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NEW MATERIALS, PROCESSES,  
AND TECHNOLOGIES

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## Radiation-Modified Polytetrafluoroethylene: Structure and Properties

S. A. Khatipov, E. M. Konova, and N. A. Artamonov

*Karpov Scientific and Research Institute of Physical Chemistry,  
ul. Vorontsovo Pole 10, Moscow, 105064 Russia  
e-mail: khatipov@cc.nifhi.ac.ru*

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**Abstract**—The scope offered by radiation technology for polytetrafluoroethylene modification was discussed. New modified materials possess enhanced antifriction and compressive parameters and are suitable for different commercial applications.

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Polytetrafluoroethylene has numerous commercial and technical applications, especially as sealing and tribotechnical material [1–5]. Low friction coefficient with respect to steel, as well as self-lubrication properties make it irreplaceable in manufacturing “dry” friction units. However, low wear resistance and high creep values prevent neat polytetrafluoroethylene from this application. Typically, polytetrafluoroethylene is used in compositions with fillers (coke, graphite, carbon fiber, fiberglass, metals and metal oxides, ultradisperse compounds, etc.) taken in various amounts and combinations [4, 5]. The wear of these composite materials is 2–3 orders of magnitude lower compared to neat polymer.

Up to the present time, preparation of compositions represented the only efficient method of modification of polytetrafluoroethylene with the aim to increase its wear resistance. As to chemical modification of this polymer, these opportunities are extremely limited by its chemical inertness, and the properties of copolymers of tetrafluoroethylene with different perfluorinated compounds are far from those of the compositions. Attempts to increase the wear resistance of polytetrafluoroethylene via radiation-induced modification at room temperature in air and vacuum did not yield practically significant results: The wear resistance increased tens of times, but in parallel the mechanical characteristics of the polymer were significantly deteriorated [5].

The major factor limiting (and in most cases preventing) the use of polytetrafluoroethylene, both neat and as part of compositions, in items intended for special technical applications under radiation exposure is an extremely low radiation resistance [1, 5–7]. Radiation exposure of polytetrafluoroethylene causes its degradation and significant reduction of strength at fairly low, from the radiation chemistry viewpoint, absorbed doses (0.1–1 Mrad, which is at least by an order of magnitude lower than the dose that causes degradation of other polymers) [1, 6]. Radiation exposure of polytetrafluoroethylene in melt at 360–380°C underlies its utilization [7]. For this reason, radiation-chemical modification of polytetrafluoroethylene was not considered as a candidate method for improving its physicochemical properties.

At the same time, comparatively recent studies [8–10] showed that radiation exposure of polytetrafluoroethylene at temperatures exceeding the melting point of its crystalline phase (327°C) yields new modifications of polytetrafluoroethylene possessing unusual properties. Extensive research efforts along this line are being currently undertaken. They are of interest both for development of radiation-induced modification methods and for preparation of new promising materials with improved and unique set of properties [11–22].

A broad spectrum of physicochemical methods was applied to elucidate the structure of polytetra-

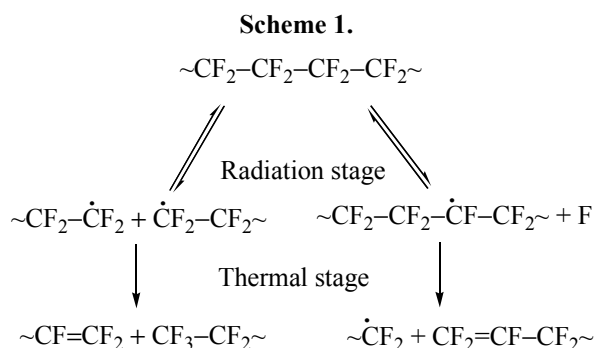
fluoroethylene modifications [12–20]. Those studies interpreted the change in the physicochemical properties of the films as due to formation of a three-dimensional network of cross links of polymer chains. Possible cross linking of polytetrafluoroethylene chains in melt under radiation-chemical exposure was discussed earlier [23–25]. Also, anomalous behavior of polytetrafluoroethylene, associated with an unexpectedly low degree of its radiation-induced degradation in melt, was mentioned in [26].

At Karpov Research Physicochemical Institute, Federal State Unitary Enterprise (NIFKhI), block samples of polytetrafluoroethylene modifications were synthesized by  $^{60}\text{Co}$   $\gamma$ -ray exposure (1.25 MeV) of polymer samples [22]. By contrast to above-mentioned studies, those samples were exposed in the presence of an oxygen impurity dissolved in the bulk of the block samples at fairly low absorbed doses (up to 20 Mrad). Those low doses caused unexpectedly strong modification of the macroscopic properties. Specifically, wear resistances and creep increased by a factor of up to  $10^4$  and up to  $10^2$ , respectively. It was concluded that the dominating role in the phenomena observed is played by morphological changes in the polymer, caused by accumulation of radiation-induced defects in the crystalline phase.

