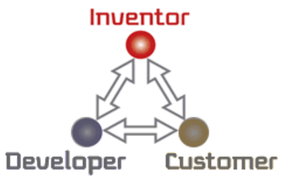
European Space Agency (ESA)

Innovation Triangle Initiative (ITI) – Proof of Concept A00012922



Design of anti-static ETFE based nanocomposites (COSMIC)

SUMMARY REPORT

Delivery date:

20 September 2013

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*Version :*01	*State :* Final	

CONTEXT

The principle of the ESA Innovation Triangle Initiative (ITI) “proof of concept” is to quickly test new products at the Lab scale in the context of space industry. The consortium is composed by an Inventor (CRP Henri Tudor), a Developer (CRP Henri Tudor) and a Customer (ThalèsAleniaSpace). ThalèsAleniaSpace is currently looking for a new generation of wires for satellite application. The main issue of the current wires, whose insulation is made of crosslinked ethylene tetrafluoroethylene (ETFE), is electrostatic charges that must be avoided. This is possible through the development of an antistatic ETFE material. To this end, one of the most relevant strategies is to disperse conductive nanofillers within ETFE matrix to create an electrostatically dissipative nanocomposite.

OBJECTIVES

The targeted bulk resistivity of the nanocomposites must be comprised between 1 x 10^{11} Ω.cm and 1 x 10^{12} Ω.cm in the temperature range -180°C to + 160°C. The objectives of the current project were to develop formulations of crosslinked antistatic ETFE-based nanocomposites with electrical resistivity fitting the targeted values. It is important to mention that a great attention was paid to the compatibilization between the ETFE matrix and the carbon-based filler. To this end, the latter were functionalized by chemical grafting with functions compatible with the chemical structure of ETFE. It was expected that modified nanofillers may reduce the electrical percolation threshold of ETFE compared to the use of as-received nanofillers. A multiphysical characterization and some first thermal durability testing were done to assess the properties of the new nanocomposites.

MAIN RESULTS

We found that the use of a certain reference of untreated carbon-based nanofiller enabled to reach the targeted ETFE electrical resistivity values of 10^{-11} ohm.cm – 10^{-12} ohm.cm for amount of filler comprised between 0.5 wt. %. and 1 wt. % (Figure 1). Unfortunately, the chemical treatment of the nanofillers did not enable to reduce the amount of required nanofiller to reach the targeted values of electrical resistivity.

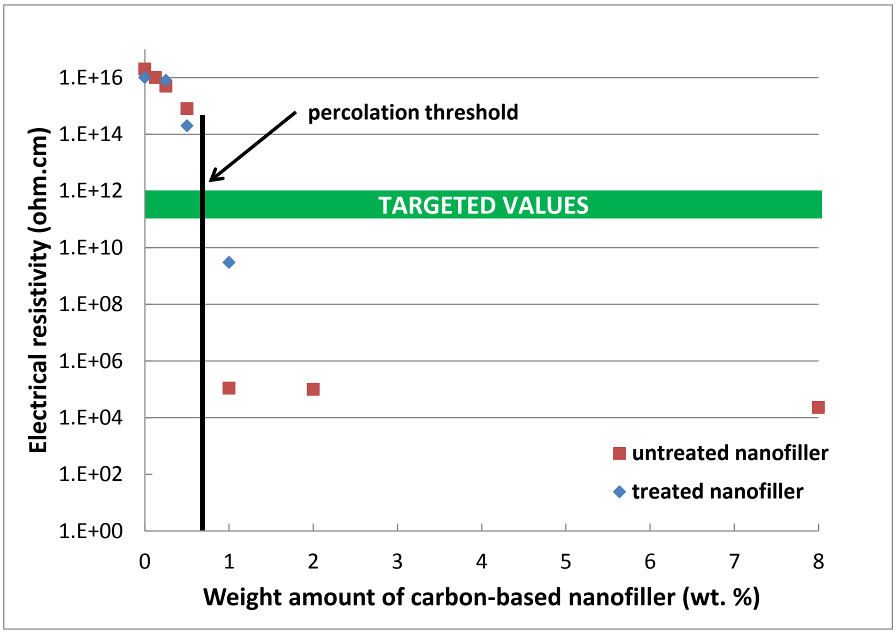

Figure 1: Impact of the weight amount of carbon-based nanofiller and of their chemical treatment on the electrical resistivity of ETFE nanocomposite

Concerning the multiphysical investigation of the materials, it was observed that the dispersion state of the nanofillers was not optimal due to the presence of aggregates. Nevertheless, using a transmission electron microscope (TEM) at high magnification, numerous areas with single nanofillers were noted explaining the low percolation threshold. As demonstrated by Fourier transform infrared spectroscopy (FTIR), irradiation-crosslinking induced the formation of the chemical bonds C=C (unsaturated molecules) et C=O (hydroperoxides transformed into carbonyls), the distribution of these chemical functions is homogeneous within the specimen section of 2 mm. These results demonstrate that irradiation-crosslinking leads to a homogeneous chemical structure from the skin to the core of the material having a thickness of 2 mm. As shown by wide-angle x-ray scattering (WAXS), the addition of carbon-based nanofiller to ETFE led to a decrease of the crystallinity index of ETFE. Irradiation crosslinking (followed stabilization by annealing) had no impact on the crystallinity for neat ETFE while that of the nanocomposite increased (Table 1).

