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Electrical conduction investigation of pre-stressed carbon/epoxy fabric laminates heated by ampere-level currents

Gang Zhou^{1*}, Ewa Mikinka¹, Xujin Bao² and Weiwei Sun³

Abstract

A study on electrical conduction of carbon/epoxy laminates has so far been conducted in an ad hoc nature without a standardised method, involving many extrinsic factors. How these factors affect electrical conduction of carbon/ epoxy laminates has not been well established. The objectives of this work are to ascertain the effects of electrical currents, temperatures, and clamping torques on the anisotropic electrical conduction of carbon/epoxy laminates. Two-probe method with solid electrodes was developed with machined carbon/epoxy laminate specimens of various dimensions. The contributions of elevated temperatures and clamping pressures to electrical conduction were investigated. Various contact conditions with or without conductive paint were examined. The relationship of electrical resistance correlating with temperature and clamping pressure was developed to aid an analysis of data trends. From the average test results of 18 groups, aided with qualitative predictions, the milliampere-to-ampere increases of current led to significant reductions in electrical conductivities in both in-plane and through-the-thickness directions. The increase in clamping torque increased the electrical conductivity values in both directions. Applying conductive paint to the contact faces did not appear to affect the contact resistance. Thus, the enhanced values of electrical conductivity from the painted specimens were attributed to their lower body temperatures, as the conductive paint at the contact faces soaked up the substantial amount of the electrical energies.

Keywords Carbon/epoxy laminate, Electrical conductivity, Temperature, Clamping torque

Introduction

The electrical conduction and current-induced thermal behaviour of carbon fibre-reinforced laminates have attracted lots of attention in the fields of lighting strike protection [1-3], electromagnetic interference shielding [4, 5], electro-thermal de-icing systems [6], composite antennas [7], high-speed rotors [8, 9], electrification

Loughborough LE11 3TU, UK

[12, 13]. The common challenges to these investigations were that (a) composite laminates were anisotropic, (b) the lack of measurement standard for electrical conduction for anisotropic composite laminates led to the adaptations from those for isotropic and homogeneous materials, (c) the specific contributions of extrinsic and intrinsic factors to electrical conductivities were completely unclear, (d) the routine use of direct current (DC) of milliamperes was not companied by any understanding of its effects on ampere-level electrical conduction, and (e) there was little information on the effect of raised temperatures on electrical conduction. This has led to not only the widely varying values of electrical conductivities for the same materials but also the use of electrical

of composite aircraft [10, 11], and damage detection



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^{*}Correspondence:

Gang Zhou

G.Zhou@Lboro.ac.uk

¹ Department of Aeronautical and Automotive Engineering,

Loughborough University, Loughborough LE11 3TU, UK

² Department of Materials, Loughborough University,

³ C-Power Ltd, Tianjin, China

conductivity values measured with constant milliampere-level DC in the aforementioned applications, which caused significant difficulties in exploring multi-functionalities of lightweight composite structures in design, analysis, and numerical modelling.

Although several endeavours on the electrical conductivities of carbon/epoxy laminates were reported [1-3,14], there is a dearth of knowledge of how current level, raised temperature, and clamping pressures, in addition to conductive paint, affect electrical conduction characteristics either individually or collectively. In particular, the fundamental effect of varying clamping torques on electrical resistance has never been reported before. The present systematic investigation focuses on examining experimentally the electrical conduction and electro-thermal behaviour of carbon/epoxy fabric laminates under various levels of electrical current and clamping torques. The objectives are to develop a systematic and in-depth understanding of electrical conduction within the volume of the specimens and at the electrode-specimen contact faces and evaluate the effects of current level, raised temperature, clamping torque, and conductive paint on the anisotropic nature of electrical conduction and electro-thermal behaviour using 2-probe method (2PM) via solid electrodes.

