ELECTRICAL CONDUCTIVITY CHARACTERISATION OF STOCHASTIC ZIGZAG PATHS IN UNIDIRECTIONAL C/PAEK

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ABSTRACT

Induction welding has emerged as a promising technology for fusion bonding of carbon fibre reinforced thermoplastic composites. The process relies on the generation of eddy currents, induced by an alternating electromagnetic field, within a composite susceptor. The eddy current density and the resulting heat generation is directly influenced by the electrical conductivity of the carbon fibre network, making accurate measurement of this orthotropic electrical property crucial for physics-based process simulations. The present study focuses on the characterisation of the in-plane transverse electrical conductivity in unidirectionally reinforced PAEK composites using the six-probe method. The investigation specifically addresses the stochastic nature of the conductive paths within the fibre networks by considering the effects of the specimen dimensions and the uniformity of the current density field. The conductivity data obtained within this study is found to be consistent with earlier characterisation efforts that employed a different analysis technique. The agreement serves as a reliable validation of the methodology used in the previous study and provides valuable insights for future research in the field of electrical characterisation.

1 INTRODUCTION

Induction welding [1,2] is an attractive technology for fusion bonding of carbon fibre reinforced thermoplastic composites. In this process, eddy currents are induced in the composite assembly by an alternating electromagnetic field, heating the parts. It is challenging to generate eddy currents in unidirectional (UD) reinforced plies, because of their low transverse electrical conductivity. Therefore, formation of significant eddy currents relies on the ply interfaces where closed-loop conductive paths are present in the fibre network through changes in stacking orientation. The advancement and broader adoption of induction welding technology in industrial applications using UD ply-based TPCs relies on improving control and gaining deeper insight into the welding process through physics-based process simulations. A key aspect in this endeavour is the thorough understanding and quantification of the electrical conductivity behaviour exhibited by UD plies, and the subsequent integration of this knowledge into constitutive models.

The intralaminar electrical conductivity of a UD reinforced ply is governed by its three principal conductivities. The principal conductivity tensor is expressed in the material coordinate system as

$$\boldsymbol{\sigma} = \begin{bmatrix} \sigma_1 & 0 & 0 \\ 0 & \sigma_2 & 0 \\ 0 & 0 & \sigma_3 \end{bmatrix} \tag{1}$$

in which σ_1 in [S/m] is the longitudinal conductivity, that of the fibre direction. Since the polymer matrix surrounding the carbon reinforcement is an insulator ($\sigma_m \approx 0$), σ_1 is given by the fibre conductivity σ_f and the fibre volume fraction v_f, that is

$$\sigma_1 = \sigma_f v_f + \sigma_m v_m \approx \sigma_f v_f, \qquad \sigma_m \approx 0 \tag{2}$$

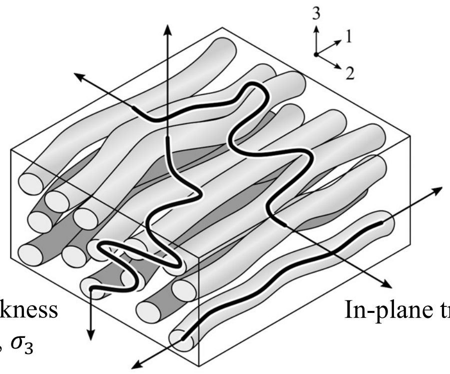

Figure 1: Schematic of longitudinal, transverse and through-thickness current flow in an UD ply

The remaining principal components of the conductivity tensor, σ_2 and σ_3, are not a direct function of the intrinsic electrical properties of the constituents, but rather driven by topology. Tortuosity and clustering of the carbon fibres causes numerous fibre-fibre contacts in the microstructure. Through these contacts, stochastic zigzag paths are available that allow for some, though typically limited, in-plane and through-thickness transverse electrical conductivity, as illustrated by Figure 1. The accumulation of resistance met at these contacts, as well as the increased path lengths of these corridors, causes roughly four orders of magnitude difference between the longitudinal and the in-plane or through-thickness transversal direction, $\sigma_1 \gg \sigma_2$ and σ_3 [3]. Hence, the electrical conductivity of UD plies is highly anisotropic in nature.