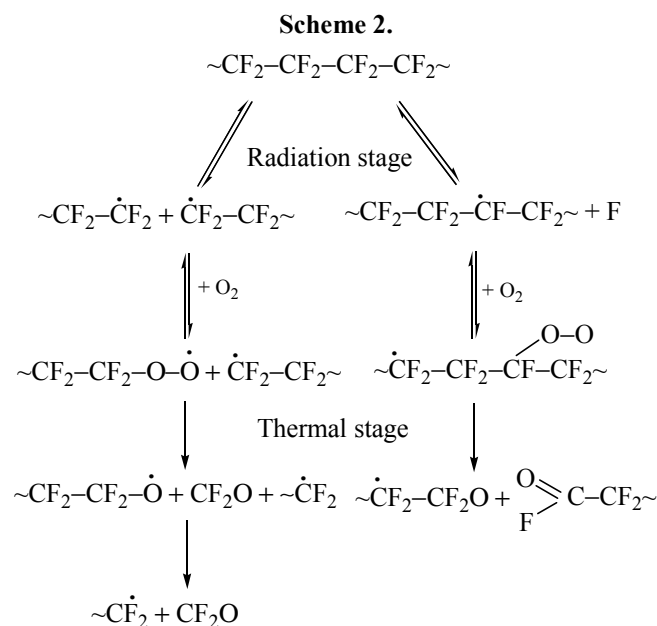
Here, we examined the structure and properties of radiation-modified polytetrafluoroethylene prepared by low-dose radiation exposure near the melting point of the crystallites.

### Structural and Chemical Features of Radiation-Modified Polytetrafluoroethylene

The main primary products yielded by ionizing radiation exposure of polytetrafluoroethylene are terminal ( $\text{CF}_2\text{-CF}_2\text{-}$ ) and middle ( $\text{-CF}_2\text{-CF-CF}_2\text{-}$ ) fluoroalkyl macroradicals resulted from detachment of fluorine atoms and polymer main-chain scission (Scheme 1). The yields of terminal to middle radicals



for radiation modification in a vacuum at room temperature are in a close to 1:10 ratio, and the total radiation-chemical yield is 0.07–0.25 (1/100 eV) [27]. In the presence of molecular oxygen, the dominating process is formation of terminal ( $\text{-CF}_2\text{-CFOO}\cdot$ ) and middle ( $\text{-CF}_2\text{-CFOO}\cdot\text{-CF}_2\text{-}$ ) peroxide macroradicals (Scheme 2) with a close to 1:1 ratio of yields and the total yield of 0.12–0.22 [27].



At room temperature fluoroalkyl and peroxide radicals in the polytetrafluoroethylene chain have very long lifetimes. At temperatures above  $100^\circ\text{C}$  they undergo thermal degradation yielding ( $\text{-CF}=\text{CF}_2$ ) terminal double bonds,  $\text{-CF}_3$  and  $\text{-CFO}$  terminal groups,  $\text{CF}_2\text{O}$  low-molecular-weight gaseous products, and perfluoroalkenes (see Schemes 1 and 2). Thermal degradation of the macroradicals localized near the chain ends yields low-molecular-weight free radicals  $\text{C}_n\text{F}_{2n+1}$  ( $\text{CF}_3\cdot$ ,  $\text{C}_2\text{F}_5\cdot$ ,  $\text{C}_3\text{F}_7\cdot$ , etc.). Reactions of atomic fluorine with the middle and terminal radicals yield middle and terminal double bonds, molecular fluorine, and fluoromethane [1, 27].

Thus, radiation exposure of polytetrafluoroethylene at high temperatures (above  $100^\circ\text{C}$ ) causes accumulation of terminal trifluoromethyl and carbonyl groups, as well as of individual terminal and middle double bonds.

It was convincingly demonstrated that radiation exposure at temperatures above the melting point of polytetrafluoroethylene causes formation of chain branchings ( $\text{-CF}\langle$ ), side branches  $\text{>CF-CF}_3$  and  $\text{>CF-}$

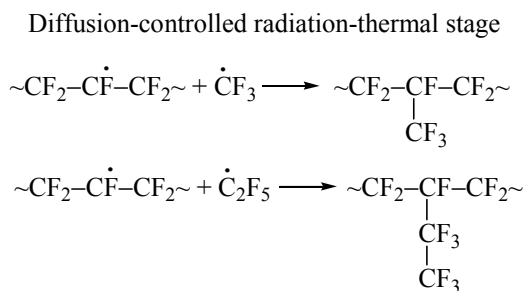
$C_2F_5$ , terminal groups ( $-CFO$ ), and double bonds ( $-FC=CF-$ ,  $-CF=CF_2$ ). Also, the concentration of terminal  $-CF_3$  groups substantially increased because of the polymer main-chain degradation [13–20]. Another group of defects, specifically polyconjugated  $p$ -bonds, is yielded by radiation exposure of polytetrafluoroethylene in melt, as suggested by the appearance of fluorescence in the visible [21].