Carbon/epoxy specimens and preparations

Carbon/epoxy laminates were composed of plain weave fabric prepregs, in which consisted of PAN-based Grafil 34–700 carbon fibres and LTM45 epoxy resin. A fabric laminate panel of 300×300 mm was laid up with 8 plies in a QI lay-up in $(\pm 45^0/0^0/90^0)_{2s}$, with a cured ply thickness of 0.428 mm. It was cured in an autoclave at 60° C under a pressure of 0.62 MPa for 16 h to have a nominal thickness of 3.4 mm. A nominal value of electrical conductivity is 55556 S/m for Grafil 34–700 carbon fibres [15] and is 1.4×10^{-12} S/m for LTM45 epoxy resin [16]. The fibre volume fraction of the laminate is 58%.

To achieve best contact between solid electrodes and specimen faces for both rectangular and square shapes, the entire surfaces of the panel were milled symmetrically to parallel on a CNC machine. This was achieved by (1) measuring the thicknesses of the panel at the ends of a longer edge to find the through-the-thickness (TTT) middle points and linking them to establish the horizontal middle line on the cross section; (2) raising the thinner end to keep the middle line horizontal; (3) milling off 0.1 mm from the upper surface; and (4) turning the panel over to repeat the same operations for the opposite surface, once parallel was secured. The thickness of the milled panel became 3.18 mm, with about a quarter of a cured ply thickness being machined off from each face. These operations achieved a geometric flatness and parallel of the two surfaces and exposed some carbon fibre filaments, though they might have some effect on the fibre volume fraction of the machined laminate. Strips of specifically selected widths were then cut using a bandsaw (Maxtile 260 with a diamond-coated platinum blade) with the two cut cross sections of each strip being milled to parallel. Individual measurement specimens of both rectangular and square shapes were finally cut off the milled strips. The nominal dimensions of the square specimens were 10×10 , 20×20 , and 30×30 mm, respectively. The rectangular specimens with a width of 10 mm had the respective lengths of 10, 20, and 30 mm. These different sizes of specimens were designed not only to evaluate contact resistance but also to examine the dimensional effect on electrical conduction (not discussed here). About half of all the specimens had conductive silver paint (commercial RS PRO 123-9911 silver electrically conductive paint with a silver content in the range of 30 to 60%) applied uniformly on their respectively designated contact faces. Four out of six main groups of all used specimens are shown in Fig. 1a-d, as the two other main in-plane groups are identical to those in Fig. 1a-b. For specimen identifications, the measurement directions are indicated as part of figure captions, as 'in-plane' or 'TTT', whereas the figure legends for individual specimens and specimen groups adhere to the following code: (1) '3d-5d' denotes the unpainted 20×10 mm coupon specimens and '6d-8d' the unpainted 30×10 mm coupon specimens for the in-plane direction data, (2) '3d-5d' denotes the unpainted 20×20 mm square specimens and '6d-8d' the unpainted 30×30 mm square specimens in the TTT direction data, (3) '3c-5c' denotes the painted 20×10 mm coupon specimens and '6c-8c' the painted 30×10 mm coupon specimens for the in-plane direction data, and (4) '3c-5c' denotes the painted 20×20 mm square specimens and '6c-8c' the painted 30×30 mm square specimens for the TTT direction data.

Measurement method for volume electrical conductivities

Due to the lack of electrical conductivity measurement methods for anisotropic fibre-reinforced composite laminates, the present 2PM was adapted from one used in ASTM D4496 [17] for moderately conductive isotropic materials. The aim is to ensure that the composite materials at both current and voltage probe contact locations were the same not only in the in-plane but also in the TTT measurements. A composite specimen with a length of l, a width of b, and a thickness of w was clamped between two solid electrode boards, as shown in Fig. 2. When electric current I with a selected value was supplied via solid electrodes and flowed in the length direction of the specimen, the total electrical

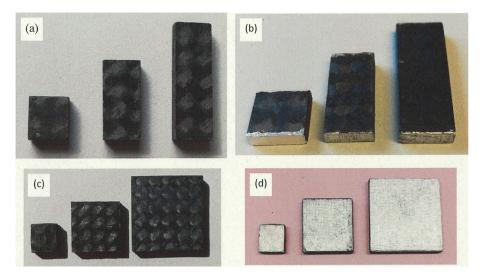


Fig. 1 Electrical conductivity measurement specimens **a** unpainted in in-plane direction, **b** painted in in-plane direction, **c** unpainted in through-the-thickness direction, and **d** painted in through-the-thickness direction

