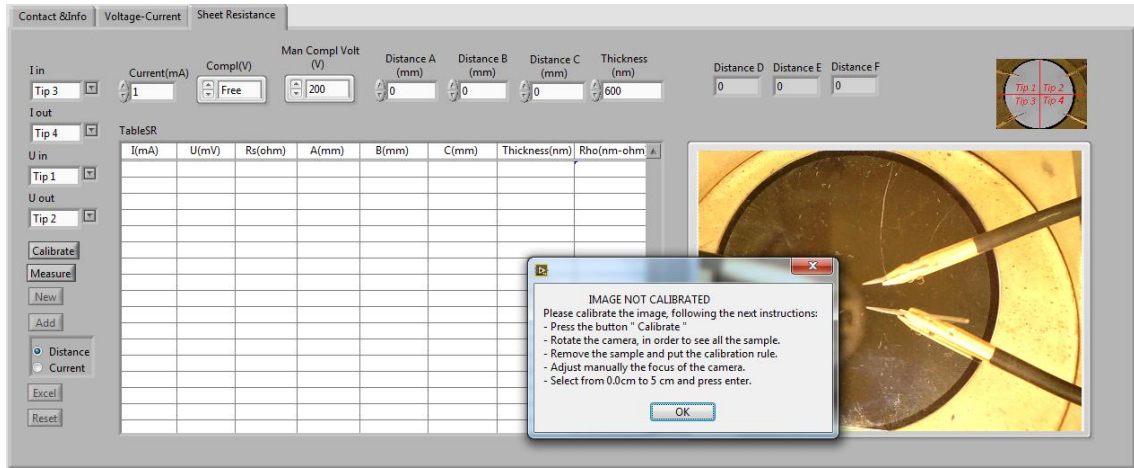


Figure 59: Sheet resistance and the calibration request

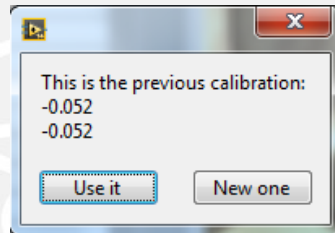


Figure 60: Default Calibration values, units in mm/pixel

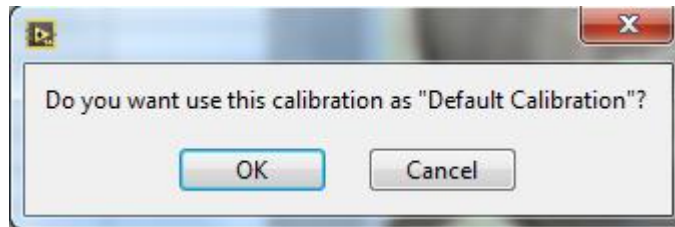


Figure 61: Final message for a new calibration

With all the buttons active, the flow diagram of the state chart is shown in red in Figure 62.

