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Electrically conductive carbon fiber / PEKK / silver nanowires multifunctional composites

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A B S T R A C T

New multifunctional composites with high through-thickness electrical conductivity were elaborated with carbon fiber (CF) reinforced PEKK filled with silver nanowires (AgNWs). Composites were fabricated by film stacking at high temperature for the first series of samples and by powder impregnation for the second series. Morphological analysis of composites was performed by scanning electron microscopy (SEM) in order to check impregnation quality of carbon fibers and dispersion of AgNWs across the ply. Electrical conductivity was measured in the out-of-plane direction of laminates and recorded as a function of the volume fraction of silver nanowires. It was observed that the presence of small fractions of AgNWs (~1.5 vol%) within the carbon fiber reinforced PEKK increased the transverse conductivity of non-filled CF/PEKK laminates by at least 3 orders of magnitude. The maximum conductivity achieved by laminates is 250 S m^{-1} . These new multifunctional composites may be used as structural high performance composites for electromagnetic shielding, or lightning strike protection.

1. Introduction

Multifunctional composites have a structural function combined with a non-structural function. Generally, the structural function is accomplished by high performance carbon fiber reinforcement. The non-structural function, including electrical or thermal conductivity, sensing and actuation or flame retardancy, can be achieved by nano or micronic particles [1–3]. Carbon fiber composites exhibit low out-of-plane electrical conductivity [4]. Improving the electrical conductivity of composite structures is required for several aerospace applications. For this purpose, various techniques were often employed to increase electrical conductivity of fiber reinforced composites. The insulating matrix was replaced by a conductive matrix [5] or substituted by a conductive particles/polymer composite [6–8]. In another approach, conductive nanoparticles like carbon nanotubes were produced at the surface of fibers fabrics [9,10]. The use of interleaved conductive veils between composites plies was also studied [11]. The first technique, which has been employed not only to increase electrical conductivity but also mechanical properties of fiber composites [8,12], was chosen for this work.

For structural composites, the use of thermoplastics in the aerospace industry represents an alternative to thermosets. The major advantages are resilience and toughness, but also easier processing and recyclability. The poly aryl ether ketone (PAEK) family offers large possibilities as matrices for reinforced continuous carbon fibers composites by providing appropriate mechanical and thermal properties for high performance applications [13–17]. Poly ether ketone ketone (PEKK) [18] exhibits high glass transition temperature T_g and high melting temperature T_m [19]. However, it has been shown that T_m strongly depends on the ratio between the terephthalate (para linkages) over isophthalate (meta linkages) isomers in the main chain, noted as the T/I ratio [20]. In a previous paper about PEKK with different T/I ratios [21], we reported that the melting point of PEKK with 100% para linkages (close to $393 \text{ }^\circ\text{C}$) decreases to about $33 \text{ }^\circ\text{C}$, $58 \text{ }^\circ\text{C}$ and $93 \text{ }^\circ\text{C}$ compared with PEKK with T/I ratio of 80/20, 70/30 and 60/40 respectively. The tailoring of T_m opens the route for composite processing at lower temperatures to avoid degradation [22].

Many works have been devoted to increase electrical conductivity in polymer materials. Carbonaceous particles such as carbon black (CB) [23], carbon nanofibers (CNF) [15], carbon nanotubes (CNT) [24,25], graphene particles [26] have been used as well as metallic particles with low aspect ratio ($\xi = \text{diameter over length}$) [27] or high aspect ratio [28,29]. Best results in terms of homogeneous dispersion, low percolation threshold and high conductivity

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were achieved with high aspect ratio metallic nanowires for low loaded composites [30,31]. For such nanocomposites, density and mechanical properties of the polymeric matrix are practically preserved.

In a previous work we reported that highly conductive PEKK/AgNWs composites can be elaborated with homogenous dispersion of the fillers [32]. Now, the aim of this work is to elaborate PEKK and PEKK/AgNWs carbon fiber reinforced composites. For this purpose we propose two different approaches to impregnate a carbon fiber unidirectional tape with a high viscosity PEKK with and without high aspect ratio AgNWs. The morphology of composites was studied by SEM. We report the electrical conductivity values of composites with different AgNWs volume fractions.

2. Experimental

2.1. Materials

2.1.1. Poly ether ketone ketone

The high performance thermoplastic PEKK (KEPSTAN 6003) ($M_w = 25\,000$) was supplied by ARKEMA in powder form ($20\ \mu\text{m}$). The T/I ratio is 60/40. Fig. 1 shows the DSC thermogram of PEKK 60/40 previously cooled at $10\ \text{°C}\ \text{min}^{-1}$, during a heating scan at $10\ \text{°C}\ \text{min}^{-1}$.

PEKK with 60% of para linkages and 40% of meta linkages has a glass transition temperature $T_g = 156\ \text{°C}$, a cold crystallisation temperature $T_{cc} = 241\ \text{°C}$ and a melting temperature $T_m = 300\ \text{°C}$. With these scanning conditions, the crystallinity ratio of PEKK 60/40 is $3 \pm 2\%$: It will be considered as a quasi-amorphous polymer. Note that PEKK 60/40 can be crystallized by an annealing process between T_g and T_m or by slow cooling from the molten state [21].

