Fracture behaviour and thermo-oxidative ageing of EPDM

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ABSTRACT

The objective of this study is to understand the ageing of the insulating elastomer coating electric cables. Accelerated thermal ageing have been realized on EPDM filled with ATH.
Experimental characterizations reveal details of the oxidation process due to the ageing, which leads to the rupture of the network chains. This phenomenon provokes the formation of two phases inside the EPDM: the network of active chains and the soluble fraction.
The decrease of the density of active chains and the increase of the free chains quantity lead in turn to a loss of stiffness of the material, and to a decrease of the failure and fracture properties.

KEYWORDS

Ageing, EPDM, crack propagation, thermo-oxidation, soluble fraction, fracture, ATH

INTRODUCTION

Insulating materials of electric cables in nuclear power plants are constituted of rubbers having good ageing resistance properties, like ethylene and propylene copolymers (EPR, EPDM …). In service, these cables are submitted to low irradiation and ambient temperatures that could reach 50°C in the reactor building.
In the presence of oxygen, the main ageing process is the oxidation, which induces modification of the chemical and, macromolecular structure of the elastomeric material, the main consequences of which are the rupture of the network chains or the creation of new crosslink nodes.
These ageing mechanisms lead to the modification of the mechanical behaviour and of the physical and chemical properties of the material. The characterization of the ageing is based on the measurements of the evolution of different parameters, and especially the tensile strain at break used to estimate the life time of cables.

The prediction of the life time and the non destructive control of cables properties by innovative methods are the focus of numerous experimental and numerical studies at EDF R&D. The objective of this contribution is to study the thermal degradation mechanisms of model material, composed of EPDM, by characterizing the evolution of the network structure during the ageing and to link this evolution to the tensile failure and crack propagation properties of the material.
To achieve this goal an accelerated thermal ageing program has been applied to a model material, constituted by an EPDM matrix filled with aluminum trihydrate fillers (ATH). Then the ageing has been characterized by physical, chemical and mechanical measurements, to investigate the degradation mechanisms that occur at the different scales of the elastomeric material.

This extended abstract is organized as follows: section 2 details the composition of the model material of the study, the experimental techniques used and the ageing conditions. Section 3 presents the changes in chemistry due to ageing. The characterization of thermal degradation of the material structure is reported in section 4. And section 5 contains the evolution of the macroscopic properties during the ageing.

MATERIAL AND EXPERIMENTAL TECHNIQUES

Model material

The model material of this work is an EPDM matrix, composed of 50 mol% of ethylene, 45.1 mol% of propylene, and 4.9 mol% of norbornene (ENB). Its degree of cristallinity is inferior to 1 %. The material is filled with 100phr of aluminium trihydrate fillers (ATH). 3phr of peroxide have been used for the vulcanization process realized at 170°C during 20min. The material doesn't contain added antioxidant.

Accelerated ageing

The samples have been thermally aged in an air ventilated ovens at 130°C, during 5h, 10h, 20h and 30h. Specimens were suspended inside the oven to avoid direct contact with the surfaces of the furnace.

Swelling measurements

Swelling measurements have been done by immersing samples of 100 to 300 mg in xylene solvent during six days at 20°C, and drying them in vacuum at 40°C.
Swelling ratio and soluble fraction have been determined using the extension of the Flory-Rehner theory done by Kraus [1].

Monotonic tensile test

Monotonic tensile tests have been performed on dumbbell samples of 50mm gauge length, 4mm width and 0.9mm thick, at 50mm/min, on an Instron machine equipped with a 1kN cell force. The strain of the samples has been determined with the displacement measurement of the machine.

Infrared spectroscopy

Infrared spectroscopy measurements have been performed with a Bruker Tensor 27, equipped with a diamond lens, using 64 scans in ATR mode with a resolution of 4 cm^{-1}, in the wavelength range of 4000 cm^{-1} - 600cm^{-1}