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# Preparation and Characterization of Polytetrafluoroethylene (PTFE)/Thermally Expanded Graphite (TEG) Nanocomposites

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## Abstract

In this study, TEG-reinforced polytetrafluoroethylene (PTFE) nanocomposites were prepared. The structural properties of the PTFE/TEG composites were then investigated using X-ray diffraction (XRD) and infrared spectroscopy (FT-IR). Subsequently, the thermal stability, thermal resistance of the composites were studied through differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA). The XRD results allowed to report a high degree of dispersion of the TEG inside of the PTFE matrix. Moreover, milling at high temperature was found to enhance the dispersion of the TEG inside of the polymer matrix. Furthermore, the results revealed an increase of the glass-transition temperature when using an increased concentration of TEG. This result confirms that the degradation of the TEG/PTFE nanocomposite occurs at a higher temperature with a greater TEG loading. These finds give an indication of the potential of TEG to improve the thermal properties and durability of PTFE, for instance with application in the field of aeronautics.

**Keywords :** Nanocomposites, Physical properties, Thermal properties

## 1. Introduction

Polymer nanocomposites are multiphase materials obtained by dispersing a filling agent with dimension in the order of nanometers into a polymer matrix. Various materials are commonly used as nanofillers, such as clay, silica, carbon-based materials. The resulting polymer nanocomposite displays enhanced properties, for instance in terms of rigidity, strain resistance, electrical conductivity, etc. In this context, a significant increase of interest has been observed in the last decade in the field of black carbon, carbon nanotubes and expanded graphite (EG) used as nanofillers of polymer materials [1][2]. The present study focuses on EG as nanofiller, and in particular EG obtained by thermal treatment, *ie.* Thermally Expanded Graphite (TEG). The latter has several advantages, including availability at relatively low cost and ease of production [3]. Numerous properties can be enhanced by adding EG as nanofiller in a polymer matrix. These improvements are well documented in the literature in the context of various applications. [4-7] have reported an improvement of the oil absorption properties by using EG mixed with magnetic sorbents. This is particularly relevant in the context of environmental issues, for instance oil spill remediation, or water purification [8]. The potential of EG has also been extensively studied in the field of energy storage in buildings, in particular regarding Phase Change Materials and thermal conductivity of building materials [9-13]. The electrical conductivity of polymers is generally low. Adding nanofillers in an amount that exceeds the percolation threshold allows to improve their electrical and thermal conductive properties for application in the field of electronics [14-18]. In this context, the potential of EG to improve the performance of Fuel cells has been reported by [19-21]. Finally, [22-25] have studied the potential of EG to improve the durability and mechanical properties of polymer materials, while [26][27] have observed an increase of flame retardation properties. Hence, numerous studies have been carried out regarding the preparation and

characterization of polymer nanocomposites. They differ by the studied materials (type of polymer and nanofiller), the preparation method for the nanocomposites, and the characterization techniques involved. Commonly studied polymers include PET [28], Epoxy [22][23], PEDS [25] and PTFE [24, 29-34]. In the present study, TEG was used as reinforcing nanofiller in a fluoroplastic (PTFE) polymer matrix, whose general formula is:  $[-CF_2-CF_2-]_n$ . PTFE, owing to its' qualities of low friction and non-reactivity, is used in a wide array of applications in the industry - from wiring in aerospace, machinery lubricant, to non-stick coating for cookware. PTFE and TEG were chosen to study the possibility of increasing the functional characteristics of PTFE, including enhancing its kinetic characteristics, elastic limit, wear resistance, etc. In fact, various studies have been carried out regarding nanocomposites based on PTFE and carbon-based nanofillers [24, 29-36]. However, these studies focused mainly on the potential of carbon nanofillers to improve the mechanical and tribological properties of PTFE. They concluded towards an enhanced durability and increased resistance to wear of the obtained nanocomposites compared to raw PTFE. In this context, the aim of the present work was to further assess the potential of TEG as filling agent for PTFE. In particular, the focus was put on the impact of TEG on the structural and on the thermal properties and degradation process of the resulting PTFE/TEG nanocomposites. The influence of the conditions when preparing the nanocomposites was as well considered. In the present document, the procedure for preparing the TEG and PTFE/TEG nanocomposites is first presented, followed by a description of the characterization methods employed and associated results.