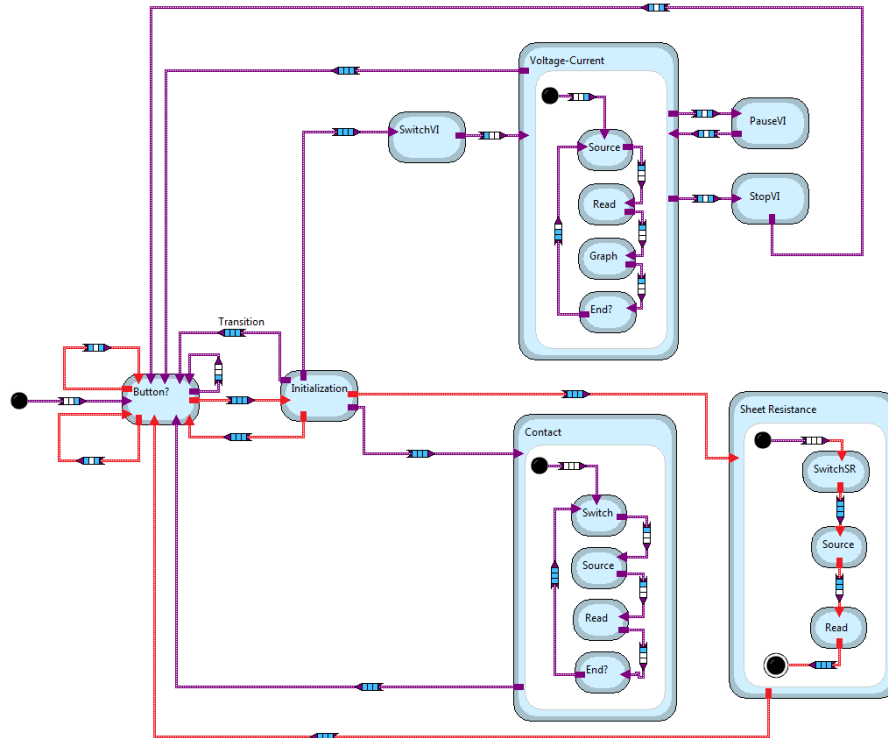


Figure 62: Possible action by pressing one button of the "Sheet Resistance" tab

Figure 63 shows the action of the "Measure" transition node, which measure the distance between two points. To measure a distance are necessary the coordinates of the two points and the parameters of calibration, which are taken from the default calibration parameters. First, the image is calibrate with the "Set Simple Calibration" VI module using the default calibration parameters. Then the previously selected points will be convert to real world units by the "Convert Pixel to Real World" VI module and finally the distance between the points is measure by "Point Distance" Vi module. The result of the measurement will be shown in a new window.

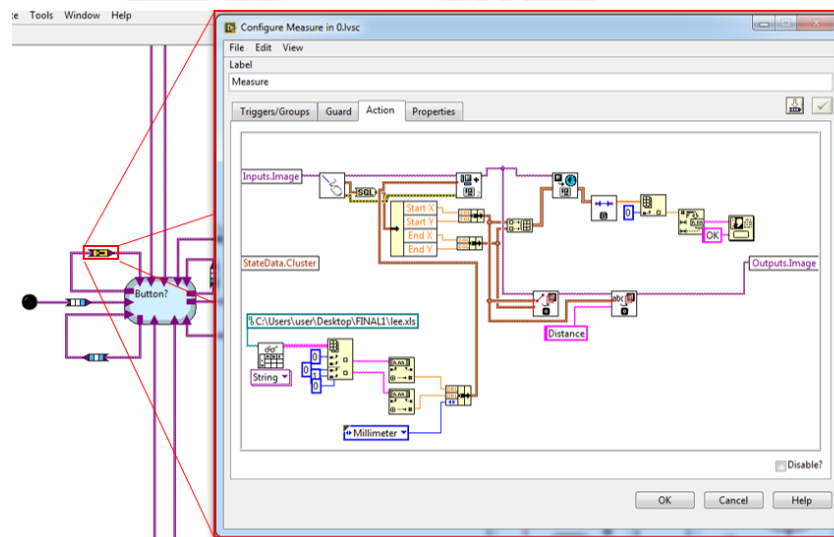


Figure 63: Action of the "Transition Node" which measure the distance between two points

4 Experiments

In order to prove the validity of the program, measurements of 6 samples of aluminum were carried out. The sample were produced with different thickness and at different temperatures during the deposition of the material. The deposition temperatures used were room temperature and 400 °C. For each temperature three different samples thickness 100 nm, 300 nm and 600 nm were produced (Table 19).The measurement was realized with variable distance, from the boundary to the center of the sample.

Table 19: Characteristic of the samples

Material	Thickness (nm)	Deposition temperature
Aluminum film	100	Room temperature
	300	
	600	400 °C

Thin film aluminum samples are shown in the Figure 64



Figure 64: Thin film aluminum samples

For these sample (Figure 64), the “Voltage VS Current” process has been performed with the same distance between the tips and -100 mA and 100mA as the minimum and maximum. The resistance shows for the samples a constant slop (resistance). That is characteristic for metals as they show a linear behavior between voltage and current (Figure 65 and Figure 66).