In earlier work of the authors [3], an efficient six-probe method was explored for characterising the electrical conductivity tensor for two commercially available aerospace-grade UD prepreg materials. An overview of the results is listed in Table 1. The study provided proof for the validity of the rule of mixtures for predicting the longitudinal electrical conductivity σ_1. Additionally, the remaining tensor properties, σ_2 and σ_3, were characterised through means of an inverse method. That is, a numerical model was employed to fit both σ_2 and σ_3 to a single six-probe characterisation experiment. However, unlike the rule of mixtures that describes σ_1 accurately, there exists no simple analytical definition for σ_2 nor σ_3 to compare the characterised properties to because of the stochastic nature of intralaminar fibre network. And, especially because both properties are fitted to a single experiment, the validity of the inverse method for obtaining the true material behaviour thus remained debatable.

Material	**Orthotropic electrical conductivity**		
	Longitudinal, σ_1	In-plane transversal, σ_2	Through-thickness transversal, σ_3
Toray AS4/PEEK, 59% FVF	38.3 ± 1.54 kS/m	10.8 ± 2.30 S/m	1.34 ± 0.40 S/m
Solvay AS4D/PEKK, 59% FVF	36.9 ± 0.47 kS/m	4.34 ± 0.57 S/m	0.70 ± 0.12 S/m

Table 1: Orthotropic electrical conductivity as obtained from the work of Buser et al. [3]

The present study addresses the characterisation of the in-plane transverse electrical conductivity σ_2 alone through direct characterisation, in contrast to the previous work. The study attempts to gain additional confidence in the inverse method employed in [3] through verification. Careful attention will be paid to the complexities involved when designing the electrical characterisation experiment. As an example, the dimensions of a test coupon determine the length of the tortuous fibres over which the statistical nature of the zigzag paths is analysed and should thus be taken into account. Also, similar to the work in [3], morphologically distinct and commercially available UD reinforced PAEK prepregs will be characterised and reflected upon.

2 MATERIALS AND METHODOLOGY

2.1 Six-probe method

This work will employ the six-probe method as the basis for characterisation. A schematic of the method is shown in Figure 2 in which a direct current (DC) I is one-sidedly applied over the width w of a specimen via the outer electrodes. It creates an electric field that is uniform across the width, but possibly non-uniform across the thickness t. Four inner probes are used for sampling the voltage response on both sides of the specimen, V_t and V_b. The parallel configuration of these two sets of voltage probes with respect to the current flow eliminates lead and contact resistance from the measurement data, which is a mayor benefit of the six-probe method.

Besides determining the in-plane conductivity σ_x, the previous study [3] utilised the non-uniform current density distribution for determining the through-plane conductivity σ_z in the same measurement run, which can be deduced from the difference recorded between V_t and V_b. The present study, however, will divert from that approach and will rather attempt to create an effectively uniform current density distribution between the inner measurement probes. That is: a uniform current density across the cross-sectional area $w \times t$ over (at least) the depicted distance δ (Figure 2).

The emergence of a fully developed uniform current density depends on several factors, including the distance between the various electrodes, the geometry of the specimen, and the degree of electrical anisotropy in the xz-plane. The electrical anisotropy ratio σ_x/σ_z is an indicator of whether the current density tends to spread evenly throughout the specimen thickness or rather remain closer to the top surface where the current is introduced. Effectively, the degree of anisotropy determines the minimum distance between the current and voltage electrodes as well as the maximum specimen thickness that can be tested for maintaining uniformity. A fully developed and approximately uniform current density is characterised by equivalent readings across the top and bottom voltage probes, such that $V = V_t \approx V_b$. Then, an analytical expression is applicable for the conductivity in x-direction that regards the current as one-dimensional:

$$\sigma_x = \frac{\delta}{wt}\frac{I}{V} = \frac{\delta}{wt}\frac{1}{R} \tag{1}$$

