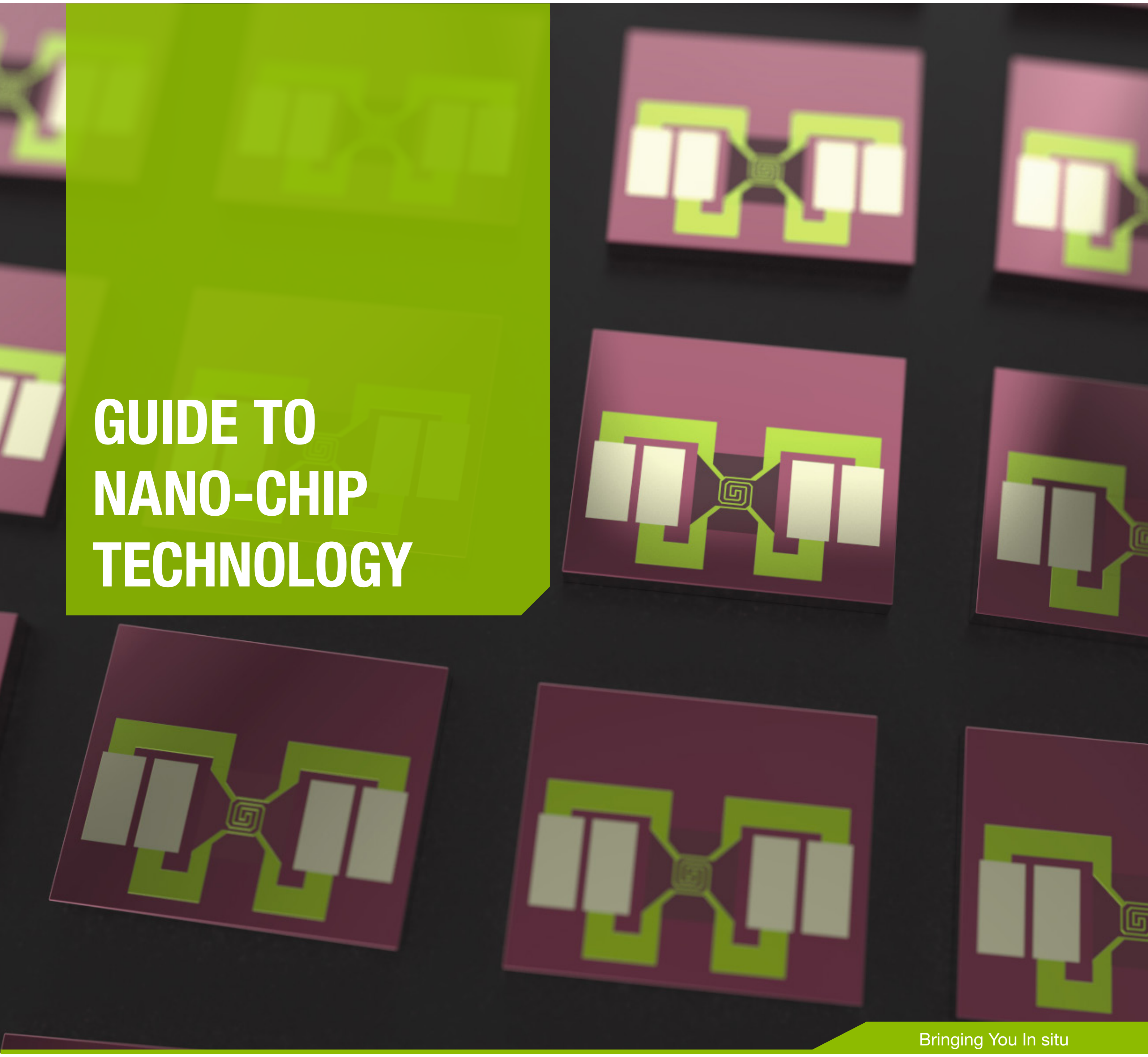
GUIDE TO NANO-CHIP TECHNOLOGY



Bringing You In situ

MICRO-ELECTRO-MECHANICAL SYSTEMS (MEMS)

