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COMPOSITES THEORY AND PRACTICE

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ELECTRICAL PROPERTIES OF ALUMINIUM-FIBRE REINFORCED COMPOSITE LAMINATES

Hybrid materials such as Fibre Metal Laminates (FMLs) containing carbon fibre reinforced polymers (CFRPs) are very attractive candidates for novel design strategies due to their specific properties. However, Fibre Metal Laminates (FMLs) may be susceptible to galvanic and electrochemical corrosion in a damp environment due to the applied metal sheets. Aluminium alloy-glass/epoxy composite FMLs exhibit high corrosion resistance. Their corrosion process is limited to the metal outer layers if they are not protected because glass fibre reinforced composites are non-conductive. Galvanic corrosion initiation is likely when a composite contains carbon fibres, owing to the electric conductivity of these fibres. Therefore, it is necessary to determine the electrical properties of the produced hybrid materials. Measurements were made to determine the surface resistivity of components and contact resistivity of the laminates. Investigations were conducted on on a polymer composite and FMLs consisting of aluminium 2024-T3 joined with GFRPs (R-glass, S-glass) and CFRP. The aluminium alloy sheet was anodized in a sulphuric acid solution (SAA process). The composite plates and hybrid laminates were cured in the autoclave process. The surface resistance of the materials was determined by measuring the drop in current using the two probe method and strip electrodes. In the laminate specimens, the electrodes were placed in the longitudinal direction between the corresponding layers. The interlaminar interface properties of these laminates were studied by measuring the contact electrical resistivity of this interface. Moreover, the variation in temperature with time during electrical measurements was recorded by means of the thermovision technique for the composite specimens. This study revealed that the aluminium oxide and GFRP-R composite are insulators with very high but negative surface resistivity. The surface resistivity of the CFRP composite is equal to about $10^2 \div 10^3 \Omega/\square$ and depends on the direction of the fibres. When the electrodes are located perpendicularly to the fibres, the surface resistivity is lower and the surface temperature increases locally. Generally the contact resistivity of this composite is ~10^3 times higher than indicated in literature. It is a result of the high quality of the prepreg and autoclave curing of the laminate. The measurements of electrical contact resistivity indicated that it is possible to obtain a dielectric interface between the aluminium alloy and carbon reinforced composite by anodizing the aluminium and applying aglass prepreg layer 0.25 mm thick. The thinner glass composite layer does not increase the in-plane contact resistivity.