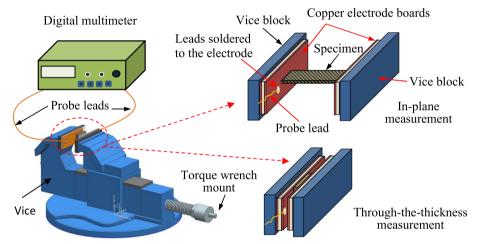


Fig. 2 Two-probe method for measurement of volume electrical conductivity

resistance *R* was composed of the resistance at the contact faces R_{con} and the volume resistance R_{vol} in the specimen, as given in Eq. (1). As the contact face resistance was generally small, as demonstrated in Electrical contact resistance section, it was customary in calculations of electrical conductivity for coupon specimens to retain only the volume electrical resistance via

$$R = R_{\rm vol} + R_{\rm con} \approx R_{\rm vol} \tag{1}$$

Values of the electrical resistance were calculated from measured voltage drops. With the known specimen dimensions, the volume electrical conductivity kin S/m was calculated by Ohm's law as

$$k = \frac{l}{bwR} \tag{2}$$

Experimental set-up for electrical conductivity measurement

A digital torque screwdriver Norbar 13850 with a range of 0.3 to 1.5 Nm and a lever arm radius of 10 mm was used to apply precise clamping torques with an accuracy of \pm 6%, along with a Wera 7000A torque wrench with a range of 1 to 25 Nm. All the applied torques up to the level of 1.3 Nm from Norbar 13850 could be converted to pressures in MPa, ranging from 1.67 to 7.22 MPa in

the in-plane measurements and from 0.06 to 1.3 MPa in the TTT measurements. An IR dual-laser noncontact thermometer RS820 with a digital display was used for rapid precise temperature measurements in a range of

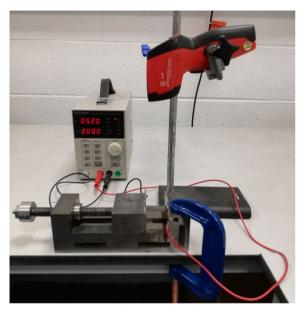


Fig. 3 Experimental set-up for electrical conductivity measurements of composite laminates

-50 to 380 °C with an accuracy of $\pm 1\%$, with the actual experimental set-up being shown in Fig. 3. Surface temperature readings at the middle of the surfaces were taken at approximately 1 min, in order to ensure that the variations of the electrical powers that were responsible for temperature rises were controlled only by the selected values of current. As a result, the corresponding temperature rises induced by the increased currents in the longest specimens may not have reached stabilising levels. Although the milliampere-level currents were routinely used in the measurements of electrical conductivities for carbon/epoxy laminates [1, 12, 13] to keep interior builtup heat to a negligible level, a range of applied electrical currents used in this work were from 0.5 to 3 A to examine the effect of the current on raised temperatures and eventually electrical conduction. The upper current limit was established via much trial and error when substantial Joule heating caused permanent damage with smoke and smell in some specimens. A total of 18 groups of specimens without and with conductive silver paint were measured and the average values of electrical conductivity from the respective groups, along with standard deviations, were presented in Table 1.

