

Influence of Gamma Irradiation on Structure and Properties of Nitrile-Butadien Rubber in Presence of Modified Nano Metals

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Abstract Polymer-based nanocomposites NBR in the presence of nanoscale zinc oxide powder were obtained the crosslinking agent disulfochlorid benzene (DSCB) and carbon black. The role of ZnO nanopowders and DSCB in NBR systems NBR+ZnO+DSCB and NBR+ZnO+DSCB+F324 with radiation, chemical exposure. With the help of physico-chemical and spectral methods shown changes in the molecular structure of the rubber NBR, in the presence of ZnO nanopowders and DSCB. Each studied nanocomposites have been identified by and the emergence of cross-linking and cross-linking yield in the rubber. It is shown that the presence of the chemical interaction between polar groups of the macromolecule BNK ($-C\equiv N$) DSCB ($-C-Cl$) nano particles of zinc oxide leads to a significant change in the density of spatial grid nanoexamples that provides an increase in the output of cross-links ($1/M_s$). Shown properties of filled nanocomposites prepared by chemical bonds (C-C) to modify the mechanical properties in the 423K and 500kGy. Especially, conductive properties of nanocomposites are identified based on NBR. It is found that the change in the value of dielectric permittivity (ϵ) and dielectric losses ($tg\delta$) material depends on chemical reactions of the polymer structure.

Keywords Butadiene nitrile rubber, Nanocomposite, Vulcanisation, Gamma irradiation, Disulfochloridebenzene

1. Introduction

An effective technique for the improving the technical parameters and the spatial grid of polymer systems is the introduction of these nanoscale inorganic modifiers. [1, 2] The action of these modifiers is often due to their influence on the process crosslinking of polymers, including the ability to form a chemical bond [3-5]. In the last decade, actively carried out the study of polymeric nanoparticles of metals, semiconductors [6-8], which is together good processability, have improved wear resistance, high hardness and elasticity. The highly developed surface, as well as the proximity of the nanoparticle size to the size of the macromolecules lead to forming nanocomposites unique electronic, electro-mechanical and physical properties are very promising for the creation of new constructional materials [9, 10] used for new equipment, solid and elastic defense materials. Significantly, the desired effect is achieved by introducing a small amount of modifier.

One of the attractive type nano modifiers are oxides of

metals (OM) [11] which are several levels were equally dispensed in the polymer type matrix. [12]

It is believed that the reason for the unique effect of OM polymer systems is their large chemical activity associated with nanoparticle size (2-100nm).

Thus, OM can have a double effect: to act as structures for the adjustable matrix or the ability of the formation of additional nodes, mesh links in elastomeric systems.

Initiating crosslinking with OM and the possibility of reproducing the particles in the polymer matrix may also determine the improvement in mechanical properties of nanocomposites. Thus, the amplification effect: increase in dynamic endurance and tensile strength was observed with the introduction of the OM in the ethylene-propylene copolymer. [13]

Recent advances in the field of processing of nitrile rubber (NBR) have led to the expansion of their modifications and allowed to receive new crosslinked elastomeric materials. The completed earlier studies of the structure and mechanical properties of various compositions NBR, showed that the NBR has a complicated structure, including C-C and C-S-C bond. [14]

Such a set of cross-linking provides large opportunities for the necessary complex by their structural adjustment properties. Several works shown that, introduction to active,

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crosslinked and reactive low molecular weight compounds (LWC) [15] results in a substantial improved in mechanical properties. However, these studies are quite limited and do not provide a complete picture of the mechanism of the observed effects and the conditions of manifestation.

NBR differ significantly greater tendency to cross-linking radiation, other than diene elastomers. Since it is cured valuable technical material can be obtained even when irradiated without additives.

Radiation-chemical synthesis of NBR in the homogeneous crosslinking, as a rule, complicated by the reactions of the polymer chains. [16] Due to the fact that the unsaturated elastomer is NBR based on the ability of the polymer molecules in principle the radiation crosslinking is carried out using a fairly wide range of substances. NBR is a convenient object for studying the impact of nano-sized powders OM, and DSCB crosslinking agent that is active polar (Cl) and an aromatic group, which allows you to assess the impact of these groups on the parameter space grid and thermal radiation vulcanizates.

