Radiation Crosslinking of Neoprene W with Diallyl Etherofmaleic Acid and Epoxy Resins

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Abstract Discusses the use of technological aspects and radiation crosslinking Neoprene W in the presence of a diallyl ester of maleic acid (DAEMA) and epoxy resin (ED-5) in the presence of metal oxides. With the help of physico-chemical and spectral methods shows the variation in the molecular structure Neoprene W in the presence of DAEMA and ED-5 after irradiation with γ-rays of 500 kGr. With analysis method of Sol-gel for each test system determined the radiation-chemical yield (RCY) and the emergence of cross-linking of cross-links in the elastomer. The dependence of the crystallinity index of the degree of stretching for Neoprene W, irradiated at 500 kGy was defined. It was found that at the radiation vulcanization Neoprene W in the presence of a bifunctional bonds epoxy resin weakly has a decisive influence on the kinetics of the process and yield crosslinking. It is shown that above 1500 kGy irradiation in the filled Neoprene observed destruction in the elastomer chain, resulting in deterioration of physical and mechanical properties of the vulcanizates.

Keywords Neoprene, Elastomer, Vulcanization, Radiation, Allyl, Sensitizer, Gel, Crosslinking, Dose, Crosslinks

1. Introduction

Neoprene W (DuPont, France) is one of the most promising elastomers for special purposes. This is due to its valuable properties high physical and mechanical properties. [1-5].

Neoprene W differs considerably greater tendency to radiation crosslinking than other diene elastomers. It vulcanized under irradiation even without additives and auxiliaries, and in the presence of low molecular weight additives can be prepared valuable technical vulcanizates [6-10].

Radiation-chemical synthesis of Neoprene W in a process homogenous vulcanization, as a rule, complicated kinetic activity in the reaction of the polymer chains [11-14].

At the crosslinking a Neoprene W exposure to ionizing radiation [15-17] as well as in their mixtures with small amounts of poly functional monomers as the allyl-methacrylate leads to the formation of high molecular weight polymer.

Due to the fact that the Neoprene W is an unsaturated elastomers, proceeding from the structure of Neoprene W and reactivity abilities sheet polymer molecules in principle carried thermal and radiation vulcanization with help a fairly wide range of substances.

The high molecular Neoprene W is also irradiated with the participation of low molecular weight organic sensitizers, triazine bondss. The activity of these sensitizers in radiation-chemical processes is low, probably due to the weak influence of the properties [14, 18, 19].

It is known that for achievement optimum properties vulcanizates requires a fairly large dose, which increases the cost, prime cost of materials and serves possibility of using radiation instead of thermal. In this regard, very relevant is the question of how reduce the radiation dose methods, necessary for obtaining optimum properties and hence improve the performance of the process [14, 17, 19, 20].

Neoprene W is a convenient object for studying the effect of the sensitizing agent which has active functional allyl group also cyclic modifier has cycle epoxide groups, which allows us to estimate the effect of these functional groups on the parameter space grid radiation vulcanizates.

Effect of contents allyl and epoxy groups to study the structure and properties of Neoprene W depends not only on the magnitude of the absorbed dose, and the activity of the solid oxide additives [21, 22].

The mechanism of radiation crosslinking Neoprene W in these crosslinking systems is still insufficiently understood.

The results of investigation of the impact sensitizer diallyl ether of maleic acid (DAEMA) and epoxy resin modifier on the kinetic and structural parameters of the grid vulcanizates exposure to ionizing radiation are set out in the article.

2. Methods and Object of Research

For radiation curing used chloroprene rubber stamp Neoprene W (French product) obtained by emulsion polymerization. By results of Fourier research spectroscopy isomeric composition of the double bonds in the investigated polymer 1,4-trans units consists 86.5%, cis 1,4-10%; 1.2-1.6% and 3,4-1%.

Crystallizability plays a big important role in the recycling of Neoprene W. Maximum speed of crystal-lization amounts 263K.

