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Methods of probe microscopy in the study of topography and elastic properties of cold-resistant elastomers

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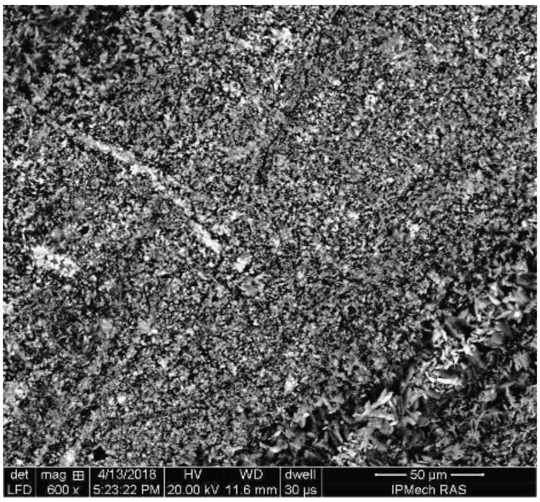
Expansion of the range of manufactured elastomers is an important task for the national economy. These materials must correspond to different operating conditions. In particular, the actual problem remains the modernization of rubber, operated in the conditions of the North Territories. One way to improve the properties is to add various modifiers to the base composition of the elastomers. In the present work, composites based on ER (epichlorohydrin rubber), which are intended to be used as seals in machine units operating at low temperatures, have been studied. The modifiers of rubber were carbon nanotubes (CNTs), added to the base composition in a different percentage.

The operation mode was modeled by tribological tests carried out on a UMT-2 friction machine under the following conditions: contact pressure varied from 0.1 to 0.3 MPa, the slip velocity varied from 1 to 100 mm/s, the bulk temperature of the test samples ranged from -25 to 22ºC

The most important estimate is the change in the surface of rubbers during operation. In this paper, we propose complex methods for studying the surface, including not only studies of topography, but also evaluation of surface properties.

The surface of the samples before and after friction tests was examined on a scanning electron microscope (SEM) - a FEI Quanta 650 device was used, operating in a low vacuum mode, using secondary and back-reflected electron detectors. Images obtained for the sample with 2 mass parts of CNTs are presented in Figure 1.

It can be seen that the topography of the sample after friction-tests has changed, areas have appeared that differ in color. However, the X-ray spectral analysis did not show any significant changes in the composition after the tests. The areas on the sample after friction-tests, differing in color, also have a close elemental composition. (It is possible that the difference in color is due to the difference in the emission parameters of the regions caused by uneven friction).

(a) 

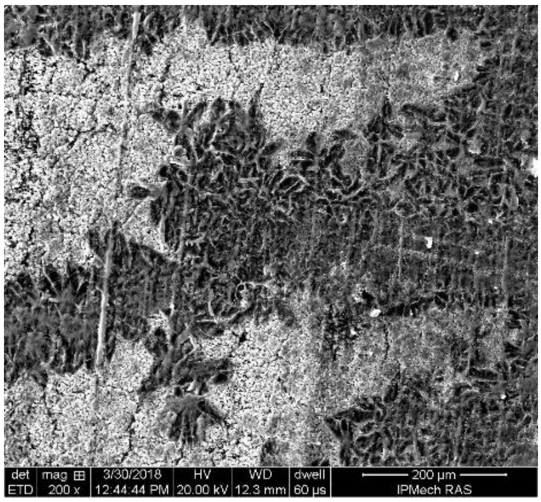
(b) 

Figure 1. SEM images (secondary electrons) of the sample surface with 2 m.p. CNT: (a) original sample, (b) sample after tribo test.

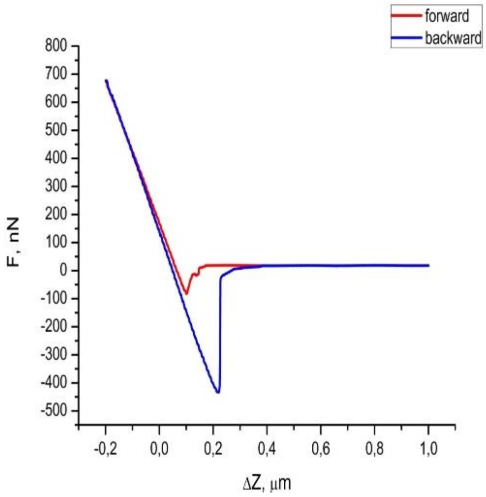


Figure 2. Force-distance curves (samp-2mp CNTs).

Table 1. Visco-elastic properties of Samples with different parts of CNTs before and after friction tests

№	ER+ CNTs	F_{ADH}, nN		E, MPa		A, nA	
		before	after	before	after	before	after
1	0 mp	158,1	37,8	111,9	76,5	2,9	4,4
2	0 mp	71,8	41	92,8	82,4	2,3	4,2
3	0,1 mp	194,1	206,3	57,2	44,4	2,6	3,5
4	0,3 mp	324,2	203,6	584,4	123,6	2,4	3,8
5	1,0 mp	139	322	309,7	144	3,0	3,9
6	2,0 mp	89,7	136	354,8	197,4	3,2	4,2
7	10 mp	238,3	99,8	2511,9	197,1	2,6	3,3

The main method of investigation was probe microscopy. Surface topography was studied at the device "Smart SPM-TM" AIST-NT (in the tipping mode, probes of the fpN10 series). The surface properties were examined on a NTEGRA Prima NT-MDT instrument (HA-NC ScanSens probe). To evaluate the adhesion, the methods of obtaining force curves (force-distance) were used; Young's modulus was estimated from the slope of the graph. Visco - elastic characteristics were studied by the method of "Power modulation". The results of the surface investigation by the SPM method are presented in Fig. 2 (for a sample with 2 mass parts of CNT, before friction-tests) and in Table. 1 (data are given for all samples before and after the tests).

The analysis of the obtained results allows making a number of conclusions:

Adhesion forces: the addition of CNTs leads to an increase in adhesion: in samples without CNTs, adhesion is small enough, adding even 0.1 parts of CNTs leads to a sharp increase in adhesion strength. However, a clear dependence of adhesion on the quantity of CNTs could not be identified. The same pattern is observed for samples after friction tests. We note that it was not possible to reveal a general pattern for the effect of friction on adhesion.

Elastic characteristics:

Young's modulus, measured from the slope of the curve: for samples without CNTs and for samples with a small value of CNTs (up to 0.1 m.p.) Young's modulus is relatively small, but with an increase in the CNTs content (starting from 0.3 m.p.) there is a sharp increase in the Young's modulus. For this concentration range, (as for the case of adhesion forces), the dependence on the number of CNTs was not revealed. After friction tests, the Young's modulus decreases for all samples.

Method of Power Modulation: The results of studying the elasticity by the modulation of force indicate that the elasticity measured by this method differs little for different samples. However, it can be seen that, after friction tests, in all cases a noticeable increase in the rigidity of the sample is observed.

In general, this investigation showed that modification of the basic composition of rubber by nanotubes significantly changes the properties of rubber. At the same time, it should be noted that the conclusions obtained do not make it possible to establish an unambiguous correlation between the elastic properties expressed in terms of the Young's modulus and the elastic parameters determined by the modulation method of force. Obviously, additional experiments are required here.

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