It should be noted that the degree of lateral uniformity, i.e. across the y-direction, can also be of concern for the validity of Equation 1. Despite the apparent line contact of the electrodes with the specimen, uneven electrical contact across that line contact or local variations in the electrical conductivity of the specimen may disturb the current uniformity. The present study, however, focuses on specimens where the fibres span the width of the specimen such that $\sigma_x = \sigma_2$. This orientation practically ensures a high degree of uniformity due to the highly conductive nature of the laterally oriented carbon fibres.

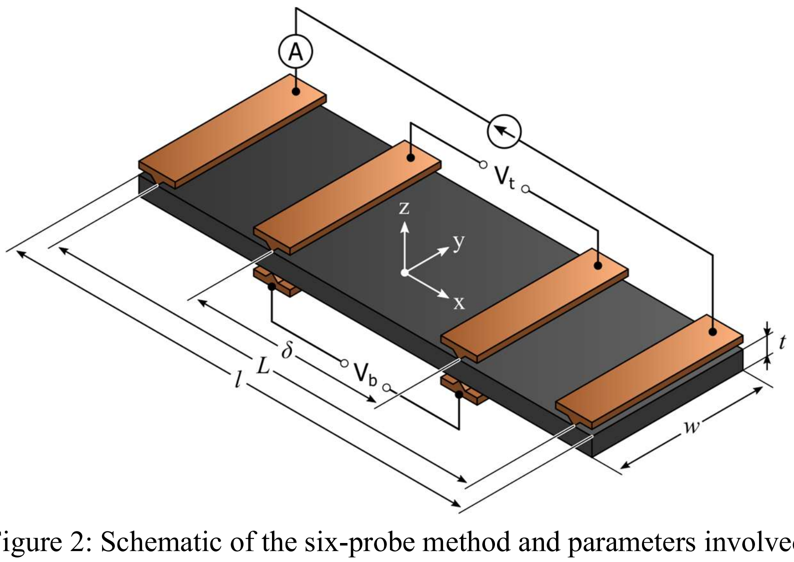

Figure 2: Schematic of the six-probe method and parameters involved

2.2 As-received prepregs

Unidirectionally carbon fibre reinforced PEEK (Toray TC1200) and PEKK (Solvay APC) with an average consolidated ply thickness (CPT) of 0.14 mm were used for this study. The pre-impregnated (prepreg) tapes contain carbon fibre from Hexcel (resp. 12K AS4 and 12K AS4D) in a volume fraction of 59%, a fibre areal weight of 145 g/m^2 and a fibre conductivity of 59 kS/m [4]. The prepregs have been selected for their prominent contrast in morphology, depicted in Figure 3, where it can be observed the prepregs differ in terms of uniformity of the fibre-matrix distribution.

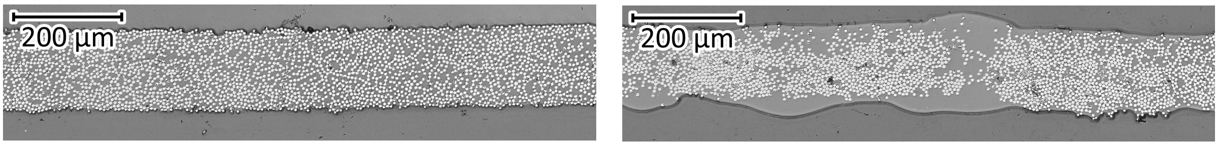

Figure 3: Micrographs of Toray PEEK (left) and Solvay PEKK (right) as-received prepregs

2.3 Specimen preparation

A hot press was employed to consolidate 12×12 in^2 laminates with a $[0]_{4s}$ fully UD lay-up using a picture frame mould. Table 2 provides a summary of the consolidation cycles used. From each laminate, 45 specimens were precision milled for electrical conductivity measurements. The specimens were 55 mm in length and either 15, 30, or 45 mm in width, resulting in a total of 15 specimens per size. The fibres were oriented in the width direction of the specimen. The specimens were extracted from various locations of the parent laminate in a manner that ensured even distribution. The arrangement can be observed by advancing to Figure 5.