Neoprene W macromolecules consist of static distribution links of butadiene and vinyl chloride. Especially in radiation chemical cross-linking Neoprene W, affect their microstructures, molecular weight, MWD (molecular weight distribution) and the gel content. That is why, prior the radiation-chemical processes in the preliminary elastomer viscosimetric method and the gel permeation chromatography (GPC) determined average and weight average molecular weight and the polydispersity (M_n = 72 231 = th. M_w=231 th.M_w:M_n = 3.2). For initiate the chemical processes used radiation sensitizer diallyl ester of maleic acid (DAEMA).

DAEMA is prepared by reacting maleic anhydride with allyl alcohol in the presence of sulfuric acid (M = 182, melting point= 385K, white liquid, soluble in aromatic hydrocarbons) One of the most important conditions for the normal flowing of radiation crosslinking Neoprene W is to provide the required molecular weight and yield of crosslinks.

Proceeding from the structure of the isoprene rubber and the reactive sites of the polymer molecules, in principle, crosslinking can carry out by using a synthetic epoxy resin. Naturally that efficiency of crosslinking process will determine overlooking Neoprene W rubber and concentration necessary for given type of vulcanizing system reactive places and type of crosslinking system.

Epoxy resins are linear polyethers, at the ends of molecular chains which are capable of highly reactive groups, and chains secondary hydroxyl groups.

The mainly part of epoxy resin received in the laboratory or industrial scale constitute resin formed when interacting of polyphenols with epichlorohydrin.

For receipt with the purpose of the activation process and the yield of crosslinking introduced into heterogeneous systems, active zinc oxide was evaluated by band gap (ΔE=3,3eV) thereby found practical application and dosage which are selected strictly in accordance with the individual characteristics at radiolysis. For holding of crosslinking have been used for 100 mas.p. of elastomer (Table 1).

Table 1.   Physical properties of components

Components	Melting temperature (K)	Molecular weight (M)
1. Diallyl efir of Malein acid $CH-C\overset{O}{\underset{O}{\parallel}}O-CH_2-CH=CH_2$ $CH-C\underset{O}{\overset{O}{\parallel}}O-CH_2-CH=CH_2$ (DAEMA)	396K	172,18
Epoxydian resin (ED-5)	Liquid Viscous 323-353K	500-650

Based on the sample weight of 1g were placed in ampoules made of glass "Ray" and evacuated for hours until a residual pressure of $1,3 \times 10^{-1}$ Pa. Radiolysis sealed vials with samples were conducting γ-rays Co^{60} with a power of 4.9 Gr/s at room temperature. The absorbed doses in the samples studied were calculated by comparing the density of the electronic and dosimetry systems [23].

On changes in the molecular weight of Neoprene W were judged by the inherent viscosity [η_{int}], measured using a viscometer Ubellohde in toluene at 298K. The calculation was conducted on equation Mark-Houwink at the value of the constant K=$1,16 \times 10^5$ and α=0,73 (toluene).

The kinetics of crosslinking was investigated by changing the number of crosslinks (n_c') and resistance rupture of the vulcanizates. Numbers of crosslinks n_c' were determined by the method of equilibrium swelling of vulcanizates in toluene and was calculated using the equation of Flory Rehnera [24].

The rate constant of radiation crosslinking was calculated graphically from to the equation kinetic equation of the chemical reaction of the first order. The apparent activation energy E was calculated by the Arrhenius equation [25]. The apparent activation energy E is defined by the number of cross linked molecules (1 / $M_{n\tau}$) are given in the below (Table 2).

Table 2.   The apparent activation energy for systems based elastomer Neoprene W by irradiation γ rays of 500 kGy (mas. p. at 100 mas.p. of elastomer)

№	Vulcanized system (mass.p)	Dose, kGy	E, kkal/mol	The amount of cross-linking molecules, (1/ Mn_τ), mol/sm³
1	Neoprene W-!00	500	14,2	1,8
2	Neoprene W- 100 + 4,0DAEMA	500	17,6	5,9
3	Neoprene W-100 + 6,0 ED-5	500	21,3	4,8
4	Neoprene W-100 + 4DAEMA+6,0ED-5	500	24,4	5,3
5	Neoprene W -100+ 4 DAEMA + 6,0 ED-5 + 5,0 ZnO	500	31,3	6,7

Changes of molecular structure of crosslinking Neoprene W was evaluated on the Fourier spectroscopy. For receiving filled vulcanizates have been used technical carbon P324. When choosing of technical carbon was paid attention on the following: particle diameter (30-33 nm), udine surface (75-85 m³/g), oil number (60-80ml/100g), density (1820 g/m³).