Keywords: CFRP, FML, surface resistivity, contact resistivity

WŁAŚCIWOŚCI ELEKTRYCZNE LAMINATÓW ALUMINIUM-KOMPOZYTY POLIMEROWO-WŁÓKNISTE

Laminaty metalowo-włókniste (FML) zawierające kompozyt wzmacniany włóknem węglowym (CFRP) są atrakcyjnym materiałem konstrukcyjnym ze względu na specyficzne właściwości mechaniczne i elektryczne. Problemem w FML jest podatność na korozję galwaniczną w wilgotnym środowisku ze względu na obecność warstw metalu. W przypadku laminatów stop aluminium - kompozyt wzmacniany włóknem szklanym (GFRP) korozja ograniczona jest tylko do zewnętrznych powierzchni metalu, jeśli nie są one zabezpieczone warstwą antykorozyjną. W laminatach wzmacnianych włóknem węglowym prawdopodobieństwo zaistnienia korozji jest znacznie wyższe z powodu przewodności elektrycznej włókien. Dlatego projektując materiały, w których włókno przewodzące pełni rolę sensora z wykorzystaniem również właściwości piezomechanicznych, konieczna jest znajomość właściwości elektrycznych komponentów oraz wytworzonych materiałów na równi z ich właściwościami mechanicznymi. W pracy przedstawiono pomiary rezystywności powierzchniowej komponentów - anodowanej w roztworze kwasu siarkowego (SAA) blachy ze stopu AW2024T3 oraz kompozytów lotniczych CFRP, GFRP-R i GFRP-S utwardzanych autoklawowo i rezystywności kontaktowej laminatów hybrydowych aluminium - CFRP oraz aluminium - GFRP-CFRP. Rezystywność powierzchniową kompozytu węglowego wyznaczano przy umieszczeniu elektrod paskowych równolegle i poprzecznie do kierunku włókien. W laminatach elektrody wklejono pomiędzy blachę i kompozyt oraz pomiędzy kompozyty węglowy i szklany, równolegle do przebiegu włókien. Podczas pomiarów rezystywności powierzchniowej mierzono zmianę temperatury, wykorzystując kamerę termowizyjną. W wyniku badań wykazano bardzo wysoką rezystywność warstwy anodowanej, porównywalną z rezystywnością kompozytu GFRP, potwierdzając właściwości izolacyjne tych materiałów. Kompozyt CFRP charakteryzuje słaba przewodność elektryczna, przy czym rezystywność jest o trzy rzędy wyższa od spotykanej w literaturze. Ponieważ badany kompozyt należy do wysokojakościowych z certyfikatem lotniczym i utwardzany był w autoklawie, cechuje się dużą jednorodnością strukturalną, znikomą porowatością i wysoką czystością powierzchni, co przekłada się na wyższą wartość rezystancji. Pomiary rezystywności kontaktowej w układzie płaskim ('in-plane") wykazały, że na granicy kompozytu CFRP z aluminium i z kompozytem GFRP rezystywność odpowiada rezystywności powierzchniowej CFRP, co świadczy o uzyskaniu bariery izolacyjnej.

Słowa kluczowe: CFRP, FML, oporność powierzchniowa, oporność kontaktowa

INTRODUCTION

Hybrid materials such as aluminium alloys with carbon fibre reinforced polymers (CFRPs) are very attractive candidates for novel design strategies due to their specific properties. Such concepts require appropriate joining technologies with regard to the mechanical behaviour and corrosion resistance of the compound [1, 2].

Fibre metal laminates (FMLs) may be susceptible to galvanic and electrochemical corrosion due to the applied metal sheets. The published data for FML laminates: glass/epoxy composite-aluminium alloy indicate the high corrosion resistance of such system [2-4]. Their corrosion process is limited to the metal outer layers if they are not protected. The layers consisting of a composite reinforced with glass fibres are non-conductive as a barrier for the corrosion process. However, galvanic corrosion initiation is possible in the case of composites containing carbon fibres, owing to the electric conductivity of these fibres. The phenomenon of electrical resistance change of composites containing carbon fibres under the influence of external loads is used as a sensor, among others for the detection of internal failures in structures [5, 6]. Structure health monitoring by means of its active components belongs to intensively developed methods based on basic physical phenomena. Therefore carbon fibres may perform two roles, i.e. as reinforcement in composites and as sensors [6]. These properties can also be used in FMLs. Therefore, research works are carried out in the scope of opportunities to use the advantages of carbon fibres with simultaneous limitation of the side effects of conductivity. The probability of galvanic corrosion is one of these adverse effects. Recently, information on research in the scope of galvanic corrosion in metal--carbon fibre contact has been published, which demonstrates the importance of this problem and the necessity for its solution. Mainly connections with aluminium are tested [7-10] but also connections with steel [11, 12] and titanium [11]. A polymer matrix composite with unidirectional reinforcement in the form of carbon fibres is characterized by bulk electrical conductivity in the direction of the fibres. Its value depends on the volume of carbon fibres [6]. This composite should have high resistivity depending on the polymer properties and on the degree of microstructural imperfection resulting from the possibility of local contact between the fibres [6, 13, 14]. The value of surface resistivity should also be similar to polymer resistivity. Metal sheets are subjected to the anodizing process. The generated oxide layer should be an electrical insulator. Therefore, in theory, the anodized layer-polymer interface should constitute a perfect barrier preventing the corrosion process in FMLs reinforced with carbon fibre. However, the real elements of aircraft and vessels may be corroded in operating conditions due to environmental factors, variable loads and the level of tolerable manufacturing imperfections. Therefore, it is necessary to determine the electrical properties and electrochemical parameters of the produced hybrid materials.