All the above-listed products, except for chain branchings, side branches, and polyenes, have long been known; they are yielded by radiolysis of polytetrafluoroethylene at room temperature as well, which suggests proceeding of similar radiation-chemical processes. However, by contrast to partly crystallized polytetrafluoroethylene, the radiolysis in melt is more homogeneous not only in the amorphous but also in the ordered meso phase. The reason is that,

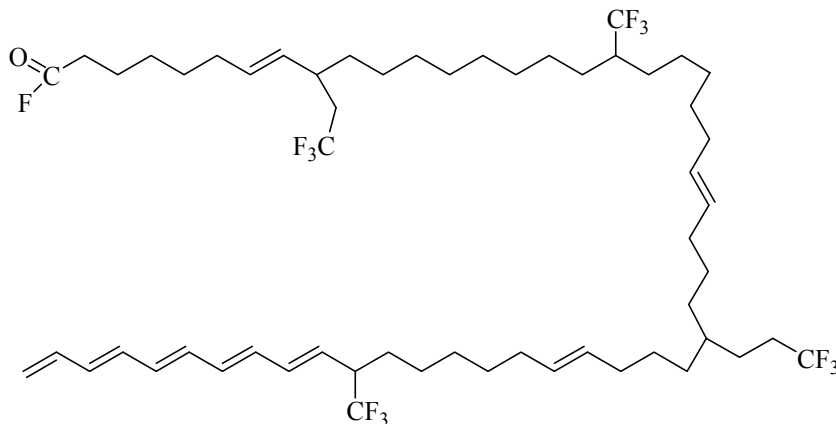
despite high melt viscosity, the gas permeability of the polymer at temperatures close to and above the melting point sharply increases [1]. Low-molecular-weight products have a higher diffusion mobility, for which reason secondary reactions of these products with macroradicals are more highly probable.

Of greatest interest are low-molecular-weight free radicals  $C_nF_{2n+1}$  whose recombination with macroradicals yields chain branchings and side branches (Scheme 3). Formation of polyenes is associated with propagation of conjugation chain via free valence migration and stabilization near the double bond [21]. As a result, accumulation of unsaturated bonds is localized within the selected segment of the polymer chain. Scheme 4 presents the generalized structure of the polymer chain of modified polytetrafluoroethylene.

**Scheme 3.**



**Scheme 4.**



The defects frozen in the crystalline phase after radiation exposure cause loosening of the crystalline core of the particles, as well as a decrease in size and an increase in the shear strain of the crystallites. All this leads to formation of physical engagements of the kinetic segments of the macromolecules, homogenization of the supramolecular structure, and decrease in porosity.

### Characteristics of Radiation-Modified Polytetrafluoroethylene

#### *Tensile Strength*

Given below are results of mechanical tests of standard initial and irradiated polytetrafluoroethylene plates and films prepared by shaving of the initial and irradiated blocks.

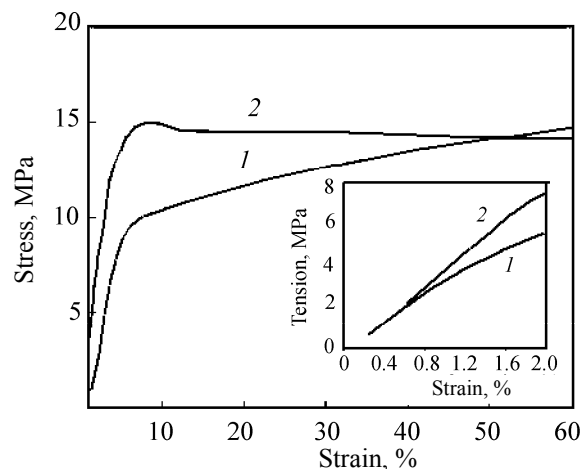
Figure 1 shows the stress–strain curves for the initial and irradiated plates when stretched at a uniform velocity of  $20 \text{ mm min}^{-1}$ . The initial plate (curve 1) exhibits a monotonic nonlinear increase in stress at low and high strains.

The stress–strain curves for the initial polytetrafluoroethylene suggest a lacking prominent fluidity limit and a homogenous strain via spatially uniform shear of the polymer chains. The initial section at low strains also exhibits nonlinear pattern, which suggests a major contribution from plastic inelastic strain. The crystalline domains do not prevent development of such strain because of a low shear strength of polytetrafluoroethylene crystallites [1]. The X-ray diffraction patterns of the 200%-stretched polytetrafluoroethylene plates suggest that, under strain, the degree of crystallinity increases from 59 to 67%. This directly evidences rearrangement of the supramolecular structure in the direction of application of the external load during creep development.

Irradiated polytetrafluoroethylene exhibits essentially different strain–strain curves: There is a maximum corresponding to necking, which is followed by a plateau associated with neck extension (Fig. 1, curve 2). The initial section exhibits a close to linear pattern.

