# TASK: TW5-TTMA-001
# ADVANCED MATERIALS: SIC/SIC CERAMIC COMPOSITES

## Deliverable: Measurement of the electrical resistivity from RT to 1000 of unirradiated 2D and 3D SiC/SiC composites

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### 1. Experimental

One of the most important aspect in the measurement of the resistance, in the high temperature range, using the classical four-probe method, is the deposition of good quality contacts, for both measuring the voltage drop across the sample, as well as for injecting the current. An important issue in the manufacturing of these contacts is represented by the diffusion of the material used for contacts in the sample, Ag in this case. In order to prevent this diffusion from occurring, a Ti layer of order of microns in thickness is deposited before the Ag layer. Also, for reasons of adhesion, a Ni layer is intercalated between the two layers. Silver paint is used to solder the Pt wire on the Ag pads. The Ti , Ni and Ag layers were deposited by means of electron beam evaporation at a pressure of $10^{-6}$ Torr. Prior to the deposition, the sample was cleaned by bombardment with $Ar^+$ ions, accelerated in a voltage difference of 3kV. The contacts were deposited on the sample using masks. The image of the sample after the deposition of contacts for longitudinal resistivity measurement is presented in Figure 1. The distance between the voltage contacts and the current ones is large in order to obtain a uniform current distribution in the portion of the sample where the voltage drop is measured. For the measurement of the resistivity in a direction perpendicular to the fibers the electrical contacts were done in the same manner as for the measurements along the fibers direction, but, in this case, the current and voltage contacts overlapped.

The experimental set-up consists of a programmable current source (Keithley) and a digital multimeter (Keithley) which are both computer controlled through the General Purpose Interface Bus (GPIB) interface and using the LabVIEW programming environment. In order to obtain the temperature dependence ($20^oC$-$1000^oC$) of the resistance of the sample, a tubular furnace was also adapted so that its temperature can be controlled by 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 $\pm 0.1^oC$. Inside the furnace, a quartz tube is 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 pump that are both computer controlled with the serial RS-232 port. All the computer programs were developed in the LabVIEW environment. In order to increase the signal/noise ratio, an optimization study has been carried out using $1\Omega$ standard resistor. Beside the classical solution for the rejection of the common noise, the moment at which the measurement is done was synchronized with the sinusoidal voltage of power supply. Under these precautions, the noise/signal ratio is less than 100ppm and the accuracy of the resistance measurement is of about ±0.05%.

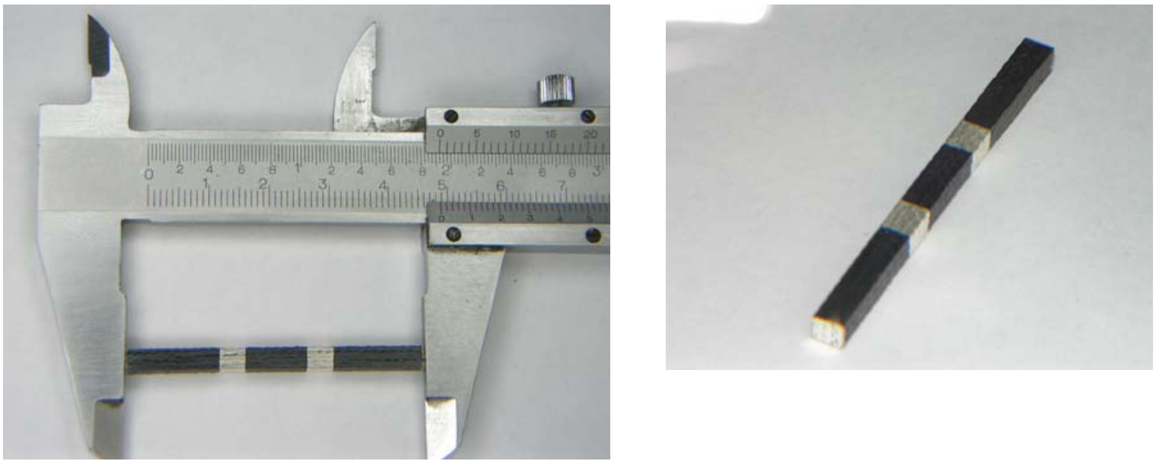

*Figure 1. The sample after the deposition of the contacts for longitudinal resistivity measurement*

## 2. Results

The electrical resistance measurements of the samples, both in-plane and in transverse direction, exhibit a thermal hysteresis, as presented in Figures 2 and 3. In order to understand this hysteretic behaviour, the samples were subjected to several thermal cycles (between RT and 1100 K) until the values of the resistance were stabilized. Afterwards, the samples were polished and new contacts were deposited. Finally, the resistance was measured again and no hysteretic behaviour was observed. Through this experiments it was demonstrated that hysteretic behaviour is due to the material itself and not to the electrical contacts. It has to be noted that hysteretic effect is stronger in transverse direction. Taking into consideration that the electrical resistance in the transverse direction is mainly due to the SiC matrix, one can presume that the material transformation, during the thermal cycle, appears in the SiC matrix. The changes in the samples can be correlated with the sublimation effect observed during the heating of the samples. It has been observed that two heating cycles are enough to stabilize the material from the electrical resistance point of view. Therefore, the data presented in this report correspond to the third thermal cycle.