The present study describes the methodology of measuring the electrical properties of components used for producing hybrid laminates as well as the electrical properties of metal-composite interfaces, intended to operate in environments characterized by electrolyte features.

MATERIALS AND EXPERIMENTAL MEASUREMENTS

Materials

The tests were carried out on the components i.e. metal sheet and composite plates and on the hybrid laminates. The configuration of the specimens is presented in Table 1.

TABLE 1. **Specimen configuration**
TABELA 1. **Konfiguracja próbek**

Specimen	Components	Number of prepreg layers	Thickness of metal/ prepreg [mm]	Specimen dimensions [mm]
Al	AW 2024 T3 (AlCu4Mg1) + SAA	-	0.5	100x150x0.5
CFRP	HexPlyAS7/M12 UD carbon prepreg (Hexcel, USA)	8	0.131	100x150x1
GFRP-R	HexPly M12TVR380/26% UD R-glass prepreg (Hexcel, USA)	4	0.255	100x150x1
GFRP-S	RC45 S-glass UD prepreg (Axiom)	11	0.13	100x150x1.5
Al/CFRP	Al + CFRP (0)	14	0.5/0.131	50x50x2
Al/GFRP-R/ CFRP	Al + GFRP-R (0) + CFRP (0)	1+12	0.5/0.255/0.131	50x50x2
Al/GFRP-S/ CFRP	Al + GFRP-S (0) + CFRP (0)	1+13	0.5/0.13/0.131	50x50x2

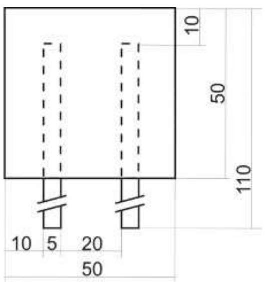
The aluminium alloy sheet was anodized in a sulphuric acid solution (SAA process). The composite plates and hybrid laminates were cured in the autoclave process using parameters recommended by the prepreg manufacturers. The permanent thickness of the hybrid laminates is necessary for further research. Moreover, it was established that only one layer of glass composite would be applied in the hybrid laminates. Therefore the number of layers of carbon composite is different.

Strip copper electrodes with the geometry described in section Electrical Resistivity Test Method were placed between the CFRP/GFRP and Al/composite during the manufacturing process.

Electrical Resistivity Test Method

The components subjected to testing - the anodized layer on the aluminium alloy, epoxy resin, glass fibres and GFRP composites - are dielectric materials. Composites reinforced with carbon fibre are conductive materials with low conductivity. The electrical properties of these materials were tested on the basis of the ASTM D257 standard [15]. These test methods cover direct-current procedures for the measurement of DC insulation resistance, volume resistance and surface resistance. From such measurements and the geometric dimensions of the specimen and electrodes, both the volume and surface resistivity of electrical insulating materials can be calculated, as well as the corresponding conductance and conductivity. In this experiment, the surface resistance of the materials was determined by measuring the drop in current using the two probe method and strip electrodes (Figs. 1 and 2). Self-adhesive copper tape (Agar Scientific) 0.01 mm thick and 5 mm wide intended for electrical contacts in SEM microscopy was used as the electrodes. The electrodes were adhesive bonded with the composite specimens in accordance with the fibre direction and perpendicular to the fibre direction, respectively. In the laminate specimens, the electrodes were placed in the longitudinal direction between the corresponding layers schematically shown in Figure 1. The interlaminar interface properties in these laminates were studied by measuring the contact electrical resistivity of this interface. The measuring set and the specimen on the measuring stand are illustrated in Figure 3.

a)