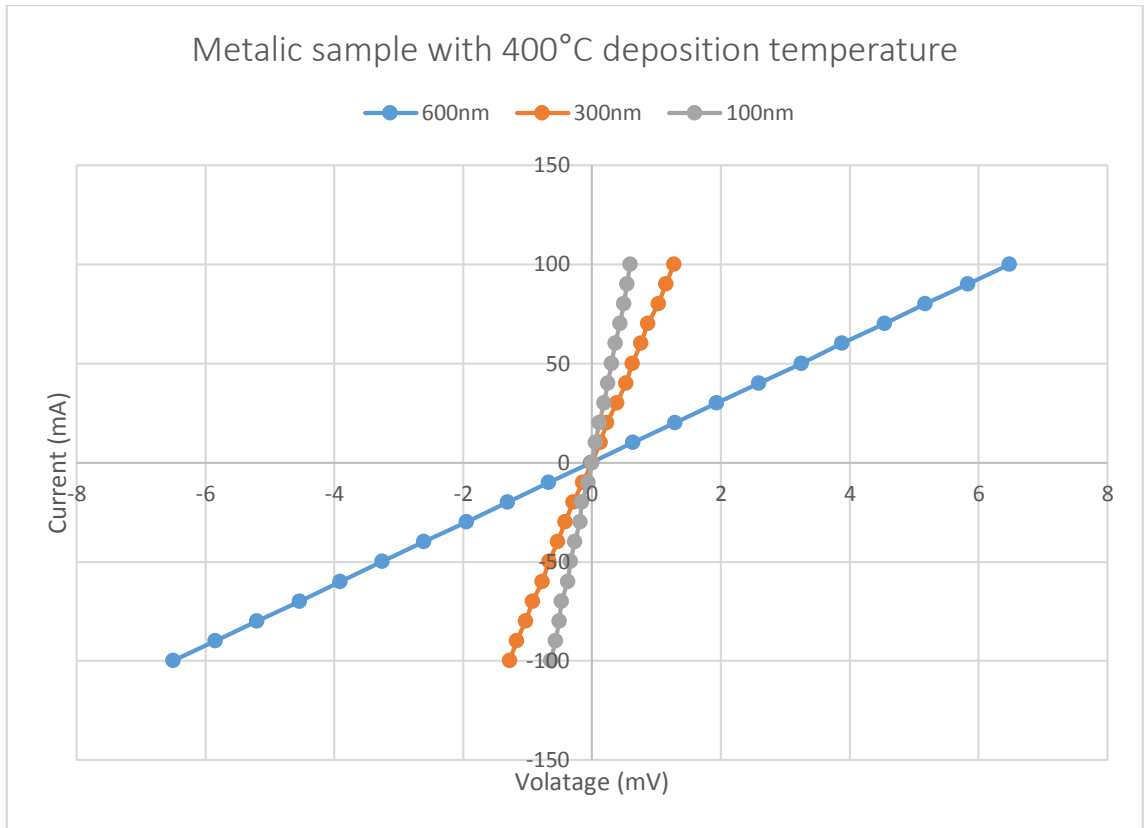


Figure 65: Graph from the test of metallic sample with 400 °C deposition temperature