Physical and mechanical properties of the vulcanizates were determined automatic bursting machine UL-In (US-made) display the results support the exchange via LAN and Internet.
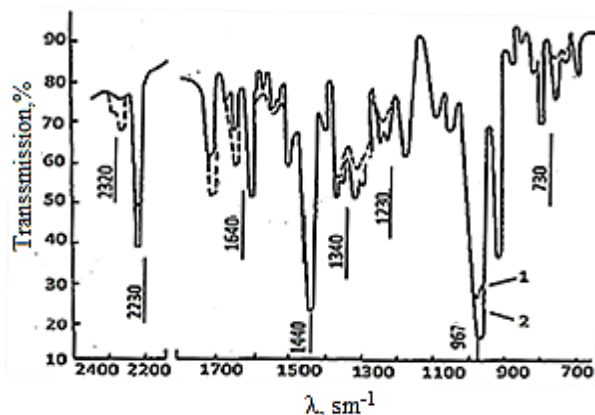
Radiation chemical yield concentration of cross-links (G_{Nc}) and the number of cross-linked molecules ($G_{M}p_{\tau}$) was determined by sol-gel analysis.

Calculation of parameters of a spatial grid of irradiated cross-linked polymer was determined by the formula Flory Renera and Charlzbi [25-27]. The index of the degree of crystallinity was determined from tension (α, %) after irradiation at a dose of 50 kGy [28].

3. Results and Discussion

Despite the large number of studies on the effects of ionizing radiation on the elastomers, it is still not yet sufficiently clear mechanism of crosslinking Neoprene W quasi-binary systems. Furthermore, in the literature there are conflicting data on the assessment of output crosslinking loss of unsaturated and other structural changes in Neoprene W by irradiation.

Reducing the intensity of the absorption bands specific to the studied sensitizers as increasing dose clearly indicates their expenditure in the crosslinking (Figure 1).

Figure 1. IR spectra were irradiated model mixtures based Neoprene W: 1-unirradiated Neoprene W; 2- Neoprene W+ DAEMA; 3-Neoprene W + ED + DAEMA + ZnO after irradiation dosa with 500 kGy

For the modern system observed a number of identical changes at the crosslinking. Reduces the intensity of the band in 1440 sm⁻¹, which belongs to deformation vibrations of C-H bond in the group -CH₂ elastomer [29] as well as the intensity of the bands 967 sm⁻¹, respectively, is due to flatness and deformation vibrations of C-H bond in the group-CH-CH -train configuration [30, 31].

Analysis of structural changes in the system of Neoprene W -DAEMA was performed by Fourier spectroscopy. Fig. 1 shows IR-spectrum compositions Neoprene W with 4.0 mas. p. DAEMA irradiated to a dose of 250 kGy. (Fig. 1)

By changes in the spectra can be seen that the decrease of the band at 970 cm⁻¹ characteristic vibration of C-H bonds in the vinyl double bond also disappears band C-C bond in the area of 1650 sm⁻¹ decreases the intensity of the band 750 sm⁻¹ corresponding to stretching vibrations of the bonds C-Cl.

Observed the emergence of a large band 1700 sm⁻¹ is a type of vibrations COO ester groups.

At the main observed change in the spectrum of Neoprene W when sharing their irradiation sensitizers and auxiliary low molecular weight compounds can be suggest at the crosslinking reaction proceeds with groups-CH, CH₂ group and the double bonds of the polymer molecule.

Visibly reduces the intensity of the absorption at 750 cm⁻¹ caused by vibrations of the bonds C-Cl [29]. This gives reason to believe that there is a place intense cleavage of chlorine molecules Neoprene W. Reducing the intensity of the bands 1230 cm⁻¹, which lies in the area of analytical absorption fluctuations CH group.

