Characterisation of electrical resistance for CMC Materials up to 1200 °C

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Abstract. Damage to thermal protection systems (TPS) during atmospheric re-entry is a severe safety issue, especially when considering re-usability of space transportation systems. There is a need for structural health monitoring systems and non-destructive inspection methods. However, damages are hard to detect. When ceramic matrix composites, in this case carbon fibre reinforced silicon carbide (C/C-SiC), are used as a TPS, the electrical properties of the present semiconductor material can be used for health monitoring, since the resistivity changes with damage, strain and temperature. In this work the electrical resistivity as a function of the material temperature is analysed eliminating effects of thermal electricity and the thermal coefficient of electrical resistance is determined. A sensor network is applied for locally and time resolved monitoring of the 300 mm x 120 mm x 3 mm panel shaped samples. Since the material is used for atmospheric re-entry it needs to be characterised for a wide range of temperatures, in this case as high as 1200 °C. Therefore, experiments in an inductively heated test bench were conducted. Firstly, a reference sample was used with thermocouples for characterising the temperature distribution across the sample surface. Secondly, electrical resistance under heat load was measured, time and spatially resolved. Results will be shown and discussed in terms of resistance dependence on temperature, thermal coefficient of electrical resistance, thermal electricity and electrical path orientation including an analysis on effective conducting cross section. Conversely, the thermal coefficient can also be used to determine the material temperature as a function of electrical resistance.

1. Introduction
In a time in which the ecological aspect of space travel is becoming increasingly important, re-usability of space-vehicles is a major concern. Building and optimizing space vehicles for re-entry is not a trivial matter as, depending on the flown trajectory, temperatures of over 3000K can be reached during re-entry. It is a severe challenge for scientists to develop materials and sensors which can withstand these extreme conditions. For gaining better knowledge of the re-entry phase and for subsequently optimizing structural safety margins, sensors are needed which can collect data and monitor the heat shield during an actual re-entry flight. This is not only important for developing better TPS, but also for detecting defects during flight. In case of minor or major defects, re-entry trajectories can be adapted and might not only be able to save a spacecraft but also save lives.

Several methods are available to monitor the condition of a heat shield in ground testing. During actual re-entry flight; however, most of these methods cannot be used as most of them need external optical access. Common heat shield instrumentation comprises temperature, heat flux and pressure sensors. There is also a range of recession sensors available or in development e.g. the Hollow aErothermal Ablation and Temperature (HEAT) sensor from Santos et. al. [1].
Two isolated wires of platinum and tungsten are wound on a kapton tube which is filled with heat shield material. During re-entry an electrical circuit between the wires is closed when the kapton chars at a known temperature.

Other nondestructive monitoring techniques for TPS include ultrasonic scanning and thermography [2]. Fibre optic sensors can be used for measuring strain and temperature by means of wavelength shifts. One such sensor was developed for the structural core of the X-38 spacecraft [2]. Other concepts use acoustic emissions and ultrasonic systems, such as timed echos of sound waves on an ablator surface [3, 4]. Carbon fiber reinforced polymers (CFRP) and ceramic matrix composites (CMC) material have been investigated for in-situ monitoring of laminar failure using a passive method of resistance measurement [5, 6, 7, 8, 9, 10]. Special emphasis of this study is that the sensor should be reusable. Therefore the approach is that the material itself, which is reusable, is being used as the sensor. It is important for health monitoring systems to know certain properties of the materials used, to determine whether the reason of a change in resistance is due to a change in temperature, strain or to damage. In this work the main focus is on the influence of the sample geometry and the temperature on the resistance, including compensation for thermally induced electric current.

2. Material
The material used is a CMC material, carbon fibre reinforced silicon carbide (C/C-SiC) as can be seen in Fig. 1. The lay-up are stacked 0/90°plies of a 2/2 twill weave. The material is highly stressable and unlike monolithic ceramics not brittle. It contains the positive properties of a ceramic, such as corrosion, abrasion and oxidation resistance, high temperature tolerance and adds to them a higher damage tolerance with a quasi ductile fracture behaviour. C/C-SiC has a long history, it has been developed since the 1980’s at the German Aerospace Center (DLR) [11] and has already seen many different fields of use, including TPS on re-entry vehicles and in combustion chambers. The C/C-SiC material is manufactured at the DLR Institute of Structures and Design with the liquid silicon infiltration (LSI) process [11].