This work aims to address these problems.

2. Methodology and Research Facility

In this work, for nanocomposites using a high-molecularly nitrile butadiene rubber (NBR) and the content of acrylic nitrile (AN) was 40% in the molecule. NBR is obtained by co-coagulation of nitrile butadiene latex in the ratio of 60:40. As a result, studies by the Fourier method of IR spectroscopy in the butadiene portion of the polymer composition of the isomeric double bonds in the elastomer investigated 1,4-isomer and 14.8% trans-1,4 isomers 70.9%.

Macromolecules rubber NBR was distributed from the static of butadiene and acrylonitrile.

Features crosslinking NBR affect their microstructure, molecular weight distribution (MWD) and gel content. Therefore, prior to the radiation-chemical processes in the elastomer pre osmometric method and penetrating gel chromatography (PGC), determined by the average number and molecular weight and polydispersity ($M_n=69$ thousand, $M_w=226$, $M_n:M_w=4,1$).

As activation crosslinking polymer used nano zinc oxide (ZnO). With the introduction of ZnO nanopowders pay attention to the dimension (20-25nm) dispersion and purity (99.8). Specific surface (250 g/m²), true density 5.606 g / sm³. Powder dosage was varied in the range 0.8-4.0 phr. The nanomaterials had been obtained from the Inc. Houston, TX, USA.

In the process of further agent (DSCB) aromatic compounds, which react readily with macromolecules NBR. DSCB is an organosulfur compound with the formula C₆H₅(SO₂)₂Cl₂ It is a colourless viscous oil that dissolves in organic solvents, but reacts with compounds containing reactive N-H and O-H bonds.

The effect of technical carbon phase was judged by the change in the rheological and mechanical properties.

With the introduction of fillers in polymer systems we have noticed a chemical reaction at the interface of the two phases, adsorption, dispersion, specific adsorption and surface geometry (78-90, 80-88 m²/g) and the impact of carbon black furnace (F324) on the formation of carbon rubber gel (CRG) in the elastomer.

Nanocomposites based on NBR which are prepared by the laboratory rollers with a fraction $f = 1: 2$. Firstly added rubber (100 phr), Then added nanopowder 5.0 phr ZnO, then at the end 3,0 phr DSCB. For filled mixture is introduced at the end of 50 wt. h. P324 filler (carbon black) mixing on rollers constituted to binary (1) and quasi systems (2, 3) 12-15 min.

1. NBR + ZnO;
2. NBR + ZnO + DSCB;
3. NBR + ZnO + DSCB + F324

Studies were carried out on films and plates (60x60 mm) obtained by compression of mixtures at 423 K, followed by cooling.

For the irradiation of the samples were placed in a glass (1 g) and vacuumed for hour, under the $1,3 \times 10^{-1}$ Pa residual pressure. All the samples were irradiated under the Co-⁶⁰ gamma-ray source at absorbed dose of 4.9 kGy at room temperature.

Absorbed dose of samples calculated by comparing the electron densities in the dosimetry systems. [17, 18]

Studies were carried out on films and plates (60x60 mm) obtained by compression of mixtures at 423K, followed by cooling.

The molecular structure and isomeric composition of nanocomposites NBR was studied by Fourier Transform *Infrared Spectroscopy* (FTIR) in the 700-3000 cm⁻¹ rubber films were prepared by applying the solution (toluene) to a substrate and evaporation of the solvent constant.

