ECCM16 - 16TH EUROPEAN CONFERENCE ON COMPOSITE MATERIALS, Seville, Spain, 22-26 June 2014

ELECTRICAL RESISTANCE HEATING – A METHOD FOR BINDER ACTIVATION IN CFRP PROCESSING?

Y. Grohmann*a, F. Zacharias*a, Dr. F. Kruse, Prof. M. Wiedemann

*aInstitute of Composite Structures and Adaptive Systems, Composite Process Technology, German Aerospace Center (DLR), Ottenbecker Damm 12, 21684 Stade, Germany
*yannis.grohmann@dlr.de and fabian.zacharias@dlr.de

Keywords: conductivity measurement, resistance heating, binder activation, carbon fiber preforming

Abstract
The preforming of dry carbon fibers (CF) is a time- and money-consuming step in the production process of carbon fiber reinforced plastic (CFRP) parts. In this step, multiple CF layers are formed into a three-dimensional shape and need to be fixed to each other. Common fixing technologies are stitching or the use of binders. Most binders need to be activated thermally. Different technologies have been developed for that (chapter 1). This paper introduces a new method for activating the binder, which is presented in chapter 2. In order to find the most important process parameters for a fast, economical and energy-efficient process, the resistance behavior of different CF materials are investigated theoretically (chapter 3) and practically (chapter 4).

List of abbreviations

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Unit</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha_C )</td>
<td>[1/K]</td>
<td>Temperature coefficient of the electrical resistance of carbon</td>
</tr>
<tr>
<td>( \rho_{CF} )</td>
<td>[( \Omega ) mm(^2)/m]</td>
<td>Specific electrical resistance</td>
</tr>
<tr>
<td>( A_{EI} )</td>
<td>[mm(^2)]</td>
<td>Contact area of the electrode</td>
</tr>
<tr>
<td>( F )</td>
<td>[N]</td>
<td>Force for compaction</td>
</tr>
<tr>
<td>( l_{CF,\perp} )</td>
<td>[m]</td>
<td>Thickness of one CF layer</td>
</tr>
<tr>
<td>( n )</td>
<td></td>
<td>Number of layers</td>
</tr>
<tr>
<td>( T )</td>
<td>[°C]</td>
<td>Temperature</td>
</tr>
<tr>
<td>( R_{20} )</td>
<td>[Ω]</td>
<td>Resistance at T=20°C</td>
</tr>
<tr>
<td>( R_{CF,\perp} )</td>
<td>[Ω]</td>
<td>Resistance of one CF layer in through-thickness direction</td>
</tr>
<tr>
<td>( R_{\text{material}} )</td>
<td>[Ω]</td>
<td>Material resistance of all CF layers</td>
</tr>
<tr>
<td>( R_T )</td>
<td>[Ω]</td>
<td>Resistance at a certain temperature T</td>
</tr>
<tr>
<td>( R_{\text{contact}} )</td>
<td>[Ω]</td>
<td>Sum of the contact resistances</td>
</tr>
<tr>
<td>( R_{\perp,n} )</td>
<td>[Ω]</td>
<td>Electrical resistance in through-thickness direction of n CF layers</td>
</tr>
</tbody>
</table>

1. State of the Art

During the last couple of years, semi-finished carbon fiber (CF) products with applied binders have come to the fore in the process of building dry CF preforms which are injected with resin by the Resin Transfer Molding (RTM) process. Once heated up to temperatures above 100°C, these binders start to liquefy and will fix adhering materials to each other while cooling down. Apart from their fixing properties, binders are also valued for their ability to improve the
stiffness of textile preforms and to influence the mechanical properties of the finished composite part. Different methods of heating up the binder have been developed over the years. Conductive or convective technologies are often used, but are too slow for high-volume production. Another common method is infrared radiation, which is used especially for the heating-up of larger surfaces. Despite the fact that induction heating is a very fast method and the thermal energy is produced to a certain extent volumetrically, it is not widely used. The reasons for this are its high costs, the limitation to electrically conductive material and the need for multiple compressed and multiaxially oriented layers of CFs. Another way to generate heat is the use of ultrasound. A disadvantage of this technology is the possible occurrence of fiber ondulations after the process.