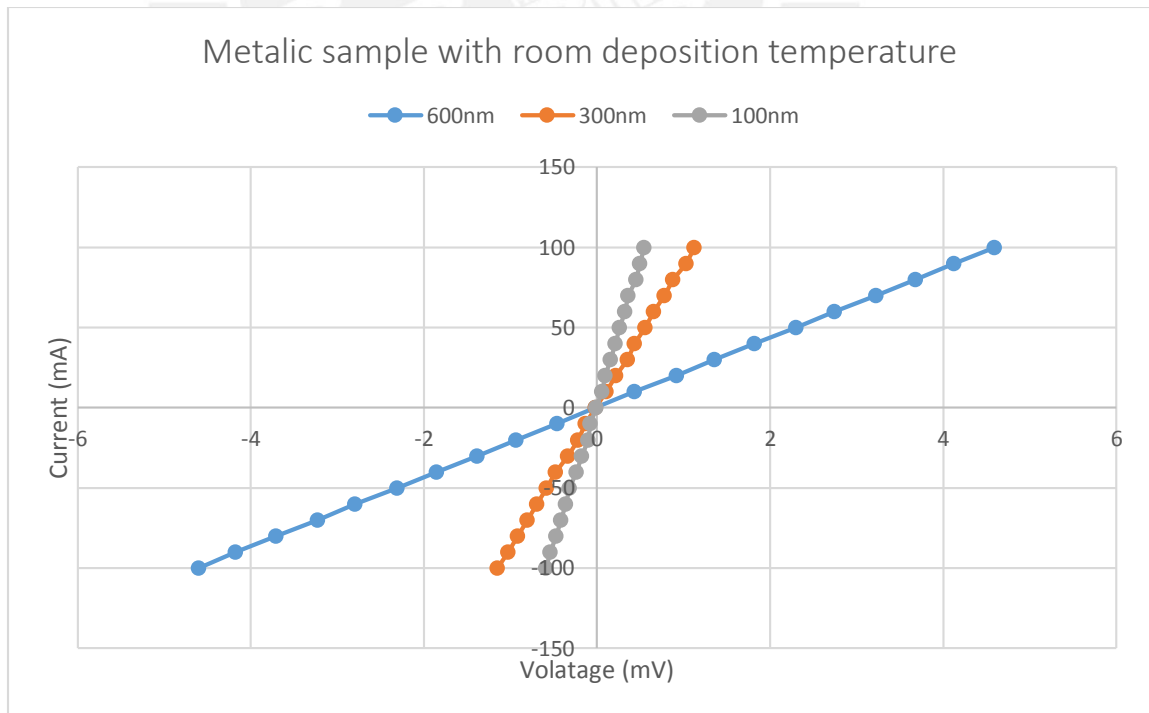
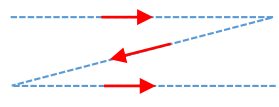




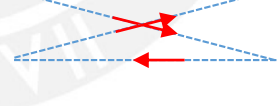


Figure 66: Graph from the test of metallic sample with room deposition temperature

As shown in Figure 65 the voltage value for the current of 100 mA for the sample with 600 nm has bigger values than the samples with 300 nm and 100 nm

For the experiment of sheet resistance the, distances between the tips has been changed gradually in order to see the variation of the resistivity with regard to the distance between the tips. Previously, several tests with different types of tip configurations have been performed (Table 20)

Table 20: Different types of tips configuration

Configuration	Tips selection	Figure
1	Current injection: 1 Current output: 2 Voltage input: 3 Voltage output: 4	
2	Current injection: 1 Current output: 2 Voltage input: 4 Voltage output: 3	
3	Current injection: 1 Current output: 3 Voltage input: 2 Voltage output: 4	
4	Current injection: 1 Current output: 2 Voltage input: 4 Voltage output: 3	
5	Current injection: 1 Current output: 4 Voltage input: 3 Voltage output: 2	
6	Current injection: 1 Current output: 4 Voltage input: 3 Voltage output: 2	

The equation used to calculate the resistivity was the relation between the horizontal and vertical resistance and the resistivity.

$$e^{-\frac{2\pi*d}{\rho}R_{MN,OP}} + e^{-\frac{2\pi*d}{\rho}R_{NO,PM}} = 1$$

Equation 13: Modified van der Pauw relationship

The variable distance were with horizontal, vertical and diagonal displacement (Figure 67), The Figure 68, Figure 69 and Figure 70 show a tendency of the resistivity to the value $4 \times 10^{-8} \Omega \text{ m}$. The value measure by the four points linear is $4.53 \times 10^{-8} \Omega \text{ m}$. These are the results of the configuration 2 and 3. Figure 71 show the tendency of the resistivity of the area defined by the position of the tips. Figure 72 show the tendency of the resistivity of the distance between the tips.

