The testing methods for anti-icing properties of ultra-thin fluoropolymer coatings

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Abstract: The paper discusses the testing methods of ultra thin film-forming fluorocontained polymer compositions, which form the surface modifying structures, long-life coatings and nanosize films, giving the treated surface anti-icing, tribological and hydrophobic properties.

1. Introduction
The world experience in the use of anti-icing film-forming compositions shows the breadth of the use of organosilicon (hydrophobic) substances, and anti-adhesives – fluorinated substances and polymers in combination with synthetic additives and inhibitors, which give a standard protective film on the surface. The use of modern film-forming lubricant compositions to obtain an ultra-thin (nanoscale, up to 100 nm thick) protective coating that preserves the geometric and electrical parameters of the surface, while reducing surface energy, friction, adhesion (particle sticking) in the absence of corrosion activity, aging, and wear can be characterized by the synergy of the resource-saving effect, which is the most important task of preserving technical characteristics, durability, and reliability.

Indeed, in some cases, fluoropolymers can be components of anti-icing film-forming compositions, since they not only have antifriction properties (reducing the friction of ice and snow), but also hydrophobic and anticorrosive properties. The research and application of fluoropolymers and compounds is a promising direction, since the molecules of fluorinated film-forming compositions in some cases can bind to the surface by chemisorption forces, which provides a long-term effect from two weeks to three years.

2. Research and analysis
The object of the study was the study of anti-icing coatings designed to reduce the adhesion of ice to cables. The following types of de-icing coatings are considered: "FLUORA-C"; "SNEGOTEK-1"; "FLUORA-D"; "Electronik A No. 50 11/20"; “Fluora CF”; "SFK-20K”.

The aim of the work is to qualitatively assess the adhesion of ice to samples of cables with and without insulation, on which the coatings indicated above are applied.

The research is carried out by conducting experiments on the separation of cables from samples with an anti-icing coating applied and without such ice frozen on their surface and comparing the obtained load values. The experiments were conducted in an accredited ice experiment pool. To study the adhesion of ice to current-carrying cables in isolation and without it, a test program was developed that allows, under specified conditions of exposure to low temperatures, to freeze samples in ice for subsequent loading and to determine the force when ice breaks off. Cables without insulation have a complex curved surface formed by the helical laying of individual wires.
Insulated cables have a smooth, rough surface. Tables 1-2 show the implemented test program for the purpose of studying the adhesive strength of ice frozen on the surface of cables with and without an anti-icing coating.

Table 1. Test program for non-insulated cables with a diameter of D=15 mm.

| № | Name of the coating         | Sample number | The area of the frozen part of the cable, cm² | Test №1 | Test №2 | Test №3 | Test №4 |
|---|----------------------------|---------------|---------------------------------------------|--------|--------|--------|--------|
| 1 | Without anti-ice coatings  | 1             | 17,4                                        | 11,3   | 11,8   | 13,7   |
|   |                            | 2             | 19,8                                        | 12,8   | 13,3   | 15,5   |
|   |                            | 5             | 20,8                                        | 11,7   | 12,3   | 14,9   |
|   |                            | 6             | 17,9                                        | 9,9    | 11,8   | 13,7   |
| 2 | Coating «Fluora-C»          | 8             | 18,8                                        | 12,2   | 10,8   | 13,2   |
|   |                            | 11            | 19,2                                        | 13,9   | 12,3   | 14,9   |
|   |                            | 13            | 20,3                                        | 12,3   | 12,8   | 16,5   |
| 3 | Coating «Snegotech-1»       | 14            | 17,0                                        | 12,2   | 10,8   | 12,7   |
|   |                            | 17            | 19,2                                        | 13,9   | 12,3   | 14,4   |
|   |                            | 18            | 17,9                                        | 10,8   | 10,8   | 12,2   |

In the ice experiment pool of the accredited center for the study of the adhesive strength of ice frozen on the surface of the cable, a technique based on the determination of the force of separation of ice from a sample of material having a cylindrical shape and a relatively small diameter was used.

Table 2. Test program for insulated cables with a diameter of D=17 mm.

| №  | Name of the coating         | Sample number | The area of the frozen part of the cable, cm² | Test №1 | Test №2 | Test №3 | Test №4 |
|----|----------------------------|---------------|---------------------------------------------|--------|--------|--------|--------|
| 1  | Without anti-ice coatings  | 3             | 18,8                                        | 12,2   | 11,8   | 12,7   |
| 2  | Coating «Fluora-C»          | 4             | 21,4                                        | 13,9   | 13,3   | 14,4   |
| 3  | Coating «Snegotech-1»       | 7             | 21,4                                        | 13,3   | 12,3   | 14,9   |
| 4  | Coating «FLUORA-D»          | 11            | 16,0                                        | 11,3   | 11,3   | 14,1   |
| 5  | Coating «Epilam Electronic-A №50 11/20» | 16 | 17,9 | 13,2   | 11,3   | 13,7   |
| 6  | Coating «Fluora-CF»         | 19            | 20,3                                        | 14,9   | 12,8   | 15,5   |
| 7  | Coating «Epilam SFC-20»     | 20            | 16,0                                        | 12,2   | 11,8   | 12,7   |

The basis of this method of experimental research is the freezing of samples in freshwater ice and their subsequent loading until the moment of separation with the mandatory registration of the value of the applied load.