Analytical predictions of volume electrical conductivities Rigorously speaking, the volume electrical resistance of a clamped composite specimen has the contributions

Measurement direction (ID ^a)	Specimen dimensions	Silver paint	No. of specimens	Av. of electrical conductivity	Standard deviation	Coefficient of variation
-	mm X mm X mm	-	-	S/m	S/m	%
In-plane 0 ⁰ (IP0-Xd)	10 X 10 X 2	No	2	5488	± 394	7
	20 X 10 X 2		3	5514	±704	13
	30 X 10 X 2		3	7056	± 1370	20
In-plane 90 ⁰ (IP90-Xd)	10 X 10 X 2		2	5848	±439	8
	20 X 10 X 2		3	5804	±463	8
	30 X 10 X 2		3	6104	± 203	3
TTT (TTT-Xd)	10 X 10 X 2		2	14.8	± 2.2	15
	20 X 20 X 2		3	13.6	± 1.6	12
	30 X 30 X 2		3	10.5	± 0.7	7
In-plane 0 ⁰ (IP0-Xc)	10 X 10 X 2	Yes	2	6171	±437	7
	20 X 10 X 2		3	7206	± 373	5
	30 X 10 X 2		3	8012	±982	12
In-plane 90 ⁰ (IPO-Xc)	10 X 10 X 2		2	6171	±437	7
	20 X 10 X 2		3	7138	± 511	7
	30 X 10 X 2		3	7290	± 635	9
ТТТ (ТТТ-Хс)	10 X 10 X 2		3	22.9	± 0.5	2
	20 X 20 X 2		4	23.0	± 1.0	4
	30 X 30 X 2		4	18.5	± 0.7	4

Table 1 Electrical conductivity test results for plain weave fabric-based carbon/epoxy laminate

^a IPO and IP90 denote in-plane 0^o and 90^o directions, respectively, TTT through the thickness direction; X takes 1–2 for 10 mm long specimens, 3–5 for 20 mm long specimens, and 6–8 for 30 mm long specimens; c for painted and d for unpainted specimens

of three components, as shown on the right-hand side in Eq. (3). The 1st part, called the residual resistance, is characterised by electrons interaction with physical imperfections of the specimens. The 2nd part, called the intrinsic resistance, is characterised by electrons interaction with phonons (lattice vibration) only. The 3rd part corresponds to the contribution of a clamping pressure to the specimens, with assumption of uniaxial compression. While the 1st part is independent of temperature, the 2nd part could react with temperature. For non-metals such as composite laminates with relatively few free electrons, the 2nd and 3rd parts become dominant.

$$R = R_0 [1 + \alpha (T - T_0) - \varepsilon_c (1 + (\nu_{21} + \nu_{12}))]$$

= $R_0 \Big[1 + \alpha (T - T_0) - \frac{P}{E_c} (1 + (\nu_{21} + \nu_{12})) \Big]$ (3)

in which R_0 denotes the resistance measured with 10 mA at room temperature T_0 of nominally 20^oC, α temperature resistance coefficient in per degree, T raised temperature, P clamping pressure, E_c either fibre-direction compressive modulus in the in-plane measurements or compressive modulus of epoxy in the TTT measurements, v_{12} and v_{21} their major and minor Poisson's ratios of the composite. The coefficient α could be either positive in conductive materials such as in the in-plane directions of composite laminates, when the resistance increased with an increase in temperature due to Joule heating, or negative in non-conductive materials (in the TTT direction of laminates) when the resistance fell with an increase in temperature [12]. With both temperature and mechanical compression being considered, the volume electrical conductivity k could be given as

$$k = \frac{\iota}{bwR_0 \left[1 + \alpha (T - T_0) - \frac{P}{E_c} (1 + (\nu_{21} + \nu_{12})) \right]}$$
(4)

Results and discussion Effects of electrical currents

As applied current was increased by at least two orders of magnitude from 10 mA up to 3 A, resistance in the inplane direction specimens increased, as expected. As a result, the electrical conductivities of both the unpainted and painted laminates decreased exponentially, as shown in Fig. 4. These trends appear to show that the values of the electrical conductivities tend to level off after the significant drops, when the currents approach 3 A, without risking Joule heating damage. In the TTT direction, the trends of variations of electrical conductivity for both unpainted and painted laminates shown in Fig. 5 are similar to those in the in-plane direction, though dropping to the level-off values is greater and more dramatic. This appears to suggest that these characteristics were attributed to the combined effect of the lower thermal conductivity of the epoxy dominated TTT direction and the much larger painted contact areas of the TTT specimens, thereby soaking up the substantial amount of the electrical energies. All these findings show futher that once applied currents were increased from miliamperes to the levels of amperes, the values of the electrical conductivities measured at the former conditions were significantly overestimated, when used for the latter conditions in both the in-plane and TTT directions.