DENS solutions

Introduction

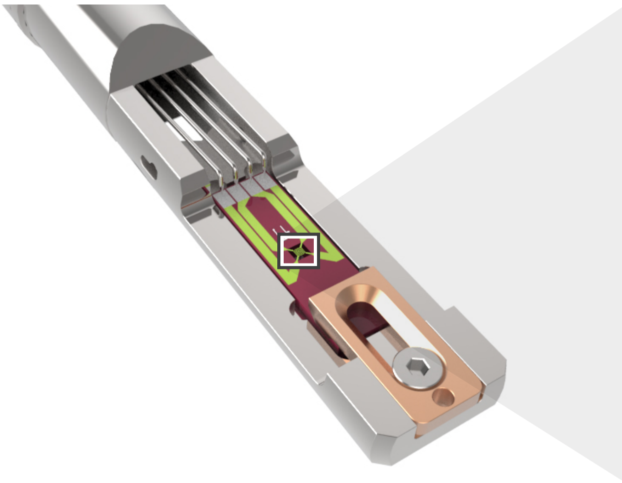
The DENSsolutions Nano-Chip is a functional consumable sample carrier that replaces traditional TEM copper grids and offers a micro-scale hotplate for heating samples. The micro-hotplate is based on the Joule heating principle of a metal spiral. Based on key functionality of a Micro-Electro-Mechanical-System (MEMS) device, Nano-Chips are fabricated to the scale required for TEM samples (300μm×300μm×600nm).

The micro-hotplate offers localized heating of TEM samples, with a room temperature drop within 300 μm from the edge of the micro-hotplate. Sample drift during heating experimentation is dramatically reduced due to < 20 mWatt required. Traditional furnace holders ordinarily require approx. 1,000 mWatt and water cooling, which contribute to poor resolution and sample instability.

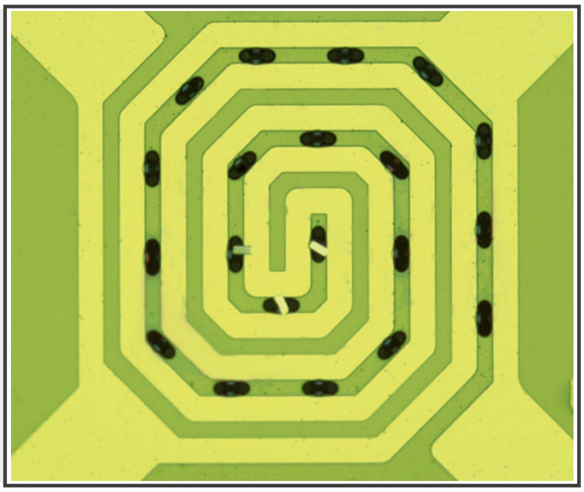
The Nano-Chip ST (RT to 800°C) and Nano-Chip XT (RT to 1,300°C) are available in a range of window materials suitable for most application requirements. Achieving accurate, fast, stable temperature control is now possible with the DENSsolutions Nano-Chip range of in situ TEM solutions.

Control Method	4 Point Resistive Feedback
Guaranteed Temperature Range	Room Temperature up to 1,300°C
Max. Temperature	1,500°C
Temperature Accuracy	< 5%
Temperature Stability	< 1°C at 1,300°C
Heat Rate	200°C per millisecond
Quench Rate	200°C per millisecond
Settling Time	< 2 seconds
Ultimate Sample Drift	0.5 nm per minute
Resolution	< 0.6 Å