The material has been qualified in plasma wind tunnel testing and during real re-entry flight such as the SHEFEX missions [12]. For use in this work the plates are cut into rectangular samples of geometry 120 mm x 320 mm x 3 mm (width x length x thickness). A C/C-SiC sample with a polished surface can be seen in Fig. 1. Further information about the production process can be found in [11] and [13]. Measurements at room temperature in previous works have shown a specific electrical resistance for C/C-SiC of approximately 38 \( \mu \Omega \text{m} \) [5].

3. Theoretical approach
Change in electrical resistance can be due to a multitude of reasons and is therefore a useful indicator for a range of effects that should be detected in a sample. Different sample sizes, geometries or contact layouts result in different electrical resistances. The electrical resistance is a function of the sample temperature. Damage in a sample from small fractures to complete destruction of a sample can be measured with electrical resistance [14]. To be able to determine the cause of a change in electrical resistance different approaches must be taken.
3.1. Effective cross sectional area

The related cross sectional area $A^*$ of the sample is important, as the panel shaped samples have a much bigger cross sectional area $A$ than a usual conducting wire has. The relation for resistance $R$

$$ R = \rho \frac{l}{A} \quad (1) $$

to specific resistivity $\rho$ is valid for electrical conductors with a much bigger length $l$ than width. The area $A$ needs to be replaced by the effectively conducting area $A^*$ for use with the wide samples in this work. The related cross sectional area $A^*$ needs to be experimentally determined. Method and results are described below.

3.2. Thermal electricity

Adding to the temperature dependence of the electric resistance for some material combinations, a nonhomogeneous temperature distribution results in thermoelectric charge. Therefore, between two measurement points a vectored voltage is measurable depending on the temperature distribution and materials used. This voltage has a large influence on the resistance measurements, as the resistance of the material is measured by measuring the voltage drop over the sample. As an example, the voltage drop of 10 V over a sample at a current of 10 A according to Ohm’s law

$$ U = R \cdot I \quad (2) $$

would result in a measured resistance of 1 $\Omega$. With an induced thermoelectric voltage of -5 V, i.e. antipodal to the measurement direction as depicted in Fig. 2 case (1), the measurement according to

$$ U_{\text{measured}} = U_{\text{sample}} - U_{\text{thermic}} \quad (3) $$
yields 15 V resulting in a falsely evaluated resistance of 1.5 $\Omega$. With an induced thermoelectric voltage of +5 V, in measurement direction as is shown in Fig. 2 case (2), the measurement yields 5 V resulting in the falsely evaluated resistance of 0.5 $\Omega$.

To derive the real resistance from measurements and to subtract the thermoelectric voltage, both resistance and voltage measurements are conducted. With those measurements the values can then be corrected mathematically, since the measuring amperage is known and the measured resistance is known. Using Eq. (2) the resulting voltage can be calculated, the vectored voltage can then be found by Eq. (3).

3.3. Temperature dependence

Furthermore, it is known that the electrical resistivity $\rho$ is the reciprocal quantity of conductivity $\sigma \sim e^{-\alpha/T}$, with $\alpha = E_g / 2k$ representing energy gap over Boltzman’s constant. The resistivity of a semiconductor is thus dependent of temperature. With $T_0$ as reference temperature and $T$ as material temperature it can be described by

$$ \rho(T) = \frac{1}{\sigma} = C (\rho(T_0)) e^{\alpha(T)} \quad (4) $$
4. Experimental set-up

Three different experimental set-ups are used in this work. All of them use the test bench for resistance measurements (WISTAM), which measures the electrical resistance of a sample time and spatially resolved. Samples with up to twelve contacts on either side can be used. WISTAM is clocking the measurements, measuring first one contact on one side with all of the contacts on the other side, one after the other, then the next contact and so on, resulting in 144 single measurements. A schematic of the measured virtual current paths is shown in Fig. 3. A detailed description of the test bench and the measuring principle can be found in [14]. The individual test set-ups are described in the following section.

4.1. Mesh Experiments

For a fundamental analysis of the measurement principle a hexagon mesh was used. On each side 24 wires for twelve contacts were soldered and connected to the test bench for four-wire-measurements of electrical resistance. The hexagon mesh was chosen because of its distinctive feature being that all distances from one side to the other are the same regardless of the path taken, as long as the length bigger than the width. As all the possibilities of current distribution have the same distance from measuring point A to measuring point B, it can be said that a difference in the measured resistance can only be a result of a difference in the related cross sectional area. As a mesh has very distinctive possible paths it is also a very good demonstrator for damaged sections, as different paths can easily be cut to show a change due to cut fibres.

