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Mechanical behaviour characterizing and simulation of polyacrylate rubber

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Materials

ABSTRACT

Purpose: of this paper is to investigate the influence of EB radiation on the mechanical behaviour of UV curing polyacrylate rubber (ACM) and to simulate its behaviour.
Design/methodology/approach: The material was irradiated by two different EB doses, 100 kGy and 250 kGy, its mechanical behaviour was investigated with the help of uniaxial, equibiaxial and planar shear experiments. The results were applied to the Ogden's Model (1972) in order to obtain the parameters to simulate the material behaviour by finite element method (FEM) and to compare experimental and FEM curves. The structure molecular changes caused by EB were investigated with the help of infrared spectroscopy.
Findings: In most cases the experimental results showed an increase in the strength at rupture and a decrease in the elongation at the rupture with increasing of radiation dose. Equibiaxial and planar shear tests presented similar behaviour like uniaxial results, in terms of elongation decrease and strength increase, with some deviations. Ogden's Model third order provided simulated curves with similar behaviour in comparison to experimental curves. The infrared spectroscopy showed different chemical group contents in the analyzed regions, surface and middle region.
Research limitations/implications: Two doses of EB radiation were applied; higher or lower doses were not investigated.
Practical implications: Improved behaviour of UV curing ACM can extend the range of industrial applications, or improve its performance in known applications.
Originality/value: Usually EB radiation has been used to modify polymeric structure and to improve thermal and mechanical polymers behaviour. Regarding like rubber materials EB is usually applied as an alternative form of vulcanization. UV is a new type of curing for polyacrylate rubbers, which are usually cured by thermal processes.
Keywords: Engineering polymers, Polyacrylate, Electron Beam, Finite Element Method

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1. Introduction

Mechanical behaviour of elastomers depends strongly on crosslink density. The cross linking of rubbers and thermoplastic polymers is a well-proven process of the improvement of the thermal properties [1]. Many authors proposed the use of EB radiation as an alternative to improve polymer properties, like PP [1], PA [2] and PE [3]. Modification of thermoplastics and rubbery materials by electron beam (EB) is a potential method for developments of new polymers and composites [4]. Usually EB radiation is presented in many papers as an alternative to vulcanize rubbers, in presence of different polyfunctional polymers. Banik [5] showed in his work the effects of EB radiation on cross linked rubbers, fluocarbon (FKM), natural rubber (NR), nitrile rubber

(NBR) and ethylene propylene dyne monomer (EPDM). EB increased the cross linking level of the elastomers analysed by Banik, changing their mechanical behaviour.

This work has the purpose to investigate the effects of EB, as post cure method, on the mechanical behaviour of a new polyacrylate rubber (ACM), cured by UV radiation instead of conventional methods. Recently, a liquid acrylic rubber technology was developed, what contains an UV sensitive cure monomer. This new technology makes possible to apply the rubber, in a liquid form and to cure it after the application, using a traditional UV curing process, ideal for gasket applications.

Polyacrylate elastomers are classified by SAE J200/ASTM D2000 to be in the DH(150°C capable) and EH (175°C capable) categories. ACM based compounds are used in products that require excellent heat and oil resistance. This is appropriate as nearly 80% of the acrylic elastomers sold go into automotive components. Polyacrylate compounds can be formulated for applications ranging in temperatures from –40°C to 190°C [6]. UV curing ACM needs a specific UV wave length to start its cure process and depths of more than 20 mm can be totally cured in seconds.

Ionizing radiation, like ER radiation, interacts statistically with outer sphere electrons, no matter what compound in the system they belong to. Most of energy of absorbed EB radiation appears as single ionizing spurs, the remaining 20% of energy is located in large, multi-ionizing spurs due to low range of final degradation energy electrons [3]. The multi-ionizing spurs are responsible for chain scission in case of polymer degradation, when submitted to EB radiation. Some broken chains, caused by multi-ionizing spurs, cannot find partners for reaction and contribute to the population of degraded macromolecules [7]. In this paper different EB doses were applied to ACM, followed by mechanical tests, in order to verify the increase in the cross linking level or polymer degradation, through their mechanical behaviour after to be irradiated.

Experimental data were used to characterize the ACM mechanical behaviour and made possible to simulate the material behaviour using finite element methods (FEM). Experimental curves were compared to simulated curves, in uniaxial, equibiaxial and planar shear strains. The material behaviour was also simulated in compression.

The mechanical tests frequently performed on rubbers are of two types: compression and tension; while the compression state is always uniaxial, tension can be applied in a uniaxial, planar or equibiaxial state [8].