*Specifications depend on your microscope & experiment

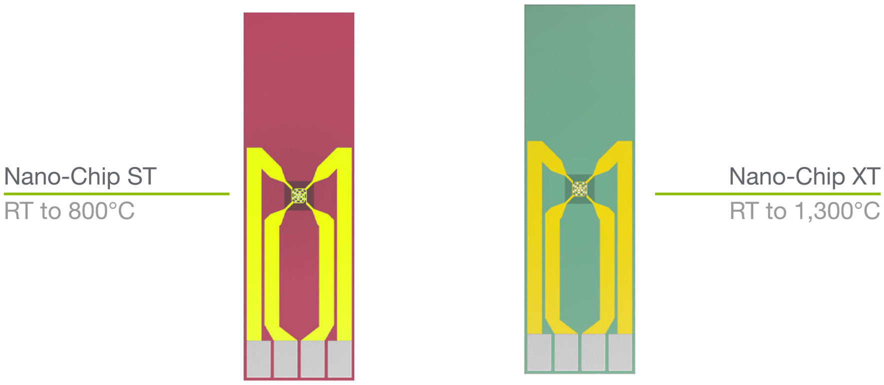


Nano-Chip in holder



Micro-hotplate with FIB samples on windows

Wildfire Nano-Chip Range



Select your Window Material:

Silicon Nitride

Silicon Nitride Support Films are the standard configuration for the Wildfire Nano-Chip range. These superior products are fabricated using Low Pressure Chemical Vapor Deposition (LPCVD). The latter results in high quality, low-stress and amorphous silicon nitride thin films.

Silicon Nitride Support Films have the advantages of being chemically inert and mechanically robust. Therefore, they can withstand harsh chemical and temperature environments (up to 1,100°C). These low scatter support films are electron transparent (<20nm thick) and they are fully compatible with oxygen plasma cleaning processes.

Carbon

Carbon Support Films have been developed to further enable nanotechnology applications and extend molecular biology research. The Wildfire Nano-Chip Carbon is a covered with a thin layer of carbon (~5 nm). These Carbon films are extremely thin and highly transparent to electrons, offering fine grain and low contrast that does not interfere with specimen structure.

During standard temperature studies (up to 800°C) or when nanoparticles are being studied and a completely amorphous / high contrast support is required, the Carbon window Wildfire Nano-Chip is the most suitable.

Through-Hole

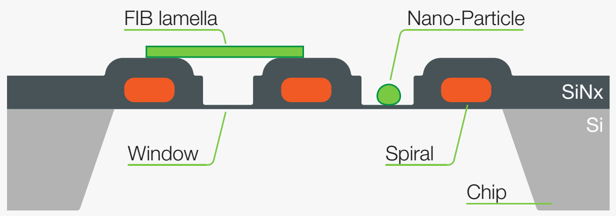
Through-Hole Nano-Chips support nanotechnology applications and FIB prepared sample research. The Wildfire Nano-Chip Through-Hole does not have any support film and offers the best contrast if your samples are large enough to span the 5 by 20 micron window. Like for all Nano-Chips, the Through-Hole has multiple windows to allow for a range of samples to be observed with the same chip.

When ultra-high temperature studies are involved or when FIB samples are being studied, the system of choice should be the Wildfire Nano-Chip Through-Hole.

Custom Nano-Chips available upon request.

Nano-Chip Design

The Nano-Chip range has been developed to achieve high resolution at elevated temperatures without reducing the value of the TEM. Preventing thermal drift during imaging is achieved through the micron size heater hotplate – called micro-heater. The Nano-Chip is made of a suspended Silicon Nitride (Si_3N_4) membrane, which the metallic spiral (micro-heater) structure is sandwiched to ensure a non-conductive surface. The heater spiral is connected by four (4) contact pads. These contact pads complete the four (4) point probe voltage and measurement system. Two (2) contact pads are used to supply the voltage and the other two (2) to measure the resistance. When the current flows through the system, the spirals resistance can be measured using Joule heating, which has a linear correlation to temperature (°C).



*not to scale

Metal or Ceramic Heater

The heater material has a crucial influence on the system's performance when varying temperature from room temperature (RT) to elevated temperatures (> 1,500°C). Two types of heater materials are commonly used by suppliers of MEMS related devices: metal heaters and ceramic heaters.

Conductivity – Metal heaters are widely accepted as one of the most reliable heating materials, which can be seen in most household electronics. This is due to the fact that the performance characteristics of a metal heater material being extremely responsive to electrical stimuli and exhibits linear conduction behaviour (RT to > 2,000°C). In comparison, ceramic conductors offer lower performance characteristics, particularly at temperatures under ~500°C. As ceramics behave as semiconductors, they exhibit non-linear conduction behaviour and proving it difficult to accurately control the response.