4.2. Edge Experiments

This mesh is also used for experiments showing that electrical resistance near the edge of a mesh is higher than in the center of a mesh. Section 3.1 described why this investigation is needed. This experiment was also conducted with C/C-SiC material. A rectangular sample was installed with two contacts, and two wires were soldered to each contact for a four-wire-resistance-measurement. The sample layout is shown in Fig. 4. As pictured the contacts were on one side of
a rectangular sample and the measurement section marked with $l$ is seen as the sample length. Therefore, the cross section $A$ is bigger as it would be in a rod. In the experiment the sample was measured and then shortened, indicated in Fig. 4 by the arrow along the red line, and afterwards measured again, repeating this process until the sample was too small to be shortened further.

4.3. High Temperature Experiments
Electrical resistivity of material samples is measured under high temperature. The measurements are carried out in the temperature range from room temperature to 1200°C. The samples are heated inductively by a hot test facility [5]. The samples are placed inside the susceptor ring during the experiment. To avoid oxidation of the carbon in the sample material, all high temperature experiments are carried out under vacuum conditions at 100 Pa with the whole experimental set-up, as can be seen in Fig. 5, being placed inside a vacuum chamber. The heating process takes less than 3 minutes. Thermocouples, a pyrometer and an infra-red camera are used to monitor the temperatures and a pressure sensor for monitoring of the chamber pressure is installed. To make sure that the sample is heated homogeneously, the highest temperature is held for 2 minutes before cutting the power. The induction coil induces current in the susceptor but also in the measurement devices. Therefore, measurements are only taken during the cooling period when the generator and therewith the magnetic field are shut off. Depending on the starting temperature this lasts for one to five hours.

4.4. Sample Layout and Test Procedure for High Temperature Experiments
The panel shaped samples are made of C/C-SiC and have 12 small drilled holes on either side, as can be seen in Fig. 6. In the holes, wires are riveted with copper rivets to connect the sample electrically. The contacts are connected to WISTAM to monitor voltage and electrical resistance of the sample. The temperature of the sample surface and the riveted contacts, which are in water-cooled clamps situated outside the graphite cylinder, are monitored during cool down, as well as electrical resistance and voltage on the sample, as there is a thermoelectric potential due to different temperatures over the sample surface.

5. Results
Preparatory measurements are mandatory that correct calculations of the high temperature experiments can be made, with all effects mentioned in the theory section being considered. One of the important measurements is to know the effective relating cross section $A^*$ as it was described previously in Section 3.1. It was observed in wide samples that near sample edges the measured resistance is higher than the theory according to Eq. (1) indicates. This is accounted to the measurement current having a limited cross section to pass across the sample. In turn, in wide sample centres, the current is not using all of the space provided. For compensation of this effect a materially and geometrically dependent correlation factor is used. With it, a measured value on the edge of a sample can be compared to a measured value in the center of the sample.

5.1. Edge effects in a mesh
To show the edge effect, a hexagon mesh was used as was shown in the experimental set up in Section 4.1. Measurements of one thread of 494 mm length gave a resistance of 323 mΩ. Eq. (1) yields with the thread diameter 0.514 mm a specific resistivity of 0.1359 µΩm of the material.
Figure 7: (a) Electrical resistance over path position of the mesh sample 1 (590 mm) and mesh sample 2 (350 mm). (b) Electrical resistance over path position of panel shaped C/C-SiC samples PH1878P-A and PH1878P-B.

Measurement of electric resistance between directly opposite contacts across the sample yields the results shown in Fig. 7 (a). The figure shows that the outer three to four measuring points have a higher resistance than the inner measuring points. This is due to the propagation of the electrical field in the mesh, which is restrained by the edge. Since the resistance is constant at the measuring points in the center of the sample, it is assumed that the resistance between measuring point 7-8 and measuring point 15-16 has reached its maximum effective related cross sectional area. As it is assumed that the current distribution in the grid is homogeneous the cross sectional area can be calculated as

$$A^* = 2(1 + x + n) \cdot A_0,$$

where $A_0$ is the cross sectional area of one wire, $n$ is the number of wires from the measuring point mentioned above to the nearer edge of the sample and $x$ the number of maximum wires effectively used. In this example with this material $n_{max} = x = 4$. As for every contact two paths can be taken in the grid, due to the nature of the hexagon grid, which has a double wound wire on both longer sides of the hexagon, the number of paths is multiplied by two. The results from Eq. (1) using the effective cross sectional area from Eq. (5) above are also given in Fig. 7 (a). The concurrence is good with deviations at the edge for Sample 1.