The intrinsic viscosity (IV) and quasi nanobinary systems determined in toluene at 293 K in a known manner Ubbelohde viscometer. The calculation was performed by the Mark Houwink $[\eta]=KM^\alpha$ value of constant $K=1,9 \times 10^4$ and $\alpha=0,64$ for toluene. [19]

Effect of OM on the parameters of the output concentration of the number of grid lines ($1/M_s$) was determined by the sol-gel analysis. Calculation of the spatial grid of cross-linked polymer parameters determined by the sol-gel analysis and by the *Flory-Rehner equation*. [20]

The mechanical properties of the vulcanizates were determined by a *universal testing machine* (UTM), in accordance with *ASTM D412*, displaying results supports communication via LAN and Internet.

Measurements of the electrical properties (ϵ' , $\text{tg}\delta$) of nanocomposites was performed using the bridge P 5083 in the frequency range of 10^2 - 10^5 Hz, which has been linked with the measuring cell clamping electrodes [21]. Samples were prepared for investigation in the form of films with a thickness of 140-160 microns and a diameter of 25 mm by a pressing operation at $T > T_c$ and $P=10^7$ Pa, followed by cooling under pressure to room temperature at a rate of 276 Grad / min.

3. Results and Discussion

Fourier spectroscopy methods describe that the ZnO particles is well distributed in the matrix of NBR, the system NBR-ZnO-DSCB characterized polarity so the process of combining NBR nano zinc oxide with DSCB mainly determined $-C\equiv N$ polar groups and C-Cl their reactivity under thermal and radiation effects.

On this basis, the main attention was paid to the intensity of the band stretching vibration of the nitrile group, located in the area of 2235 cm^{-1} (Fig. 1) and are characterized by exceptional stability, both in frequency and form.

FT-IR spectra of nanocomposite in presence of zinc oxide shown that the new absorption peaks distinctly at 3223 cm^{-1} , 852 cm^{-1} , 745 cm^{-1} , and 477 cm^{-1} assigned to the presence of metaloxide in the nanocomposite.

The emergence of new bands in the region adjacent to 2240 cm^{-1} is probably due to the formation of complex compounds of groups $-C\equiv N$ and zinc chloride. [22]. Visibly reduces the absorption intensity in the range of 750 cm^{-1} due to the vibrations of C-Cl [23, 24]. This gives reason for the believing that during heating and irradiation (500 kGy) holds intensive elimination of chlorine from DSCB molecules. On cleavage of said chlorine, apparently, decrease in band intensity and 1230 cm^{-1} , which are in the field of analogical IR absorption study substituted aromatic structures [25-27].

We can assume that established in the literature looking [28], it is expected that the introduction of polymer systems ZnO nanopowders in the radiation-chemical reactions is cleaved from the molecule DSCB chlorine atoms, which

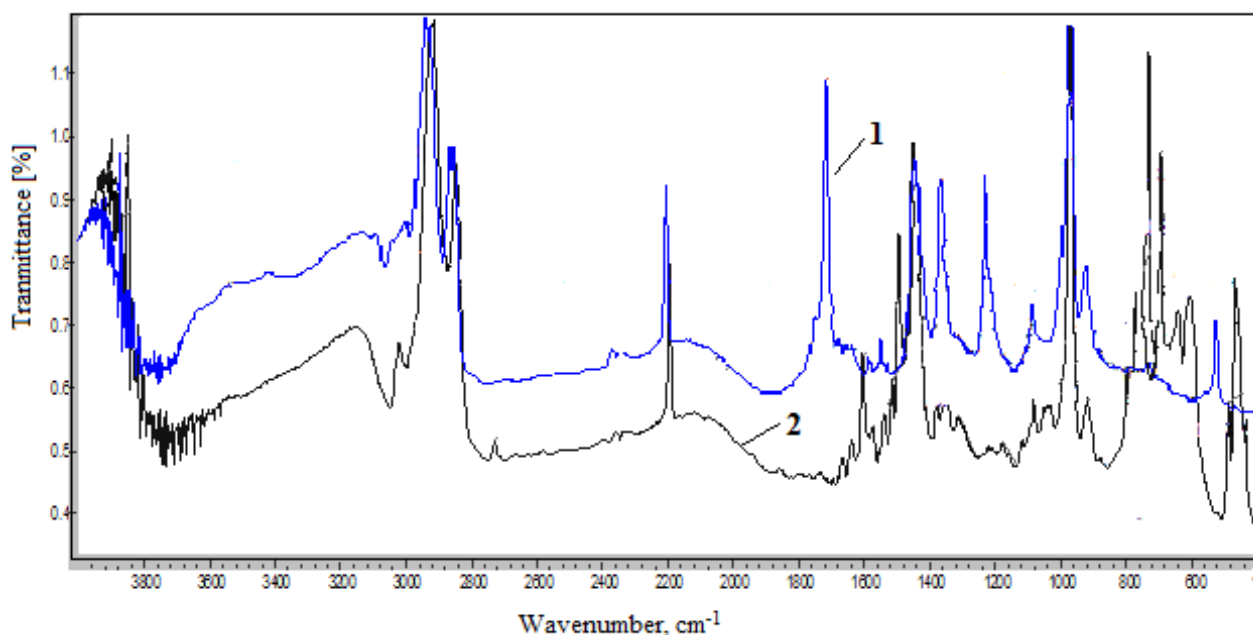
interacts with the zinc oxide, resulting in branched structures are formed. Complex heterogeneous structure of the obtained nanocomposites is confirmed by the results of the study of the rheological properties of crosslinked NBR.

