Nanofilled EPDM composite for aerospace applications

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Abstract: *O-rings are designed to be seated in a groove and compressed during assembly between two or more parts, creating a seal at the interface. The joint may be static, or (in a few circumstances) may have relative motion between parts and O-ring (rotating pump shafts and hydraulic cylinders, for example). Starting from the functionality of O-rings, the component materials can be described. Synthetic rubber, EPDM (ethylene-propylene diene monomer) is one of the most popular, versatile and long-lasting elastomeric material that is suitable for a wide range of applications, such as those in the aerospace domain, presenting a good behavior in extreme temperatures and high ozone resistance. The paper presents an experimental study using EPDM − Nordel 4760, butylic rubber (IIR)− Butyl 268 and clorobutylic rubber (Cl-IIR) − Chlorobutyl HT 1066 and a nanometric agent. As the targeted application of these materials is a specific one for extreme temperatures conditions, a testing matrix was developed, aimed to qualify their technical performance. The mechanical resistance of elastomers recommends the quality of its nanomodified version through superior performance. The structure of the analyzed materials proved to be unaltered when subjected to low temperatures, exhibiting good mechanical performance suitable for the targeted application.*

Key Words: *O-ring, aerospace, mechanical testing, nanosilica*

1. INTRODUCTION

Elastomers are a class of materials with quite distinct properties from all other solid materials. They are highly elastic − capable of being stretched many times their original length, and upon release, quickly revert to their original state. Their ability to significantly deform, and hence conform to the geometries of adjacent surfaces, makes them ideal for use in seals, sealants, gaskets, and shock absorbing applications [1].

The EPDM rubber is a type of elastomer characterized by a good isolation and a good thermal resistivity and environmental stability. This elastomeric material is an M-class rubber, distinct by chemical formula which is ethylene, propylene and non-conjugated diene terpolymer [2]. In addition to the above listed features, EPDM has excellent heat resistance, oxygen tolerance, weather fastness and anti-aging properties, as well as good chemical resistance, electrical insulation, low temperature performance, thus being the perfect candidate for aerospace application [2]. In recent years, the EPDM rubber became increasingly attractive for applications in the space field due to its low prices, great mechanical characteristics and physical properties [2].

However, in order to obtain certain physical and mechanical properties of EPDM based products for commercial application, this elastomer often needs to be modified by adding different filler agents into its composition.

Studies in the last years analyzed the effect of fillers [3], the most used ones being silica [4-6], and carbon black [7, 8].

Making a comparison between the two, silica yields a higher resistance and better wet grip combination with a lower rolling resistance than the carbon black [9], especially when used with compatibilizing agents [10, 11].

It is a great challenge to achieve the sealing interface between two pressurize modules of Low Impact Docking System (LIDS) developed by National Aeronautics and Space Administration (NASA). This is due to the androgynous nature of LIDS.

The synergistic effects of atomic oxygen (AO), ultraviolet and particle radiation, together with debris are the most commune problems along with operating environment (which includes the temperature range) to which the seal is expected to be exposed when performing docking/undocking operations [12].

The base condition for an O-ring seal and gasket with application to the launch vehicle must be able to withstand and dampen the shock and vibration loads placed on assemblies. The service life of launch vehicle is short but critical.

They must perform as specified for the several minutes of powered ascent, until they are jettisoned along with their booster [1].

The current paper describes the obtaining and characterization of EPDM based materials modified with nanosilica.

The results indicate that the nanomodified materials exhibit superior mechanical performance suitable for the aimed application, while maintaining an unaltered structure after subjection to low temperatures.

2. EXPERIMENTAL PROCEDURES

2.1 Materials

The experimental program consisted of creating a test matrix that covers the need to identify the mechanical characteristics, the fragility, the structural changes and the morphology of the materials used in the study. The component materials used to obtain the samples are described in Table 1.

Table 1. The properties of the component materials used to obtain the samples in the study

Component name	**Component properties**
EPDM rubber − EPDM Nordel 4760 (Dow Chemical Company)	− Mooney viscosity 70 ML_{1+4} at 120°C − 70% ethylene and 4.9 wt %5-ethylidenenorbornene (ENB) − 0.88 g/cm^3 density − 10% cristallinity − 170,000 molecular mass
Butyl rubber (IIR) − Butyl 268	− Mooney viscosity 51 ML_{1+8} at 125°C − 1.70 mol% unsaturation grade