5.2. Edge effects in C/C-SiC

Further experiments in C/C-SiC samples also show this edge effect, as shown in Fig. 7 (b). Therefore, experiments were conducted to determine $A^*$ in C/C SiC as described in Section 4.2.

In Fig. 8 the measured electrical resistance is shown over the sample width $b$, a function of which is the cross sectional area $A = b \cdot h$ indicated by the blue line. The calculated resistance with Eq. (1) is shown as the red line in Fig. 8. It can be seen that while the sample is wider than 40 mm the measured resistance is constant. This indicates that, as mentioned above, the current is not using all of the space provided. With width smaller than 40 mm the measured resistance
Figure 8: Measured and calculated resistance of a sample which was shortened for each measuring point over the sample length.

starts rising and below a width of approximately 25 mm it is nearly coincident with the theory from Eq. (1).

This effect of a rising resistance near an edge, as shown in this section and Section 5.1, can now be used to calculate an effective cross sectional Area $A^*$ in a C/C-SiC sample. $A^*$ can be calculated with the specific resistance of the material and the lowest measured resistance in the middle of the sample using Eq. (1). The effective sample width $b^*$ can be calculated using

$$A^* = b^* \cdot h$$

with the sample height $h$. $b^*$ consists of two parts

$$b^* = b_1^* + b_2^*$$

whereas $b_1^*$ is a fixed value independent of the contact location, only dependent of the sample and the sample material, and $b_2^*$ is dependent on the contact position. $b_2^*$ can be calculated with an experimentally determined quadratic formula

$$b_2^* = -8.282x^2 + 0.119x + 0.041$$

whereas $x$ is the distance of the contact to middle of the sample for samples with a width of 0.12 m as used here. The calculated values for the electrical resistance for each contact pair using Eq. (8) is shown in Fig. 7 (b) and show a very good correlation of measured and calculated values.

5.3. Damage in a mesh

When all paths in a mesh can be compared, irrespective of their location, damage to a mesh can be both detected and located. The hexagon mesh from Sections 4.1 and 5.1, is used. Areas are cut out during measurements, as can be seen in schematic pictures of Fig. 9. Green and violet colours indicate current lines along the upper and lower edges of the sample. The violet line
Figure 9: Plot of resistance change and plot of resistance during cutting of connections in a grid, which is shown schematically in the upper pictures.

The diagram of Fig. 9, shows resistance $R$ and its deviation $\Delta R = R(t) - R(t-1)$ over cuts and respectively time. When a cut is made the different measurement pole permutations correspond to the cut depending on their location. This can be seen in the different peaks in the lower section of Fig. 9. The first cut is at the right of the grid, far away from the violet line. Therefore, for the cuts 4/1 to 4/7 the measurement at points 1 and 2 (solid green curve) corresponds to the measurement points 1 and 2, which is a straight line from left to right at the top of the grid. The green line corresponds to the measurement points 23 and 24, which is a straight line from left to right at the bottom of the grid. The experiment is conducted by measuring the electrical resistance across the sample while cuts are being made along the regions indicated by the white space and the arrows in the schematic pictures.
responds with peaks, which means a rise in the electrical resistance, while the violet line does not respond at all. Apart from their last incisions, the subsequent cuts at 5/1 to 5/7 and 3/1 to 3/7, right next to the location of the first cut, do not induce much change of resistance. This is explained by the current path between the measurement points not being strongly influenced. The cuts at locations 36/1-7 and 40/1-7, at the left edge of the mesh, in turn do receive a resistance response.

On the upper right hand side, in a dashed green line, a potential current flow at the end of the experiment is shown in the pictogram. The lower middle area of the grid is completely removed, in the upper part are two long cuts, which each then only leave one connection in the grid. The cutting of the first of those mentioned two long cuts, cut 22/14 to cut 22/09 in the diagram in Fig. 9, can be seen from the 5000th second. The resistance response to the cuts can be seen immediately and rises with each cut. Both, the violet and the green, are affected and therefore both are rising with each cut. The last cut, 22/9 leaves only one connection and has therefore the biggest effect with the highest difference in resistance before and after the cut.