The increase absorption in the 1650-1750 cm⁻¹, in our opinion, happens on account of the education of various oxidation products elastomer. The highest efficiency of a sensitizing action is also shown combination DAEMA and ED-5 when administered of zinc oxide.

The observed radiation at the influence of too absorbed doses (1000 kGy) significantly increases the background that interferes with conduct quantitative comparisons. That is why in the work we have shown only the impact of doses of 250 kGy.

Can make the conclusions of irradiated model systems (Neoprene W + DAEMA + ED-5 + ZnO) following conclusions:

On the radiation crosslinking process significantly affects the chemical structure of macromolecules in the presence of some low molecular components (particularly sensitizers stitching). Quantitatively radiation crosslinking of polymers can be characterized by the magnitude of the output of the molecular weight.
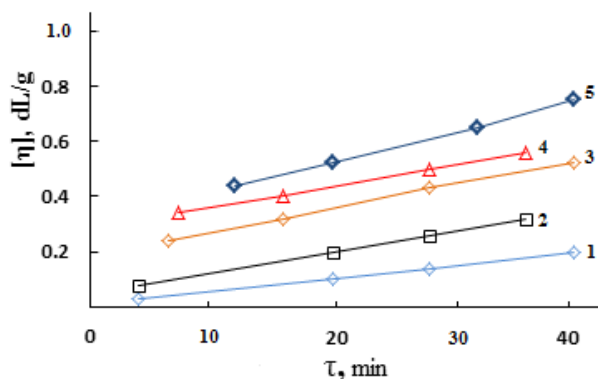
For determine the radiation crosslinking of Neoprene W low molecular weight components we studied some kinetics of vulcanization. Studies of viscosity and molecular weight of Neoprene W duration of irradiation (D = 250 kGy) showed that the polymer molecules represent yourself few cross-linked structures (Figure 2).

For individual samples obtained elastomers characteristic viscosities and obtained for all elastomeric compounds are low. Similar results were obtained with involving of zinc oxide.

Effect of zinc oxide, DAEMA and ED-5 with Neoprene W, on the kinetics of vulcanization mixtures from the duration of irradiation showed that the molecular weight is 0.8×10^5 dg / l.

Matching of all the properties of the vulcanizates from our side allows us to assume that the inclusion compounds

obtained in Neoprene W represent three-dimensional grid, double-stitched for sites chains. In favor of this is supported by the fact that the obtained elastomer composites do not crystallize and have an average molecular weight of low intrinsic viscosity and good solubility. As firmly established in the literature view [2, 8] that the cross-linking occurs when radiation effect arise to the active atoms in an allyl group. Number allylic chlorine considered elastomer is approximately the same (0.5-0.7% of the total chlorine content) [2]. One would have expected that it will be vulcanization under the effect of radiation with the close speed and till the equal degree of crosslinking, which is not really there. Investigated Neoprene W have various crosslinking speed, that exemplified by radiation curing at 500 kGy. (Tabl. 2). Limiting the output value of cross-linked molecules $1/M_{n\tau}$ is also different.

Figure 2. Dependence of the magnitude the characteristic viscosity Neoprene W, from the time of crosslinking: (D=250 кGy) 1-Neoprene W; 2- Neoprene W + DAEMA; 3-Neoprene W + ED-5; 4-Neoprene W + DAEMA + ED-5; 5-Neoprene W + DAEMA + ED-5 +ZnO
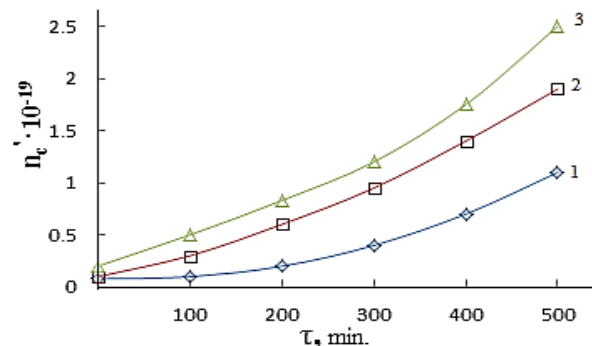
Consequently, the cross-linking is carried out not only due to the allyl chloride, apparently, in cross-linking adopts participation of some mobile chlorine atoms or other reactivity of the centers, which may appear in the vulcanization process, as the kinetics of radiation vulcanization Neoprene W is described by a bullet order and limit cross-linking It is not achieved.