Response Rates – When applying a voltage to a circuit, the time needed to change from temperature X to temperature Y, is identified as the 'response rate'. Metals and ceramic materials differ drastically in their response time. Metals offer optimal performance between RT and 1,300°C as times ramping and quenching happens in < 1 second. In comparison, ceramics are commonly observed to be between 6 and 7 times slower.

Thermal Stabilization Time – When heating any material, the entire heated mass will interact with its environment to achieve energy equilibrium. The thermal stabilization time is identified as the time needed to balance the energy input and energy loss. Reaching this equilibrium, which causes drift issues, is best when stabilized as quick as possible. Nano-Chips typically stabilize at standard temperatures within 2 seconds. In comparison, ceramic heaters can take from 2-10 minutes for equivalent temperature steps.

Control Loop Measurement – Fast response rates are important for achieving effective feedback control loop measurement of temperature. The feedback control of a heater is measured and corrected in real-time by correlating the resistance to the temperature of the heater current. It requires both the voltage and the corresponding current to be measured accurately and simultaneously. The shorter response time results in a faster feedback loop, which in turn improves stabilization and overall performance. Long response times slow the feedback loop and in some cases can cause failure of the feedback mechanics as the speed cannot follow the environment's change. Due to metal heater materials stabilizing much faster and its linear response, the overall performance of the control loop is optimized

Extreme Temperatures – Metal heaters can have close to uniform performance throughout the whole working range to 1,500°C. Experiments requiring temperatures above 1,500°C cause degradation of the heater as the melting point is approached. In comparison, ceramic materials in these extreme temperatures offer a higher working range in excess of 1,500°C due to the chemical stability in this range.

Temperature Control

Studying dynamic in situ events requires the temperature control to be immediate and accurate. Measuring and controlling temperature on the micron scale is not an easy task and this problem has been tackled by using a closed loop 4-point-probe resistive measurement. Nano-Chips measure locally the electrical resistance of the micro-hotplate and acts both as a Joule Heater and a Thermal Resistive Sensor.

There are two common methods to measure temperature of the heated material.

Power Dissipation Measurement

Measuring by means of ‘power dissipation’ can be a very straightforward method if the environment around the heated material is well defined. However, a large error in temperature accuracy can occur if the environment is not exactly defined. This has to do with the fact that the actual temperature of the heated material is related to the energy equilibrium between energy input and energy output.

If a material is consistently being heated by means of joule heating, an equilibrium will occur between the energy that went into the material and the energy that is dissipated out, which can be defined as:

Q in = Q out

If we would make this more specific, it can be written as: all the energy going into the material is equal to all the energy dissipating out of the material.

Q joule heating = Q convection+ Q radiation + Q conduction

If the energy loss is well defined, the temperature of the material can be determined as directly proportional to the power dissipation. A problem occurs if the energy loss is not well defined, as is unfortunately very common in real-life applications:

The energy which is lost due to conduction is often unknown.

For example: the type of sample and the amount sample loaded onto the heater also influences the amount of energy loss due to conduction, leading to an extra error in the measurement.

The energy which is lost due to convection can fluctuate.

For example: the vacuum inside your TEM can fluctuate which influences your experiment. A more extreme case would be for an Environmental TEM (ETEM) where there is significant heat loss due to convection.

The only way to solve this is to calibrate the heated material for each possible (fluctuating) environment which might occur and adjusting the power vs temperature relation. A very cumbersome approach and still prone to errors as the different power vs temperature ratios can only be defined in finite increments.

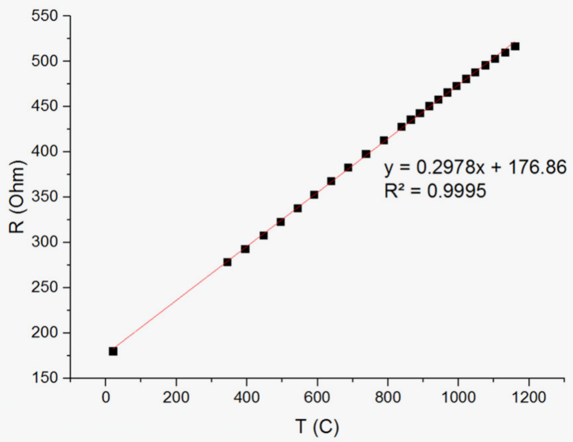
An alternative and more robust solution can be found in the approach to measure the temperature by means of measuring the change in electrical resistance of the material.