Rubber is unique in being soft, highly extensible, and high elastic [9]. Because of its molecular structure, rubbers exhibit a non linear mechanical behaviour, stress-strain relation, along its high strain range. The classical theory of elasticity can be used to describe accurately just small deformations, for large deformations other treatment for the relation stress-strain must be used.

Rivlin [10] developed a general treatment of stress-strain of rubber-like materials, assuming only that the material is isotropic in elastic behaviour in the unstrained state, and incompressible in bulk [9]. This treatment suggests that the strain measures can be given by three strain invariants:

$$I_1 = (\lambda_1)^2 + (\lambda_2)^2 + (\lambda_3)^2$$
$$I_2 = (\lambda_1)^2(\lambda_2)^2 + (\lambda_2)^2(\lambda_3)^2 + (\lambda_3)^2(\lambda_1)^2 \qquad (1)$$
$$I_3 = (\lambda_1)^2(\lambda_2)^2(\lambda_3)^2$$

Rivlin [10] proposed a strain energy density equation to describe the elastic behaviour for large deformations in function of I_1, I_2 and I_3, where the amount of energy stored elastically in unit volume of material under strain is specified by λ_1, λ_2 and λ_3. Because of the considered incompressibility of the material $I_3 = 1$, so that the strain energy density function W could be expressed in forms of I_3 and I_3. The general form of Rivlin's strain energy density function W is presented as the Equation 2.

$$W = \sum_{i,j=0}^{\infty} C_{ij} (I_1-3)^i (I_2-3)^j \qquad (2)$$

Other Models were developed after Rivlin's Model, like Gent [10], Arruda & Boyce [11] e Ogden [12]. Few Models like Ogden, Gent work well with multiple types of test data. The Ogden's Model, Equation 3, was used in this work to simulate the ACM behaviour under uniaxial, equibiaxial and planar shear deformations, in order to compare the results against the experimental data and under compression, in order to predict the material behaviour under such condition. The success of the Ogden-type strain-energy function, coupled with the difficulties which had been encountered in achieving comparable results with strain-energy functions of the Rivlin-type [13].

$$W(\lambda_1, \lambda_2, \lambda_3) = \sum_{p=1}^{N\infty} (\mu_p/\alpha_p)((\lambda_1)^{\alpha p} + (\lambda_2)^{\alpha p} + (\lambda_3)^{\alpha p} - 3) \qquad (3)$$

Uniaxial tests are very common for testing like-rubber materials. The main purpose is to provide a pure tensile strain, where $\lambda_1 = \lambda$, $\lambda_2 = \lambda_3 = (\lambda_1)^{1/2}$, to reach such conduction the length of the specimen must be much longer than the thickness and width, the material must be considered incompressible and isotropic, usually backbone specimens are used in this kind of test.

Equibiaxial tests generate a pure state of strain, where $\lambda_1 = \lambda_2 = \lambda$ and $\lambda_3 = 1/(\lambda_1)^2$, equivalent to uniaxial compression tests, but more accurate and using a more complex kind of experiment.

Planar shear test causes, because the material is nearly incompressible, a state of pure shear in the specimen at a 45 degree angle to the stretching direction [11], where $\lambda_1 = \lambda$, $\lambda_2 = 1$ and $\lambda_3 = (\lambda_1)^{-1}$. The most significant aspect of the specimen is that it is much shorter in the direction of stretching than the width. The objective is to create an experiment where the specimen is perfectly constrained in the lateral direction such that all specimens thinning occur in the thickness direction [14].

Infrared Spectroscopy is usually used to verify changes, or chemical group variation, in polymeric molecular structures, caused by EB radiation.

2. Experimental

2.1. Material

ACM rubber sheets, with dimensions of 150 mm x 150 mm x 2 mm, were obtained through low pressure injection process. The material, in liquid form, was injected at room temperature of 25±2°C into a silicone mould, which was exposed to UV radiation, according to supplier specification, promoting the material cure.

2.2. Specimens irradiation

The electron beam irradiation of the injected ACM sheets was carried out at room temperature of 25±2°C, at the Institute of Nuclear and Energetic Research in São Paulo, by an electron beam accelerator Model DC 1500 / 25 / 4 of the Radiation Dynamics, Inc. Doses of 100 kGy and 250 kGy were applied. The Table 1 shows EB accelerator and process specifications.