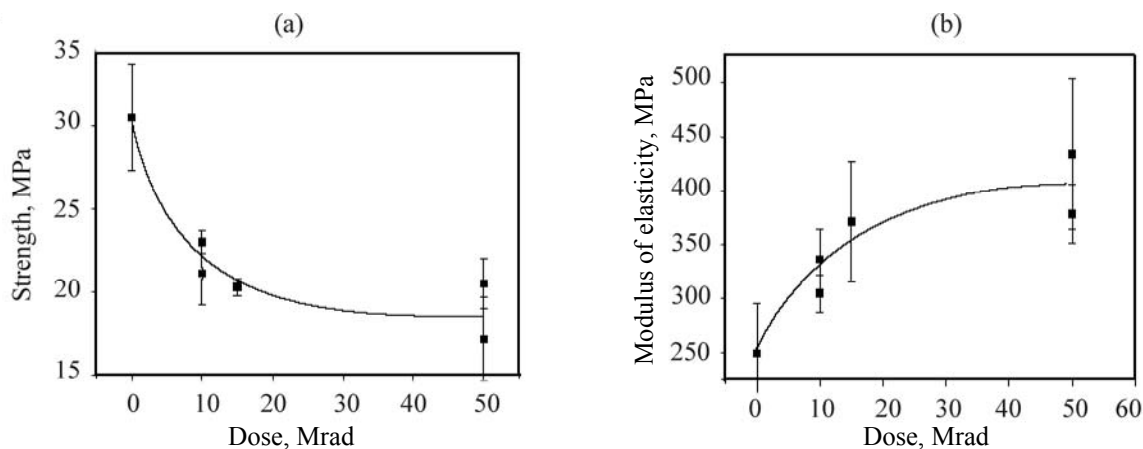
Figure 2 presents the results of tests with the film samples. It is seen that, with increasing radiation dose, the modulus of elasticity estimated from the slope of the strain–stress curves in the initial section (typically at 0.5–2% strains) tends to increase, and the strength, to decrease.

These data suggest that radiation exposure disturbs the perfect crystalline domains of the polymer, thereby

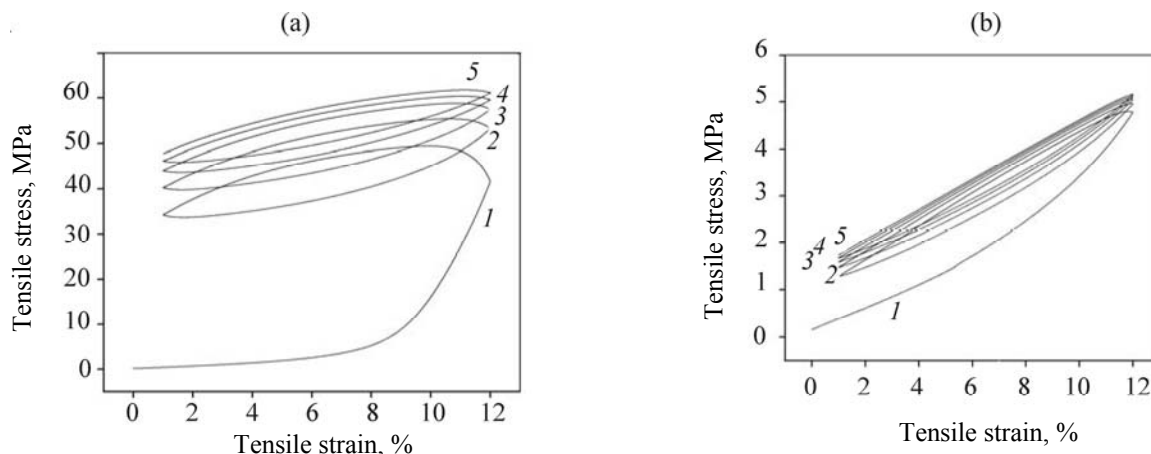


**Fig. 1.** Stress–strain curves for (1) initial and (2) radiation-modified polytetrafluoroethylene plate (3-mm-thick). Stretching velocity  $20 \text{ mm min}^{-1}$ ; absorbed dose 200 kGy.

complicating homogeneous sliding in them. Barriers to creep development in such plastic polymer as polytetrafluoroethylene with a prominent inelastic component of strain cause the modulus of elasticity to appreciably increase. Hindered homogeneous strain favors necking and transition to non-Newtonian flow in the region of the plateau. During stretching the neck narrows till the sample ultimately fails, without spreading over the entire test portion of the sample; the tensile strength is much below that of the initial polymer. Upon achieving the fluidity limit the strain is 7–10% at absorbed doses of 10–50 Mrad. The relative breaking elongation is 100–250%. It should be noted that the tensile strength of radiation-modified polytetrafluoroethylene is close to those typical for commercial polytetrafluoroethylene-based compositions: F-



**Fig. 2.** (a) Strength and (b) modulus of elasticity of polytetrafluoroethylene film (150- $\mu\text{m}$ -thick) vs. absorbed dose. Stretching velocity  $10 \text{ mm min}^{-1}$ .