Table 1: Impact of crosslinking (X) and addition of nanofiller on the crystallinity of ETFE measured by WAXS

Material	Xc (wt. %)
ETFE	29.5
X-ETFE	30.1
ETFE/nanofiller 99/1	21.1
X-ETFE/nanofiller 99/1	27.4

It was observed by dynamical mechanical analysis (DMA) that irradiation-crosslinking had no significant effect on the evolution of the elastic modulus of the materials with temperature, whatever the materials was. With increasing the content in nanofiller, the elastic modulus increased up to an amount of 0.5 wt.%, then the modulus decreased. Values of elastic modulus at -170°C, 25°C and 160°C are reported in Table 2.

Table 2: Elastic modulus of the materials obtained at 1 Hz by DMA

Temperature (°C)	Elastic modulus (MPa)					
	ETFE	X-ETFE	X-ETFE/ nanofiller 99.75/0.25	X-ETFE/ nanofiller 99.5/0.5	X-ETFE/ nanofiller 99/1	ETFE/ nanofiller 99/1
-170	4132	4067	3941	4884	4239	4099
25	1062	1071	1066	1252	1071	1140
160	160	180	205	287	185	329

The thermal stability of ETFE increased with the irradiation-crosslinking and further increased by combining irradiation-crosslinking and adding nanofiller, as indicated by the increase of the onset temperature by thermogravimetric analysis (TGA) (Figure 2).

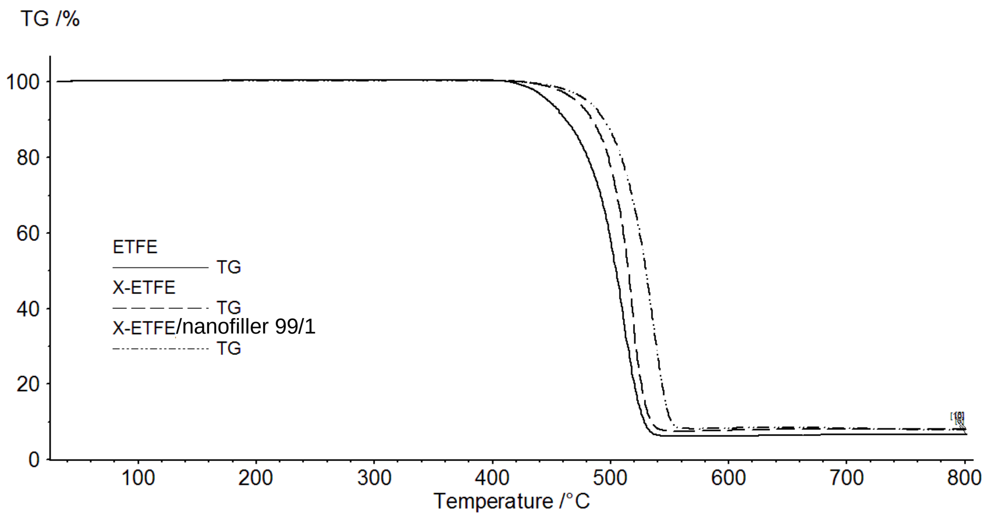

Figure 2: Thermogravimetric analysis of ETFE, crosslinked ETFE (X-ETFE) and ETFE nanocomposite (with composition 99 wt.% ETFE / 1 wt.% carbon-based filler)

The first thermal cycling from -60°C to 150°C revealed that after 100 cycles, no significant modification of colour, shape, crystallinity was noted, while surprisingly thermal stability of ETFE and crosslinked ETFE increased while that of the crosslinked nanocomposites was not modified.

CONCLUSIONS AND PERSPECTIVES

The use of untreated carbon-based nanofiller enabled to reach the targeted ETFE electrical resistivity values of 10^{-11} ohm.cm – 10^{-12} ohm.cm for amount of filler comprised between 0.5 wt. %. and 1 wt. %.

The chemical treatment of the nanofiller did not enable to decrease the required amount of nanofiller to reach an antistatic behaviour. The characterization step of the project revealed that the dispersion state of the nanofiller was not optimal due to the presence of aggregates. The chemical, thermal and mechanical properties of the materials were measured with attention focused on the impact of irradiation-crosslinking and of the addition of nanofiller on the properties of ETFE. The first thermal cycling from -60°C to 150°C revealed no or little impact of the thermal cycling on the material properties after 100 cycles.

The next step of this project can rely on an optimization of the chemical treatment methodology of the carbon-based nanofiller and on an optimization of the dispersion state of the latter to reduce the electrical percolation threshold. Furthermore, the scale-up and industrial implementation of our lab-developed formulation and processing methodology may be investigated. In particular, some wire prototypes could be fabricated and tested based on wire specifications and space specifications in terms of thermal resistance, atomic oxygen resistance, and UV irradiation resistance.