## **2. Experimental procedure**

### **2.1.Preparation of TEG**

The raw material used for the preparation of TEG was natural graphite, which was oxidized by sulfuric acid. The latter enables to optimize the expansion volume of the graphite after thermal treatment [37]. Graphite oxide was then exposed to thermal treatment in the apparatus (Fig. 1), at a temperature of  $\sim 1000^{\circ}\text{C}$  during 1 minute, which gives the best results in terms of expansion volume according to [38]. An apparatus for the production of TEG has been developed in the context of the present study. The apparatus is presented on Figure 1 ; it allows the preparation of TEG at a rate of 1.5 kg/h. A gas flow rate valve is used to control the combustion and maintain the combustion chamber at the desired temperature. The developed apparatus has numerous advantages: its design is technologically relatively simple and it can be easily carried around. This makes it highly relevant for instance for applications requiring onsite production of TEG, such as oil spill remediation. The aspect of the TEG obtained with the apparatus and used for the samples is depicted on Figure 2, as well as the microstructure of the TEG obtained by scanning electron microscope.

Figure 1. View of the apparatus for direct production of TEG at a rate of 1.5 kg/h. 1: Combustion chamber. 2: Container for graphite supply to the combustion chamber. 3: Raw graphite transport system. 4: Gas flow rate control valve. 5: TEG outlet. 6: Gas bottle. 7: Power supply.

Figure 2. Aspect of the TEG produced with the experimental device: macroscopic view (left), microstructure (middle) obtained by scanning electron microscope; pure PTFE (right).

## **2.2.Preparation of PTFE/TEG nanocomposites**

The PTFE/TEG nanocomposite samples were prepared by manual milling. An analytical balance was used to weight the PTFE and TEG, which were then sieved and mixed together

by vigorous manual milling in a mortar during approximately one hour. Three different mass fractions of TEG were considered for the samples: 2.5wt %, 5wt %, and 10 wt%. These values were chosen such as to obtain a composite material in the percolation threshold. In addition, two different thermal pre-treatment were considered, *ie.* milling at ambient temperature (around 23°C), and milling at a temperature of 160°C . For the samples milled at a temperature of 160°C, the PTFE and TEG were first mixed in a heated crucible until the mixture reached the temperature of 160°C (sample temperature measured by IR thermometry). Then, the mixture was milled in a mortar in a similar manner than for the samples obtained at ambient temperature. Thus, a total of 6 samples were obtained and characterized. The aspect of the PTFE used for the samples is depicted on Figure 2.

### **2.3.Characterization methods**

The following methods have been used in order to study the morphological and thermal properties of the samples. X-ray diffraction (XRD) of the composites was carried out using a XPERT-PRO diffractometer system, with CuK $\alpha$  radiation at 45kV and 40mA in order to assess the structural properties of the PTFE/TEG nanocomposites. In particular, XRD allowed to follow the impact of the addition of TEG on the crystallinity of the obtained nanocomposite material. The measurements were carried out at a temperature of 25°C, with a copper anode. The sample was examined within the interval  $2\theta = (20\dots70)^\circ$ . The Fourier Transform Infra-Red (FT-IR) spectroscopy measurements were conducted using a Jasco FT/130 IR-6300 (Jasco Analytical Instruments, Easton, MD, USA) instrument to further study the structure of the material. The investigation was carried out in the frequency range of  $(600\dots4000) \text{ cm}^{-1}$ , with a resolution of  $4\text{cm}^{-1}$ . The scanning speed was 2mm /sec. The ATR method was used, with a TGS detector. Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) measurements were performed in order to study the thermal properties of

the samples. They were performed simultaneously for all samples, with a Jupiter STA 449 F3 calorimeter by NETZSCH (NETZSCH, Selb, Germany). An Al<sub>2</sub>O<sub>3</sub> crucible was used. The samples were studied in the temperature range (24...320)°C, at a heating rate of 10K/min. All TGA-DSC measurements were carried out under an Argon atmosphere with flow rate of 20.0ml/min. The mass of the studied samples were measured as follows - for the samples milled at T=23°C: 33.95mg (10wt % TEG) 29.10mg (5wt % TEG) 35.86 (2.5wt % TEG). For the samples milled at T=160°C: 25.10mg (10wt % TEG) 30.05mg (5wt % TEG) 27.22mg (2.5wt % TEG).