Clorobuthylic rubber − Chlorobutyl HT 1066	− Mooney viscosity 38 ML_{1+8} at 125°C − 1.26 mol% unsaturation grade
Trimethylolpropanetrimethacrylate − Luvomaxx TMPT DL 75 (TMPT)	− melting point -25°C − boiling point: 200°C − 22% percentage of ash − pH 9.2 − 1.36 g/cm^3 density − 75% active ingredients
Dibenzoyl peroxide Perkadox 14-40B (Akzo Nobel Functional Chemicals)	− 1.60 g/cm^3 density − 3.8% active oxygen content − 40% peroxide content − pH 7
Antioxidant agent − Pentaeritritol tetrakis (3-(3,5-di-tert-butil-4-hidroxyfenil) propionate Irganox 1010	− melting point 40°C − 98% active ingredients
Nanofiller	− Nanometric batch with specific surface of 350-410 m^2/g − SiO_2 content higher than 99.8%

2.2 Processing and testing methods

The mixture was obtained by machining in a Brabender internal mixer and producing semi-finished products in the form of rubber blends. The experimental parameters nanoelastomers were: rotation speed of 10/150 rotations/ min, working temperature ranging from 110 to 190°C, mixing time of about 10 min.

The tests were carried out on samples obtained in a mixer, through a process that consisted of combining different raw materials into a homogeneous paste. The specimens' specific dimensions are based on the type of test.

Tensile strength and tearing strength tests were carried out with a Schopper strength tester with testing speed of 460 mm/min, using dumb-bell shaped specimens according to ISO 37/2012, and angular test pieces (Type II) according to EN 12771/2003, respectively. The elastomer nanocomposites morphological characteristics were investigated by SEM using a QUANTA 250 FEI scanning electron microscope equipped with an EDS module, in low vacuum conditions.

For low temperature fragility tests the climatic chamber used was IV Ilka STBV-1000 type. For the fragility evaluation the elastomer samples were tested at low temperature (temperature range of −200ºC ÷ +200ºC, associated with depressurization, pressure of 1.5 Torr in 30 minutes) after which they were tested to impact, to observe deformable behavior. The morphology structure was investigated by an optical microscope MEIJI 8520, 40X and 100X. The obtained samples had the following formulations:

Table 2. The different formulations of the obtained samples

Component	**Control sample**	**E-B**	**E-Cl-B**	**E- S**	**E-B-S**	**E-Cl-B-S**
EPDM Nordel 4760	100	95	95	100	95	95
Buthyl	-	5		-	5	-
Cl-Buthyl	-		5	-	-	5
TMPT	3	3	3	3	3	3
Irganox 1010	0.5	0.5	0.1	0.5	0.5	0.5
Nano-SiO_2	-			2	2	2

3. RESULTS AND DISCUSSIONS

The evaluation of the properties of composite materials is generally different from that of metals. Depending on the nature of the material and the type of application, a test plan is designed to assess their characteristics according to specific standards [4].

3.1 Mechanical tests

The elastomeric composites with different formulations were mechanically studied at room temperature according to SR ISO 37/1997. From the mechanical measurements for elongation at break, in the case of EPDM samples mixed with nanosilica, it results that in the case of E-Cl-B there is a decrease by 17% compared to the control sample and in the case of E-Cl-B-S samples it decreases by 14% (Fig. 2) [13].

The breakage strength for E-Cl-B samples drops by 45%, and in the case of E-Cl-B-S it decreases by 54%. EPDM-based blends mechanical results for butyl rubber and nanosilicate are presented in the following histograms.