Kinetics of radiation vulcanization in the presence of Neoprene W can be seen that the magnitude of the output n_b' stay on little unchanged over the entire time interval. The rate and degree of crosslinking at 25 min. practically does not decrease as compared without additive. Furthermore, in the initial period, crosslinking is accelerated. It is perfectly clear from the kinetic curves change in viscosity of the mixture after irradiation (Figure 3, curves 2).

Consequently, acceleration of cross linking is possible with the introduction of cyclic groups such as a molecule of epoxy resins represents simple linear polyethers, at the ends of molecular chains which have reactive epoxy groups.
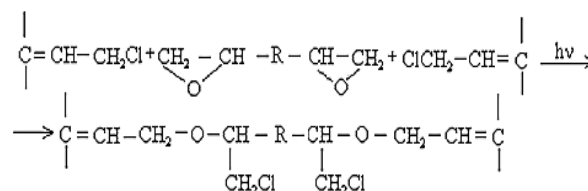
At the radiation vulcanization Neoprene W, epoxy resins should be viewed not as a vulcanizing agent, but as a modifying agent. Because they bind with the elastomer, but not weakly shows a decisive influence on the kinetics

without the degree of crosslinking. (Figure 3, line 2) Since, resins are difunctional compounds; they may be attached to a molecule of the elastomer and providing sufficiently long and flexible cross-links. Linking is done by opening the epoxide cycles.
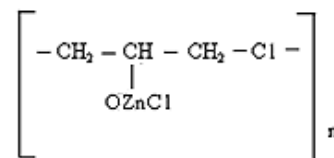

Figure 3. The kinetics formation of output number of cross-linking (n_c') at the vulcanizing Neoprene W (D = 500 kGy): 1-Neoprene W; 2-Neoprene W + ED-5; 3-Neoprene W + DAEMA

It is difficult in the present time to give preference to which or accession resin to the elastomer at the radiation. It can be count only firmly enough established binding resin with an elastomer opening of the epoxide ring in the possibility formation of cross-links.

However, the effect of resin crosslinking on the process Neoprene W slightly and is at the same rate and to the same level as that involving DAEMA. Therefore provide for the participation of the double bonds of the polymer will be visible properly.

Enough firmly certain action of the epoxy resin and zinc oxide on the process of emerging cross-linking was found. This can be explained by the interaction of an epoxide with zinc chloride to form compounds type-

Some increase in cross-linked molecules in the presence of zinc oxide compared mixtures without oxides (Tab.2.) and close values $1/M_{n\tau}$ to settlement suggest the possibility that the presence of zinc oxide crosslinking is emptive for allylchloro, in the formation of new active centers suppressed oxides metals or products of their transformation. A good proof of this assumption is that the investigated elastomers values E, at the metal oxide vulcanization are very close.

When administered in the system of metal oxides first occurs intramolecular hydrogen chloride, resulting in formation of conjugated double bonds, [2], which are then DAEMA is grafted and is sewn in the molecule, with participation of ES formed cyclically crosslinks.

If this incarceration is valid, that offered low molecular substances at the interaction with the double bond, should raise the rate and degree of crosslinking.