Table 1.
EB accelerator and irradiation process specification

EB accelerator specification	
Energy range	0.5 – 1.5 MeV
Current range	0.3 – 25 mA
Beam power	15 kW
Irradiation process specification	
Electron energy (selected)	1.013 MeV
Beam current (selected)	4.54 mA
Process	Conveyor
Conveyor speed	6.72 m / min
Dose rate	22.42 kGy / s
Dose by cycle under the beam	5 kGy

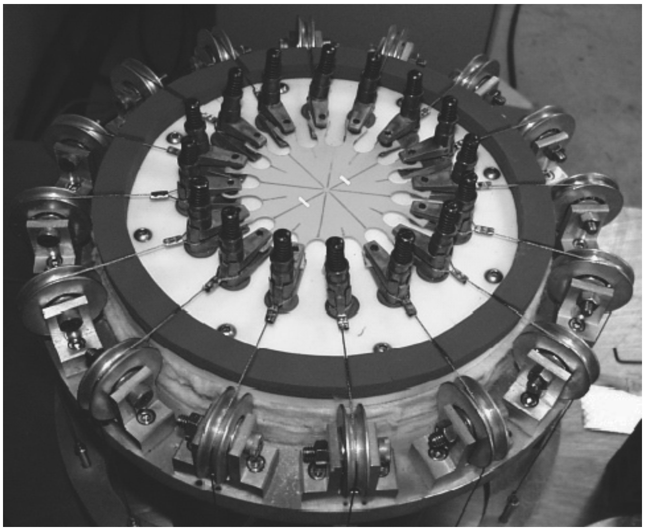

Fig. 1. Equibiaxial testing machine, Miller [14]

2.3. Mechanical behaviour

The polyacrylate mechanical behaviour, and the electron beam influence on it, was investigated using three types of mechanical experiments: uniaxial, equibiaxial and planar shear extensions. Three specimens of each material condition, 0 kGy, 100 kGy and 250 kGy, were tested according to the three experiment types.

Uniaxial extension specimens were obtained, from the inject sheets, according to ASTM Die-C. The tests were carried out using ASTM 412-6a method, in an Instron 5567 testing machine, equipped with a 5 kN load cell, at a crosshead speed of 500 mm/min, at room temperature of 25±2°C, using a contact extensometer for strain measurement.

The equibiaxial extension specimens were obtained and the tests were carried out by Axel Physical Testing Services, in Michigan, USA, according to method presented by Day and Miller [15]. The testing machine is shown in Figure 1, a crosshead speed of 30 mm/min was applied, at room temperature of 25±2°C. The force transmitted by the 16 grips to a common load plate was measured using a strain gage load cell, a laser non contacting extensometer was used to measure the strain.

The work of Day and Miller [14] was used as reference to define the planar shear specimen dimensions and test method. Flat ACM sheet, with dimensions of 150 mm x 30 mm x 2 mm, was stretched in uniaxial extension, at room temperature of 25±2°C , causing a pure shear strain. The initial distance between the grips was 10 mm, the by laser extensometer analysed an initial distance of 3,35 mm and the crosshead speed was 5 mm/min, Figure 2.

Uniaxial compression state was simulated considering friction coefficient of 0.8, room temperature of 25 °C, specimens with diameter of 13mm and 6.3 mm high, using ANSYS software. Uniaxial state simulation was carried out to define a friction coefficient applicable to compression state. The purpose was to verify a range which would not cause variations in the simulated curves under compression state.

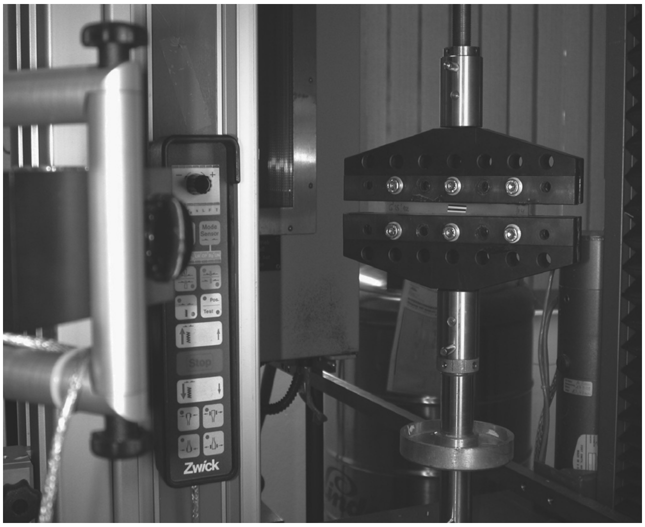

Fig. 2. Planar shear testing device

2.4. Infrared Spectroscopy

ATR spectra of the ACM sheets in the different conditions, without irradiation and with three irradiation doses, were taken using a Spectrum One FT-IR Perkin Elmer spectroscope, the ATR method (Attenuated Total Reflectance) was used. Two different regions of the samples were analyzed, surface and the middle region, in order to verify the chemical groups present in both regions. The spectra were obtained by absorbance, which offers a linear rate between the peaks and the functional group contents in the regions, according to the Lambert-Beer Law. The direct relation between peaks and functional groups contents makes possible to investigate the amount changes in chemical groups like >C=O, >C=C< and C-O-C.