**Fig. 3.** Strain of (a) initial and (b) radiation-modified polytetrafluoroethylene plate (3-mm-thick) under cyclic stretching. (1–5) Cycle nos.; maximal and minimal load in cycle 12 and 1 MPa, respectively; loading-unloading rate 4 MPa min<sup>-1</sup>. Absorbed dose 20 Mrad.

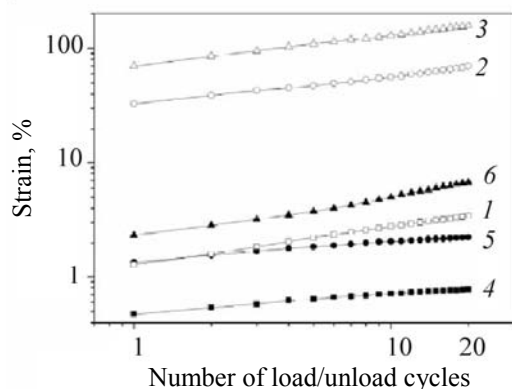
4K20 (KL-1 foundry coke 20%), F-4S15 (alumino-borosilicate glass powder 15%), and F-4UV15 (Gralen carbon fiber 15%).

#### Tensile Creep

Creep examinations of radiation-modified polytetrafluoroethylene consisted in measuring the strain of samples under cyclic loading (Fig. 3). The strain–stress curves for the initial polytetrafluoroethylene (Fig. 3a) exhibit an ascending spiral pattern. Upon the maximal strain is achieved in the first loading cycle and the load is decreased to a minimum, the sample does not regain its initial size, i.e., inelastic

(or slowly reversible) plastic strain is observed. Each subsequent loading/unloading cycle causes both the total strain and its irreversible component to increase. With increasing number of cycles the reversible component of strain decreases from 20% in the first to 15% in the second cycle. The reversible strain accounts for 25% total strain.

The behavior of the irradiated plate under cyclic loading is basically different: Both the total strain and its irreversible component (Fig. 3b) tend to considerably decrease. The reversible strain is weakly dependent on the number of cycles, and its proportion in the total strain increases to 72%.



**Fig. 4.** Strain of (1–3) initial and (4–6) modified polytetrafluoroethylene plates (3-mm-thick) vs. number of loading/unloading cycles (in logarithmic coordinates). Absorbed dose 20 Mrad. Maximal and minimal load in cycle 12 and 1 MPa, respectively; loading/unloading rate 4 MPa min<sup>-1</sup>.

Figure 4 and Table 1 present the measured inelastic strains for 20 cycles at different maximal loads in the cycle. Figure 4 shows that strain virtually linearly varies with the number of cycles in all cases. Hence, under the actual test conditions the time dependence of the strain adheres to a power law,  $\varepsilon \sim t^\alpha$ , and the creep rate decreases with time as  $\dot{\varepsilon} \sim \alpha t^{\alpha-1}$ . As seen from Table 1, for the polytetrafluoroethylene modifications examined the creep rate decreases 4-fold relative to the initial material at a load of 4 MPa, and nearly 30-fold, at a load of 14 MPa. The parameter  $\alpha$  changes less considerably, and at the load of 14 MPa it is higher for the irradiated than for the initial polytetrafluoroethylene. The reason is that, for the modified sample, 14 MPa is close to the breaking load, while for the initial plate it makes only 50–60% of the breaking strength. At identical relative loads the creep of the irradiated material is much lower than that of the initial polytetrafluoroethylene.

**Table 1.** Creep rate of the initial and radiation-modified polytetrafluoroethylene plates in relation to the maximal load in the cycle. Plate thickness 3 mm; absorbed dose 20 Mrad. ( $\alpha$ ) Power index in the time dependence of the strain under cyclic loading:  $\varepsilon \sim t^\alpha$ , and  $V_{20} = (d\varepsilon/dt)_{20}$ , creep rate by the end of cycle no. 20

Parameter	Initial sample				Modified sample			
	load, MPa							
	4	8	12	14	4	8	12	14
$\alpha$	0.17	0.32	0.25	0.27	0.06	0.17	0.17	0.36
$V_{20}$ , %/min	0.08	0.43	14.78	60.04	0.02	0.07	1.35	2.25

With increasing absorbed dose the creep rate for the modified polytetrafluoroethylene tends to monotonically decrease, specifically, 27-fold relative to the initial sample at the dose of 20 Mrad and 52-fold at the dose of 40 Mrad. However, an increase in the dose also causes the strength of the polymer to decrease. Hence, modification is most efficient at intermediate radiation doses.