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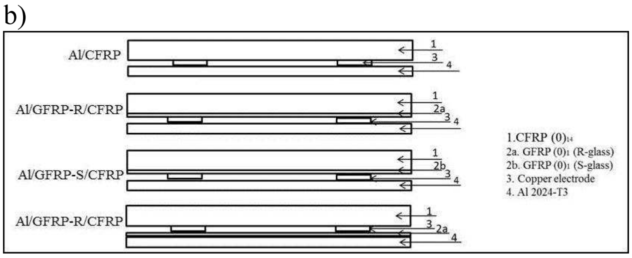


Fig.1. Diagram of laminate and electrode configurations for contact resistivity measurements: a) top view, b) side view

Rys. 1. Schemat konfiguracji laminatów i elektrod do pomiarów oporności kontaktowej: a) widok z góry, b) widok z boku

The measurements were carried out by means of a potentiostat (Atlas 0531, Atlas-Sollich, Poland) (Fig. 3a). Variations in current versus time were recorded at a constant predetermined potential. Measuring was continued until stabilization of the current level. Furthermore, the variations in temperature on the specimen surfaces were recorded in the course of electrical measurements by means of a thermovision camera (OPTRIS PI450) (Fig. 3a). Moreover during determination of the contact parameters, the voltage was scanned from 0.5 V to 1 V at the rate of 1mV/s and current density vs. voltage was recorded.

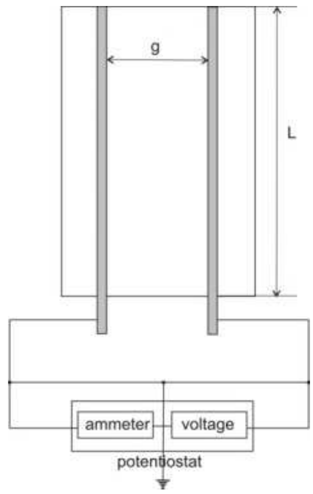
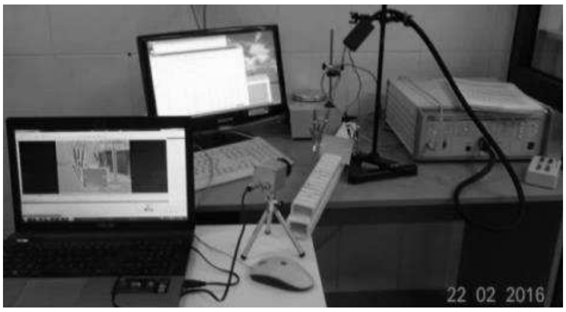


Fig. 2. Connection diagram for surface resistivity measurements; g - distance between electrodes, L - efficiency length of electrode

Rys. 2. Schemat połączenia do pomiarów oporności powierzchniowej; g - odległość pomiędzy elektrodami, L - efektywna długość elektrody

a)



b)

Fig. 3. Experimental setup of electro- and thermovision camera testing (a) and specimen in measuring circuit (b)

Rys. 3. Stanowisko do pomiarów elektrycznych i termowizyjnych (a) oraz próbka w obwodzie pomiarowym (b)

The surface resistance was determined using Ohm's law (1) and the surface resistivity was calculated according to equation (2):

$$R_s = \frac{U}{I_s} \quad (1)$$

$$\rho_s = \frac{P}{g} R_s \quad (2)$$

where: R_s - measured surface resistance in ohms, U - fixed voltage in [V], I_s - measured current in [A], ρ_s - surface resistivity in ohms (per square), P - efficiency length of electrode in [mm], P = 2L according ASTM standard [15], g - distance between electrodes in [mm].

Contact resistivity ρ_c was calculated according to equation (3):

$$\rho_c = R_c A_c \quad (3)$$

where: R_c - in-plane measured contact resistance in ohms, A_c - surface contact in [cm^2].

RESULTS AND DISCUSSION

The tests of the electrical properties of the components used for the production of hybrid laminates and the tests of electrical properties of the metal-composite and composite-composite interface were commenced with temperature measurement in the course of 8V DC voltage application for the tested CFRP, GFRP and anodized 2024T3 aluminium alloy.