Resistance Measurement

Using the electrical resistance of a material as a temperature indicator offers substantial benefits over power dissipation as it can be used to operate independently from changes in external environmental as it is an inherent material property.

At the basis of this method is the Thermal Coefficient of Resistance (TCR) of the heated material, which is the internal property of material. The TCR describes the relative change of resistance that is associated with a given change in temperature. Having in mind positive TCR, this basically means that electrical resistance of a material will increase due to increase in temperature.

If the TCR of a material is known, it can be used to control the temperature in any environment, as it is completely independent of energy losses. Finding the TCR of the heater material is the main goal of our calibration procedure.

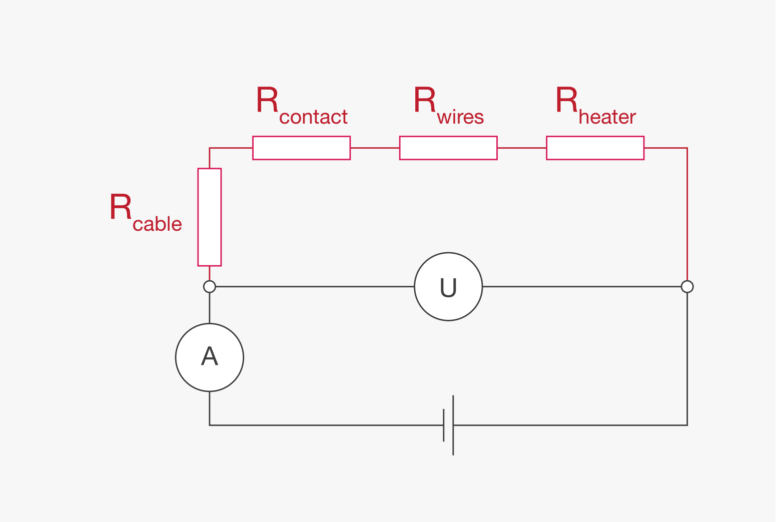


The TCR of many metals are commonly known to follow a linear behaviour (see graph below), which facilitates temperature control and greatly improves time response. For ceramics this method is much more difficult to use as they behave as semiconductors and thus follow non-linear behaviour in terms of resistance vs temperature.

For example, a heater set to 500ºC in 10-7 mbar vacuum will result to the 'power dissipation' of the heater of approximately 6 mWatts and 'resistance' measure to be typically 350 ohm. Whereas, a heater set to 500 ºC in 10-2 mbar gas environment, the measured 'power dissipation' of the heater can be as high as 19 mWatts. However, the measured 'resistance' of the heater remains the same at 350 ohm.

For the above reasons, DENSsolutions uses the 'resistance' temperature indicator across the whole Nano-Chip range.

2 contact measurements (old system)



Using the two contact resistance measurement, the total resistance of the entire electrical loop is measured, including the resistance of the micro-heater, the resistance of the cables that connect the heater to the outside regulator plus the contact resistance of various cables (Rcable), contact resistances between the chips and needles (Rconact) and lead wire resistance (Rwires). Having all these resistances being part of the measurement loop introduce measurement errors (see image).

Why is this a problem?

The problem is twofold, i.e. static and dynamic. First of all, additional higher resistances will create an offset in the calibrated TCR value of the heater and, as a result, the measured temperature of 2 contact MEMS device will be overestimated. The larger the Rcontact, Rcable and Rwires the larger the overestimation. Secondly, there is a dynamic part, e.g. changing Rcontact and Rcable during the experiment will introduce additional temperature error.

As the resistance of the cables are often in an environment in which the temperature can fluctuate and differ from that of the micro-heater (note that some parts of cables are outside microscope), the non-constant resistance will introduce an error in the measurement.