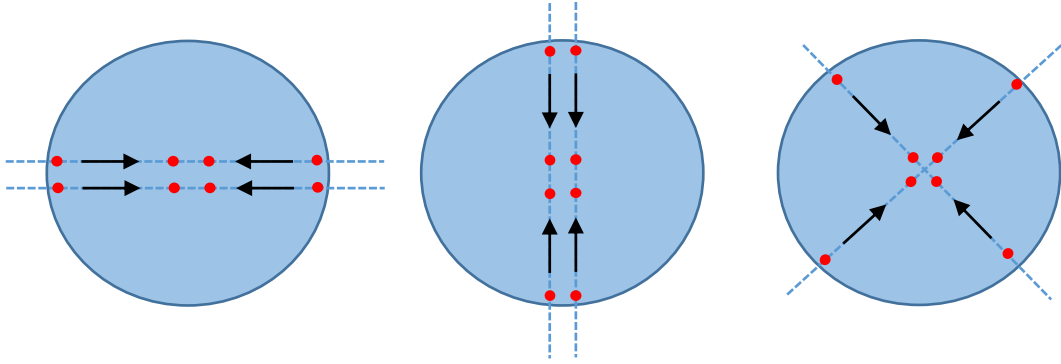


Figure 67: Displacement of the distance horizontal (a), vertical (b) and diagonal (c).