2.1.2. AgNWs silver nanowires

Silver nanowires were elaborated through the polyol process. Ethylene glycol (EG) was used as a solvent and a reducing agent. AgNO_3 was used as the precursor and poly vinyl pyrrolidone (PVP) ($M_w = 55,000$) served as the capping agent. The reaction was carried out at $160\ \text{°C}$ in a round bottom balloon. The elaboration of silver nanowires through the polyol process was first described by Sun et al. [33]. This process allowed us to obtain AgNWs with an average diameter of $190\ \text{nm}$ and an average length of $41\ \mu\text{m}$ [32]. The mean aspect ratio of AgNWs is 215. After a cleaning process, the

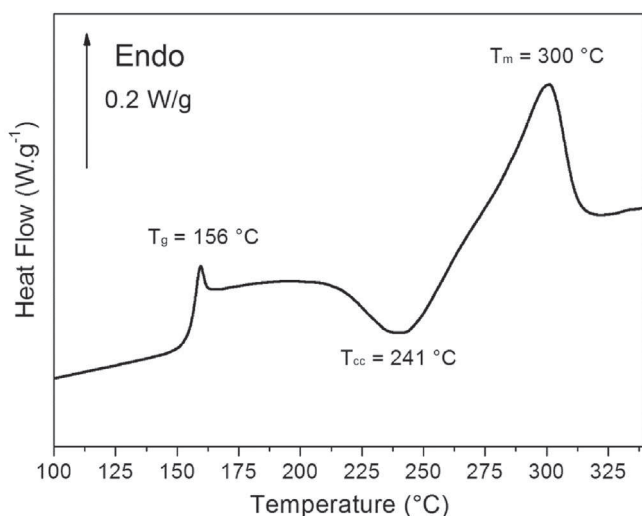


Fig. 1. DSC thermogram of PEKK 60/40 at $10\ \text{°C}\ \text{min}^{-1}$.

AgNWs can be stored as a suspension in water or ethanol. Clean AgNWs in ethanol are show in Fig. 2.

2.1.3. Carbon fiber

High performance carbon fiber (CF) unidirectional tape (Fig. 3a) was used to elaborate composites. The carbon fiber tapes were woven by a glass fiber reinforced polyamide thread each centimeter to prevent fraying on the edges. It was supplied by Toray under the trade name T700S-12K. Its density according to technical data sheet is $1.8\ \text{g}\ \text{cm}^{-3}$.

2.2. Experimental

2.2.1. Scanning electron microscopy

Morphological analysis were performed by SEM. Analysis were carried out on a JEOL JSM 6700F instrument equipped with a field emission gun. Images were collected under $10\ \text{KeV}$ accelerating voltage. A drop of AgNWs in suspension in ethanol was deposited in a SEM pin for observation. Dispersion of AgNWs within the reinforced carbon fiber composites was studied in the backscattered electron mode in order to enhance the contrast between organic matrix and metallic nanowires. Fracture surfaces of composites were observed after being cryo-fractured in liquid nitrogen.

2.2.2. Transverse electrical conductivity

For through-thickness electrical conductivity measurements of laminates, samples were first silver-coated to ensure good electrical contact and then placed between two circular electrodes of $10\ \text{mm}$ in diameter. The surface resistance R was measured using the four-wire configuration with a Keithley 2420 SourceMeter. The bulk resistivity ρ was calculated by:

$$\rho = \frac{R \times S}{e}$$

where S is the electrode surface and e is the thickness of the sample. The dc conductivity σ_{DC} was given by:

$$\sigma_{DC} = \frac{e}{R \times S}$$

3. Elaboration of CF/PEKK and CF/PEKK/AgNWs laminates

3.1. Film stacking

Film stacking is a commonly used technique to impregnate carbon fibers at laboratory scale [34,35] and industrial scale [36]. Carbon fiber tape was laminated between layers of PEKK or PEKK/AgNWs and then melted and pressed to drive the resin between the fibers. PEKK films were produced by pressing PEKK powder, at $1\ \text{MPa}$ and $340\ \text{°C}$ for $5\ \text{min}$. PEKK/AgNWs films were obtained by pressing PEKK/AgNWs pellets using the same experimental parameters. The elaboration of PEKK/AgNWs pellets is described in a previous work [32]. Thin films between 80 and $120\ \mu\text{m}$ were obtained. Carbon fiber reinforced laminates were elaborated by pressing the sandwich configuration at $10\ \text{MPa}$ during $10\ \text{min}$ at $350\ \text{°C}$ (Fig. 3b).