Laws of change of nano-molecular-weight polymer compositions vary depending on the time of crosslinking. For samples, pure NBR viscosity output is not high. In the binary system, (2) the yield viscosity is 0.8. In the three-component system, in the presence of DSCB, there is a jump in the solution concentration and viscosity yield was 1.2.

Adding DSCB increases the yield viscosity, probably due an increase in the density of the nanocomposites.

Character of change of the molecular weight (MW) in the irradiated nano compositions also different. The irradiation dose of 500 kGy is no formation of a spatial network. When dissolved in toluene initial rubber has not been subjected to irradiation of gel fraction after radiation doses of 150-300 kGy elastomer becomes completely soluble, indicating degradation of the molecular chains.

The observed dependence of the viscosity of the course of the absorbed dose (50-500 to Gy) in nano compositions can be attributed to small crosslinking rubber. The intrinsic viscosity ($[\eta]$) in a system increases: (NBR-ZnO=0.3; NBR-ZnO-DSCB=0.8) with increasing doses above 500 kGy intrinsic viscosity falls. It should be noted that not require a high dose for the conducting radiation crosslinking nanocomposites. Probably, the DSCB and nano-ZnO played active role by the combined act in the process.



1. Sample obtained by heat action (423K x 40')
2. The sample was obtained by irradiation (D = 500 kGy)

Figure 1. Fourier transform infrared spectroscopy (FTIR) system of nanocomposite filled thermal and irradiated nanosamples (NBR + DSCB + ZnO)

Table 1. The basic properties and structural parameters of the thermal (426K x 40') and radiation (D = 500 kGy) samples of activated zinc oxide nanopowders

Content of nanopowders of zinc oxide in rubber, mass/part	Part of sol fraction, (S)%	The average molecular weight of the fraction sol, (M_{n_s})	The average molecular weight of the chain section between the cross-links ($1/M_s \times 10^5$), mol/sm ³	The average molecular weight of the cross-linked polymer ($1/M_{n_r}$), mol/sm ³	Tensile strength, (σ_r), MPa	Elongation at break, (ϵ), %
T=426K x 40'						
0	0,40	5140	3,0	0,77	6,0	800
1,8	0,46	5300	6,2	1,36	8,0	790
2,6	0,52	5780	7,5	1,27	10,0	780
3,6	0,54	6320	8,4	1,18	11,0	770
4,2	0,72	6900	9,1	1,11	12,0	760
D=500 kGy						
0	0,28	2750	0,8	0,20	-	-
1,8	0,31	3190	1,9	0,55	-	-
2,6	0,38	3330	2,7	0,40	3,0	980
3,6	0,44	3550	3,2	0,34	4,0	920
4,2	0,49	3720	3,4	0,27	6,0	810

Laws of the kinetic theory of high elasticity does not apply to the characteristics of the structural parameters of the vulcanization of elastomers grid due to reduced conformational entropy of the molecular chains in the polymer system - OM, due to the interaction of the chains with the surface of the metal oxide.