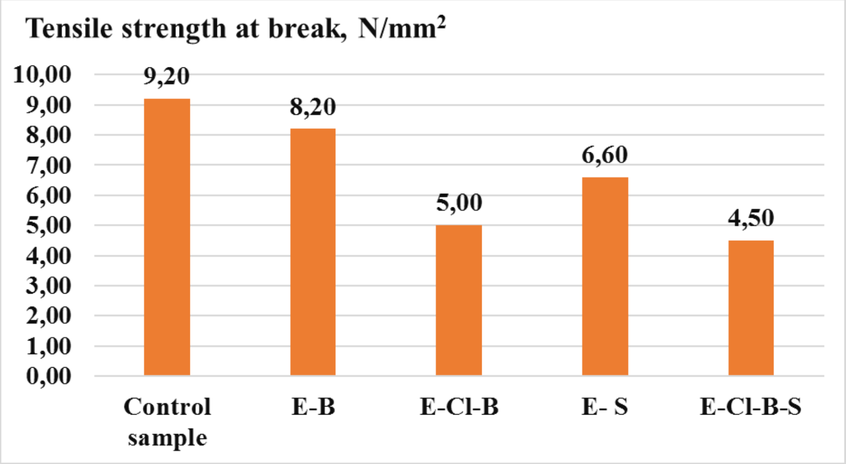

Fig. 1 – The elastomeric composites tensile strength at break

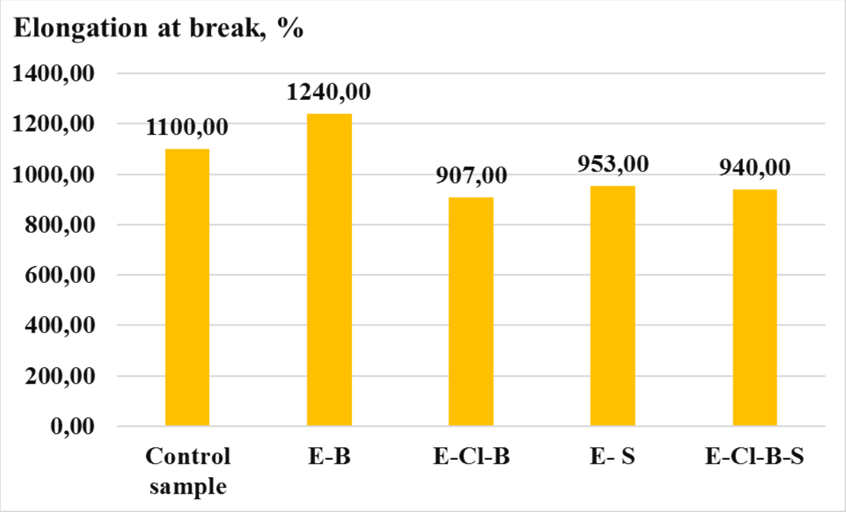

Fig. 2 – The elastomeric composites elongation at break

3.2 Fragility tests

Fragility tests were intended to identify the lowest temperature at which the elastomeric product showed no sign of fragility when subjected to a shock test under given condition. The fragility tests were performed according to STAS 8204-73 (Fig. 3 and Fig. 4). The samples presented a good behaviour at the macroscopic level and showed no sign of fragility at when tested for the chosen temperature.

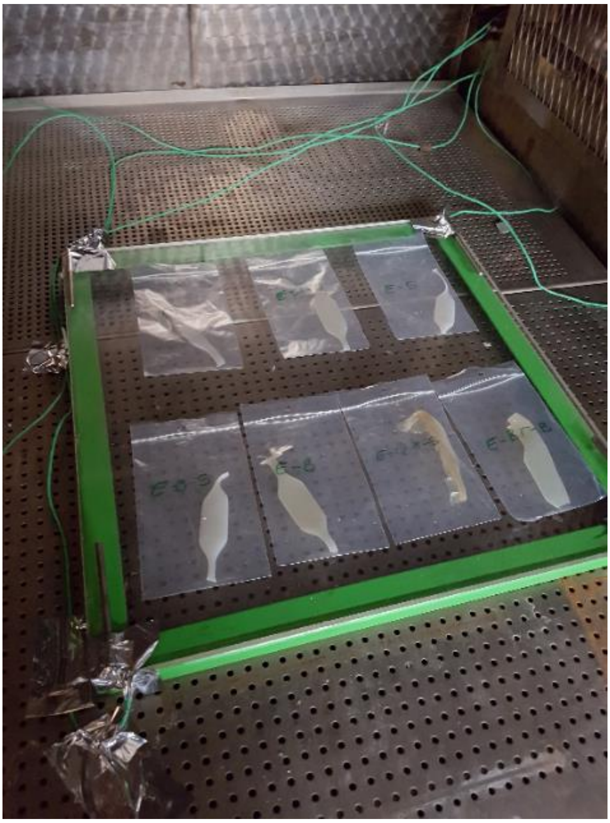

Fig. 3 – The samples batch with mounted thermocouples, prepared for testing at -70°C

Fig. 4 – The samples during testing at -70°C

3.3. Optical microscopy

For a more precise analysis optical investigation was performed to highlight the morphological changes after an impact test at cryogenic temperature. It can be noticed that there are no significant changes in the morphology structure.

Fig. 5 reveals relatively uniform surfaces with a porous morphology in the case of E-Cl-B samples presented in Fig. 6.