3.2. Powder impregnation

Since PEKK was synthesized in powder form, the powder impregnation technique was well suited for elaboration of reinforced carbon fiber laminates. This technique was employed to impregnate glass fibers [37] and carbon fibers [38,39] by powdered

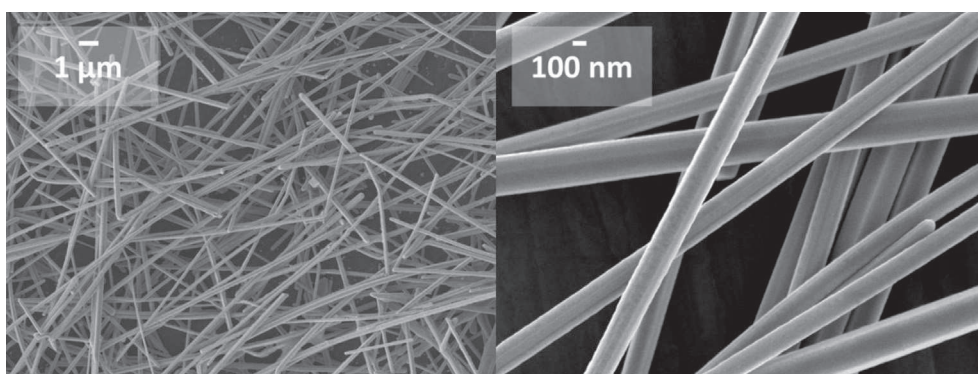


Fig. 2. SEM images at two magnifications of clean AgNWs.

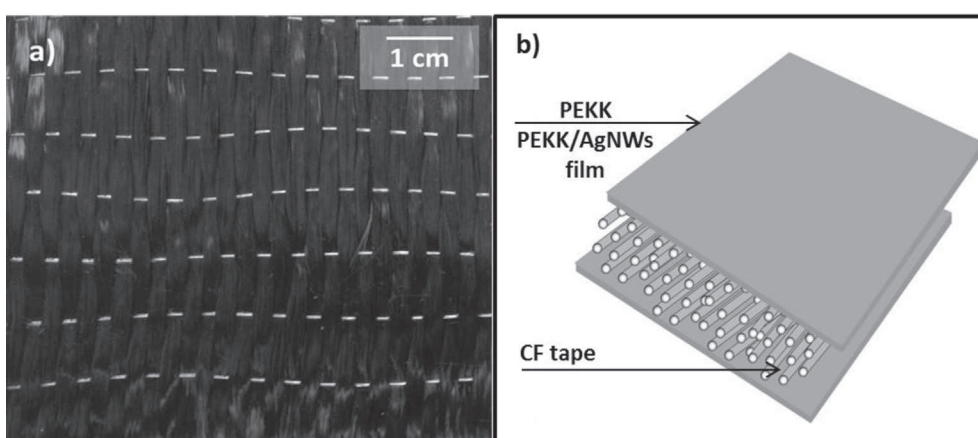


Fig. 3. a) Carbon fiber unidirectional tape b) Composite preparation by film stacking.

thermoplastics, including those with high viscosity e.g. PAEKs [40,41].

With this technique, the main purpose was to incorporate PEKK powder and AgNWs inside carbon fiber tape, achieving an close contact between fibers, polymer powder and silver nanowires prior to the melting of PEKK, as it is schematically outlined in insert on Fig. 4 where the elaboration process of carbon fibers reinforced laminates is presented.

The schema 1 (Fig. 4) shows the carbon fiber (CF) vertically maintained and cleaned by air flow to remove impurities. The insert of Fig. 4 gives a representation of of the impregnated fiber and Fig. 5a shows an image of the swelling of the carbon fibers tows due to the incorporation of the PEKK powder and metallic nanowires.

PEKK/AgNWs powder was elaborated by mixing AgNWs in suspension in ethanol with PEKK powder. For obtaining a homogenous powder, the mixture was sonicated and ethanol was evaporated by a rotary evaporator [32]. PEKK powder or PEKK/AgNWs powder was mixed with ethanol. The weight ratio (PEKK or PEKK/AgNWs over ethanol) ranges from 0.2 to 0.3. The mixture was sonicated and mechanically stirred for a few seconds for achieving good dispersion and avoid agglomerates. The schema 2 illustrates the impregnation device. The suspension was applied on each face of the carbon fiber tape through a fluid stream of 500 μm width. A flow rate and pressure are necessary to open and spread the carbon fiber to facilitate the incorporation of polymer powder and nanowires.

The next step is illustrated in 3 (Fig. 4). Then, samples were dried at 90 °C between 5 and 10 min to evaporate ethanol. Fig. 5b exhibits

the close contact between PEKK powder, CF and AgNWs in the dried sample.

The final stage 4 (Fig. 4) involved hot pressing of samples at 350 °C at 10 MPa for 10 min to achieve the impregnation process. The thickness of fabricated laminates were between 300 and 500 μm.

Previous work on PEKK/AgNWs nanocomposites has shown low percolation threshold of 0.6 vol% [32]. According to this and in order to obtain highly conductive composites, the content filler of laminates was superior to 1 vol%. Two different assemblies were elaborated: a simple one with a single ply and another assembly with two laminates superimposed at 0°/± 90°.