From the submitted material be seen that vulcanizates Neoprene W obtained by the action of ionizing radiation on the structure is much more complicated.
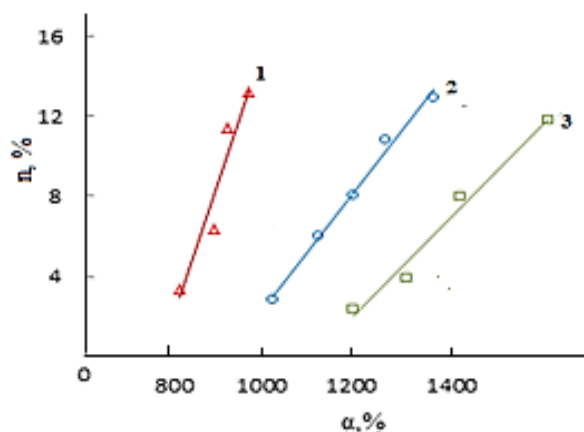
One of the characteristics of features radiation vulcanization of Neoprene W is the formation of a sufficiently large number of durable C-C bonds in participation crosslinking agents.

As shown by change unsaturation elastomer Neoprene W, is defined with Fourier method spectroscopy resulting in dense grids, which are compares with the mechanical properties and crystallization of vulcanizates, the value of change unsaturation value is within the accuracy of the method. The received output of the effective concentration of the cross links at 500 kGy is equal to $n_c' = 6 \times 10^{-19}$ cm^3.

The use of such dosages for the comparison is justified, since the rate of change of unsaturation remains constant over a wide range of doses.

Observed changes in the intensity of the absorption bands of 780 cm^{-1}, corresponding to the double bond in the 1,4-cis configuration. Changes in these bands may be caused by the cis-trans isomerization of the elastomer, and the costs of the double bonds.

For all doses the degree of crystallinity increases linearly with extension of the sample 1-4 his suspenders (Figure 4), which allows to characterize the intensity of crystal formation for given concentration slope of tangent of the angle annoy tilt of the straight abscissa.
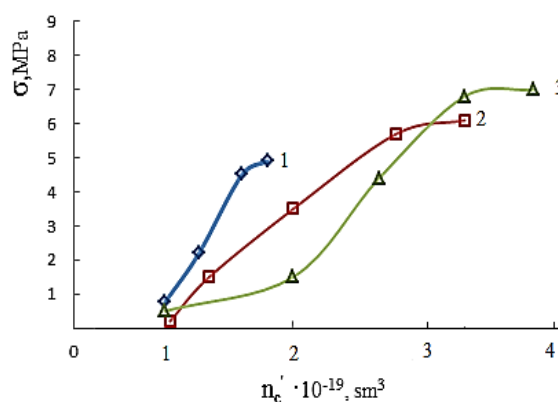

Figure 4. The dependence of the crystallinity index of the degree of stretching for Neoprene W (1-4) irradiated at 500 kGy Direct correspond to different values $n_c' \cdot 10^{-19}$, sm^3 ; **1**-1,8; **2**-2,4; **3**-3,9

With increasing irradiation temperature (373 K) the intensity of crystallization samples is lowered.

Thus, increasing the radiation dose to 500 kGy, leads to additional destruction of vulcanizates. Therefore, the destruction is not the cause of observed changes in the properties of vulcanizates.

Application of cross-linking agent DAEMK and a synthetic resin introduction of metal oxide in an elastomeric mixture results activation of the output effective crosslinks. With the output rise crosslinks increases the strength and elongation with increasing crosslinks decreases.

Above 1500 kGy irradiation samples observed the destruction in the polymer chains that, results the deterioration of strength of vulcanizates. (Figure 5)
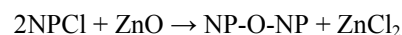

Figure 5. Addictiontensile strength (σ) output concentration of crosslinks in Neoprene W irradiated at a dose of 500 kGy. **1**-Neoprene W +DAEMK + ZnO; **2**-Neoprene W + ED-5 + ZnO; **3**-Neoprene W + DAEMK + ED-5 + ZnO

As shown above, zinc oxide is widespread and almost obligatory component at the radiation vulcanization of neoprene elastomers. Thus, consideration of mechanism Neoprene W, can serve as their role in the vulcanization process.

In most of the work with the metal oxide plays the role of acceptor of hydrogen chloride. But it is not always possible to confirm the experiment. However, by the known method managed to identify with the application of extraction alcohol-toluene mixture, making it possible to accurately determine the content of metal chlorides (Table 3).