The principle of direct electrical resistance heating of dry CF preforms has come more into focus within the last few years. Remaining challenges for this technology are an even distribution of heat throughout the preform and getting good electrical contacts to the fibers.

2. A new approach to electrical resistance heating of CF preforms

At the Institute of Composite Structures and Adaptive Systems of the German Aerospace Center (DLR) in Stade, Germany, a new method for binder activation is being developed. Similar to spot welding of metals, only a locally restricted part of the preform is heated. In order to concentrate the electric flow, and thus the heat generation, on a small spot, the fiber preforms are compressed and contacted by an electrode arrangement in through-thickness direction of the CF stack (see Figure 1 (left-hand side)). When voltage is applied, the fibers heat up in a completely volumetric way caused by the Joule effect. As a result, a balanced distribution of heat in through-thickness direction can be expected even for thick preforms. Hence, the method allows a locally restricted fixing and consolidating of thick multilayer preforms with binder in milliseconds. Important process parameters are the resistance of the material and all influences on it, which are investigated and discussed in the following chapters.

Figure 1: Resistance model of multilayered CF materials in through-thickness direction
3. Principles in modeling the resistance of CF in through-thickness direction

Carbon is an electroconductive material, where Ohm’s law can be applied. The specific resistance $\rho$ of CF varies from $7 \frac{\Omega \text{mm}^2}{m}$ [1] to $17 \frac{\Omega \text{mm}^2}{m}$ [2].

While there is an international standard for testing the resistance of CF in longitudinal direction (ISO 13931), the testing of the resistance in through-thickness direction of multilayered fiber stacks is not standardized. In the following sections, a theoretical model and a test set-up for the determination of CF resistances in through-thickness direction is described.

3.1. Influences on the electrical resistance of CF in through-thickness direction

Pressure

Preliminary tests have shown that the pressure has a major influence on the resistance in through-thickness direction of CF layups. Due to the flexibility of dry CF products, higher pressures cause a compaction of the CF materials. This improves the contact in between the CF layers and to the electrodes and leads to a decrease in resistance. Increasing pressure can reduce the influence of isolators like toughener fleeces or binder coatings.

Temperature

The resistance’s temperature dependency can be expressed by formula (1), where $R_{20}$ is the resistance at $20^\circ\text{C}$ and $T$ the target temperature. The temperature coefficient for carbon is $\alpha_c = -0,5 \times 10^{-3} \frac{1}{K}$ [3]

$$R_T(T) = R_{20}(1 + \alpha_c(T - 20^\circ\text{C})) \quad (1)$$

As a result, the theoretical resistance drop of carbon at a temperature of 120 $^\circ\text{C}$ is just 5%.

Electrodes

Metal surfaces (e.g. copper electrodes) are not perfectly even. Instead, they have a certain surface roughness and an impurity layer (see Figure 1). Impurity layers (e.g. binder residue) reduce the area of contact and thus increase the resistance.

Time

It could be observed that the electrical resistance dropped over time, which is likely to be caused by the settling effect of loose fiber material that arranges itself into the closest packing possible. It is to be expected that after applying a very high pressure and releasing it to a certain extent, the resistance will be lower as compared to the case of just applying this kind of pressure.

Layup and orientation

The number of CF material layers directly relates to the sum of resistances between the layers. Therefore, the total resistance will be higher with more layers between the electrodes. Depending on the material used, isolating layers like toughener fleece and binder will increase the total resistance. It is assumed that the orientation of two adjacent fiber layers also has an impact on the resistance.
3.2. Resistance model of multilayered CF materials in through-thickness direction

The resistance of multilayered CF material in through-thickness direction can be modeled as a series connection.

