# TASK: TW4-TTMA-001
# ADVANCED MATERIALS: SiC SiC Ceramic Composites

***Deliverable*: Measurement of electrical resistivity unirradiated composites along different directions, up to 1000$^0$C**

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## 1. Contact deposition

The four-point resistance measurement, as its title suggests, implies the presence of four contacts: two contacts through which a current is passed through the sample, and two from which the voltage drop across the sample is measured.

In order to achieve uniform deposited contacts on the sample, evaporation of the contact material (Ag, Au) by means of electron beam was used. A special set-up was developed for the actual contact deposition. This set-up is presented in Figure 1, and it consists of a small electrical engine, used for the continuous rotation of the sample during the deposition, and a mask used for the patterning of the contacts.

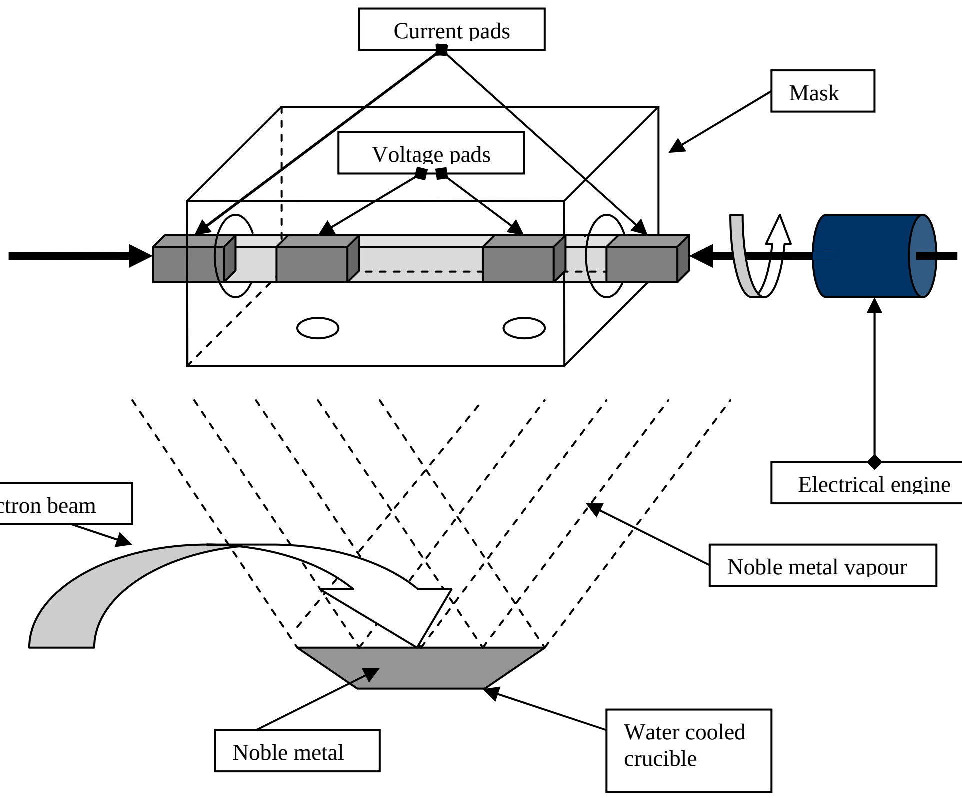

*Figure 1. Experimental set-up for the deposition of electrical contacts on SiC/SiCf*

Prior to the deposition the sample is subjected to ion beam cleaning. With the evaporation chamber vacuumed, argon is released into the chamber, the pressure reaching the value of approximately $6*10^{-2}$ Torr. The argon ions are accelerated towards the sample and the chamber at a potential difference of 3kV, producing the cleaning of the sample.