#### Compressive Creep

Compression tests validated major decrease in creep and appearance of the elastic component of strain in radiation-modified polytetrafluoroethylene. In these tests we determined the total strain  $\varepsilon_S$  of cylindrical samples for 24-h load exposure, as well as the residual (irreversible) component of strain  $\varepsilon_{\text{irrev}}$  24 h after the load was removed. At a load of 14 MPa under 24-h exposure the modified rods exhibit virtually totally elastic strain. The residual strain is low, 2% only, against 75% for the initial material (Table 2). At a load of 28 MPa these parameters were estimated at 62 and 22% for the initial and modified polytetrafluoroethylene samples, respectively.

Thus, the creep rate for the modified polytetrafluoroethylene under compression remains much lower than that for the initial sample.

#### Thermomechanical Properties

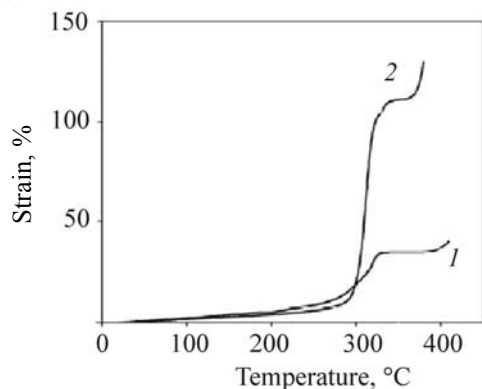
The above-said suggests that a decrease in creep and appearance of elastic response in radiation-modified polytetrafluoroethylene can be associated with accumulation of radiation-induced defects in the crystalline phase. In this connection, it was of interest to examine the temperature dependence of strain for the modified polytetrafluoroethylene samples at temperatures close to and above the melting point of the crystallites. We obtained such dependences for 100- $\mu\text{m}$ -thick film samples at a heating rate of 2–3 deg  $\text{min}^{-1}$  (Fig. 5).

The run of the thermomechanical curves suggests a sharp increase in strain with temperature approaching the melting point of the crystallites. For modified polytetrafluoroethylene the strain jump strongly exceeds that for the initial material. After the melting point is passed through, a plateau is observed: The strain is temperature-independent. At close to 400°C temperatures the thermomechanical curves exhibit a second jump associated with development of breaking strain.

Thus, by contrast to the modified samples in the crystalline state, for which the total strain at a given load is much lower than that for the initial samples, the total strain of the modified sample in the melt region is much higher. To determine the ratio of irreversible to reversible components of the strain, we recorded thermomechanical curves under sample unloading. Figure 6 shows the total and irreversible strains vs. the radiation dose. It is seen that an increase in the total strain of the modified samples is accounted for primarily by the reversible component. With increasing radiation dose the irreversible strain in the melt region changes insignificantly, and the reversible strain increases substantially. For the initial samples

**Table 2.** Compressive strain of the initial and modified polytetrafluoroethylene sample. Samples are rods 10 mm in diameter and 20 mm in height. Irradiation dose 20 Mrad,  $\varepsilon_S$  is total strain at 24-h load exposure;  $\varepsilon_{\text{irrev}}$ , irreversible strain 24 h after removing the load; and  $\varepsilon_{\text{irrev}}/\varepsilon_S$ , proportion of irreversible strain

Parameter	Initial sample		Modified sample	
	load, MPa			
	14	28	14	28
$\varepsilon_S$ , %	16	40	8	23
$\varepsilon_{\text{irrev}}$ , %	12	25	0.2	5
$\varepsilon_{\text{irrev}}/\varepsilon_S$ , %	75	62	2	22



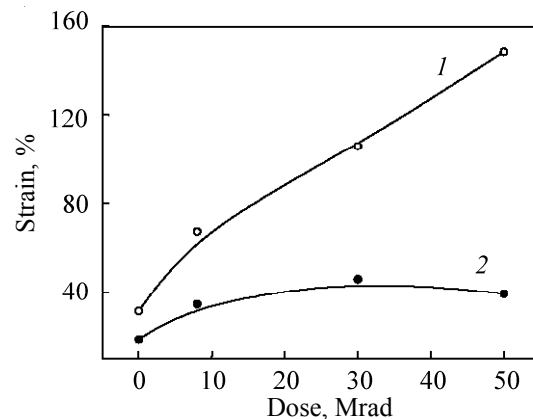
**Fig. 5.** Temperature dependence of strain for (1) initial and (2) radiation-modified polytetrafluoroethylene film. Film thickness 100  $\mu\text{m}$ ; load 0.625 MPa; absorbed dose 20 Mrad.

the reversible strain was estimated at 40%, and for modified samples, at 80%. Thus, in the molten state the modified samples behave like vulcanized rubbers, specifically, the reversible strain is fairly high, over 100%.