4. Results and discussion

4.1. Morphology of composites

Morphological analyses, described in the following, were performed by SEM on fracture surfaces from cryo-cut samples. SEM images were recorded perpendicular to the direction of fibers. Specific attention will be paid to impregnation quality as well as carbon fibers and silver nanowires distribution through laminates.

4.1.1. Morphological analysis of composites elaborated by film stacking

Fracture surfaces of unfilled laminates (CF/PEKK) elaborated by film stacking are presented in Fig. 6.

In Fig. 6a and b, the entire thickness of the laminate is represented, while Fig. 6c shows an area in the center of the composite.

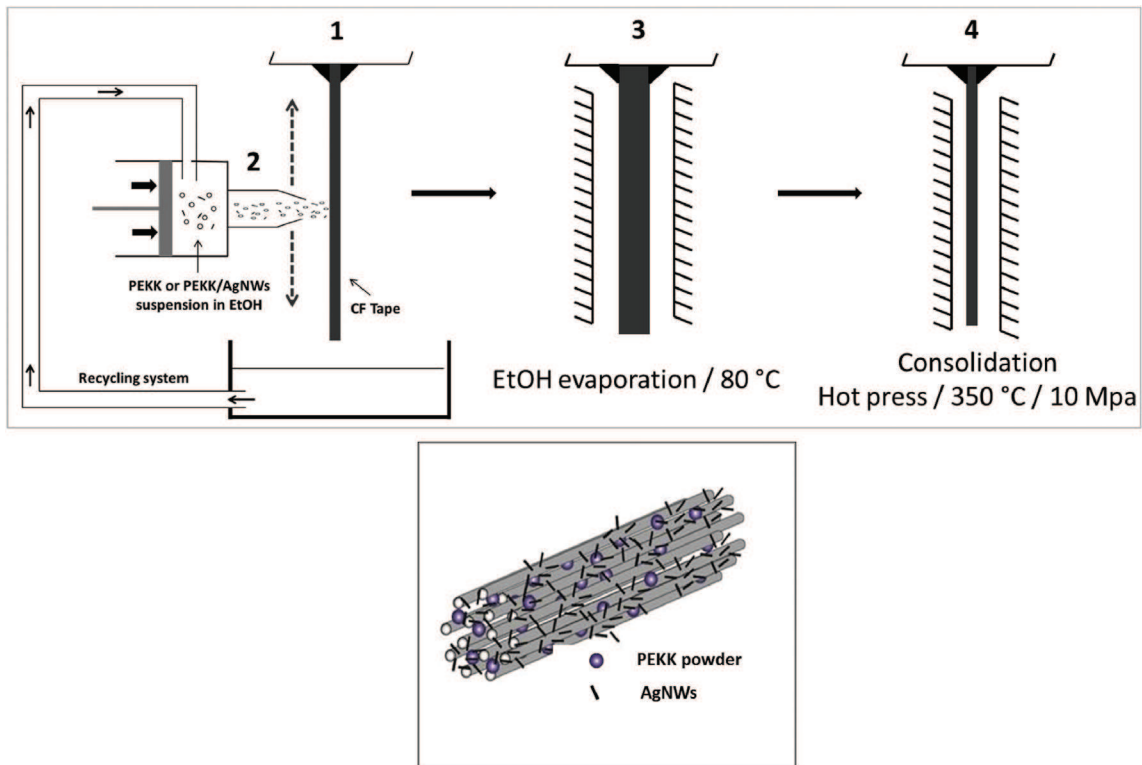


Fig. 4. Laboratory scaled powder impregnation process.

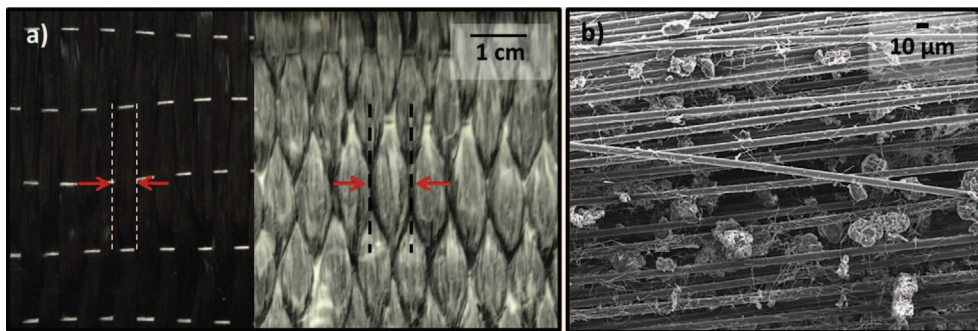


Fig. 5. Powder impregnation process: a) evidence of 0 swelling of carbon fibers tows after the incorporation of PEKK powder and AgNWs, b) close mixing between different materials prior to the melting of the polymer.