The decision to use different specimen widths in this study was deliberate and based on one of the limitations of the previous study [3], which only used a single specimen width of 45 mm. The width of a test coupon constrains the length of the fibres over which the stochastic nature of the electrically conductive zigzag paths can be analysed. The contact incidence between 'aligned' fibres is the limiting factor in facilitating transverse current flow. A reduced fibre length can lead to a decrease in the quantity of fibre-fibre contacts within the specimen, consequently lowering the probability of establishing an electrically conductive pathways between the terminal and the ground. A minimum fibre length is therefore required for the coupon to represent a homogenised state. In studies by Tse et al. [5] and Athanasopoulos and Kostopoulos [6], it was observed that specimens featured a constant transverse electrical conductivity for fibre lengths of 6 and 8 mm, respectively, for the tested materials. Their findings have informed the decision of the authors to only go as narrow as 15 mm in the present work.

Material	**Consolidation cycle**			
	Temperature	Pressure	Dwell	Cooling rate
AS4/PEEK	385 °C	20 bar	20 min	5 °C/min
AS4D/PEKK	380 °C	20 bar	30 min	5 °C/min

Table 2: Consolidation parameters of the 8-ply laminates

2.4 Electrical conductivity experiments

A six-probe test fixture was developed and manufactured following the schematic depicted in Figure 2. The spacing of the inner electrodes δ and outer electrodes L were set to resp. 20 mm and 50 mm for all tests. An electrically insulating alignment bracket, fabricated from polylactic acid (PLA), was utilised to secure and centre the specimens with respect to the probes. Contact pressure between the specimen and the electrodes was established through toggle clamps.

A Tenma 72-13360 DC power supply was used to perform a pre-programmed voltage sweep from 5 to 60 V with increments of 5 V. At each increment, voltage was maintained for 1 second. Under these conditions, Joule heating in the specimens was not observed with thermocouple monitoring, hence measurements are regarded isothermal. A TiePie HS6D differential oscilloscope was used for continuous acquisition (at a 1 kHz sample rate) of the potential difference over the measurement probes terminals. Additionally, the acting current was measured with the oscilloscope as well, by acquiring the voltage drop over a shunt resistor connected in series between the power supply and the load. In further analysis current and voltage data was taken as the average over each one-second increment of the voltage sweep. Measurements were performed twice, with the specimen mounted upside-down in the second run. The reasoning for and implications of this decision will be detailed in the results section, but for now it is worth noting that the alignment bracket ensured that the specimen alignment of the second run matched the former.

3 RESULTS AND DISCUSSION

3.1 I-V linearity analysis

The voltage sweep performed during each characterisation experiment can be used to verify the isothermal Ohmic behaviour of the specimens. Figure 4 shows the typical plot of such analysis along with a magnified section. The exemplary I-V curve clearly shows a constant resistance, the slope of the regression. Therefore, Ohm's law can be considered valid for the studied case, which underlies Equation 1. All test pieces were found to satisfy the linearity condition eventually. For the few cases where linearity was not apparent at first, it rather indicated incorrect use of the measurement equipment. Hence, even from a practical point of few, it highlights the importance of performing a linearity check to ensure accurate and reliable measurement data.