The adhesion researching were carried out using a specially designed and manufactured container divided into 20 cells with a size (length x width x height) of 100x100x70 mm. In each cell, a technological hole is made, in which a sample of the cable is placed. Before starting the experiment, each cable sample is frozen into the ice in its cell to a certain height, which ensures the same conditions for lateral constriction of the samples and the geometry of the surfaces. Since the ice build-up in the cell is stochastic, the area of the frozen part of the cable is measured during the experiment. The bowl of the ice pool is partially located inside the refrigerator and is separated from the side by an air lock. The refrigerator system provides uniform cooling of the air above the pool to minus 29°C. Recording devices and equipment are located in a special thermally insulated room inside the pool.
The process of obtaining a uniform layer is explained by the fact that the macromolecules of this type of surfactant are polar, ordered (during activation) by hydrophilic / hydrophobic ends and have a dipole magnetic moment. The polar part of the macromolecule, falling into the field of the surface of the part, is oriented in the directions of the force field of the part [1], its polar part is fixed to the part by adsorption bonds, and the non-polar part remains outside above the part. The surface morphology was studied using the NTEGRA probe nanolab by NT-MDT. The morphology of the monomolecular layer - the surface of Langmuir-Blodgett films obtained by applying a thin-layer coating-is studied by atomic force microscopy, the profilogram is shown in figure 1. When visualizing, it is easy to see that a layer of oriented molecules is formed on the coated surface, which not only radically change the surface energy: polymer fluorine compounds form the so-called Langmuir-Blodgett structures in the form of a thin-layer coating and a uniform monolayer.

![Figure 1](image1.png)

**Figure 1.** The profilogram of the non-coated and coated surface.

The wettability (hydrophobic) angle was measured by various available methods to determine the wetting edge angle, where values for different compositions were obtained in the range from 91 to 124 degrees. To measure the wetting angle, an electronic goniometer LK-1 with a digital camera Levenhuk C130 and software processing was used.

To prepare ice according to the proposed method, it is necessary to spray water in the container cell, first lowering the temperature in the ice chamber. The required ice thickness is achieved by filling each cell of the container with fresh water after pre-freezing the sample. For 8 hours, the cable samples are frozen in ice at a temperature of minus 16°C, and for 24 hours, the container is stored at a temperature of minus 6°C. The photos of the testing stand and the freezing process (samples) are shown in figure 2.

![Figure 2](image2.png)

**Figure 2.** Testing stand.

The tests were recorded on a video camera and filmed with a digital camera, with the help of which the overall picture of cable separation from the ice was recorded from the best angle.
Before starting the experiment, the most important technological task is to control the thickness of the resulting ice. To do this, a number of standard measurements are made using a ruler. All measurements were carried out with the help of trusted measuring instruments. Table 3 shows the list of measuring devices used in the experiment and their main characteristics.

### Table 3. List of measuring instruments.

| №  | Name of Dynamometer                        | Certification | Measurement range, basic accuracy characteristics | Measured parameter                      |
|----|--------------------------------------------|---------------|-----------------------------------------------------|-----------------------------------------|
| 1  | Measuring channel with load cell T 50C3    | certified     | The measurement range of $F_x$ is 50-500 N with a probability of 0.95 no more than ±0.1 % | Resistance force $F_x$                  |
| 2  | Cymometer "Polyus-8"                       | certified     | Frequency conversion range 0.1-16.5 kHz, the relative error - 0.1% | Cymometer-to-analog converter           |
| 3  | Force-measuring sensor, model U2B          | certified     | Range from 100 till 1000 N                          | Tension /compression force              |

It is known [2] that the charge of the molecules allows to reduce the surface energy of the material, to be adsorbed, chemisorbed by the hydrophilic tail, and the hydrophobic tail remains on the surface, which creates an ultrathin film.

The results of the evaluation tests of distribution of the values of the ice strength on separation in the study of ice adhesion are shown in figure 3, where the X-axis shows the name of samples, the Y-axis shows tear strength MPa.

![Figure 3](image-url). Distribution of the values of the ice strength on separation in the study of ice adhesion.

### 3. Conclusions

1. The results of experimental studies show a steady decrease in the adhesion of ice to the surface of cables when using ultra-thin coatings "Fluora-C" and "Fluora-D". The decrease in adhesion is expressed approximately in a twofold decrease in the strength of the ice to break off. The film-forming composition "Fluora CF" also effectively reduces the adhesion of ice to the surface of cables with
insulation, but does not significantly reduce the strength of ice to break off for cables that do not have insulation.

2. The coatings "Epilam Electronik-A" and "Epilam SFK-20K" showed similar results to those obtained during the studies of samples that do not have any coating. Therefore, according to the results of the conducted studies, their use to reduce the adhesion of ice is ineffective.

3. According to experimental data, the use of the "Snegotech-1" coating leads to an increase in the force required to break the ice from the cable surface, so use to reduce the adhesion of ice to the surface of current-carrying cables in some cases doesn’t have a positive effect.

4. During the preparation of experiments to measure the force of ice separation from cable samples, different duration of the phase transition at the stage of their freezing was found. Samples coated with "Epilam Electronik-A" and "Epilam SFK-20K" according to experimental studies have low thermal conductivity. It is expressed in an increase in the freezing time of water, during which liquid droplets are observed to drain from the surface of the cables. The "Fluora-C" coating significantly increases the freezing time of water, which required additional cycles of its spraying to form a crust at the base of the container cell, where the cable sample is located. The other coatings considered did not show any effects different from those observed at this stage for uncoated samples at the stage of freezing the samples in ice.

According the strong connection between the ultra-thin coating and the surface of the test samples, it was possible to repeat the tests several times and get similar results. The developed technique can be used to analyze and simulate the resource of ultra-thin fluorinated coatings to solve the problem of icing.

References
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