SEM images reveal a poorly impregnated carbon fiber tape where bare fibers and large voids are observed. PEKK does not penetrate in the ply and stays at the surface of the carbon fiber tape (Fig. 6b). In fact, it flows between carbon fiber tows as is shown in Fig. 6a), creating a heterogeneous laminate with poor polymer regions.

Filled laminates (CF/PEKK/AgNWs) elaborated by film stacking are presented in Fig. 7. SEM images were collected in the back-scattered electron mode.

An analogous morphology is observed for both filled and unfilled laminates. PEKK shows limited penetration into carbon fiber tows. The poor wetting of carbon fibers obtained by the film stacking technique might be due to the high viscosity of PEKK.

4.1.2. Morphological analysis of composites elaborated by powder impregnation

CF/PEKK laminates elaborated by powder impregnation are presented in Fig. 8 at different magnifications.

The powder impregnation technique allows us to prepare

laminates practically free of void. An intimate contact between the materials before the melting of PEKK, was necessary to achieve high impregnation quality and optimized wetting of carbon fibers. In addition, uniform random distribution of fibers within the laminate was also achieved. Morphological analysis also reveals low delamination.

The wetting of carbon fibers was also achieved with PEKK filled with silver nanowires (Fig. 9). SEM images reveal homogeneously dispersed silver nanowires all across the laminate thickness (Fig. 9a) in the resin-rich regions as observed by film stacking but also inside the carbon fiber tows: SEM images show a continuous path across the ply. The dispersion of nanowires within the laminate is similar to the dispersion of AgNWs after fracture, previously reported in PEKK/AgNWs nanocomposites [32].

At higher magnification and inside carbon fiber tow (Fig. 9c and d), SEM analysis shows fully wet carbon fibers and shows the presence of AgNWs at the fiber/matrix interface, creating continuous electrical paths within the laminate.

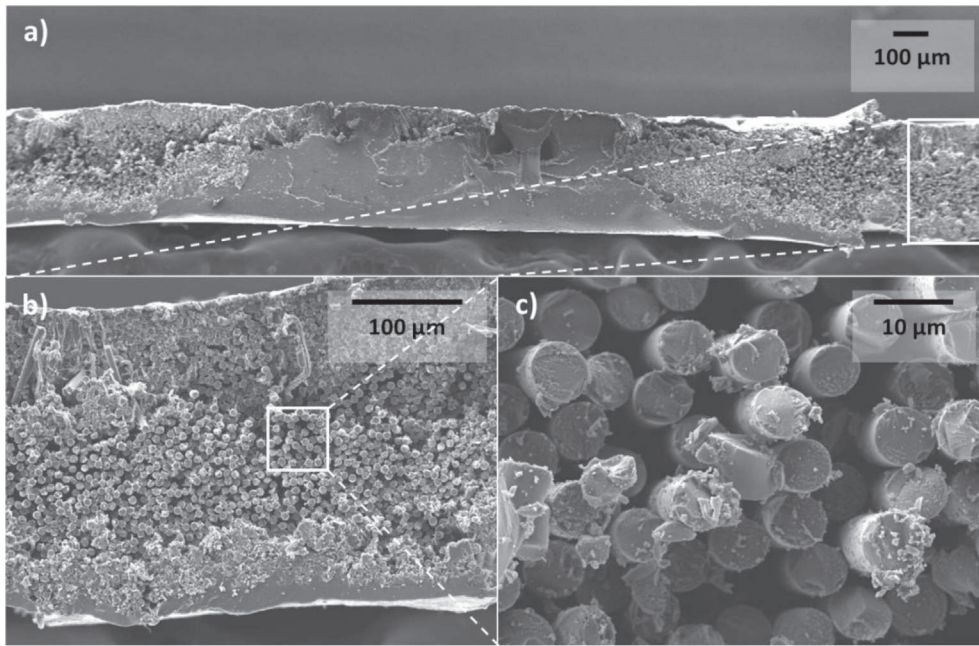


Fig. 6. Fracture surfaces of CF/PEKK laminates elaborated by film stacking at different magnifications.

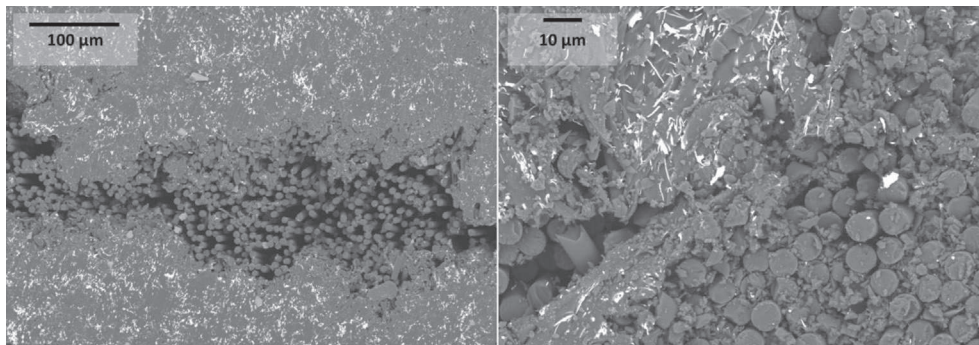


Fig. 7. Fracture surfaces of CF/PEKK/AgNWs laminates elaborated by film stacking at different magnifications.