3. Results and discussion

3.1. Uniaxial tests

The Table 2 shows the mean strength at break increase and a decrease of the mean elongation at break of the ACM. EB doses up to 250 kGy have not caused the degradation of the elastomer and consequent decreasing of mechanical properties, what could proceed in case of a high amount of chain scission.

Table 2.
Mean strength and elongation at break under uniaxial state

Dose (kGy)	Mean strength at Break (MPa)	Mean elong. at break (%)
0	4.80	191.63
100	4.93	156.46
250	5.09	120.06

The Figure 3 shows the comparison and respective increase of the modulus at 100% elongation with increasing the EB radiation dose. The elastic modulus is directly related to the elastomer cross linking level, indicating an increase of cross linking level with increasing the dose. The greater the cross linking level, the greater the cross linking level becomes [9].

The similarity between the three experimental curves, Figures 4, 5 and 6, obtained in each dose condition show a good experiment capability.

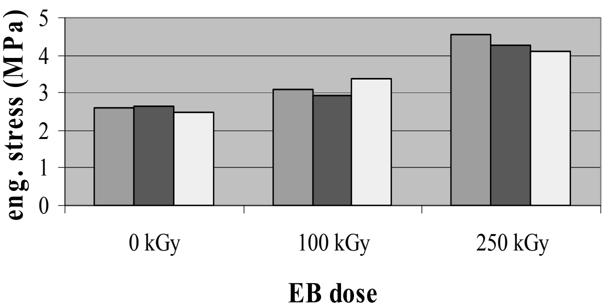

Fig. 3. Elastic modulus at 100% elongation

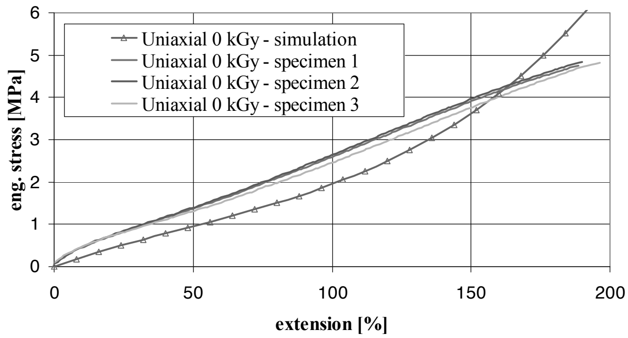

Fig. 4. Uniaxial curves of non irradiated samples - 0 kGy

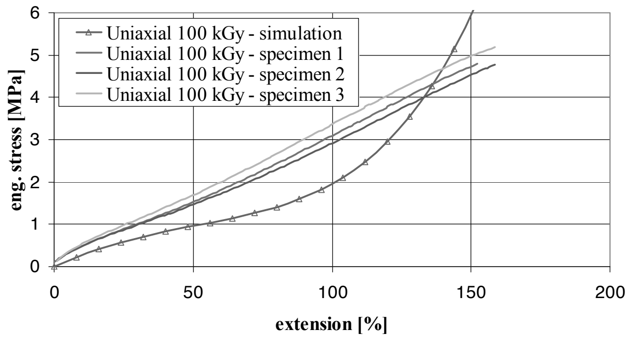

Fig. 5. Uniaxial curves of irradiated samples - 100 kGy

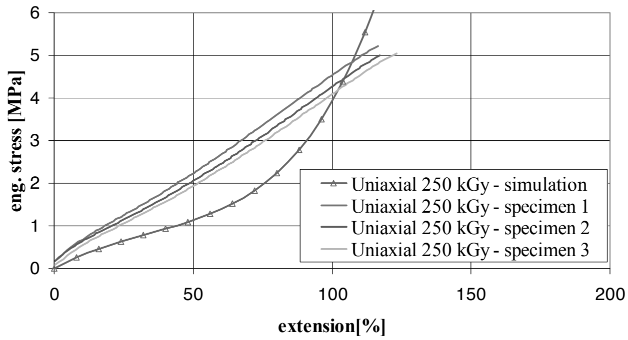

Fig. 6. Uniaxial curves of irradiated samples - 250 kGy

The simulated curve, in non irradiated condition, exhibits a nearly behaviour to the experimental data up to an elongation of around 150%. For higher radiation doses, the simulated curve presents a distanced behaviour from the experimental curves, indicating a softer simulated behaviour under pure tensile state in comparison to experimental data. An increase of cross linking level could explain the proximity to linear elastic behaviour in pure tensile state, contradicting the expected and verified with simulated non linear behaviour under such condition.