Electrical contact resistance

The responses of the contact faces of the carbon/epoxy specimens to a current were complex, as the contact faces (without conductive paint) were discrete periodically non-conductive. These non-conductive regions in the contact faces were not affected by the current but the current constricted to the conductive carbon fibres could raise an electrical contact resistance [10]. The most common practice in the measurements of electrical

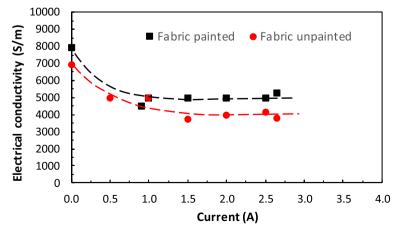


Fig. 4 In-plane electrical conductivities of fabric carbon/epoxy laminates without and with silver paint with 2Nm clamping torque

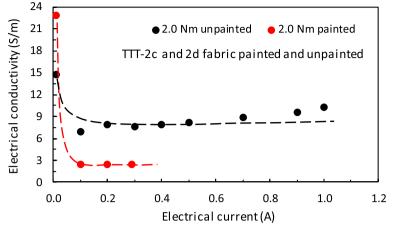


Fig. 5 Through-the-thickness electrical conductivities of carbon/epoxy laminates

conductivities of carbon/epoxy laminates is to apply conductive paint or paste on the contact faces of the specimens to minimise such envisaged resistance. Since the 'partial' electrical conduction on the electrode-contact faces reflects the true nature of the material, the use of conductive paint may dissipate thermal energy to lead to the lesser increase of temperature in the volume of the specimens, thereby showing the enhanced values of the electrical conductivities. Thus, it is very important to estimate the levels of contact resistance, with respect to the nominal levels of volume resistance of the present carbon/epoxy laminates.

Figure 6 shows that in the in-plane directions, the measured resistance data plotted against their length-toarea ratios form a linear trend for the specimens of three different lengths. In the figure, both the unpainted (triangle symbols) and painted (circle symbols) specimens are almost indistinguishable. Both intercepts on resistance axis at zero length/area ratio were taken to be about the same average of 0.01 Ω contact resistance and were just a few percent for these specimens, like the case in [1]. These results exhibited that the use of conductive paint on the contact faces reduced the contact resistance only by a very small amount, providing that the electrical conductance path network in the measurement direction was formed by a conductive medium like carbon fibre tows in the present case.

In the TTT direction, the measured conductance (reciprocal of resistance) data plotted against their areato-length ratios form also linear trends for each group of three different areas, as shown in Fig. 7, though the unpainted and painted group data are clearly separated. Since both group data tend to intercept the vertical conductance axis at the same location, the average of 0.28 Siemen for the two could be used, just like the in-plane case. Nevertheless, as the contact areas in the TTT measurements were much larger than those of the inplane ones, these data appear to suggest that the relative

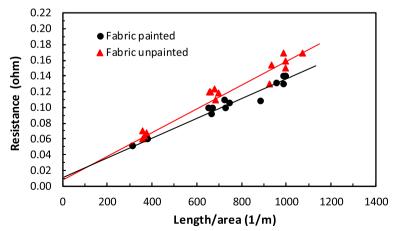


Fig. 6 Electrical contact resistance at interfaces in in-plane direction of carbon/epoxy laminates

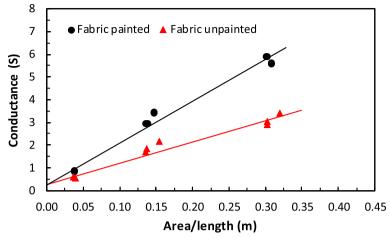


Fig. 7 Electrical contact resistance at interfaces in TTT direction of carbon/epoxy laminates

contribution of the contact resistance to the total in the TTT direction could be much greater than that in the inplane direction. This was because the interior conduction path network in the TTT direction was attributed to a random combination of breakdown of tiny resin gaps and 'fibre-to-fibre contact', which could be dominated by the former. These results also suggest that the contact resistance in the TTT direction (at zero area) was the same as the differences of conductance between the unpainted and painted groups at all non-zero locations, as the interior conduction path network did not change.