### **3. Results and discussion**

#### **3.1. Structural characterization**

##### **3.1.1. XRD**

The structural properties of the PTFE/TEG nanocomposite was first investigated using XRD. The XRD pattern of the PTFE/TEG samples is shown on Figure 3, while the corresponding measured values are presented in Table 1 and Table 2. Two broad peaks can be observed at 2 $\theta$  around 26.5° and 54.6°. These peaks correspond to the (002) and (004) peaks, which are respectively the X-ray diffraction of carbon in the PTFE matrix, and also in TEG. The XRD patterns reveal that a high dispersion of the TEG nanofillers within the PTFE matrix was achieved. In fact, the peak at 2 $\theta$  around 54.6° (004) is barely noticeable and its relative intensity is low for all samples (see Table 1 and Table 2). This behavior gives an indication that milling allows an appropriate dispersion of TEG, and is thus an acceptable method for the preparation of PTFE/TEG nanocomposites. The effect of milling temperature on the TEG dispersion is noticeable as well. For a given TEG concentration, the relative intensity of the (004) peak diminished when milling at T=160°C compared to milling at T=23°C; see Table 1 and Table 2. This trend was observed for all samples. Thus, increasing the milling

temperature improved the dispersion of TEG within the PTFE matrix. It can also be observed from the intensity of the (002) peaks that the crystallinity rate was enhanced when the TEG concentration increased, and that it was particularly high for the 10% TEG sample. Furthermore, it is possible to determine the size of the crystals which compose the sample by applying the Scherrer relation to the different X-ray diffraction peaks [39]. The width of the (002) peaks is quite important for all samples, which is an indication of the presence of crystals of small size in the samples (here in the range of 47 to 55 nm) according to Scherrer's equation [39], see Table 1 and Table 2.

Figure 3. XRD pattern of the PTFE/TEG nanocomposite at different percentage of TEG mass fraction for the samples milled at T=23°C (ambient temperature) and at T=160°C.

Table 1. XRD results for the samples milled at T=23°C

Table 2. XRD results for the samples milled at T=160°C

### 3.1.2. FTIR

The structure of the samples was further investigated using Fourier Transform Infra-Red spectroscopy. The obtained results are presented on Figure 4. The dominant IR bands obtained were 1123 cm<sup>-1</sup>, 966 cm<sup>-1</sup>, 1194 cm<sup>-1</sup>, 585 cm<sup>-1</sup>, 661 cm<sup>-1</sup>, 1283 cm<sup>-1</sup> and 2358 cm<sup>-1</sup>. Based on the general formula of PTFE:  $[-CF_2-CF_2-]_n$  and on the literature, the following assignments have been made: CF<sub>3</sub> symmetric stretching (1123 cm<sup>-1</sup>) [40], CH out-of-plane deformation of trans double bonds (966 cm<sup>-1</sup>) [37], CF<sub>2</sub> stretching (1194 cm<sup>-1</sup>) [40][42][43], CF stretching (1283 cm<sup>-1</sup>) [43], CF<sub>2</sub> wagging (585 and 661 cm<sup>-1</sup>) [43], and CO<sub>2</sub> (2358 cm<sup>-1</sup>) [44].