Correction equations of the theory of high elasticity made it possible to use them primarily for qualitative evaluation grid density of elastomer with nanooxide [29]. In applying the equilibrium voltage method [30], taking into account factors increase the deformation of the rubber phase or equilibrium swelling with the introduction of the amendment was surfing the obtained values of the concentration grid circuits nano oxide in all elastomers than comparable vulcanizates without nanopowders.

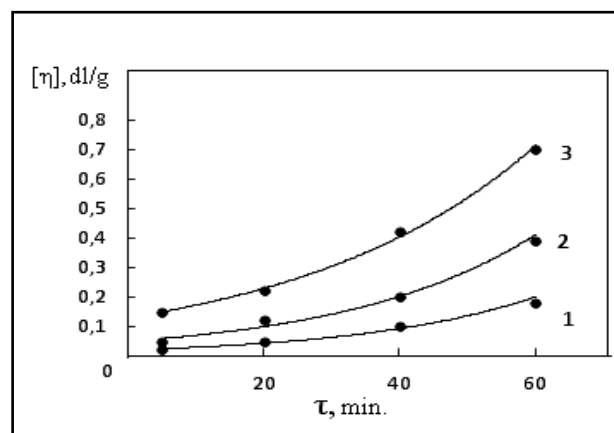
To determine the concentration of crosslinks in elastomers can be used static theory of the structure of nets position [31], not taking into account the physical condition of the polymer chains. According to this theory, the mesh structure depends on the type formed by crosslinking sites, the molecular weight and nature of the molecular weight distribution (MWD) of the polymer. Such an attempt was made earlier [32], but it is not possible to determine the concentration of mesh chains and the degree of degradation of the molecular chains in rubber vulcanizates oxide.

The study of swelling of the films in solution (toluene) gave the opportunity to study the structure of the grid nanocomposites (NBR-ZnO-DSCB) obtained after heating (423K x 40') and irradiation (D=500 kGy). Presented in table 1 shown that the nanocomposite sol fraction depending on the degree of nano ZnO and increases vulcanizate OM content in the number of grid lines ($1/M_s$) per unit volume of crosslinked polymer increases. A number of cross-linked molecules ($1/M_{n_r}$) decreases due to consumption of double bonds, but the formation of cross-links in one sm³ vulcanizate.

When performing calculations irradiated nanocomposites used the procedure described in [32]. For each composition of the polymer films was calculated measurement value of the structural parameters.

Number sol fraction soluble even at 500 kGy radiation doses of 0.49% is reached and above 500 kGy polymer becomes completely insoluble, indicating that crosslinking of the polymer molecules chains. It follows from these results that with increasing content nano zinc oxide nanopowder number of grid lines ($1/M_s$) increases according to their activity.

It is known [33] that the ability of activating metal oxides (OM) depends not only on the size of the powders, but also on the type of organic crosslinking agent. Therefore, it was of interest to elucidate the role of nanopowders OM differ in activity by crosslinking with NBR DSCB. Many researchers [34] activates the ability nanooxides associated with their interaction with the hydrogen chloride released during the reaction with the rubber cross-linking agent.

**Figure 2.** The kinetics of change of the intrinsic viscosity polymer systems of warm-up duration: 1. NBR; 2. NBR-ZnO; 3. BNK-ZnO-DSCB

This particularly applies to special-purpose rubbers crosslinking disulphochlorid benzene aromatic compounds. However, in the case of butadiene-nitrile rubbers role nanopowder metal oxides, probably much more complicated, because some of them may react with nitrile polymer [35]. The results showed that crosslinking NBR with DSCB, absence ZnO nanopowders is slow and crosslinking degree is not high (Fig. 2).