3.2. Equibiaxial tests

The Table 3 presents the mean strength and mean elongation at break for specimens tested under equibiaxial tension condition, showing an increase of the mean strength at break and a decreasing of the elongation at break with increasing the EB radiation dose, confirming the elastic modulus increase shown through the uniaxial results with increasing the irradiation dose.

Experimental curves of non irradiated specimens, Figure 7, exhibit a closer behaviour to the simulated data, emphasizing the experiment capability and good applicability of the Ogden's Model to the non irradiation condition.

After irridating the specimens with 100 kGy their experimental curves kept the identified closer behaviour and similarity to simulated curve, as showed in Figure 8, for extension rates up to 30%. In this condition, the simulated curve exhibits a distance from the other curves in the region where the specimens failed in the tests.

Regarding 250 kGy condition, one of the equibiaxial specimens developed a more brittle behaviour, influencing on the mean curve, used to obtain the Ogden parameters and consequently on the simulated curve.

Table 3.
Mean strength and elongation at break under equibiaxial state

Dose (kGy)	Mean strength at Break (MPa)	Mean elong. at break (%)
0	1.05	48.29
100	1.14	37.87
250	1.30	29.25

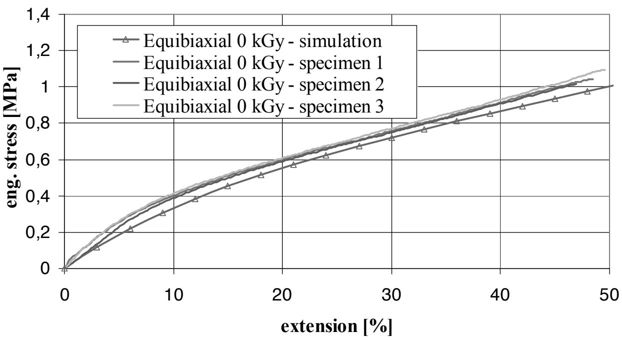

Fig. 7. Equibiaxial curves of non irradiated samples - 0 kGy

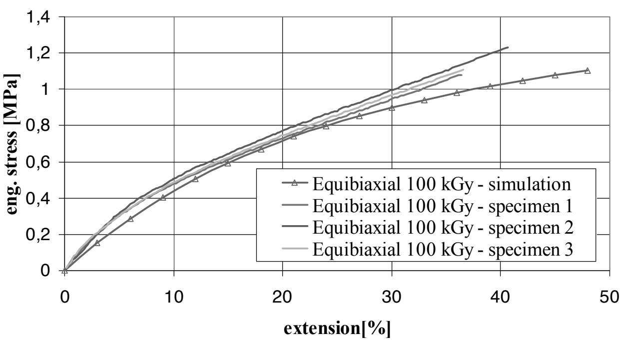

Fig. 8. Equibiaxial curves of irradiated samples - 100 kGy

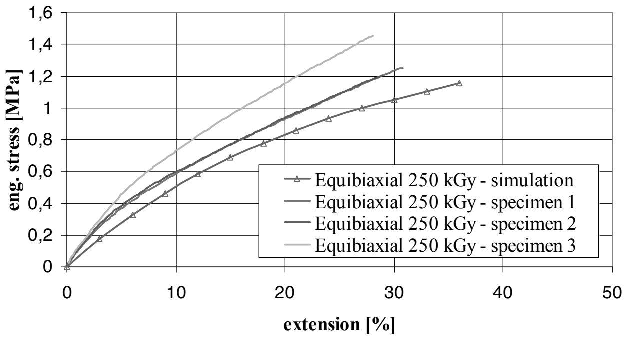

Fig. 9. Equibiaxial curves of irradiated samples - 250 kGy

It is important to take in consideration that even with the more brittle mean experimental data, presented in Figure 9, the simulated curve presents a soft behaviour in comparison to the experimental data, keeping the tendency presented in equibiaxial tests.