## 2. Experimental set-up for the resistance measurement

Resistivity measurements are very important for material characterization. Resistivity is measured indirectly, the physical quantity that is actually measured is the electrical resistance, and knowing the geometry of the sample its resistivity can be calculated $\left(R = \rho \frac{l}{S}\right)$. The simplest way to measure the sample's resistance would be to inject a current and measure the voltage drop across it. In doing so, however, one also measures the resistance of the contacts, plus the contact thermoelectrical voltage. In order to eliminate the measurement of these quantities, and make more accurate experimental determinations, the four-point resistance measurement technique is used. This method eliminates the measurement of the contact resistance, and by changing the direction of the current one can also eliminate the contact thermoelectrical voltage.

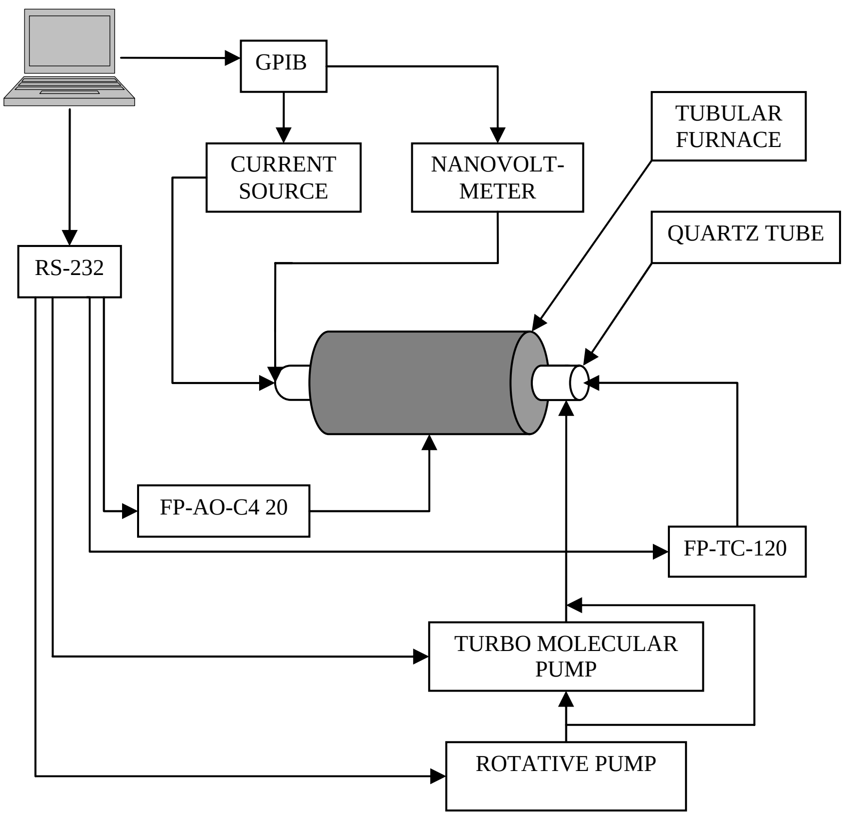


*Figure 2. Experimental set-up for the electrical resistance measurement.*

The experimental set-up consists of a programmable current source (Keithley) and a digital multimeter (Keithley) which are both computer controlled through General Purpose Interface Bus (GPIB) interface and using the LabVIEW programming environment. The computer programs the current source to send a current, records the voltage drop, programs a current in the opposite direction, records the new voltage drop, makes the appropriate calculations, determines the resistance and saves it in a text file.

In order to obtain the temperature dependence (20$^o$C-1000$^o$C) of the resistance the sample, a tubular furnace was also adapted so that its temperature can be controlled by the computer via National Instruments FieldPoint current output and thermocouple input module, and a Eurotherm TE10A power controller. The computer uses a PID algorithm to control the furnace temperature, for which the parameters were tuned to give an accuracy of ±0.1$^o$C. Inside the furnace, a quartz tube can be placed. The tube serves to make determinations of the resistance in vacuum or in nitrogen or argon atmosphere. The vacuum is obtained by a preliminary vacuum and a turbomolecular vacuum pomp that are both computer controlled with the serial RS-232 port. All of the computer programs were developed in the LabVIEW environment.