Effects of raised temperatures

Raising applied currents to the ampere levels induced significant Joule heating, as anticipated. Although the temperature rises could occur at both electrode–specimen contact faces and within the volumes of the specimens over 1-min duration, the latter was much more dominant part so that the temperature measurements were taken only at the middles of the specimen surfaces. In the inplane measurements, the used specimen surfaces were one of the machined lamination planes, whereas in the TTT measurements, the used specimen surfaces were one of the machined cross sections. Over time t (in seconds), heat H in Joules was obtained from

$$H = I^{2} t R_{0} \left[1 + \alpha (T - T_{0}) - \frac{P}{E_{c}} (1 + (\nu_{21} + \nu_{12})) \right]$$
(5)

From Eq. (5), theoretically, the increase in current could result in a parabolic increase of electrically induced thermal heat. In Fig. 8, the variations of raised surface temperatures with increase in electrical current were shown for the in-plane electrical conductivities. Under various given levels of clamping torques (same symbols with different colours) and with two different lengths (same colours with different symbols), a parabolic trend of the surface temperatures was established consistently from

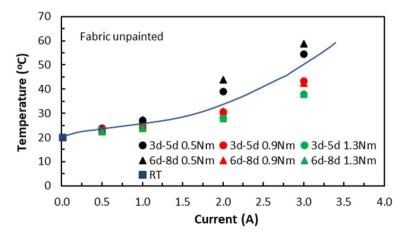


Fig. 8 Variations of raised temperature with current on the laminate surfaces of unpainted in-plane specimens

10 mA to 3 A, with a limited initial rapid rise. There were a couple of attempts of taking the current levels beyond 3 A, which led to the destruction of the specimens with temperatures exceeding 120^{0} C (glass transition temperature of epoxy). Temperatures in interior carbon fibres were expected to increase with the increased current levels due to Joule heating and thus epoxy separating them was also heated. As epoxy is thermally insulative, the rate of the temperature rises was significantly slower than that of carbon fibres, leading to the nonlinear parabolic trend. Similar trends were also reported in [10].

The painted specimens shown in Fig. 9 show the same type of responses, with an even smaller sensitivity to the clamping torques or specimen lengths. This strongly suggests that the nonlinear parabolic trend of the raised temperatures was independent not only of the presence of conductive paint at the contact faces but also of the specimen length, which indirectly confirms that the 1-min duration could be sufficient for the specimens with the chosen dimensions to reach isothermal state. Moreover, the temperature levels in the painted specimens were noticeably less than those of the corresponding unpainted specimens shown in Fig. 8. This appears to suggest that the conductive paint on the contact faces, acting as a heat sink, could have dissipated some heat, which led to the lower body temperatures in the specimens.

In the TTT direction, both the unpainted and painted laminates in Figs. 10 and 11 show the same type of responses, respectively, except that the initial rapid rises that are weak in the in-plane direction responses are much more pronounced. These findings suggest that this temperature–current relationship was independent of fibre direction, in addition to conductive paint. This further confirm that heat transfer in the volume of carbon/epoxy laminates was dominated by the epoxy resin, rather than the electrical conductance path network, which were completely different in the in-plane and TTT directions. Nevertheless, the relatively wide data bands at

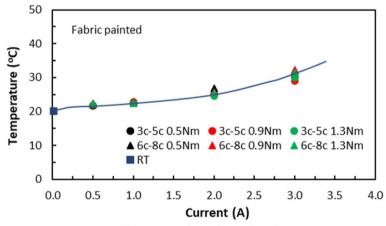


Fig. 9 Variations of raised temperature with current on the laminate surfaces of painted in-plane specimens