The role of radiation crosslinking processes zinc oxide Neoprene W (NP) assumes that the metals oxides directly by crosslinking oxygen transmission, i.e. total:

$$2NPCl + ZnO \rightarrow NP\text{-}O\text{-}NP + ZnCl_2$$

For obtaining elastomeric materials at radiation crosslinking requires the presence of technical carbon (filler), which is also the simultaneously amplifiers. Optimal filling of the filler content was 50 mas. p. on the 100 mas. p. Neoprene W.

Table 3.   The impact of the dose of radiation crosslinking on the process of the chemical composition of Neoprene W in the presence of 5.0 mas. p. ZnO

Thecrosslinking system	Dose, kGy	The chlorine content in the sample, %			The number of cross-linked molecules on one crosslinks $(1/ Mn_{\tau)},$ mol / sm^3
		Overall	Associated	In mole Including in ZnCl$_2$	
Neoprene W +DAEMA+ ZnO	150	85	14.2	3.4	1.27
	500	72	50.8	12.6	2.2

Table 4.   Effect of γ-irradiation on physical and mechanical properties of filled rubbers based on Neoprene W

Composition mixtures (mass/p. on 100 mass/p. elastomer)	Dose, kGy	Tensilestrength, MPa	Hardness index, Shore A	Elongation at break, %	Residual elongation, %
Neoprene W+DAEMA+ED-5+ZnO	500	12	30	780	38
	1000	14	35	760	35
	1500	16	40	640	23
	1700	14	45	620	22

As can be seen from the obtained results (Table 4) the introduction of the filler has a marked effect on the physical and mechanical properties of radiation vulcanizates. At the irradiation dose of 1000 kGy hardness at vulcanizates increases, and the elongation decreases. Strength properties of irradiated vulcanizates depend on the absorbed dose. At a dose of 1500 kGy optimum strength is 15 MPa. (Tabl. 4). In the elastomers containing 40 mas.p. of technical carbon , the breaking strength decreases with 1500 kGy of absorbed dose and then increases sharply. Elongation at break and hardness vary monotonically with increasing absorbed dose, regardless of the dosage of technical carbon.

At sufficiently high doses (3000 kGy) elongation and hardness are almost independent of the content of technical carbon. Obviously, in this case the radiation crosslinking Neoprene W so great that reinforced impact of filler no time to manifests.

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4. Conclusions

Results of analyzes and individual properties from the structure of Neoprene W and the vulcanizing group (DAEMA, ED-5) should make two remarks. First, studies have shown that for heterogeneous radiation vulcanizates Neoprene W elastomers fundamentally observes the same regularities that have been installed in the works [2] and other researchers on the examples vulcanizates of general purpose elastomers.

Second, the absence in a number of cases, the characteristics of structure of the radiation vulcanizates accounts to limit the influence on the properties type of low molecular group (DAEMA, ES). Probably, occur to the formation of intramolecular of crosslinks.

Question on the reaction of epoxy resins in Neoprene W at the radiation crosslinking is much more complicated. They can disconnect hydrogen chloride and form a α-Chloroxy compound. It is also possible accession of epoxy resin to the double bond.

Crosslinking metal oxide provides the highest rate of radiation-chemical yield of the number of cross-linked molecules and efficient crosslinks. The highest exposure dose leads them melting. Thus, for example, at equal concentration of crosslinks oxide vulcanizates have a fine-grained structure and interval dose of crosslinks 500-800 kGy. This is apparently due to the presence bonds of the C-C or C-O-C and a large share of movable labile bonds in the oxide vulcanizates.

Between strength and concentration of crosslinks, as the expected result, there are close to a linear relationship (Figure 3).

The density of the vulcanization grid, in the presence of zinc oxide, do not strongly influences on the strength of

rubber. Vulcanized elastomeric mixture (W + Neoprene DAEMA + ED-5 + ZnO) is significantly different in strength from other vulcanizates. It is shown that the filling of the elastomer in the presence of technical carbon static gap strength and the Shore hardness increases with increasing irradiation dose (Table 4) at the same time decreases relative elongation. Above 1500 kGy observes a gap at the polymer mesh.

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