The electrical resistivity along the fiber direction is presented in Figure 4. One notices that the resistance decreases when the temperature increases. This is a normal behaviour taking into account the fact that SiC is a semiconductor. However, this decay is not quite an exponential one and the value of the resistivity, calculated on the geometrical dimensions of the sample, is quite low with respect to pure SiC. It is known that SiC is a wide band-gap semiconductor with band gap values between 2.8-3.0 eV, so one would expect higher values for the resistivity. This low value can be explained by the fact that during the fabrication of the SiC/SiCf composite material the SiC fibers have a carbon coating of about 100nm. Therefore, the resistivity along the fiber direction has an important low metallic resistivity. Due to the percolative character the electrical current passes mainly through the carbon coated SiC fibers. Thus, the electrical resistivity along the fibers is very low.

In Figure 5 is presented the electrical resistivity measured in transverse direction. In contrast with the in-plane resistivity, in transverse direction the sample can be modelled by a multilayer structure consisting in an alternation of low resistivity layers (fiber layer) and a high resistivity layer (SiC semiconducting layer). In this case the current is forced to pass through the SiC layer and, as a result, the resistivity has a strong semiconducting component. This explains that the value of the electrical resistivity in the transverse direction is more than two orders of magnitude greater with respect to the in-plane direction. Moreover, the semiconducting behaviour of the transverse resistivity is more prominent. Another important aspect regards the effective area in the transverse direction, which is more reduced due to greater contribution of the voids in this geometry. The voids are intrinsic to the fabrication technology (chemical vapor infiltration).

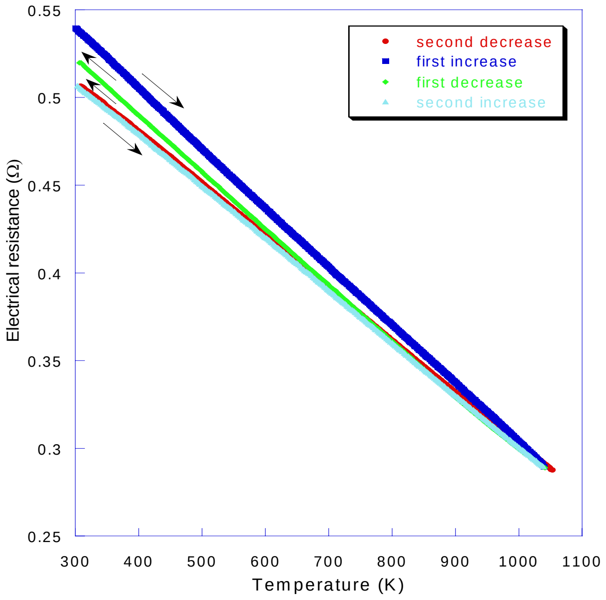

*Figure 2. The hysteretic behavoiur of the in-plane electrical resistivity during the first two thermal cycles*

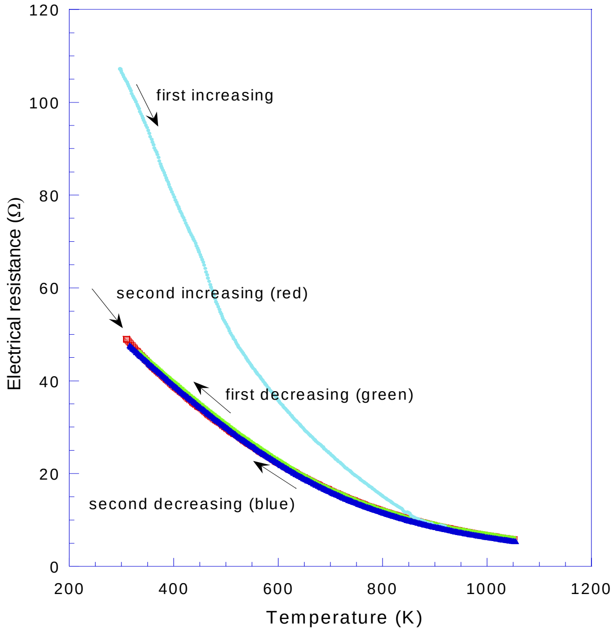

*Figure 3. The hysteretic behaviour of the transverse electrical resistivity during the first two thermal cycles.*