As a result of the thermovision measurements, infrared images were obtained together with the values of the examined surface temperature and its distribution. A thermal imaging camera can produce a colour-scaled image made up of pixels that represent individual temperatures. Thermal imaging records only surface temperature.

On the basis of the results of temperature measurements (Fig. 3), the thermal imaging with changing colours shows that the areas around the copper electrodes indicate a temperature increase. C The curves presented in Figure 4 illustrating temperature distribution measurements at individual points of the specimens show the first 60 seconds after the voltage was applied. Figure 4a illustrates aluminium with the anodized layer and visible spots from 3 various points. No temperature variations were observed. It is also illustrated by means of a curve in the bottom part of this figure. The temperature remained constant during the measurement time and is equal to 20°C (equal to room temperature). Similar behaviour was observed in the case of the GFRP-R composite where even the temperature curves are represented by a straight line almost along the whole length. The behaviours of the CFRP composite with fibres arranged in the longitudinal and lateral direction to the current direction are different. Figure 4c illustrates a CFRP composite with fibres arranged in the direction of the electrodes and temperature curves commencing at 21°C and beginning to increase simultaneously at each of the 4 points. However, in Figure 4d it is clearly visible that the temperature is different at individual points (CFRP fibres arranged perpendicularly to electrodes). In this case, the distribution is shown for 5 measuring points. In the case of points arranged in the central part of the CFRP composite (points 1,2,3), the temperature is constant and is equal to 20°C. However, points 4 and 5 are characterized by a sudden temperature increase, particularly in the case of point 4 with the curve showing the temperature of 28°C and the temperature of 24°C at point 5. Points 4 and 5 are situated in the vicinity of the conductive copper tape. After 45 seconds, the temperature decreases. Colour changes within the electrodes are visible at these points on the thermogram. The local increase in temperature around the electrodes is indicative of the occurrence of local current leakage caused by the higher conductivity of these areas or of composite dipole polarization with a short relaxation time (several tens of seconds).

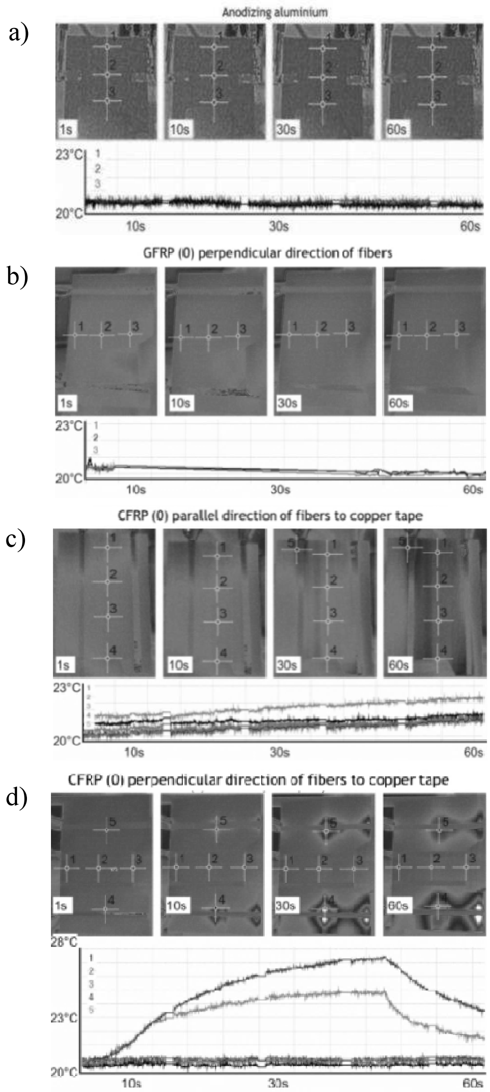


Fig. 4. Views of thermovision camera: a) aluminium alloy with sulphuric anodizing, b) GFRP-R, c) CFRP parallel direction of electrodes to fibres, d) CFRP perpendicular direction of electrodes to fibres