The total resistance in through-thickness direction comprises the sum of the contact resistances $R_{contact}$ and the material resistances $R_{material}$, as expressed in Formula (2).

$$R_{\perp n} = R_{contact} + R_{material}$$  \hspace{1cm} (2)

$R_{material}$ is the sum of the material resistances of all CF layer. The material resistance of one CF layer ($R_{CF\perp}$) can be approximated via formula (3), where $\rho_{CF}$ is the specific resistance, $l_{CF\perp}$ is the thickness of one CF layer and $A_{El}$ is the cross section of the electrode.

$$R_{CF\perp} = \frac{\rho_{CF} l_{CF\perp}}{A_{El}} = \frac{10 \text{ $\Omega \text{ mm}^2$}}{m} \cdot \frac{0.15 \cdot 10^{-3} m}{702 \text{ mm}^2} \approx 2.14 \cdot 10^{-6} \Omega$$  \hspace{1cm} (3)

If all layers are alike, the total material resistance can be calculated by multiplying the material resistance of one CF layer ($R_{CF\perp}$) with the number of layers used ($n$). Tests in which the material was exposed to high-power electrical heating and high compression forces showed that the resistance of one CF layer is close to zero and several orders of magnitude smaller than the contact resistances.

$$R_{material} = n \cdot R_{CF\perp} \ll R_{contact}$$  \hspace{1cm} (4)

For a small number of layers $n$, $R_{material}$ is therefore neglected in this model.

Depending on the layup, the contact resistance $R_{contact}$ is made up of different combinations (see Table 1).

<table>
<thead>
<tr>
<th>$R_{El-CF}$</th>
<th>Contact resistance between the electrode and the CF layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_{El-CF_{TF}}$</td>
<td>Contact resistance between an electrode and a CF layer separated by a toughener fleece</td>
</tr>
<tr>
<td>$R_{CF-CF}$</td>
<td>Contact resistance between two CF layers</td>
</tr>
<tr>
<td>$R_{CF-CF_{TF}}$</td>
<td>Contact resistance between two CF layers separated by a toughener fleece</td>
</tr>
</tbody>
</table>

Table 1: Possible contact resistances

The resistance of a multilayered CF layup $R_{\perp n}$ can be calculated as the sum of different contact resistances (see Formula (5)). Depending on the layup, $a, b, c, d$ equal the quantity of contact resistances.

$$R_{\perp n} = a \cdot R_{El-CF} + b \cdot R_{El-CF_{TF}} + c \cdot R_{CF-CF} + d \cdot R_{CF-CF_{TF}}$$  \hspace{1cm} (5)
4. Experiments and results

The verification of the resistance model and the influence of the process parameters requires a test rig that allows repeatable test conditions.

4.1. The test rig

The test rig is designed to analyze all relevant process parameters. One of the main parameters, the compaction force, is supplied by a pneumatic cylinder. The cylinder pressure is controlled by a pressure regulating valve, while the cylinder speed is reduced by manual flow-control valves.

The CF material is placed between two copper electrodes. In order to assure parallel surface alignment, at least one of the electrodes is mounted with a spherical bearing. Different types of electrodes were used during the testing.

The resistance itself was measured dynamically with a benchtop power supply as well as through static control checks with a multimeter. All analog signals for controlling, regulating and measuring the test rig’s parameters were translated from and to a computer via USB data conversion boxes. An additional temperature sensor was installed to monitor temperature changes during testing.

4.2. Method of measuring

The measurement starts when the cylinder is activated. The upper electrode moves down and the resistance of the fiber layup is measured as soon as the circuit is closed. The resistance is calculated by applying a voltage and measuring the current. In order to record settling effects of the fiber layup and their influence on the resistance, the measurement lasts for 30 seconds. The final resistance is the calculated mean value of the last 2 seconds of the resistance measurement. The main machine parameters, which have been controlled and measured, were the cylinder’s pressure as well as voltage and current. Based on [9], the environment’s temperature was also logged. For all resistance measurements, a current limitation of 0.5 A had been set. For one set of parameters, 10 measurements have been conducted.