Accelerating crosslinking polymer systems NBR-DSCB observed only in the presence of nano ZnO, characterized by the highest reactivity, the number of crosslinking NBR molecule increases approximately two time (Fig. 3).

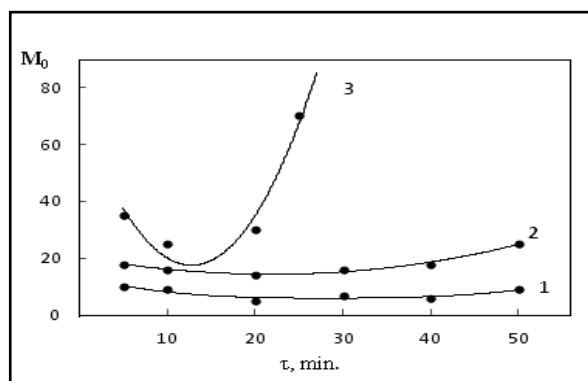


Figure 3. Kinetics of change in viscosity nanocomposites NBR-DSCB and DSCB+NBR+ZnO in the time depended crosslinking (423K x 40') 1. NBR; 2. NBR-DSCB; 3. NBR-DSCB+ZnO

Obviously, when the NBR of the nanoparticles is crosslinked, ZnO plays the role of a hydrogen chloride acceptor, which is released upon the interaction of the NBR and the crosslinking agent (DSCB). This is confirmed by the formation of zinc chloride in the system during the heating of the nano compositions (Table 2.).

Table 2. Change in zinc chloride content when NBR is crosslinked with Benzene disulfo chloride (DSCB)

Time of stitching, min	The content of zinc chloride when NBR is crosslinked with DSCB
10	0,57
20	0,70
40	0,78
60	0,80

Removal of hydrogen chloride from the sphere of reaction shifts the latter side to crosslinking and is one of the reasons for the formation of cross-links in the system.

The effect of nano-sized powders on the thermal properties of the NBR as a whole has not yet been studied sufficiently. One of the reasons for this is the complex production of metal powders.

For elastomeric mixtures based on NBR used in aggressive media, the main parameters are the stability of the temperature and hydrocarbon media (fuels and oils) in the temperature range -40 + 423 K. Relations with this, of particular interest are the study of the thermal characteristics of nanocomposites. The thermal characteristics of the

mixtures were determined on a TGA/DSC derivatograph with an IR spectrum at a heating rate of 15 Grad / min.

Analysis by the derivatograph of nanocomposites (NBR+DSCB+ZnO) shows that the beginning of the mass loss in the sample decreases. However, the reaction rate of the decomposition of the nano composite is small and the time during which the sample from the mixture of rubbers loses 10% of the initial mass. Almost does not differ from the indicators for samples from pure NBR. The release of additional heat when the sample is heated allows one to assume the possibility of the reaction of the main chain of NBR on the residual double bonds.

Table 3. Change in zinc chloride content when NBR is crosslinked with disulfo chloride benzene (DSCB)

The content of nanopowders of ZnO in rubber, phr	The temperature of the onset of mass loss, K	The loss temperature is 10% of mass, K	Maximum temperature of decay, K
0	661	699	721
1,5	561	623	683
2,5	564	624	685
4,0	561	603	688

According to the existing concepts [36, 37], the necessary condition for the enhancement of nanocomposites is the formation of a structural grid (chain structures) of carbon black (CB), on which the molecular chains of rubber are adsorbed and the spatial grid is formed in nanocomposites.

The electrical dielectrics behavior in an electric field is determined by such characteristics as specific properties of nanocomposites ultimately depend both on the nano-sized particles of both structural grids, and on the interaction of these nets, i.e. From the number of bonds: NBR-DSCB-ZnO-F324 (CB).