These data suggest that radiation-induced defects cause strengthening of the intermolecular bonding of the polymer chains in the crystalline domains of the polymer. In particular, cross links of the polymer chains can be formed. However, it was found earlier [14] that the yield of cross links at 100 Mrad is not high (0.2/100 eV) and tends to decrease with decreasing dose (at 50 Mrad cross links were not detected). Also possible is scission of the polymer chains into trifluoromethyl and carbonyl terminal groups, unsaturated bonds, and short-chain side branches [14, 16–20]. Such defects make chains with rigid helical configuration more flexible, break the symmetry of the crystallites, and complicate sliding of the chain segments relative to one another and shear strain of the crystallites as a whole.

#### *Wear Resistance of Radiation-Modified Polytetrafluoroethylene*

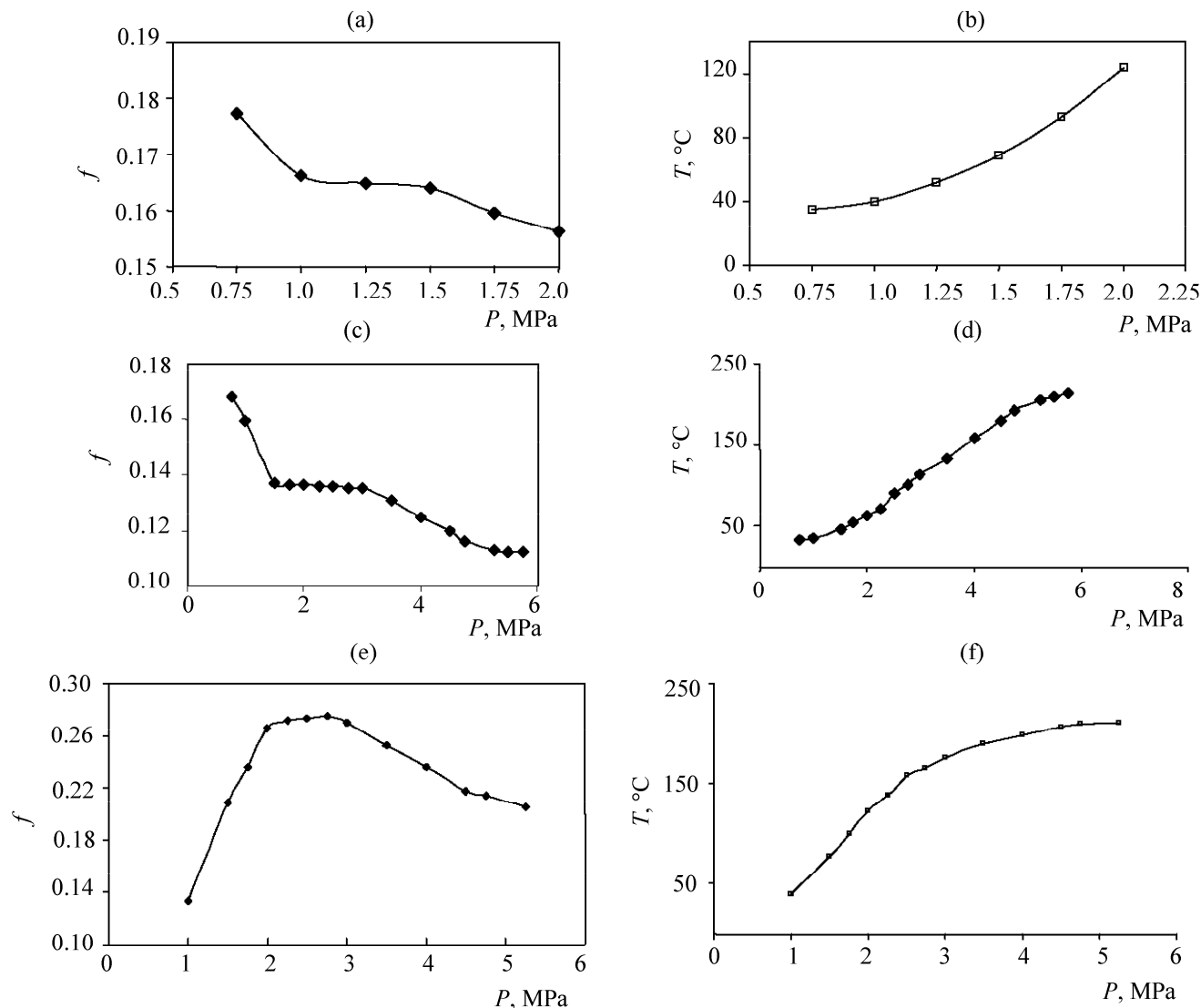
Radiation-induced modification has the most prominent effect on wear resistance. We carried out wear resistance tests by pin-on-disk kinematic scheme (at 1  $\text{m s}^{-1}$  velocity and 2.5 MPa load) and revealed the following. The wear of radiation-modified polytetrafluoroethylene samples decreases by a factor of  $10^4$  relative to the initial polymer and is lower than those of extensively applied polytetrafluoroethylene-based compositions F-4K20, F-4UV15, F-4S15, and F-4KS2 [4, 22].