4.2. Through-thickness electrical conductivity of composites

Through-thickness electrical conductivity of carbon reinforced composites was determined at room temperature for filled and unfilled composites prepared by both techniques i.e. film stacking and powder impregnation. Results are discussed in this section.

Characteristics of PEKK and unfilled CF/PEKK laminates, used as reference materials, are listed in Table 1.

PEKK is an insulator with a dc conductivity of $8 \times 10^{-14} \text{ S m}^{-1}$ that drastically increases by 12 orders of magnitude with the presence of carbon fibers. Conductivity of unfilled CF/PEKK laminates fabricated by film stacking is 10^{-1} S m^{-1} . Unfilled CF/PEKK laminates elaborated by powder impregnation have a conductivity of 10^{-2} S m^{-1} . These values are in the range of those of carbon fiber reinforced polymers [4]. It is interesting to note that the conductivity is higher by one decade for laminates elaborated by film stacking. This difference can be explained by two factors: a higher volume fraction of carbon fibers on one hand and a direct contact between carbon fibers as observed in Fig. 6, on the other hand. The main objective of this work is to improve this low through-thickness electrical conductivity of carbon reinforced laminates by introducing metallic nanowires.

Transverse conductivity of filled laminates elaborated by both film stacking and powder impregnation are compared with PEKK/AgNWs nanocomposite at the same volume fraction of nanowires taking as reference the constitutive materials of the composite (Fig. 10). Accordingly, such evaluation avoid the uncertainty due to different porosity inherent to processing.

Beyond the percolation threshold, PEKK/AgNWs nanocomposites reach a conductivity of 100 S m^{-1} for a volume fraction of 2.5% of silver nanowires. Composites elaborated by film stacking exhibit significant increase in the transverse conductivity due to the addition of nanowires from 10^{-1} S m^{-1} for the unfilled composite to 70 S m^{-1} for the one ply filled laminate. Regardless of the low quality of composites elaborated by film stacking with large voids, conductivity is high. In fact, conductivity is mainly governed by resin-rich zones where AgNWs are homogeneously dispersed (Fig. 7) at the surface of the carbon fiber tape and in the inter-tow zones. In the two plies assembly, the conductivity level is maintained (68 S m^{-1}) indicating a good distribution of nanowires in resin-rich areas at the interface of the plies. However conductivity remains lower than that of PEKK/AgNWs nanocomposites (100 S m^{-1}). Differences are explained by the heterogeneity of the laminate with large voids and resin-rich areas (Fig. 7).

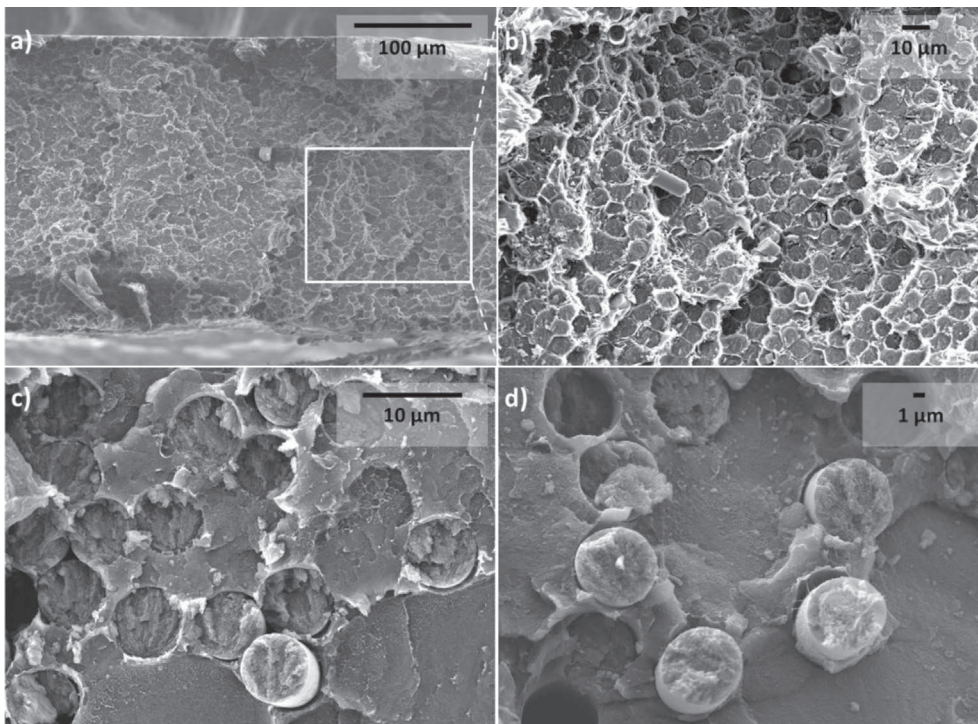


Fig. 8. Fracture surface of CF/PEKK composites elaborated by powder impregnation.

