Microscopy of composite materials based on carbon fibre

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Abstract. The work is devoted to a composite material consisting of carbon fibres in a carbon pitch-based matrix. Due to its high wear and heat resistance, this material is widely used in aviation brake systems. Two samples with different impregnation of fibres were studied. It is shown that their tribological properties are different. A complex method of microscopy (optical, scanning electron, and scanning probe microscopies) was used to study the friction surface and inner structure of the samples. In the sample with the best properties, the fibres have a regular ribbed side surface, fibre bundles are located parallel to the friction surface, matrix around the fibres has a denser structure. It is also shown that in this sample wear rates of the fibres and the matrix are the same. Thus, a small change in the production process leads to a noticeable change in the structure, which determines the operational properties.

1. Introduction
The production and application of composite materials, and in particular, carbon-fibre materials, have significantly increased in recent years [1-3]. It is known that carbon fibre-based composites in a carbon matrix demonstrates high wear resistance and heat resistance, which makes them useful in the production of brake discs in aircraft industry [4,5]. Obviously, the properties of such materials depend on the structure of the composite materials [6]. It is known that the required characteristics are achieved only under certain conditions for the production of these materials, and even small changes in production technology can lead to a significant deterioration in properties [7].

This paper is devoted to investigation of the correlation of fibre manufacturing technology with the structure and tribological properties of the resulting composite material.

2. Materials and methods

2.1. Samples
Composite materials based on graphitized fibre in a carbon matrix were studied. Samples were provided by JSC “AK Rubin”. The original fibres had a diameter of about 7 microns, and were grouped into parallel bundles. The standard technology used includes impregnation of fibres with a special compound (so-called “apret”), high-temperature annealing (graphitization of fibres), followed by pouring by a carbon pitch-based matrix.

Using this technology, two types of samples were prepared (No. 1 and No. 2). The difference in their preparation consisted in different impregnation compound composition and lower fibre annealing temperature for sample No. 2.

Tests for wear resistance were carried out on the IM-58 friction machine. A study of mechanical properties was also carried out using bending tests.
2.2. Microscopy
A complex method of microscopic studies was used to investigate the surface of the samples [8,9]. An optical microscope (OM) “Neophot-2” with an image observation system in digital format “Image Scope” was used in this work. Electron microscopic studies were carried out on a scanning electron microscope (SEM) “FEI” Quanta-650 (an accelerating voltage of 25 kV) with a secondary electrons detector. The cuts (slides) of the samples were also studied by SEM. It should be noted that in this work the energy dispersive X-ray spectral analyser was not used, since all the samples had almost the same elemental composition. Moreover, the main chemical element of the composite was carbon, which could be determined by this method with a high error. For obtaining the high-resolution surface images, the scanning probe microscope (SPM) study was performed using a Smart SPM-TM. A taping mode was used (the resonant frequency – 250 kHz), the fpN10 cantilevers were used (the force constant about 10-20 N/m and the curvature of the tip about 20 nm). This microscope was equipped by the integrated optical camera for the preliminary selection of the area for investigation. The maximum scanning field was 100x100 microns.

3. Results and discussion

3.1. Tribological properties
The tribological tests were done by the sample manufacturer (JSC “AK Rubin”) and showed the significant difference in wear resistance: for sample No. 1 this parameter was significantly higher than for sample No. 2. It can be assumed that small changes in the production process can affect the properties of the composite components and significantly change their tribological properties. At the same time, other operational parameters of the studied samples (mechanical properties of the initial carbon fibres, bending and strength properties) were approximately the same for both samples. The main part of the work was devoted to identifying the differences between these samples by microscopy methods.

3.2. Optical microscopy
At the first stage, samples were studied with a low magnification. The resulting images are shown in Figure 1. Analysis of these images indicates that the fibres are grouped into bundles. It is found that the fibre bundles have different diameters – for example, in sample No. 1 it is 50-100 microns, while in sample No. 2 it is 150-200 microns. Such a difference can be explained by the conditions of obtaining: a change in the structure of apret leads to the fact that the fibre bundles of sample No. 2 are worse divided into narrow groups of fibres.

![Figure 1. Optical microscopy of the sample surface: a - sample No.1; b - sample No.2.](image)
Figure 2. Images (optical microscopy) of sample No. 1 surface after tribological tests.