The equibiaxial curves developed an evident non linear behaviour in all irradiation conditions. Considering uniaxial and equibiaxial results the Ogden's Model exhibits closer behaviour to experimental data, where the non linearity is more evident and under non irradiated and irradiated with 100 kGy conditions, it means, lower cross linking and lower modulus in comparison to the 250 kGy state.

3.3. Planar shear tests

Comparing non irradiated to irradiated with 250 kGy specimens, in general the changes caused by EB radiation, on the ACM under planar shear state, followed the same tendency as shown under uniaxial and equibiaxial states, the increase of EB dose increased the strength and decreased the elongation. Specimens irradiated with 100 kGy presented a particular behaviour under pure shear state, the strength increased in comparison to non irradiated condition, but no elongation decreasing was caused. In Figures 10, 11 and 12 is possible to verify simulated curves running closed to experimental data in all radiation condition, for all tested extension rates.

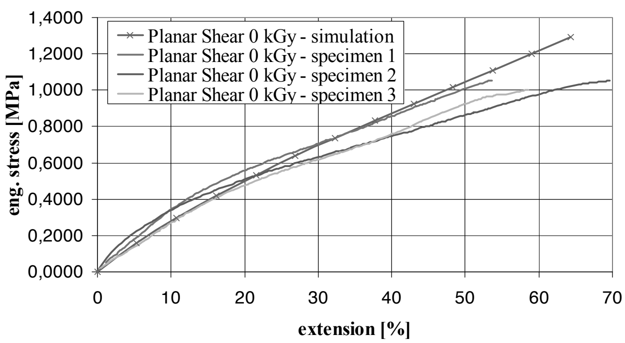

Fig. 10. Planar shear curves of non irradiated samples - 0 kGy

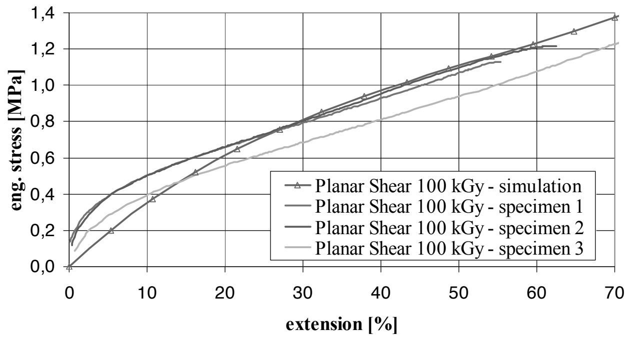

Fig.11. Planar shear curves of irradiated samples-100 kGy.

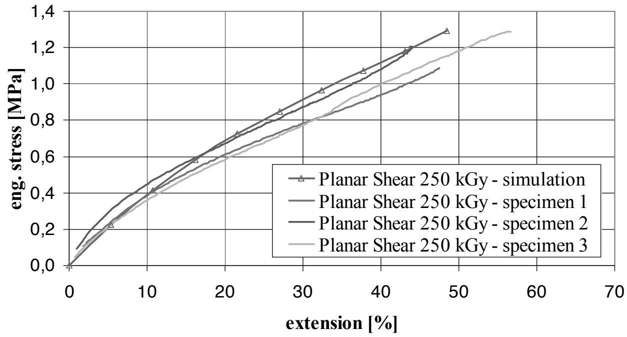

Fig. 12. Planar shear curves of irradiated samples - 250 kGy

Regarding simulated data, the obtained curves exhibit a more brittle behaviour than the experimental data. The particular behaviour in elongation, associated to the higher strength behaviour after 25% of extension, confirms the particular behaviour of the irradiated ACM under pure shear strength.

Table 4.
Mean strength and elongation at break under planar shear state

Dose (kGy)	Strength at Break (MPa)	Mean elong. at break (%)
0	1.03	61.17
100	1.20	63.13
250	1.20	49.29

3.4. Compression stress simulation

The experimental data applied on the ANSYS software generated the Ogden's Model parameters, Table 5, becoming possible to provide the uniaxial, equibiaxial, planar shear and compression simulated curves. Uniaxial compression tension exhibits similar behaviour to equibiaxial tension, but when working with compression tests or simulations is needed to consider the friction coefficient between specimen and test device. As no reference of ACM friction coefficient was found, three simulations were carried out, using different coefficients, in order to identify the influence of such data on the simulation and to define the acceptable value to be applied on the compression tension simulation.