The magnification in Figure 4 captures small deviations from the fitted linear regression. Here, the aforementioned second upside-down measurements play an important role in the interpretation of the data. Focussing solely on the first measurement, labelled somewhat arbitrarily as 'face up', it can be observed that a lower voltage reading is detected with the bottom probes, indicating non-uniformity of the current density. One could easily assume that the test design prevents the current from properly spreading over the thickness of the specimen, if it were not for the behaviour recorded with the second 'face down' run. Rather, the second run, with the specimen flipped upside-down, displays the bottom probe voltage to exceed the top probe voltage at each measurement increment. The observed behaviour caused by the flipping of the specimen is thought to be a result of the stochastic nature of the fibre contact incidence that drives the transverse electrical conductivity of the specimens. If one side of the specimen were to feature conductive paths of overall lower resistance, the current would distribute unevenly across the specimen accordingly. Flipping the specimen upside-down would therefore display the same non-uniformity, but mirrored, as is the case in Figure 4. The state described can be considered quasi-uniform, as it is the closest approximation to uniformity that the specimen can achieve under the given conditions. Consequently, each specimen response has been homogenised as the closest linear fit through both measurements.

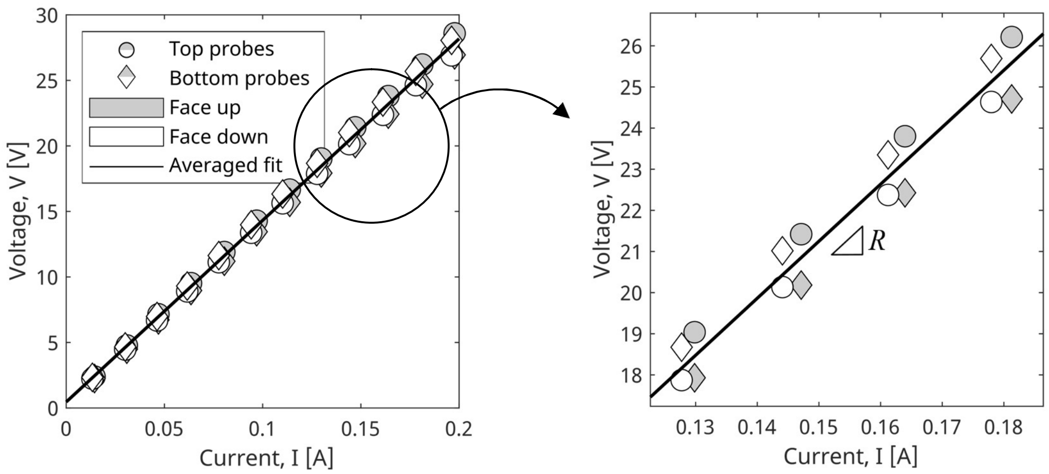

Figure 4: Typical current and voltage data of an 8-ply PEEK specimen of 30 mm width

3.2 In-plane transverse electrical conductivity

The resistances recorded, according to the procedure depicted in Figure 4, have been used as input for Equation 1 to obtain σ_2 (i.e. σ_x). The recorded values are spatially plotted in Figure 5, based on the original location (and size) of the specimen in the parent laminate.

First of all, the results show that the impact of varying specimen widths on electrical conductivity is minimal when compared to the rather significant material variations observed within a single size-group of specimens. In fact, neighbouring specimens, particularly along the x-direction, exhibit a similar magnitude of electrical conductivity.

Significant variations in material properties were mainly observed among neighbouring specimens in the y-direction. It is typical for unidirectional prepreg tapes to have greater variations in properties across the width of the tape, including roughness profile, while they remain relatively consistent along the fibre direction. The eight plies utilised for laminate consolidation were consecutively taken from a roll and laid-up without staggering. Therefore, it is not surprising that the electrical conductivity remains relatively consistent along the fibre direction (the x-direction) compared to the variations in electrical properties that occur transverse to the fibre orientation (the y-direction). The consistency in the properties recorded along the fibre orientation serves as evidence for the precision of the characterisation methodology employed. It indicates that any significant variations observed are due to the inherent material morphology rather than limitations of the test equipment or methodology.

