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# Electron beam irradiation effects on the mechanical, thermal and surface properties of a fluoroelastomer

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#### Abstract

Fluoroelastomer can be used as a sealing material for different purposes. The aim of this work is the evaluation of the effects of the ionizing radiation of an electron beam (EB) on the mechanical, thermal and surface properties of a commercial fluoroelastomer containing carbon black and inorganic fillers. The material was irradiated with overall doses between 10 and 250 kGy. Tensile strength (stress and strain at break), hardness (Shore A) and compression set were evaluated. Thermal behavior was evaluated by thermogravimetric analysis and differential scanning calorimetry. Surface modifications were inspected using scanning electron microscopy (SEM) and optical microscopy. The experiments have shown that EB irradiation promotes beneficial changes in the fluoroelastomer tensile strength behavior while compression set remain constant and the glass transition temperature increases. The SEM micrographs have shown compactness in the irradiated samples, although optical observations showed no surface morphology changes. © 2007 Elsevier B.V. All rights reserved.

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#### 1. Introduction

Elastomers are specified in a variety of industrial, automotive and aerospace applications where large volumes of high quality seals, tubes and hoses are needed to contain or transport a variety of gases and liquids [1]. Commercial fluoroelastomers were introduced in 1957 to meet the needs for high-performance sealing materials. Since then, the use of fluoroelastomers has spread out for many other applications, mainly due to their excellent properties such as resistance to high temperatures, resistance to the attack of chemical substances including oils, fuels and mineral acids, and low permeability to many substances [2].

A typical composition of fluoroelastomers includes, besides the polymer, a curing or cross-linking agent, metal oxides, fillers, processing aids and other additives. These additives are incorporated in order to assure good processing characteristics and specific properties [3].

As in case of many other polymeric materials, ionizing radiation has a variety of effects on fluoropolymers. It may cross-link them, cause chain scission or modify their surface structure [4]. Quite often, these effects occur simultaneously, and the final result will depend on the material chemical structure, type of radiation, dose rate and total absorbed dose. In general, compounds from fluoroelastomers irradiated at optimum conditions attain better mechanical properties and thermal stability than non-irradiated

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chemical cured systems [5]. There are also studies showing the influence of polyfunctional monomers on the structural changes of fluoroelastomers induced by electron beam (EB) [6].

The aim of this work was to evaluate the effect of the ionizing radiation of EB on the mechanical, thermal and surface properties of a fluoroelastomer sealing material, which was previously obtained by a conventional curing process.

#### 2. Experimental

#### 2.1. Samples

The fluoroelastomer studied in this work was a commercial product obtained from two monomers, vinylidene and hexafluoropropylene, containing also specified percentages of polymeric material, carbon black and inorganic fillers such as magnesium oxide and calcium hydroxide. Mechanical and thermal experiments were carried out with specific samples obtained from molded plates (150 mm × 150 mm). Surface investigations were carried out on the O-ring samples (internal diameter of 18.4 mm; cross-section area of  $5.3 \text{ mm}^2$ ).

#### 2.2. EB irradiation conditions

Samples were irradiated with high-energy electron beam at the IPEN-CTR facilities using a model JOB-188 Industrial Dynamitron Electron Beam Accelerator of 37.5 kW (1.5 MeV-25 mA). The irradiation processes were carried out at a dose rate of  $11.2 \text{ kGy s}^{-1}$ , and the overall applied doses were 10, 25, 50, 75, 100, 125, 150, 175, 200 and 250 kGy.

#### 2.3. Solubility tests

The solubility in acetone of non-irradiated and irradiated samples was evaluated. The samples were weighed before and after remaining 24 h immersed in the solvent. Before each weighing step, the samples were adequately dried.

#### 2.4. Thermal analysis

TG/DTG curves were obtained using a Thermogravimetric Analyzer TGA7 (Perkin Elmer, Inc.) in the temperature range from 50 °C to 900 °C with a heating rate of 10 °C min<sup>-1</sup>, under a dynamic nitrogen atmosphere in the temperature range from 50 °C to 650 °C, and under synthetic air atmosphere from 650 °C to 900 °C, using samples of about 5 mg weight. The amounts of fluoroelastomer, carbon black and fillers were determined according to ASTM D6370-99. DSC curves were carried out using a DSC-50 differential scanning calorimeter (Shimadzu Corp. Japan) in the temperature range from -40 °C to 80 °C at a heating rate of 10 °C min<sup>-1</sup>, under dynamic nitrogen atmosphere using samples of about 20 mg weight.

#### 2.5. Mechanical tests

Tensile strength measurements were carried out in an Instron Universal testing machine model 5565 in accordance to ASTM D1414-78. Hardness was evaluated using a Type A durometer Woltest model SD300 according to ASMT D 2240-86. Compression set measurements were done in an appropriate device for compression set tests under constant deflection according to Method B in ASTM D 395-85.

### 2.6. Scanning electron microscopy (SEM) and optical microscopy

SEM micrographs of the surfaces from fractured samples were obtained using a JXA-6400 model scanning electron microscope (JEOL). Optical images of non-irradiated and irradiated O-rings were registered using a Reichert-Jung POLYVAR MET light optical microscope, attached to a CCD Color Camera model KC-512NTX (KODO).

#### 3. Results and discussion

The results obtained in the solubility tests are shown in Fig. 1. The data indicate that there is a progressive decrease in the solubility as a function of the applied radiation dose. This is in agreement with the fact that higher radiation doses increase the cross-linking degree of polymeric materials and consequently the sample solubility decreases.

Representative curves of thermal behavior of non-irradiated and irradiated samples evaluated by means of TG and DSC are shown in Fig. 2. The TG experimental data allow verification that the irradiation process, in the range of applied doses, does not affect the composition of the samples. Therefore, the amount of fluoroelastomer and carbon

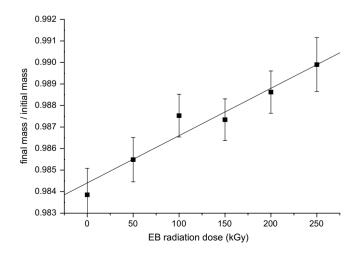


Fig. 1. Final and initial mass ratio from the solubility test as a function of the applied dose.

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