When varying the friction coefficient from 0,05 to 0,5, shown in the Fig. 13, the simulated uniaxial tension curve shows an increase of the elastic modulus and strength, indicating the influence of this change on the results. Comparing the curves obtained considering coefficients of 0.5 and 0.8 is possible to verify that such level of variation can be neglected, confirming the applicability of the 0.8 value as simulation parameter.

Table 5.
Ogden parameters for non irradiated, 100 kGy and 250 kGy states

Dose (kGy)	μ_1	α_1	μ_2	α_2	μ_3	α_3
0	-12.52	4.10	5.29	4.34	7.810	3.83
100	-129.24	4.45	59.77	4.60	70.38	4.30
250	-1005.20	4.43	487.65	4.50	518.77	4.36

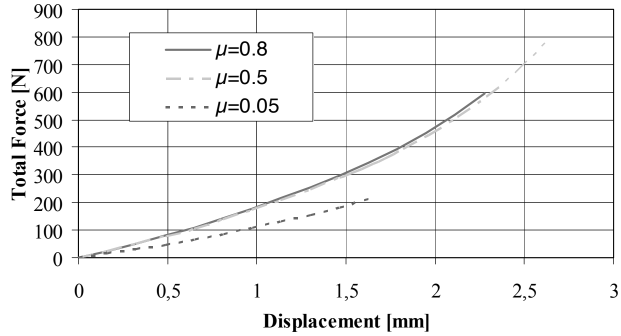

Fig. 13. Different friction coefficients for uniaxial simulation using 2 order Ogden Model

Under compression, ACM presented the same behaviour as under other conditions, regarding the radiation influence, an increase of strength for the same level of strain with increasing the radiation dose was noted.

To increase the strength under compression, for defined strains, could be a solution for industrial applications, where this property is required, like sealing by high performance solid gasket. By EB irradiated ACM could provide higher sealing capacity (strength) for the same deformation in comparison to non irradiated ACM. Sealing elements used in nuclear plant sealing present influenced behaviour because of ionizing radiation presence [16].

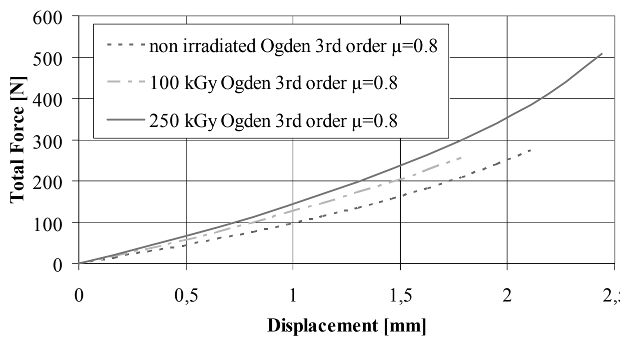

Fig. 14. Simulation of uniaxial compression state

3.5. ATR Infrared spectroscopy

Wave length peaks and related chemical groups in ACM are shown in the Table 6. Each peak indicates the content of specific content of its related chemical group in ACM, the peak high change indicates the change of such content.

Fig. 15, 16 and 17 compares the molecular structure of non irradiated ACM on surface and middle region of specimens for different EB doses. Different peak highs at 1632 cm-1 indicate different contents of >C=C< and consequently different molecular structures in both regions. The amount >C=C< groups is higher on the middle region, indicating the unsaturated state and possibility to increase cross linking level through complementary post cure method.

The second more important change was verified in the content of >C=O variation in both analysed regions, in all conditions the content of >C=O is higher in the middle region of the sample. In case of UV curing in presence of oxygen, or without the required mould, the >C=O level on surface would increase because of possible oxidation process.

Comparing the wave length peaks obtained for each applied radiation dose, in the both regions, is possible to note a decrease of >C=C< on the surface region with increasing of dose. The same occurs in the middle region, but only for 100 kGy dose.

Table 6.
Peak positions and assignment of peaks in the infrared spectra of ACM rubbers.