Rys. 4. Obraz z kamery termowizyjnej: a) stop aluminium anodowany w kwasie siarkowym, b) GFRP-R, c) CFRP, elektrody równoległe do kierunku włókien, d) CFRP, elektrody prostopadłe do kierunku włókien

The measurement of surface current density versus time was performed and illustrated by means of the curves in Figure 5. It was observed that in the case of the CFRP composite, the curves tend to achieve a stable state. Initially, at a predetermined voltage they are characterized by a visible jump and then they stabilize at the level about $5.65 \cdot 10^{-2}$ A/cm^2 in the case of fibres arranged along the electrodes (Fig. 5a), and in the case of fibres arranged perpendicularly to the electrodes, the curve clearly stabilizes at the value of $5.2 \cdot 10^{-2}$ A/cm^2 (Fig. 5b). A good correlation in time between temperature and current is observed (see Fig. 4c, d and Fig. 5c, d). This material is slightly conducting (dissipative). In the case of the aluminium and GFRP-R specimens, an unstable but very low current is recorded with density values oscillating between 0 and $-1 \cdot 10^{-10}$ A/cm^2 for aluminium (Fig. 5c), and between the upper limit of $2 \cdot 10^{-10}$ A/cm^2 and lower limit of $-3 \cdot 10^{-10}$ A/cm^2 for GFRP-R (Fig. 5d). These measured values are characteristic for insulators.

due to the energy consideration [18]. Average negative resistance is a result of current oscillation phenomena in the vicinity of zero (Fig. 5c, d). Negative resistance has been previously observed for a number of materials, including polymer films and metal films and was explained in terms of the effect of voltage or current on the conduction mechanism [19, 20].Wang et al. [18] report an apparent negative resistance phenomenon in which the entire current–voltage characteristic is a straight line with a negative slope passing through the origin of the coordinates. They observed this phenomenon in composites containing continuous carbon fibres, such that the pressure during composite fabrication is unusually high. Wang et al. [18] and Lee et al. [19] described the probable mechanisms of this phenomenon in dielectric polymers and fibre composites. The surface resistivity of the carbon fibre composite indicates the too low conduction of this composite, lower than in literature for similar composites (compare Tables 2a and 3).