**Fig. 6.** Strain of the polytetrafluoroethylene film (100  $\mu\text{m}$ ) vs. absorbed dose at temperatures above the melting point: (1) total strain and (2) irreversible component of strain.

One of the performance characteristics of the material in a friction unit is the  $pV$  factor. It is believed that, for numerous materials, the load multiplied by the sliding velocity in the friction region is a universal value over a broad range of values. Hence, knowing the limiting value  $pV_{\text{lim}}$  it is possible to find the limiting values of the velocity or load, depending on which parameter is set by the specific operation conditions. For determining  $pV_{\text{lim}}$  the load applied to the tested sample or the velocity in the friction region is gradually increased, and this is paralleled by recording the running values of temperature in the friction region and friction coefficient. Experiments are run till sample fails, as ascertained from a sharp increase in wear. We carried out such tests with polytetrafluoroethylene and its modifications in the bush-shaft mode at 1  $\text{m s}^{-1}$  sliding velocity.

Figure 7 shows that the friction coefficient and temperature in the friction region vary with the load for the radiation-modified polytetrafluoroethylene, as well as for the initial and radiation-modified F-4KS2 (filler Cobalt Blue, 2%) compositions. (We omit the data for the initial polytetrafluoroethylene because, under the experimental conditions, it fails at  $<1$  MPa loads). It is highly indicative that the radiation-induced modification of F-4KS2 composition causes substantial decreases in the friction coefficient and temperature in the contact area and nearly three-fold increase in  $pV_{\text{lim}}$ , which was estimated at 2.0 and 5.75  $\text{MPa m s}^{-1}$  for F-4KS2 and its modification, respectively. For radiation-modified unfilled polytetrafluoroethylene  $pV_{\text{lim}}$  increases to values characteristic for the best antifrication compositions, and the temperature in



**Fig. 7.** Friction coefficient  $f$  and temperature in the contact area vs. load  $P$  for (a–d) (a, b) initial and (c, d) radiation-modified F-4KS2 composition and (e, f) radiation-modified polytetrafluoroethylene. Absorbed dose 20 Mrad.

the friction region at comparable loads is much lower [28].

#### *Corrosion Activity of Radiation-Modified Polytetrafluoroethylene*

Of much importance for modified polytetrafluoroethylene intended for antifriction and sealing applications is interaction with the counter-body surface. Samples shaped as tapes (thickness 1 mm, size 30×70 mm), prepared by shaving of the modified polytetrafluoroethylene block (radiation dose 20 Mrad), were subjected to corrosion tests in contact with various metals. Eight testing cycles were carried out; each cycle involved heating of the sample to 60 and

250°C and keeping for 12 h, followed by treatment in a moisture chamber for 6.5 days.

Visual inspection of the surface of the sample after the tests (Table 3) showed that, when the metal materials contacted modified polytetrafluoroethylene at 60 and 250°C, no corrosion enhancement was observed for AMG-6M aluminum alloys, plated D16AT, D16AT with anode-oxide coating, chemically passivated Cr18Ni10Ti stainless steel, nonplated and chromoplated 30CrMnSiA structural steel, and OT4-1 titanium alloy.

Thus, the chemical stability of polytetrafluoroethylene remains virtually unaffected by modification,



**Table 3.** Results of corrosion tests for alloys in contact with radiation-modified polytetrafluoroethylene (radiation dose 20 Mrad)

Sample tested	Sample surface condition after tests	
	control surface	surface contacting the polymer
Tests at 60°C		
Aluminum alloy AMG-6M, nonanodized	Slight uniform corrosion	Slight uniform corrosion, pittings
Aluminum alloy D16T, nonplated, anodized	No changes	No changes
Aluminum alloy D16T, plated	Mirror surface fogging up	Mirror surface fogging up
Aluminum alloy D16T with anode-oxide nichrome coating	No changes	No changes
Cr18Ni10Ti stainless steel, chemically passivated	No changes	No changes
30CrMnSiA structural steel	Single pittings and caverns up to 1 mm	Single pittings and caverns up to 1 mm
30CrMnSiA structural steel, chromoplated	No changes	No changes
Titanium alloy OT4-1	No changes	No changes
Tests at 250°C		
	No changes	No changes
30CrMnSiA structural steel	Single pittings and caverns up to 1 mm	Single pittings and caverns
30CrMnSiA structural steel, chromoplated	No changes	No changes
Titanium alloy OT4-1	No changes	No changes

and modified polytetrafluoroethylene, like its initial analog, is suitable for application at temperatures up to 250°C in contact with stainless steels, chromoplated structural steels, and aluminum and titanium alloys.

### CONCLUSION

Radiation-chemical treatment is promising for modification of polytetrafluoroethylene. Radiation-modified polymer has improved performance characteristics and compares favorably with composite materials in antifriction and sealing applications.

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