This shows that the nearer a virtual current path is to a damaged section the more it will affect the measured electrical resistance. It is not necessary for the damaged section to be directly in the virtual current path. A bigger impact, like the cut that removed the lower part of the grid, shown as ‘Cut horizontally’ in Fig. 9, also affected the measurements from measuring point 1 and 2. Even though the virtual current line, the violet line in the pictures of Fig. 9, is not directly affected, a rise in resistance can still be measured. However, it is lower than the directly affected green line.

In Fig. 9 the cumulated resistance change of the different cuts can also be seen in dashed and dash-dotted lines. An additional small dashed line is shown (red), which corresponds to the measurement points 11 and 12. From 700th second, the red line shows less resistance (32 mΩ)

![Figure 10: Electrical resistance for all current paths during testing.](image-url)
than the green, 35 mΩ, and violet, 38 mΩ, lines. This is due to the edge effect, described in Section 5.1, as the virtual current line of the measuring points 11 and 12 is in the center of the grid. During the experiment, the green line, which is more deeply affected by the cuts starts rising, which can also be seen by the corresponding peaks of the resistance change ΔR. The red line starts rising earlier than the violet line, as it is nearer to the cuts and therefore affected more by them. As all of the 144 virtual current paths are affected differently, depending on the distance between the area of defect and the virtual current path as well as the impact of the defect (e.g. size), the location of the defects can be traced to the appropriate areas.

5.4. Defects in C/C-SiC
Damage can also be detected in C/C-SiC, as is shown in Fig. 10. A defect was inserted near measurement point 12 of Fig. 3. All connections with measurement point 12 were deeply affected and group together after the damage was inserted between the 100th and 200th second in the upper area. Going from measurement point 10 to 8 and 4 the effect shows less in the data and the measurement areas group clearly together. This shows that the behaviour of the material can be compared to the behaviour of the mesh. This experiment is described in detail in [14] and shall not be discussed here further.

5.5. High Temperature Experiments
Further characterizations are carried out in an inductively heated test bench, described in Section 4.3. In Section 3.2 the effect of thermal electricity and its influence on resistance measurements was described. Fig. 11 shows the experimental data during the cooling process of a C/C-SiC sample PH1878P-A. The data recording started at a temperature of 1154°C and ended at a temperature of 400°C. The potential difference of the center measuring points 7 to 8 was reverse to measuring points 9 and 10, resulting in mirrored data as shown by the voltage curves in Fig. 11. The red and purple lines show the measured resistance of both connections. Resistance and voltage measurements were done alternately. The measuring device is using a fixed current of 10 mA, and the voltage drop over the sample can therefore be calculated using Ohm's law, \[ U = R \cdot I. \] Using this voltage with Eq. (3) and the measured voltage yield the voltage drop over the sample without thermal electricity. This can now be converted again using Eq. (2) into resistance values. The result represents the true material resistance and is shown as the dark blue and turquoise lines in Fig. 11. The curves show a linear behaviour of increasing resistance over decreasing sample temperature.

With the measured data the temperature coefficient of the sample PH1878P-A can be calculated with Eq. (4) and \( \alpha = 207 \) [5] to \( C = 2.843 \cdot 10^{-5} \pm 1.118 \cdot 10^{-6} \).

6. Conclusion
Theories for determination of the related cross sectional area as well as for behaviour near sample edges were presented. With a representative hexagonal mesh these effects were further analysed and calculations were shown. The findings are transferred to the heat shield material C/C-SiC. Thermal electricity in hot samples was discussed and data processing to eliminate this effect was shown. With this knowledge, experiments in an inductive heated test bench were conducted up to 1200°C showing this thermal electric effect. Measurements of electric resistance and voltage were performed, discussed and analysed. The true material resistance was calculated and shown and the thermal coefficient of the electrical resistance for the used sample was determined.

The results lead to a better understanding of electrical effects in samples and allow the test bench to be enhanced for more accurate measurements, such as double measurements in reversed directions to eliminate thermal electricity effects. They also confirm established theories and give a better insight into the used material for characterisation of material properties.
Figure 11: Voltage, resistance measured and corrected over maximum temperature of sample PH1878P-A.

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