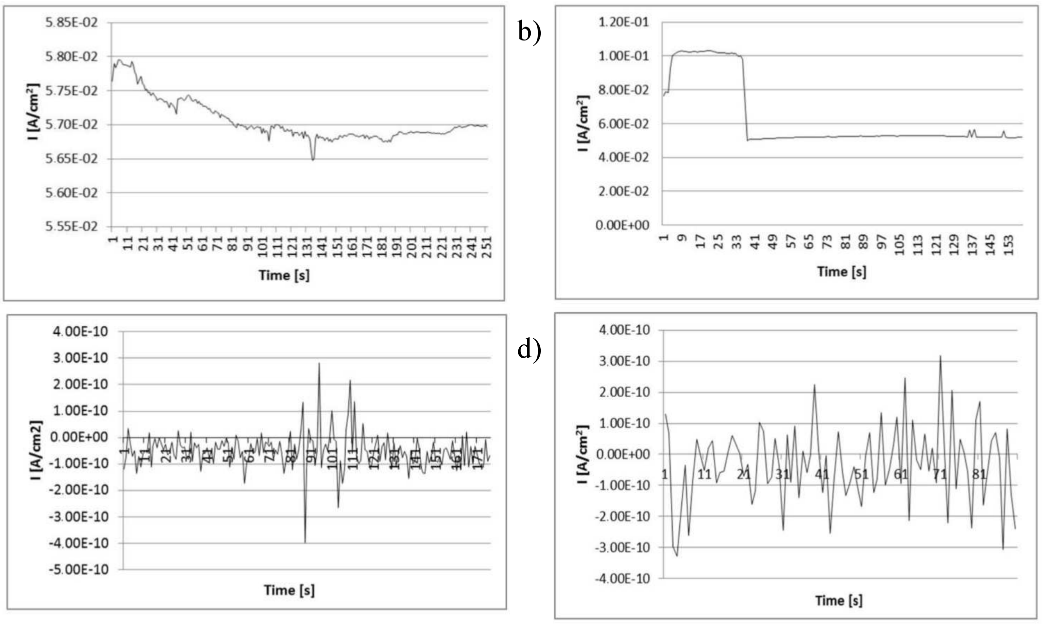


Fig. 5. Curves illustrating surface current density versus time: a) CFRP, electrodes parallel to fibre direction, b) CFRP electrodes perpendicular to fibre direction, c) aluminium anodized in sulphuric acid, d) GFRP-R

Rys. 5. Wykresy zależności powierzchniowej gęstości prądu od czasu: a) CFRP, elektrody równoległe do kierunku włókien, b) CFRP, elektrody prostopadłe do kierunku włókien, c) aluminium anodowane w kwasie siarkowym, d) GFRP-R

Tables 2 and 3 contain the values of electrical surface resistivity for components and in-plane contact resistivity for all the configurations of the laminate specimens, respectively. It can be seen that the aluminium oxide and GFRP-R composite are insulators with very high but negative resistivity. The anodized aluminium interface - GFRP-R in the hybrid laminate is characterized by very high negative resistance as well as resistivity (see Tab. 3). The GFRP-S composite is also an insulator but its surface resistivity is lower and positive. The absolute value of anodized aluminium resistance is close to the values available in literature [16, 17]. This is apparent negative resistance because true negative resistance in the formal sense is not possible

The difference in resistivity is observed in the parallel and perpendicular directions of the fibre. The electrical properties of polymer matrix composites reinforced with carbon fibres depend not only on the electrical properties of the matrix and fibres but also on the current direction in the ratio to fibre direction and on the fibre volume fraction (V_f). The resistivity of carbon fibres used in the prepregs is equal to about $1.7 \div 1.8 \cdot 10^{-3}$ $\Omega \cdot$cm [21, 22]. The electrical resistivity values for polymers are typically equal to 10^{14} up to 10^{17} $\Omega \cdot$cm [21] or higher [23]. Macroscopic anisotropic resistivity is possible if the electrical isotropic fillers are distributed anisotropically in the matrix [24, 25] or if the electrically anisotropic fillers are distributed in the isotropic matrix [24, 26]. This can be seen in carbon fibre polymer materials where the epoxy matrices are extremely insulating and can have a resistivity in the order of 10^{22} $\Omega\cdot$cm [23]. In the case of unidirectional specimens, the high resistivity of these epoxies has a proportional effect on the electrical conductivity of the material in the direction of carbon fibres. Using the rule of mixtures and the percent volume of non-conducting epoxy and carbon fibres, it is possible to determine the fibre direction conductivity [5]. The same is not true for the directions perpendicular to the fibre direction or through the thickness of the composite. The research by Angelidis et al. [27] showed for unidirectional carbon fibre reinforced polymers that the electrical conductivities in these two directions are 10^{-3} and 10^{-4} times lower than in the fibre direction. This is because of the fact that the epoxy is a non-conducting material, the carbon fibres are not perfectly straight and have some contact with each other in the transverse and thickness directions.