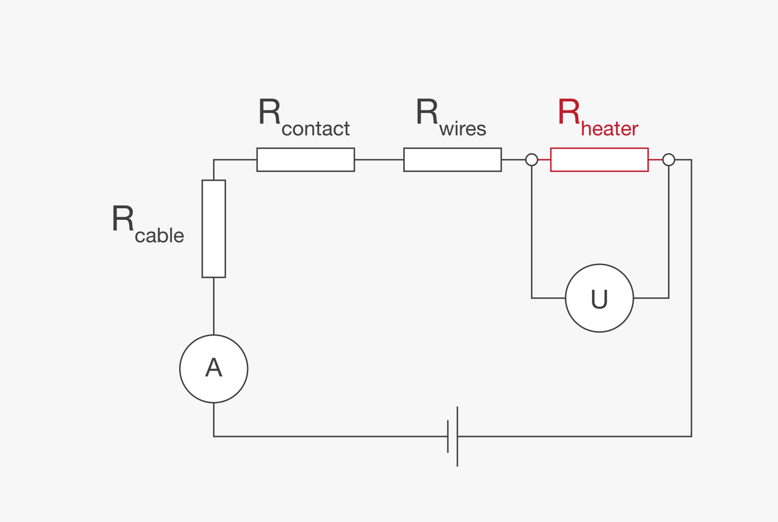
Secondly, the material of the cables and the micro-heater is often different, meaning that it will interact differently to fluctuating temperatures and thus make the measurement even more difficult to control.

A common technique to connect the holder to MEMS device is through contact pads using cable connected needles in the holder. Every time these needles are connected, the contact resistance can vary, leading to an extra measurement error when using the 2 contact system, as the resistance is non-consistent.

All MEMS devices use small lead wires to connect the micro-hotplate to the needles. The surface area of these lead wires define its electrical resistance. However, slight variation of the surface area of the lead wires from chip to chip due to errors in the micro-fabrication process can induce errors in resistance measurement.

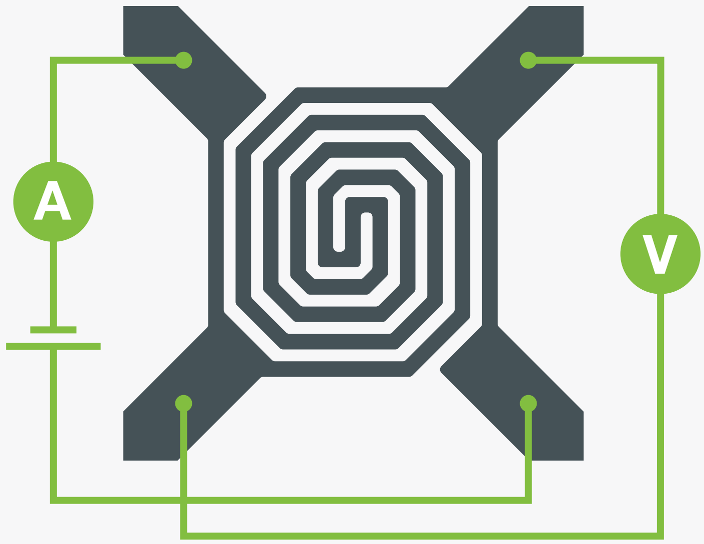
For the above reasons, the 2 contact resistive measurements technique is seen as ineffective and an outdated form to be used for modern experimentation.

4 contact measurements (modern system)



The four (4) point probe technique allows the measured resistance to be used as an accurate indication of the temperature of micro-heater. This is a significant advantage for precise electrical measurements needed in micro-heaters.

Nano-Chips use a four (4) contact resistance measurement method in order to define electrical resistance of the micro-heater. It is commonly accepted amongst the electrical engineers community that this method is the most accurate technique to locally measure a materials electrical resistance. This method excludes the influence of the cable resistance, uncertain contact resistance and lead wires resistance, by using a separate pairs of current-carrying and voltage-sensing electrodes to make more accurate measurements than the simpler and more usual two-contact sensing.



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