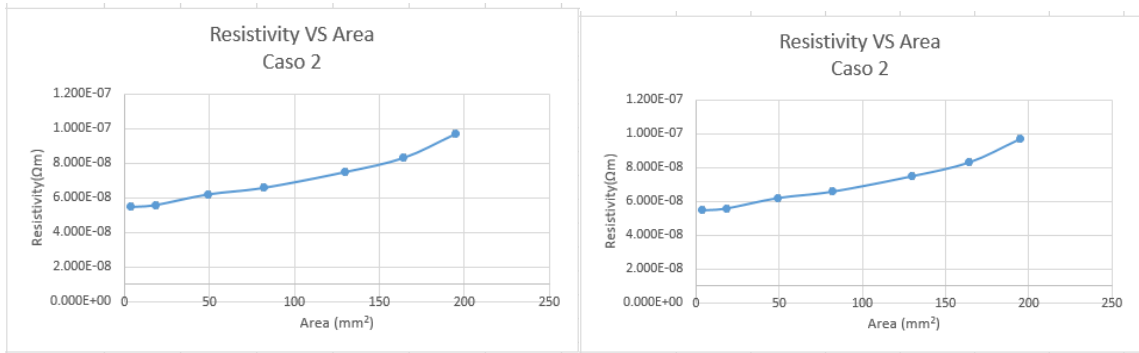


Figure 68: Tendency of the resistivity for Horizontal displacement

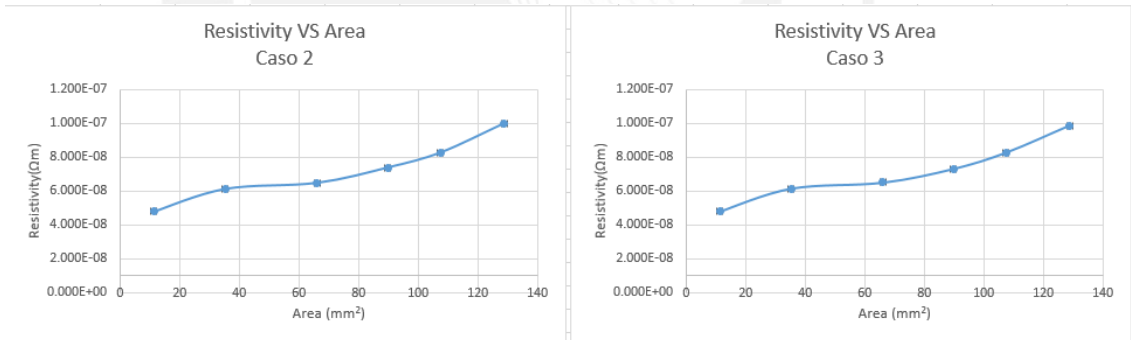


Figure 69: Tendency of the resistivity for vertical displacement

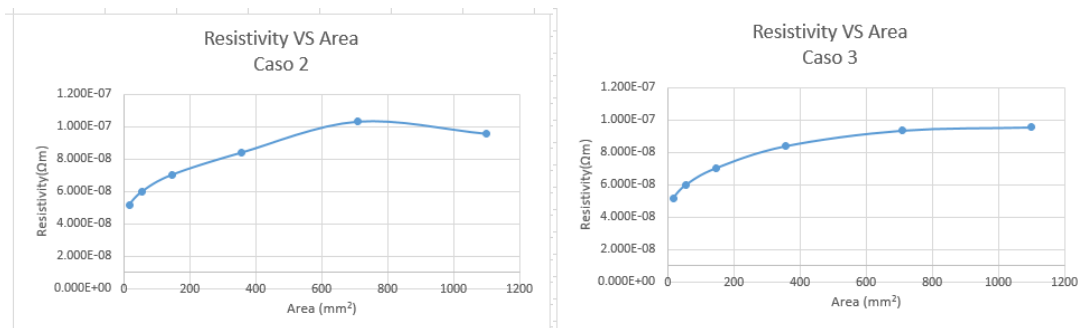


Figure 70: Tendency of the resistivity for diagonal displacement

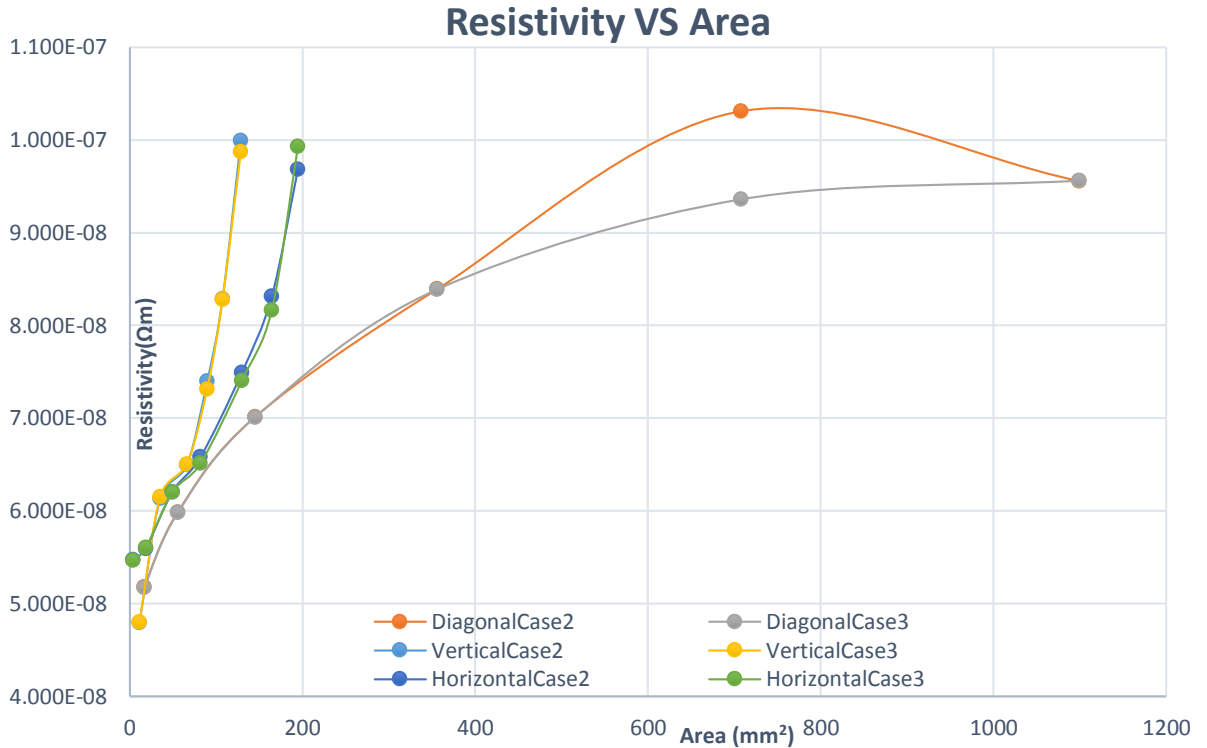


Figure 71: Comparison of the configuration 2 and 3 for all the displacement in terms of the Area