Microscopic studies with high magnification were carried out for detailed study of the differences in the samples. The obtained results for different areas of the samples are shown in Figures 2 and 3.

Figure 3. Images (optical microscopy) of the surface of samples No. 2 after tribological tests.
Figure 4. SPM images of the surface of samples No. 1 (a-topography, b-section profile) and No. 2 (intopography, g-profile section). The profiles are indicated along the highlighted lines.

Analysis of these images showed that there are two types of fibre arrangement at the surface of sample No. 1. There are areas with fibres arranged parallel to the friction surface (regions of fibre bundles) and areas with a small number of randomly arranged fibres (regions of randomly reinforced matrix). At the same time, three types of fibre arrangement can be clearly distinguished at the surface of sample No. 2. Two types are the same as already described; the third type of fibre arrangement is their unparallel orientation to the friction surface. The appearance of areas with unparallel orientation of fibres is a consequence of the higher ability of the fibre bundles of sample No. 2 to bend (unlike bundles of sample No. 1, which split and break when flexed).

3.3. Probe microscopy
The SPM images of the surface and the surface profiles are shown in Figure 4. It is seen that the nature of the surface topography of the samples correlates with the data of optical microscopy. In addition, the method of SPM profilometry showed a significant difference in the surface topography of samples No. 1 and No. 2 and evaluated the pattern of surface wear in these cases.

Thus, the analysis and comparison of areas, in which the fibres lie parallel, showed that in samples No. 1 the surface relief is flat, the fibres and matrix surface are at the same level. So, it may be concluded that the fibres and the matrix wear at the same way. At the same time, sample No. 2 has a distinct relief: the fibres lie considerably below the level of the matrix surface (0.3-0.4 μm). The reason of this effect is lower wear resistance of the fibres.
3.4. Electronic microscopy

Further studies were carried out using SEM. The images of the sections (slices) of samples No. 1 and No. 2 are shown in Figure 5. The fibres sections and the main matrix could be distinguished at both figures. It is easy to see that in sample No. 2 the fibres stand out in the matrix: they have a rather sharp border with the matrix, while the matrix around the fibres have a looser structure. For sample No. 1, the fibres and the matrix are practically merged, and the matrix itself is rather dense. The reason for this was identified in subsequent measurements.

Individual fibres for both samples were investigated using SEM with the highest magnification for samples No. 1 and No. 2. Figure 6 illustrates the difference of fibres. In sample No. 1, it consists of a large number of flat plates, which are layers of graphite. The side surface of these fibres consists of thin edges of graphite layers. Sample No. 2 fibre consists of a smaller number of thicker layers, having side surface, with cracks and deep cavities. These features have a great influence on the process of fibre/matrix interface formation and properties of matrix around fibres.

Figure 5. SEM images of slices: a - sample No. 1, b - sample No. 2.

Figure 6. SEM images of individual fibres: a - sample No. 1, b - sample No. 2
This difference in fibre structure may be explained by the features of the manufacturing process: different annealing temperatures and impregnation compounds. This difference in technology also leads to a change in the shape and number of crystallite particles covering the fibres (these crystallites are formed during the thermal decomposition of arapet).

It can be assumed that the nature of the surface of the fibres, the state of the graphite layers strongly affects the properties of the fibres. These factors, as well as the presence of particles on the surface of the fibres, can greatly influence the interaction with the surrounding matrix. As a result of a change in the surface structure of the fibre, a change occurs in the layers of the matrix adjacent to the fibres. Obviously, the structure of the matrix layers surrounding the fibre depends on the structure of the fibre surface. This is clearly demonstrated by slice images of samples No. 1 and No. 2. It is clearly visible that the fibres in the cross section of the material in sample No. 2 have a sharp border with the matrix, and the layers of the matrix around the fibres have a loose structure. At the same time, in sample No. 1, the fibre-matrix boundary is practically indistinguishable, and the matrix itself has a rather dense structure. Obviously, all this also affects the tribological properties.

4. Conclusion
The features of sample preparation determine the shape of the fibre, the surface of the fibre, and its interaction with surrounding matrix and orientation of fibres in the matrix. Different orientations of the fibres in matrix determine sample’s friction properties. Friction properties are also connected with correlation of wear-resistances of fibres and matrix. Small changes in fibre production technology lead to significant changes in the structure and properties of the resulting material (fibre-matrix composite) [10-13].

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