It can be observed that the intensity of the peaks increased with an increase of TEG concentration. Similar peaks were observed for the different TEG concentrations considered, at the exception of  $2358\text{ cm}^{-1}$  ( $\text{CO}_2$ ), which was absent for both the 2.5wt% and 5wt% TEG samples milled at ambient temperature, and which can certainly be assigned to atmospheric  $\text{CO}_2$ . When increasing the milling temperature to  $T=160^\circ\text{C}$ , the same peaks were observed but with increased intensity, except for the 2.5wt% TEG sample. Furthermore, low intensity IR bands appeared in the range ( $1500\text{-}1750\text{ cm}^{-1}$ ) and ( $3500\text{-}4000\text{ cm}^{-1}$ ) which were not present for the samples milled at ambient temperature. These peaks may correspond to byproducts created as a result of the increased milling temperature, and corresponding among others to  $\text{CH}_2$ ,  $\text{CH}_3$  deformation ( $1458\text{ cm}^{-1}$ ) [40]. This latter observation is congruent with the low fusion temperature of PTFE.

Figure 4. Fourier Transform Infra-Red spectroscopy of PTFE/TEG nanocomposites with different TEG mass fraction (2.5 wt%, 5 wt% and 10 wt%) for the samples obtained by milling at  $T=23^\circ\text{C}$  and  $T=160^\circ\text{C}$ .

### **3.2. Thermal characterization**

#### **3.2.1. DSC**

The effect of the inclusion of TEG on the thermal behavior of the nanocomposites during heating was examined by DSC. The DSC thermograms are presented on Figure 5, both for the samples milled at  $T=23^\circ\text{C}$  and at  $T=160^\circ\text{C}$ .

For the samples milled at ambient temperature, the curves corresponding to the different TEG loading display a similar behavior. From ambient temperature to  $100^\circ\text{C}$ , the DSC of the 5wt% TEG nanocomposite sample is superior to the two other samples. At about  $100^\circ\text{C}$ , a change of behavior occurs between the curves associated with the 5wt% TEG and 10wt% TEG samples.

From approximately 100°C onwards, the heat flux of the 10wt% TEG nanocomposite is significantly higher compared to those of the other two samples. The exothermal is particularly important with the increase of the concentration at 320°C. The  $I_{2.5}/I_{10}$  and  $I_5/I_{10}$  ratios are respectively equal to 30% and 70%. The 2.5wt% TEG sample displays a DSC inferior to the other two samples over the whole temperature range. Moreover, the concentration of TEG was found to impact the value of the glass-transition temperature. In fact, for the samples milled at  $T=23^\circ\text{C}$ , a glass-transition temperature of 195°C was observed for 2.5wt% and 205°C for 5wt% TEG, while it reached approximately 208°C for 10wt% TEG. A similar trend was observed for the samples milled at  $T=160^\circ\text{C}$ . Thus, the addition of TEG is responsible for an increase of the glass-transition temperature. This indicates that the degradation of the PTFE/TEG nanocomposite occurs at a relatively higher temperature with a greater TEG loading, and confirms the potential of TEG to improve the thermal properties of PTFE.

When the samples undergo milling at 160°C during one hour, the concentration of TEG induces as well an increase of the DSC curves and glass-transition temperature. However, a change of behavior is observed for the heat flux curves. In particular, a steep increase of the heat flux is observed for the 10wt% TEG sample above the curves of the other two samples, and the intersection between the curves corresponding to 5wt% and 10wt% which was observed at 100°C for the sample milled at ambient temperature totally disappeared. In this case, the  $I_{2.5}/I_{10}$  and  $I_5/I_{10}$  ratios hold the value of 7% and 50%, respectively. In addition, by comparing the DSC results for the samples milled at  $T=23^\circ\text{C}$  and at  $T=160^\circ\text{C}$ , it can be observed that a higher milling temperature seems to slightly reduce the glass-transition temperature, with values of approximately 190°C for 2.5wt% TEG, 195°C for 5wt% TEG and 200°C for 10wt% TEG. This result is an indication that milling at a lower temperature would be preferable in order to ensure the best resistance to heat for the TEG/PTFE nanocomposites.

Figure 5. Differential scanning calorimetric diagrams of PTFE/TEG nanocomposites with different TEG mass fraction (2.5 wt%, 5 wt% and 10 wt%) for the samples obtained by milling at  $T=23^{\circ}\text{C}$  and at  $T=160^{\circ}\text{C}$ . Heating rate of  $10^{\circ}\text{C}/\text{min}$ .