In Figure 2 the block diagram of the set-up is presented.

In order to increase the signal/noise ratio, an optimization study has been carried out using 1Ω standard resistor. Beside the classical solution for the rejection of the common noise, the moment at which the measurement is done was synchronized with sinusoidal voltage of power supply. Under these precautions, the noise/signal ratio is less than 100ppm and the accuracy of resistance measurement is of about ±0.05%.

### 3. Experimental results

The temperature dependence of the electrical resistance of SiC/SiCf composites is presented in Figure 3. The measurement was carried out in vacuum $10^{-6}$ Torr. The semiconducting behaviour is clearly seen in the range from room temperature to 300 $^o$C. At temperatures higher than 300 $^o$C a deviation from the exponential law has been noticed. In our opinion this deviation is most probably due to the presence of interfaces SiC-SiCf in the composite material. In order to determine the resistance at the SiC-SiCf interface, resistance measurement in the low temperature range (65K-300K) is necessary. For this purpose, a measuring cryostat is under preparation in our laboratory. In the low temperature range the contribution of the SiC-SiCf interface is negligible with respect to the intrinsic semiconducting resistance. Therefore, from the low temperature data, one can derive the parameters of the exponential behaviour. Taking into account that in the high temperature range the measured resistance has two contributions: one semiconducting and the other one due to interfaces, we can separate the interface contribution.

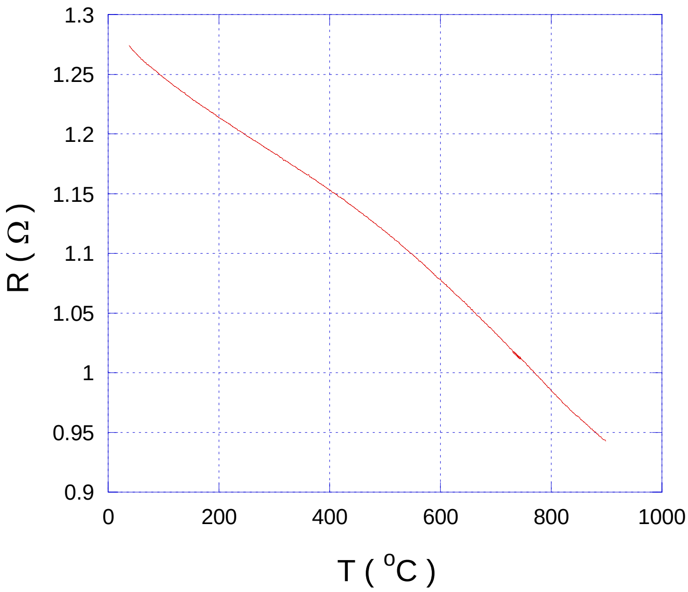


*Figure 3. Temperature dependence of the electrical resistivity of SiC/SiCf composite.*

It is worthwhile to mention that during the measurements at high temperature (T>500 $^{o}$C), a sublimation effect was observed. In order to investigate the nature of the sublimed material Energy Dispersive X-Ray Spectroscopy (EDX) is to be carried out. Anyway, in the limit of experimental accuracy, the measurements reproducibility is not affected by the sublimation effect.

## Conclusions

Summarizing, the RvsT measurements of SiC/SiCf composites have been performed in the temperature range from room temperature (RT) to 900$^{0}$C. The electrical resistivity, determined taking into account the dimensions of the sample, decreases from 1.15x10$^{-3}$Ω.m at RT to 0.85x10$^{-3}$Ω.m at 900$^{0}$C. The estimated accuracy for the electrical resistivity determination is of about ±1%. At temperatures higher than 300$^{o}$C a deviation from the exponential law has been noticed. In our opinion this deviation is most probably due to the presence of interfaces SiC-SiCf in the composite material. In order to determine the resistance at the SiC-SiCf interface, resistance measurement in the low temperature range (65K-300K) is necessary. On the other hand, further investigations are necessary in order to elucidate the observed sublimation phenomenon.