Wave number (cm-1)	Chemical Group
2980	-CH stretching vibration of O-CH2-CH3
2926	-CH2 stretching vibration of O-CH2-CH3
1740	>C=O
1632	>C=C<
1378	-CH3 deformation of O-CH2-CH3
1160	C-O-C stretching
1153	R-CO-R symmetric deformation
851	C-O-C deformation

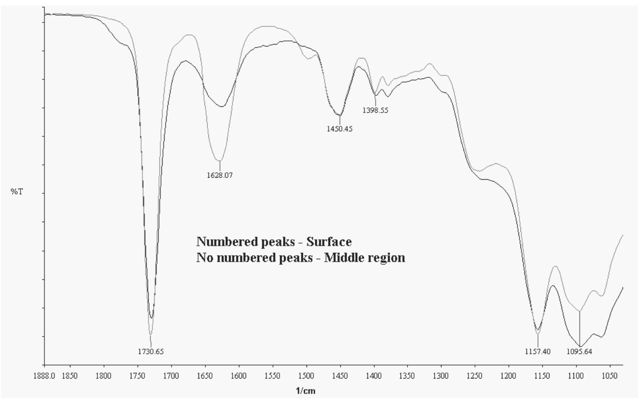

Fig. 15. Infrared spectra of non irradiated samples

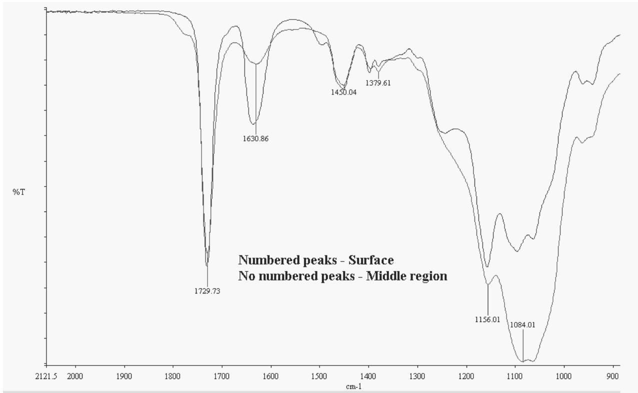

Fig. 16. Infrared spectra of irradiated samples – 100 kGy

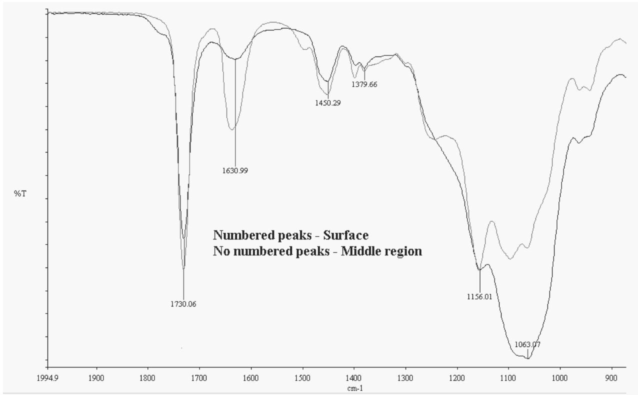

Fig.17. Infrared spectra of irradiated samples – 250 kGy

Content of >C=O and C-O-C also decrease in surface region with increasing of dose. In the middle region the variation of >C=O and C-O-C contents follow the same dynamic as for >C=C< contents, showing that different EB doses cause different structural changes. The more accentuated change is verified for 1095 cm-1 wave length, which is related to skeletal vibration. The absorbance level of this wave length increases drastically in the surface region by increasing the EB dose. The middle region presented high amplitude of peaks, according to the still verified particular behaviour of 100 kGy irradiated specimens.

The verified molecular structural changes can be explained by different reaction mechanisms, like aerial oxidation, chain scissioning, ether formation, recombination and cross linking. All these mechanisms occur simultaneously when irradiated by EB.

4. Conclusions

From the experimental and simulated data analysis is possible conclude:

Uniaxial experimental analysis shows an increase of ACM elastic modulus at 100% elongation when irradiated by electron beam with doses up to 250 kGy. In comparison to on irradiated ACM, the modulus increased 31,25% and 59,77%, respectively, when irradiated with 100 kGy and 250 kGy.

EB radiation caused an increase if cross linking level in the UV curing ACM.

Radiation dose of 250 kGy always caused mean elongation at break decrease and mean strength at break increase in comparison to non irradiated ACM.

Six parameters Ogden's Model does not represent a close behaviour of simulated data to experimental data in uniaxial tension state for UV curing ACM, especially in EB irradiated states.

In equibiaxial and planar shear states the six parameters Ogden's Model shown a close behaviour of simulated curve in comparison to experimental data.

Ogden's Model shows better accuracy where non linear elastic behaviour is evident.

No significant changes are caused on simulation by varying between 0.5 and 0.8 the friction coefficient.

Chemical group changes occur in different levels on surface and in the middle region of the specimens. Many chemical reaction mechanisms are caused simultaneously by post cure by EB, like oxidation, chain scission and recombination with C-O-C formation.

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