### 3.2.2. TGA

The degradation kinetics of the PTFE/TEG nanocomposites were then studied using Thermogravimetric Analysis. The mass reduction versus time obtained from the TGA measurements are presented on Figure 6. The highest temperature for which the samples were tested was  $320^{\circ}\text{C}$ . The reason for that is that total calcination of the sample occurred at a higher temperature, as found by the authors in a previous work [24]. In the latter work, a DSC analysis of carbon-nanotubes enriched PTFE displayed an intense peak around  $330^{\circ}\text{C}$ , and became then unusable. Thus, it was not possible to study the residual weight of the different samples in the present study.

For the PTFE/TEG samples obtained by milling at ambient temperature, a very low weight loss was observed for all samples (less than 2% in all cases), even up to a temperature of  $320^{\circ}\text{C}$ . For the sample with 2.5 wt% TEG, a plateau can be observed which indicates an almost constant sample weight. The measured weight loss increased along the TEG concentration for the other samples. For the PTFE/TEG samples milled at  $160^{\circ}\text{C}$ , the measured weight loss was similarly relatively low over the whole temperature range (less than 2.5% in all cases). Regarding the impact of TEG loading on the weight loss, the tendency is however inverted compared to the samples milled at ambient temperature. In fact, the curves obtained for 5 wt% and 10wt% TEG are practically identical; which is an indication that similar mechanisms are involved. However, for the sample containing 2.5 wt% TEG, a decreasing TGA curve can be observed over the whole temperature range, with a significant

decrease compared to the other two samples beginning from 100°C onwards. The higher thermal conductivity observed from the TGA measurements for the samples obtained by milling at T=160°C compared to the samples milled at T=23°C can be explained by the greater dispersion of the EG nanofiller inside of the polymer matrix which was already observed during the structural analysis. Thus, the impact of the TEG concentration on the thermal properties of the PTFE/TEG nanocomposite could be clearly observed as well as the influence of the temperature during milling. For the samples milled at a temperature of 160°C, the addition of TEG allowed to reduce the thermal degradation of the polymer nanocomposite. This phenomenon was further emphasized by an increase of the concentration of nanofiller. Finally, a concentration of 2.5 wt% TEG appeared to give the best thermal performance for the samples milled at ambient temperature.

Figure 6. Weight loss versus temperature for the PTFE/TEG nanocomposites (heating rate of 10°C/min) for 2.5 wt%, 5 wt % and 10 wt % TEG; samples obtained by milling at T=23°C and by milling at T=160°C.

#### **4. Conclusion**

A mobile apparatus was conceived for the production of Thermally-Expanded Graphite (TEG), and TEG-reinforced polytetrafluoroethylene (PTFE) nanocomposites were prepared by manual milling. The obtained samples were then characterized in terms of structural and thermal properties. The XRD results revealed that an acceptable dispersion of the TEG nanofiller inside of the polymer matrix was obtained, and that increasing the temperature during milling enhanced the TEG dispersion compared to milling at ambient temperature. Furthermore, the DSC measurements gave indications of a relationship between the glass-transition temperature and the concentration of TEG in the samples; an increased

concentration of TEG leading to a higher glass-transition temperature. However, a higher milling temperature seemed to slightly reduce the glass-transition temperature, which indicates that milling at a lower temperature would allow to ensure the best behavior regarding thermal degradation for the PTFE/TEG nanocomposites. The influence of the TEG loading on the thermal properties of the PTFE/TEG nanocomposite was further studied with TGA. The thermal performance was found to vary depending on the temperature during milling. For the samples milled at a temperature of 160°C, the addition of TEG allows to reduce the thermal degradation of the polymer nanocomposite, and even more so when the concentration of nanofiller increases. However, a concentration of 2.5 wt% TEG and milling at ambient temperature gave the best thermal performance for the samples. Thus, the milling temperature should be chosen accordingly to the desired application for the PTFE/TEG nanocomposites. The present work could be further extended in order to assess the potential of TEG to improve the properties of PTFE, for instance in terms of mechanical and electrical properties.

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