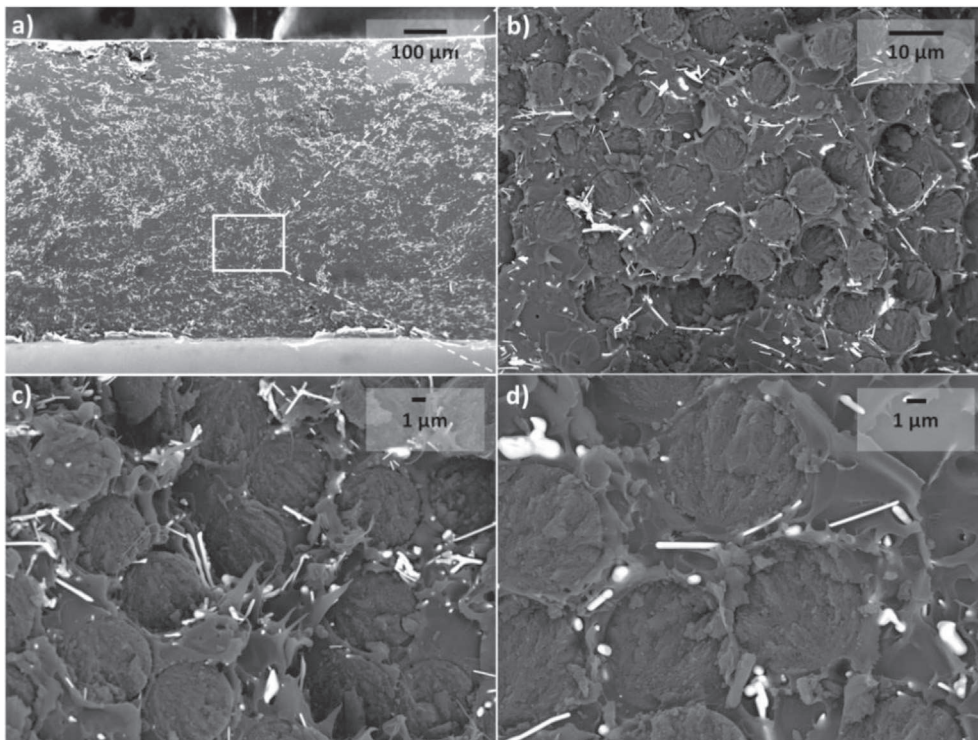


Fig. 9. Composite FC/PEKK/16 wt% AgNWs.

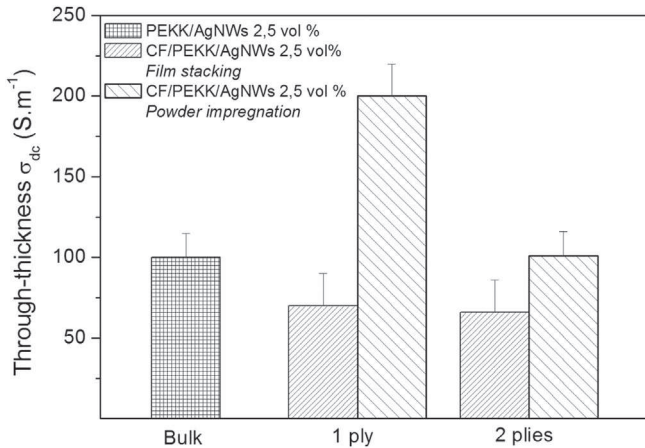
Laminates elaborated by powder impregnation exhibit higher conductivity (Fig. 10). Ag nanowires increase the conductivity by 4 orders of magnitude in the thickness direction of the unfilled composite (Table 1): For the one ply configuration, the laminate containing 2.5 vol% has a conductivity of 200 S m^{-1} . This high level

of conductivity is consistent with the hypothesis of continuous paths through the thickness of the ply, as it was observed from morphological analyses (Fig. 9). Besides, SEM images also show a homogenous dispersion of nanowires. Moreover multifunctional composites reach the same values of conductivity as well-dispersed

Table 1

Characteristics of reference materials.

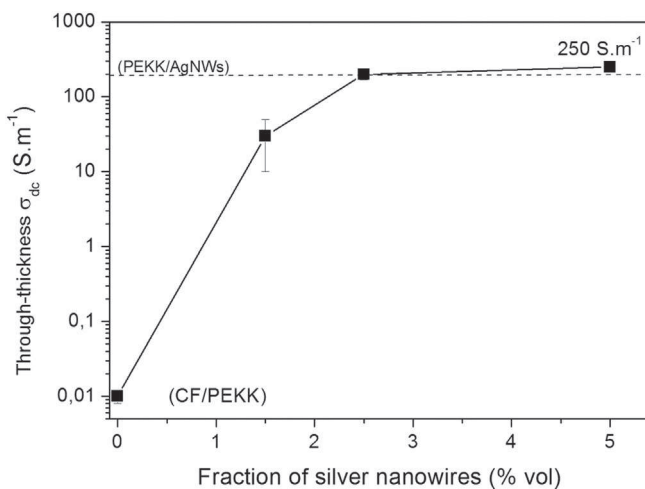
| Materials | PEKK matrix | CF/PEKK film stacking | CF/PEKK powder impregnation |
|---|----------------------------------|------------------------------|-----------------------------|
| Vol% of CF | 0 | 60 ± 5 | 50 ± 10 |
| Through-thickness conductivity ($S m^{-1}$) | $10^{-14} \pm 5 \times 10^{-15}$ | $1.5 \times 10^{-1} \pm 0.1$ | $3 \times 10^{-2} \pm 0.01$ |