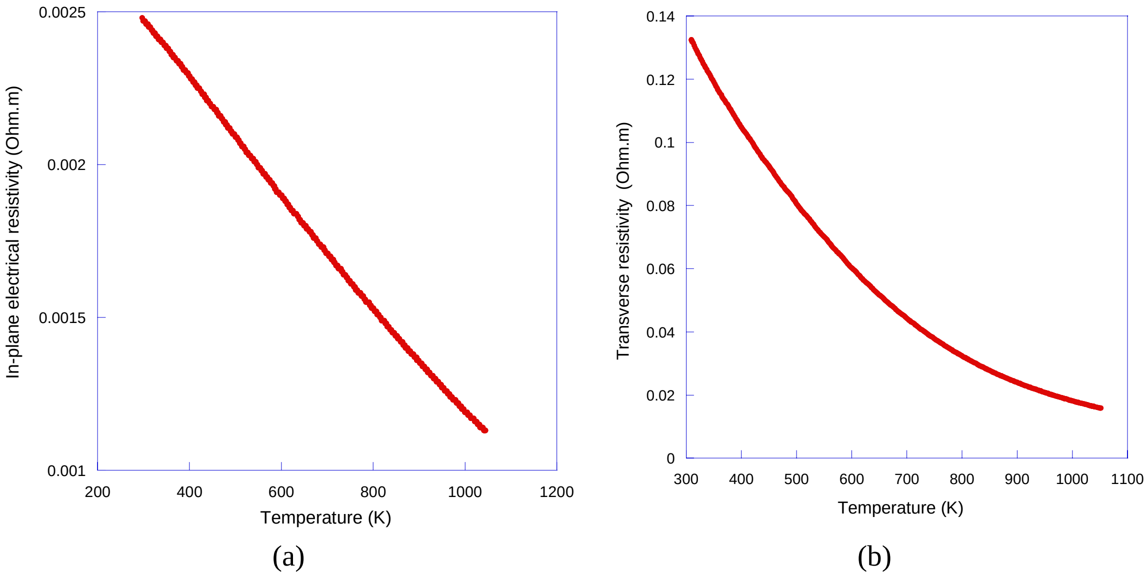

*Figure 4. Electrical resistivity of the 2D SiC/SiCf composites (a) along the SiC fibers and (b) in transverse direction.*

In Figure 5 the electrical conductivity, both in-plane and transverse direction, versus temperature is presented in a logarithmic scale.

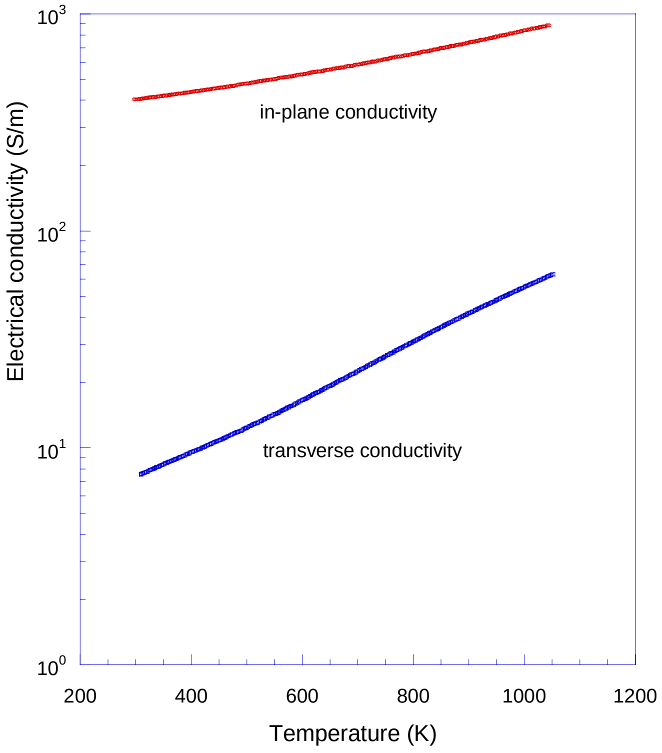

*Figure 5. The electrical conductivity along the SiC fibers and in transverse direction in a logarithmic scale.*

## 3. Conclusions

Summarizing we can conclude the following:

- The experimental set-up and the technology for the deposition of the electrical contacts are appropriate for the measurement of the electrical resistivity of the SiC/SiCf composites in the temperature range from RT to 1000 $^0$C.

- The temperature dependence of the electrical resistivity for 2D SiC/SiCf composites was measured both along and in perpendicular direction with respect to the fibers.
- Electrical resistivity must be correlated with the spectrometric data in order to elucidate the nature of the sublimation product.
- For the future a study regarding the influence of the microstructure on the electrical resistivity of SiC/SiCf composites would be very useful.

## 4. Foreseen activities

The following activities are remaining for 2006:

- Further electrical resistivity measurements for 3D SiC/SiCf composites are to be carried out. For the time being the 3D samples are not available.
- The electrical characterization of 2D and 3D irradiated SiC/SiCf composites