Based on the bandwidth of the colormaps in both plots and the comparison in Table 3, it is evident that the typical conductivities of the direct and inverse method align. This is particularly notable considering the large variation in properties illustrated by Figure 5. In contrast to the previous study [3], which examined specimens comprising 32 and 64 plies, the present study employed 8-ply lay-ups. The results suggest a homogenised behaviour in terms of specimen thickness, although potential effects may arise when studying even thinner lay-ups.

Material	**In-plane transverse electrical conductivity**	
	Direct method	Inverse method
Toray AS4/PEEK, 59% FVF	6.11 ± 3.35 kS/m	10.8 ± 2.30 S/m
Solvay AS4D/PEKK, 59% FVF	3.31 ± 1.01 kS/m	4.34 ± 0.57 S/m

Table 3: Comparison of in-plane transverse electrical conductivities σ_2 as measured with the direct method (present study) and the inverse method detailed in [3].

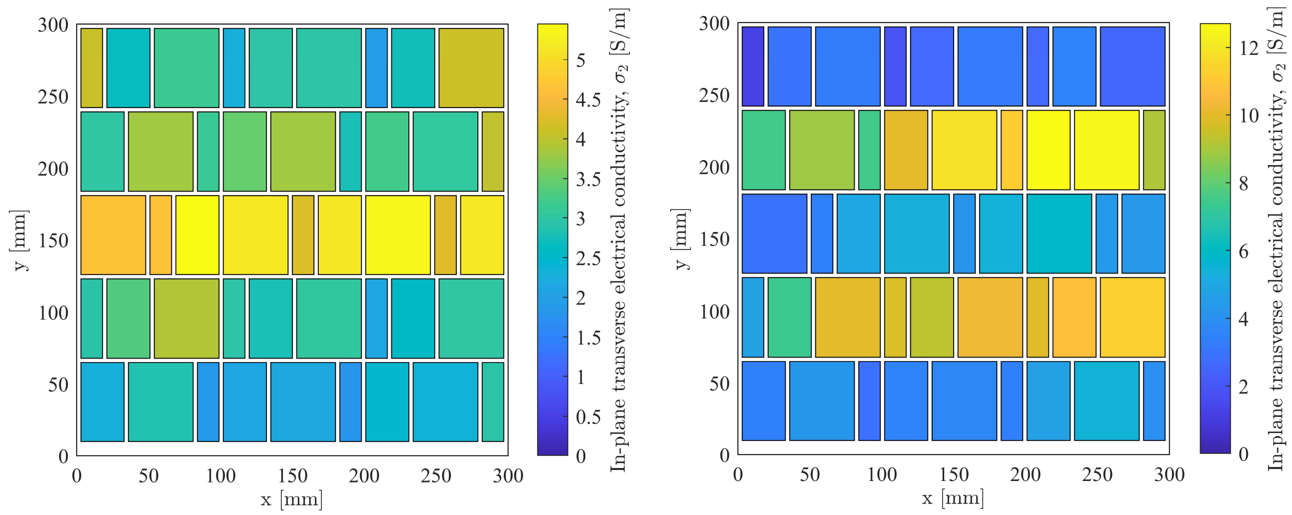

Figure 5: Spatial plot of the quantified in-plane transverse electrical conductivities with respect to their location in the parent laminate. Left: Solvay PEKK. Right: Toray PEEK. The fibre orientation aligned with the x-axis.

4 CONCLUSION

Electrical conductivity experiments were performed on UD carbon fibre reinforced PEEK and PEKK specimens using a six-probe method. Although a different thickness and widths of the test coupon were used, the electrical conductivity in in-plane transverse direction was found in agreement with earlier reported studies. The work therefore demonstrates a reliable characterisation procedure for obtaining the homogenised electrical conductivity of stochastic zigzag paths in UD carbon fibre reinforced PAEK materials.

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