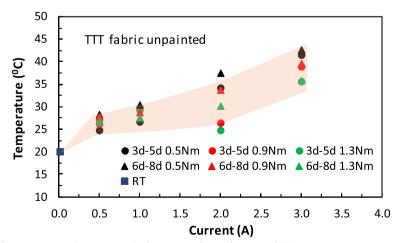


Fig. 10 Variations of raised temperature with current on the laminate surfaces of unpainted TTT specimens

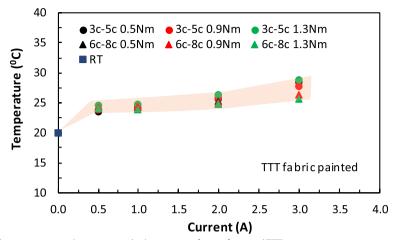


Fig. 11 Variations of raised temperature with current on the laminate surfaces of painted TTT specimens

the given currents (same colours with different symbols) indicate that there was some moderate effect related to the contact areas of the specimens.

In Figs. 12 and 13, the moderate temperature rises induced by increasing current decreased the in-plane electrical conductivities of the laminates significantly due to the corresponding increases in the electrical resistance. Nevertheless, such decreases settled quickly to the nearly constant levels for each group of the specimens for the moderate increase in temperature. The unpainted specimens had the slightly greater reductions than the painted ones. As a result, these responses reduced the ability of the laminates to dissipate heat by electrical conduction.

In the TTT direction, the thicknesses of the specimens became the measurement lengths, which remained constant, whereas their areas varied from 10×10 mm, 20×20 mm, to 30×30 mm. In Fig. 14, the large

unpainted specimens (3d-5d for 20×20 mm and 6d-8d for 30×30 mm) show, somewhat, little influence of clamping torques or areas, with the raised temperatures of data groups reaching the similar levels. From Fig. 15, the painted large specimens reached the substantially lesser temperatures than the groups with the smaller contact areas. All in all, once the body temperatures in the specimens started rising even by the moderate amounts, the TTT values of electrical conductivities reduced significantly down to the constant levels. The unpainted specimens had the greater reduction than the painted ones, due possibly to that the more thermal energies were available.

Effects of clamping pressures

Equation (4) predicts that the increase in clamping pressure at a given temperature should result in the increase

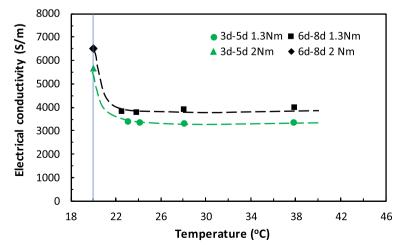


Fig. 12 Electrical conductivities of unpainted carbon/epoxy laminates in in-plane direction

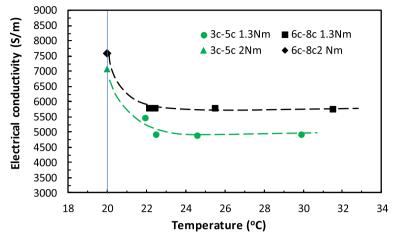


Fig. 13 Electrical conductivities of painted carbon/epoxy laminates in in-plane direction

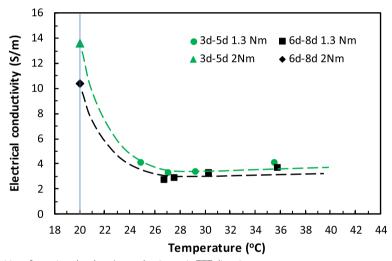


Fig. 14 Electrical conductivities of unpainted carbon/epoxy laminates in TTT direction