TABLE 2. **Surface resistance and resistivity of components (a) and in-plane contact resistance and resistivity in laminates (b)**

TABELA 2. **Opór i oporność powierzchniowa komponentów (a) i opór i oporność kontaktowa w laminatach (b)**

a)

Specimen name	R_s [Ω]	ρ_s [Ω/□]
Al (SAA)	$-8.7\cdot 10^{11}$	$-1.01\cdot 10^{11}$
CFRP II (perpendicular direction of fibres to electrode)	$4.1\cdot 10^{2}$	$8.21\cdot 10^{2}$
CFRP ⊥ (parallel direction of fibres to electrode)	$2.2\cdot 10^{2}$	$1.33\cdot 10^{3}$
GFRP-S	$4.27\cdot 10^{6}$	$2.56\cdot 10^{7}$
FRP-R	$-9.16\cdot 10^{11}$	$-1.83\cdot 10^{12}$

b)

Specimen and place of electrode	R_c [Ω]	ρ_c [$\Omega\cdot$cm^2]
1 - CFRP/copper electrode/Al	$3.5\cdot 10^{0}$	$2.85\cdot 10^{1}$
2 - CFRP/GFRP- R/copper electrode/Al	$-2.3\cdot 10^{11}$	$-1.84\cdot 10^{12}$
3 - CFRP/GFRP-S/copper electrode/Al	$2\cdot 10^{1}$	$1.62\cdot 10^{2}$
4 - CFRP/copper electrode/GFRP-R/Al	$2.43\cdot 10^{1}$	$1.95\cdot 10^{2}$

TABLE 3. **Electrical parameters of CFRP laminates by [18, 23, 28-30]**

TABELA 3. **Parametry elektryczne laminatów CFRP wg [18, 23, 28-30]**

Current direction	R_v [Ω]	R_c [Ω]Wang [29]	ρ_c [$\Omega\cdot$cm^2] Wang [18]	ρ_v [$\Omega\cdot$cm] Abry [30]
CFRP II	3÷13 Yang [23] 5÷150 Lin [28]	$3\cdot 10^{-1}$	$-2\cdot 10^{-3}$	$2.93\cdot 10^{-3}(0.6V_f)$ $3.7\cdot 10^{-3}$ $(0.5V_f)$
CFRP⊥		$4.7\cdot 10^{-1}$	$1.2\cdot 10^{-1}$	$4.16\cdot 10^{0}$ $(0.6V_f)$ $1.3\cdot 10^{1}$ $(0.5V_f)$

After the autoclave process of metal-composite consolidation, the high surface resistivity of the anodizing layer and GFRP-R composite is maintained (Table 2b). Therefore, the in-plane contact resistivity of the carbon composite/dielectric layer depends on the resistivity of this composite. The galvanic cell creation between aluminium and CFRP is blocked by the GFRP-R layer and by the oxide layer. However, the stability of the anodizing layer in a wet environment must be examined because of the observed current oscillation (Fig. 5c). Layer porosity can be one of the causes of this phenomenon.

A significant difference is observed in the contact resistivity value between the specimens with glass fibre R and S composite (specimens 2 and 3 in Table 2b). For R-glass 0.255 mm thick, the value is equal to $1.84\cdot 10^{12}$ $\Omega\cdot$cm^2, in the case of S-glass of 0.13 mm, this value is equal to $1.62\cdot 10^{2}$ $\Omega\cdot$cm^2 only. It means that the use of a thin layer of S-glass does not lead to the effect of dielectric isolation. Therefore it is necessary to examine the microstructure of this laminate in order to identify the reason for failure.

CONCLUSIONS

Aluminium oxide and GFRP-R composites are insulators with very high but negative surface resistivity. The surface resistivity of the CFRP composite is equal to about $10^2\div 10^3$ Ω/ and depends on the direction of the fibres. When the electrodes are located perpendicular to the fibres, the surface resistivity is lower and the surface temperature increases locally. Generally, the contact resistivity of this composite is $\sim 10^3$ times higher than that described in literature as a result of the high quality of the prepreg and laminate curing in an autoclave. The electrical contact resistivity measurements demonstrated that it is possible to obtain a dielectric interface between the aluminium alloy and carbon reinforced composite by means of the anodizing process of the aluminium and the glass prepreg layer 0.25 mm thick. A thinner layer of glass composite does not lead to an increase in in-plane contact resistivity.

Acknowledgements

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