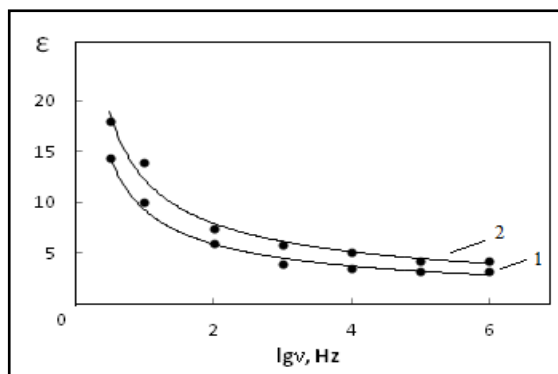
Elastomeric materials obtained by thermal and radiation cross-linking are typical electric strength, dielectric constant and dielectric loss.

The study of the electrical properties of nanopolymer samples is necessary to evaluate their performance properties. In addition, this makes it possible to establish the effect of chemical structure and heat on nanocomposites on these properties.

As a result of the study of the samples, typical frequency dependences of the dielectric constant were obtained (Fig. 4), from which it follows that the dielectric permittivity of filled nanocomposites varies in the low-frequency region, it is evidently related to displacement on the surface of the samples, which leads to cross-linking of the mesh chains.

This suggests that the observed phenomena can be explained by the nature of the interaction, both between the filler particles and between the fillers and the polymer matrix.

The temperature dependence of the dielectric loss ($\text{tg}\delta$) increases with rising temperature. In the future, the increase in temperature does not have a significant effect on the form of the temperature dependence of $\text{tg}\delta$. (Fig. 5 the curve)



1. Sample obtained by heat action (423K x 40')
2. The sample was obtained by irradiation (D=500 kGy)

Figure 4. Frequency dependences of the changes in the permittivity of filled nanocomposites (NBR + DSCB + ZnO + F324)

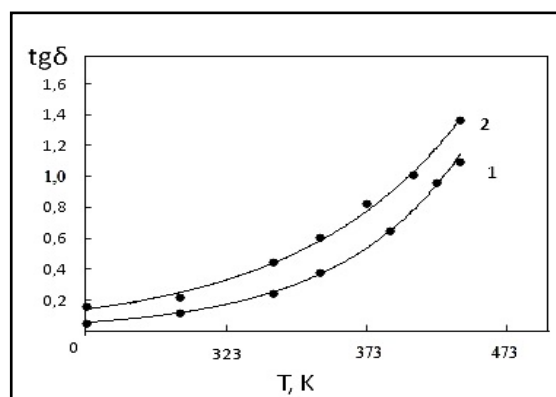


Figure 5. Temperature dependence of dielectric losses filled thermal

The analysis of the temperature dependences of the conductivity of stress-conducting nanocomposites that are strained in different degrees leads to the conclusion that a significant value in determining the properties of nanocomposites not only has the particle size of ZnO, both between ZnO particles and between the filler and the polymer matrix.

4. Conclusions

Thus, polymeric nano composite materials were prepared in the presence of an additive of a nanoscale zinc oxide powder containing a crosslinker of DSCB and carbon black. Introduction of nanopowders zinc oxides causes an acceleration of the process of NBR crosslinking. The highest binding speed of nanocomposites and the greatest number of cross-links in the mixture is achieved by the introduction of DSCB.

In all probabilities, their ability to polarize the double bond of both the polymer and the cross-linking agent, which leads to the formation of metal-containing polymeric nanocomposites characterized by a uniform, narrow dispersed distribution of ZnO nanoparticles 20-25 nm in the polymer matrix. At the same time, it should be noted that the activating ability of the ZnO nanopowder is influenced by the specific surface area.

The data obtained indicate that when the nanopowder ZnO is used together with technical carbon in the polymer, the mechanical properties of the nanocomposites. The reason for the increased strength can be as a large density of the polymer mesh, and the presence of transverse bonds of adsorption nature.

These effects of gamma irradiation on the nature of the radiation-chemical yield of crosslinking in nanocomposites are not large compared to thermal crosslinking.

The carried out researches allow to assert, that change of size of dielectric constant (ϵ) and dielectric loss ($\text{tg}\delta$) depends on a density of a grid of nanocomposites of a specific surface of technical carbon, ZnO and an irradiation dose.

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