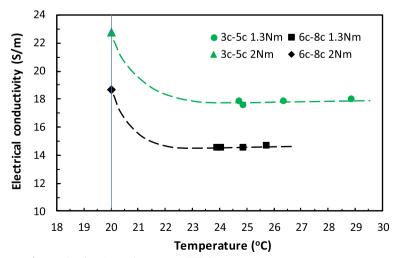


Fig. 15 Electrical conductivities of painted carbon/epoxy laminates in TTT direction

in electrical conductivity. Figure 16 shows the variations of the in-plane electrical conductivities with increase in clamping torque in a range of 0.5 to 1.3 Nm for the unpainted laminates. The established trends are linear and consistent, with the longer specimens (30 mm with triangle symbols) showing the greater values, as expected. The painted laminates in Fig. 17 exhibits the similar linear trends but with the smaller slopes. Although the contact faces of all the present specimens were carefully machined to be flat and parallel, when using solid electrodes, the values of electrical conductivities from the unpainted laminates could vary as much as 50% in the present torque ranges.

The variations of the TTT electrical conductivities with clamping torques were shown in Fig. 18 for the unpainted laminates. While a line trend is still easily established, showing little current sensitivity (same symbols with different colours), again. All these characteristics are very much observable from the painted specimens in Fig. 19.

Conclusions

The electrical conduction and current-induced thermal behaviour of carbon/epoxy fabric laminates were investigated using 2PM via solid electrodes to ascertain the effects of currents, temperatures, and clamping torques on the anisotropic electrical conduction of machined specimens with or without conductive paint. The findings suggest that the milliampere-to-ampere increases of current led to the significant reductions in

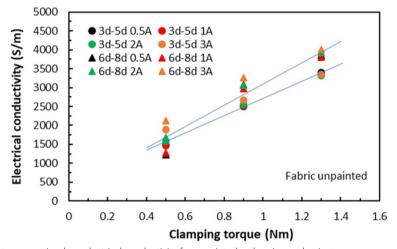


Fig. 16 Effect of clamping torque on in-plane electrical conductivity for unpainted carbon/epoxy laminates

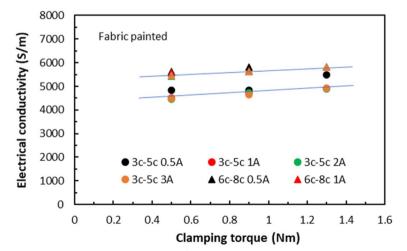


Fig. 17 Effect of clamping torque on in-plane electrical conductivity for painted carbon/epoxy laminates

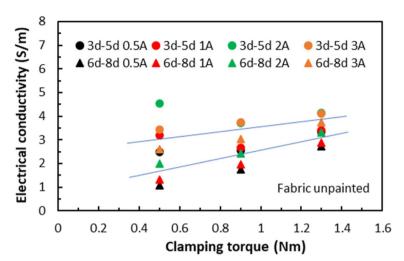


Fig. 18 Effect of clamping torque on TTT electrical conductivity for unpainted carbon/epoxy laminates

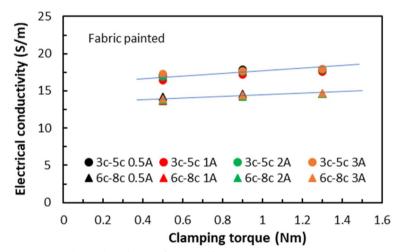


Fig. 19 Effect of clamping torque on TTT electrical conductivity for painted laminates

the electrical conductivity values in both in-plane and TTT directions. The rises of temperatures resulted in the similar reductions due to the increased resistance. The increase in clamping torque increased linearly the electrical conductivity values in both directions. Applying conductive paint to the contact faces did not appear to affect the contact resistance. Since the extrinsic factors such as clamping torques and use of conductive paint did not change the conduction path networks, the measured volume electrical conductivities are apparent values and therefore the values of the electrical conductivities obtained without paint should be used. Shall the 2PM via solid electrodes be developed into a measurement standard in the future for electrical conductivities of continues fibre reinforced composite laminates, a minimal clamping torque of 1 Nm be recommended with the minimum square dimensions of 20×20 mm in the TTT direction.

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Authors' contributions

GZ: conception, design and execution of the work, data acquisition, interpretation and analysis, manuscript drafting and revision, funding acquisition; EM: data acquisition and analysis; XB: design and execution of the work; WS: conception and funding acquisition. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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