4.3. Tested material

The fibers used for the testing were Toho Tenax intermodular strength (IMS) fibers with an aerial weight of 194 g/m² per layer. Each layer was supported by a TA1900 toughener fleece with an aerial weight of 5 g/m², which is used to increase compression after impact performance. Three different non-crimped fabrics (NCF) were in use: a Triax (-45°, 0°, +45°), a Biax (+45°, -45°) and a unidirectional (UD) (0°) NCF. In order to analyze the influence of the toughener, another UD material without the fleece was tested. All of them are coated on one side with EPR05311, an epoxy-resin-based binder with a thermoplastic behavior.

4.4. Results

It could be demonstrated that pressure, time and layup have a major influence on the electrical resistance of CF preforms. The graphs (Figure 2 - Figure 5) show that the variation of values is increasing when more layers are tested and lower pressures are applied. Throughout the entire testing, the toughener fleece proved to be a major factor in increasing the resistance.
Figure 2: Total resistance after 30 seconds over number of layers for different materials at $5 \times 10^5$ Pa.

Figure 3: Total resistance over time for different materials at $5 \times 10^5$ Pa.

Figure 4: Total resistance over surface pressure for different materials.

Figure 5: Individual contact resistances over surface pressure.
Figure 2 shows the linear relation between the resistances of different semi-finished CF products over the number of layers. The graph indicates that the number of layers respectively the number of contact resistances has a linear impact on the total resistance. The resistance of the stack composed of UD has a higher resistance then the ones composed of Biax or Triax, even though the number of layers is equal. A reason for this phenomenon could be different layer orientations, extra binder between UD layers or the way the layers are stitched together.

Figure 3 shows that after applying pressure, the resistance will asymptotically drop over time to a pressure-dependent minimum. The higher the pressure, the lower this minimum gets. For low pressures, resistance drops up to 20% and more and was still falling after a period of 30 seconds. With higher pressures, the minimum resistance was reached much faster. A pressure of 30·10^5 Pa resulted in a nearly constant resistance after 5 seconds.

Figure 4 illustrates how the resistance of different semi-finished CF products asymptotically decreases with higher pressures until all products reach a similar resistance of about 200 Ωmm². Biax has a higher resistance than Triax, which cannot be explained by the model from section 3.2. Maybe this behavior is caused by the different orientations of adjacent CF layers or different amounts of binder applied during the production process.

Figure 5 shows that all contact resistances involved decrease with higher pressures. Every time a CF layer is separated from another CF layer or an electrode by a toughener fleece at pressures under 10·10^5 Pa, the resistance is especially high. The contact resistance is lowest between two CF layers that are flexible and conductive.

4.5. Error analysis

The test set-up was designed to measure resistances and heat development during the spot welding of multilayered carbon fiber stacks. This is why, the resistance was measured via a laboratory power supply with a high-power output. Since resistivity changes were expected during the heating up of the fiber stacks, the measurements were executed with the lowest power output possible. However, test results showed that even small output variations of only a few watts already had a strong influence on the measured resistance. The fact that the influence was much stronger than expected, led to the conclusion that further tests with a closed-loop control with online heat measurement ought to be performed.

Another strong influence that was also expected to be much smaller was the contamination of the electrodes. Especially during test runs with higher temperatures where one of the electrodes has contact with the binder, the electrode needs to be cleaned after every test run in order to receive accurate results.

5. Conclusion and outlook

The tests showed that the total resistance of a fiber stack can be approximated by the contact resistances between the CF layers and to the electrodes equals. As insulators, the binder, the knitting yarns and especially the toughener fleece had the strongest influence on the contact resistance. They also were the main reason for measurement deviations.

The results of this paper show that the total resistance of a dry CF preform in through-thickness direction could be controlled by applying well-measured forces to the contact electrodes. It could be proved that time and layup has a strong influence on the resistance.
The relations between power input, heat generation and resistance change will be the objective of future research. Furthermore, the development of hot spots or heat-development irregularities has to be investigated. Another factor that will be examined closer in the future is the effect of different layer orientations on the resistance.

At the Institute of Composite Structures and Adaptive Systems of the German Aerospace Center (DLR-FA) in Stade, a fully automated RTM process for the production of 100,000 airplane frames per year is being built. In the future, this heating technology can be used during the complex preforming process.

References