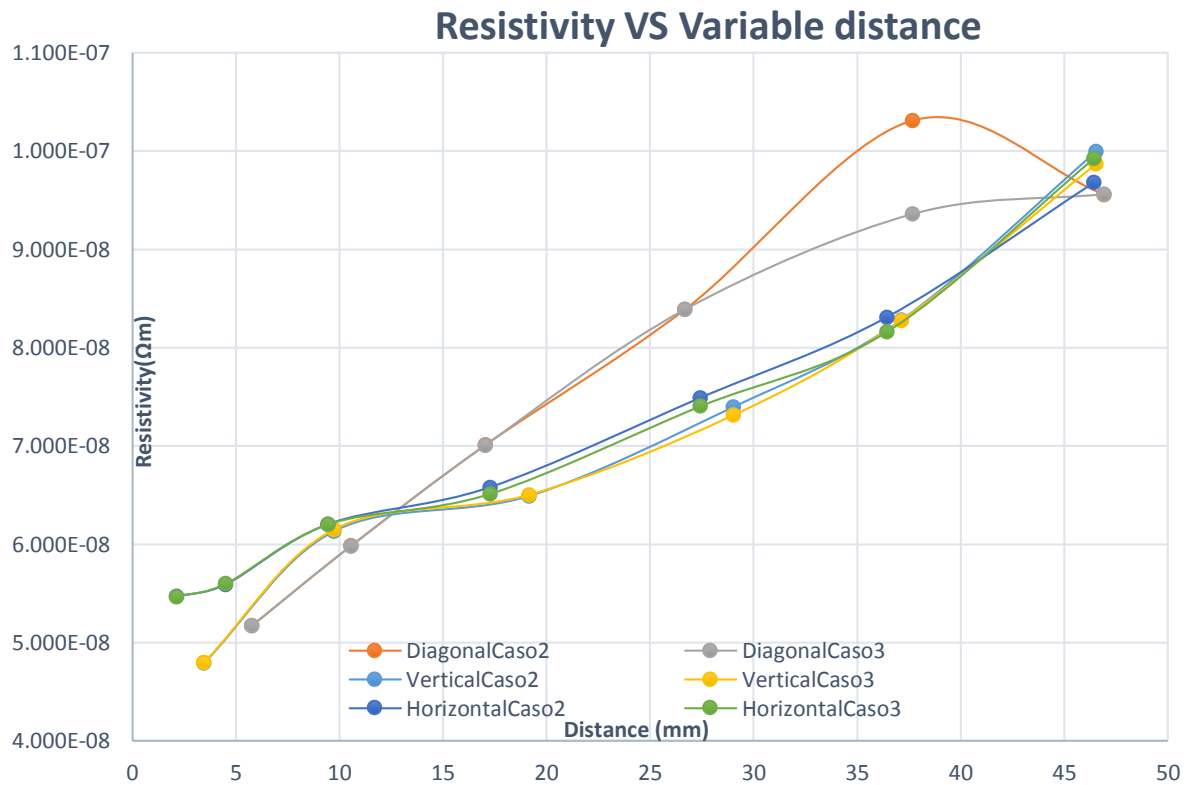


Figure 72: Comparison of the configuration 2 and 3 for all the displacement

5 Conclusions

In order to study the electrical properties, a program in LabVIEW was implemented a 4 probe measuring system, the program is implemented by the modified van der Pauw method, and the measurements has been collected with the Keithley devices. It was shown the dependency of the resistivity on film the thickness. It was also shown the dependency of the resistivity on the deposition temperature by a comparison between the samples with the same thickness.

It was implemented interface, which allows the user the calibration of the camera for the measurement of the distance between the tips.

It was also verify the validity of the modified van der Pauw method by a comparison the resistivity calculated with the resistivity calculated with the four point linear.



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