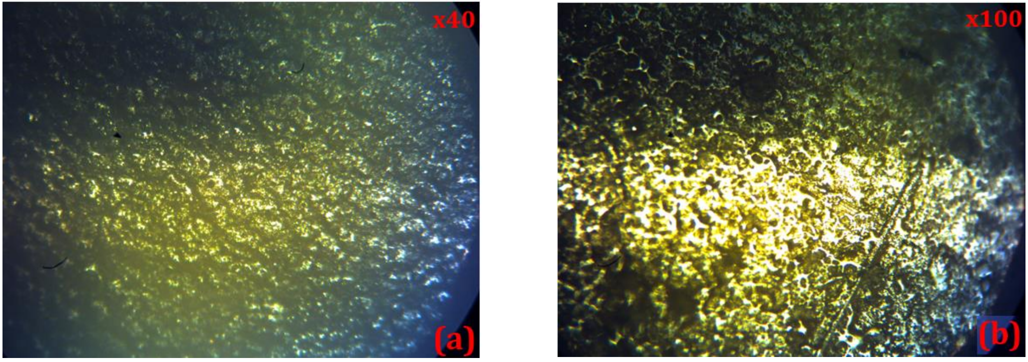

Fig. 5 − Optical images of Control sample: (a) before freezing at -70°C, (b) after freezing and impact test

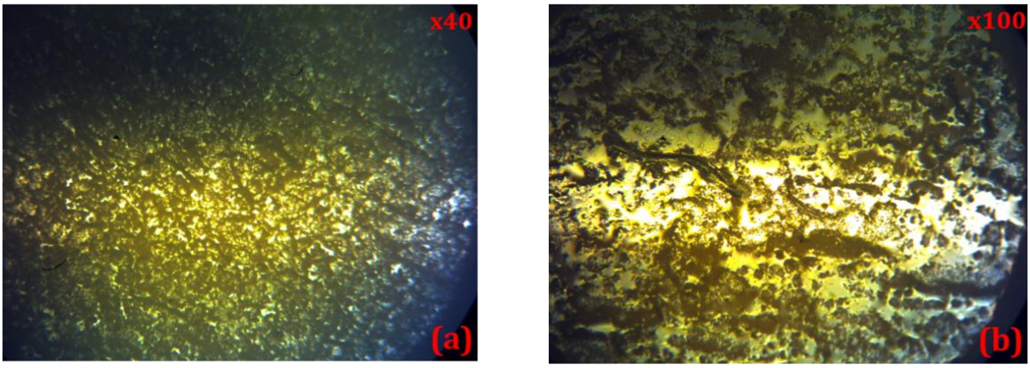

Fig. 6 – Optical images of E-Cl-B sample: (a) before freezing at -70°C, (b) after freezing and impact test

3.4 Scanning electron microscopy (SEM analysis)

SEM analysis was performed on the samples in order to evaluate the morphology structure and homogeneity. Fig. 7 presents the image of E-Cl-B-S sample at different magnification levels in which the specific morphology of elastomers can be observed, highlighting on the random edges of the analysed surface.

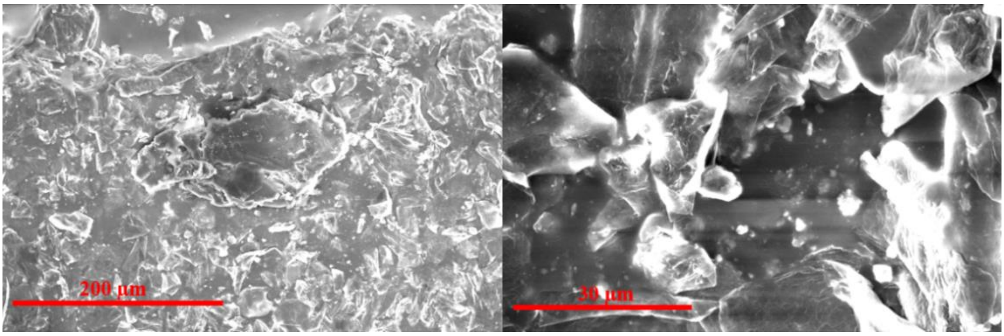

Fig. 7 – Micrographs of E-Cl-B-S sample at different magnification level

4. CONCLUSIONS

In this paper, different formulation elastomeric materials were analysed from mechanical point of view, at cryogenic temperature. After testing the samples was morphologically and structurally investigated.

The lab-scale tests were designed to define seal nanomaterials, determine the mechanical characteristics and evaluate their behaviour when exposed to cryogenic temperature.

The study found that EPDM elastomers with nanosilica powder addition can improve their mechanical performance at specific temperatures.

The study indicates the preliminary experimental results according to the test standards to verify the improvement generated by nanometric agents in the elastomeric matrix. Corroborating the data from the preliminary tests obtained in this stage, it can be concluded that nanosilica filled elastomeric materials have good low temperature behavior without signs of fragility at this temperature. Samples still show elastic behavior after cryogenization without morphological changes after impact.

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