**Fig. 10.** Through-thickness electrical conductivity of PEKK/AgNWs nanocomposite [32] and CF/PEKK/AgNWs composites elaborated by film stacking and powder impregnation with 2.5 vol% AgNWs.

PEKK/AgNWs nanocomposites [32], suggesting that in the multifunctional composites homogenous dispersion was also achieved. For the two plies assemblies, laminates also have a high conductivity of $100 S m^{-1}$. Despite the complexity of this material and the interface between the two plies, conductivity is unmodified, indicating that continuous paths across ply interface still exist.

Conductivity measurements at room temperature were also performed in CF/PEKK laminates with different content of AgNWs. Results are reported in Fig. 11.

The introduction of silver nanowires in the CF/PEKK laminates induces a percolation mechanism (Fig. 11). Since the intrinsic conductivity of AgNWs is higher than conductivity of CF, percolation is the leading behaviour. Conductivity of the unfilled CF/PEKK laminates with 50 ± 10 vol% of CF elaborated by powder

**Fig. 11.** Electrical conductivity in the thickness direction of one ply CF/PEKK/Ag NWs laminate.

impregnation increases from $10^{-2} S m^{-1}$ to $2.5 \times 10^2 S m^{-1}$ when volume fraction of silver nanowires increases from zero to 5 vol%. This behaviour is consistent with the electrical behaviour of PEKK/AgNWs nanocomposites [32]. This data confirms that the electrical conductivity observed in CF/PEKK/Ag NWs is governed by a percolation mechanism.

In multifunctional composites where carbon nanotubes were introduced to enhance through-thickness conductivity, low values of $0.2 S m^{-1}$ were reported by Lonjon, despite a homogenous dispersion of CNT within the matrix [6]. For interlayered SWCNTs Kim reported a conductivity of $1.8 S m^{-1}$ [42] and for nickel-coated SWCNTs Chakravarthi reported a value of $0.1 S m^{-1}$ [43]. High levels of conductivity were achieved with MWCNT grown by CVD on alumina fibers in epoxy matrix [44]. With high performance thermoplastic matrix, the high level of conductivity reached in this work for CF/PEKK/Ag NWs corresponds, to our knowledge, to the highest conductivity reported in the literature for CF/thermoplastic/nanoparticles structural composites.

5. Conclusion

Multifunctional composites with high electrical through-thickness conductivity have been prepared with poly ether ketone ketone PEKK, unidirectional carbon fiber CF tapes and silver nanowires AgNWs elaborated by polyol process. First, due to the high viscosity of PEKK, low quality laminates with poor wetting of fibers and large voids were prepared by film stacking at high temperature. Second, the powder impregnation technique allows us to fabricate laminates of optimum quality, with good fiber distribution and satisfactory wetting of fibers.

The powder impregnation fabrication was achieved by a rapid and simple protocol described in this work. Prior to the melting of the resin, a matrix/fiber continuity is observed. Morphological analyses show that in composites prepared by film stacking, PEKK simply flows in the inter CF tows areas creating heterogeneous composites, while powder impregnation allows us to prepare homogenous material with good dispersion of nanowires in the intra and inter tows zones.

The achieved transverse conductivity of filled laminates (CF/PEKK/AgNWs), elaborated by powder impregnation, has been compared with the conductivity of unfilled laminates (CF/PEKK) fabricated using the same technique. This conductivity is significantly improved by 4 orders of magnitude by incorporating silver nanowires. Conductivity of low-filled laminates increases till $200\text{--}250 S m^{-1}$ for 2.5 vol% of AgNWs. Such high conductivity values are mainly due to the good quality of nanowires homogeneously dispersed across the ply thickness. To our knowledge, this value corresponds to the highest conductivity reported in the literature for this kind of multifunctional composite. Moreover, the conductivity of the two plies assembly composite reaches $100 S m^{-1}$, indicating that continuous conductive path across the interface can be maintained. Results of this work may be used for the design of future aeronautical composites structures requiring high transverse electrical conductivity.

We are convinced that a scale up of the applied process to impregnate carbon fiber herein used can be performed: The technology involved in the impregnation process remains simple. A

good impregnation can be achieved rapidly between room temperature and 80 °C. Finally it doesn't requires the use of harmful chemicals or big volume of chemicals.

Acknowledgments

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