Development of a method of hydroerosive saturation of liquids with microparticles of target materials using ultra-jet

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Abstract. The article deals with the questions of assessing the applicability of the method of ultra-jet diagnostics of carbon plastics. In the work, phenomenological ideas about the process of destruction of carbon plastics under the action of a high-speed-jet of liquid are considered. The experimental part of the work is related to the implementation of the diagnostic effect of the ultra-jet on the surface of the carbon fiber reinforced plastic, the assessment of the geometric parameters of the hydraulic caverns and the qualitative assessment of the state of the fibers and the adhesion in the binder-fiber structure. In this experiment, carbon fiber samples were used both in the initial state and after the inhibitory effect of x-ray radiation. Such a technique made it possible to carry out a comparative analysis of the samples and evaluate the information content of the ultra-jet diagnostics method as applied to the materials under consideration.

1. Introduction
One of the most promising areas in the development of modern technology is the use of composite materials [1]. These materials require a special approach and the formation of new scientific and technical solutions in the development and creation of methods and means of assessing their operational reliability. This is due to the wide variety of types of such materials, the specific features of the structures made of them, manufacturing technology, a random change in the physio-mechanical and strength characteristics, and a large variety of types of defects that arise during the manufacturing process [2].

Composites are characterized by technological stresses arising in the elements at the stages of their manufacture. In addition, these materials in most industries operate under static and dynamic loads [3]. In constructions made using polymer composite materials (PCM) and especially carbon plastics experiencing dynamic loads, in order to obtain complete and reliable information about their condition, it is necessary to take special control and safety measures, for example, in mission-critical products: rocket and space technology, aircraft, shipbuilding, various transport structures, etc [4].

It should be specially noted that ensuring the safe operation of polymer composite material products is an important and urgent task [5].

Despite the fact that composite structures have many advantages, they can sometimes have various defects, such as delamination between composite layers, the waviness of composite layers or bends, in which at least partial twisting of the composite bundle on the upper part occurs, resulting in inner curls are formed in the composite structure. Considering that some of these defects can be detected
as a result of visual inspection of the composite structure, many defects can be located in the inner part of the composite structure, therefore, they cannot be detected during visual inspection [6].

In this regard, non-destructive methods for monitoring and diagnosing such structures are of great importance. They allow you to objectively determine the actual state of the structure, evaluate the reliability of their operation and give recommendations for its repair or restoration.

Because of this, many verification technologies have been developed, using, for example, X-rays [6], ultrasonic signals [7], and others for a detailed study of the structure of a composite structure [8]. Although these validation technologies can detect many defects, such as layer delamination, other defects that can be caused by disorientation or displacement of structural fibers, it is more difficult to detect defects in the resin of a composite construction.

Attempts to solve this problem with the help of flaw detection using various methods - ultrasonic, radio wave, etc. did not lead to the desired results. This is due to several reasons [9]:
1. Flaw detection methods can detect macrodefects, while a violation of the decrease in strength can be caused by microdefects (microcracks, micropores, etc.) and a number of other factors, for example, a violation of the composition of the material during application of force loads, a violation of manufacturing technology, etc.
2. Microdefects, which cause a decrease in reliability, are mainly formed in the process of loading the controlled structure with some loads (static or dynamic force, internal pressure for cylinders, etc.), and flaw detection methods mainly do not allow non-destructive testing in the process loading structures.
3. When controlling complex spatial structures, or objects that did not occupy the entire field of view of the recording system, along with informative parameters, interference is recorded that significantly reduces the reliability of the control results.

It is necessary to take into account the fact that during the production and operation of carbon fiber products, areas may appear where there is a significant weakening or disappearance of adhesion in the transition zone between adjacent layers of material, which leads to a significant deterioration in strength and performance [10]. In case of violations of the technological process, adhesion in the binder-fiber pair is also possible and a decrease in adhesive strength. Therefore, it is extremely important to enrich the existing arsenal of diagnostic technologies with new methods that would allow us to study the micro and macrostructure of carbon fiber reinforced plastics, indirectly assess the adhesive strength [7].

However, for polymer composite materials, adhesive bonds determine their quality, being its most important indicator. The determination of these bonds and the obtaining of objective information on adhesion in the binder-fiber structure are an important diagnostic task.
At present, methods of uneven tearing [10], in which the load is applied to the edge of the joint, due to which failure occurs non-uniformly, have become most widespread in testing materials for adhesive strength. The advantage of such methods is the ability to study adhesion fluctuations in different areas of the test sample.

The disadvantage of existing methods is the need to manufacture special samples, not high measurement accuracy, complexity of implementation and the availability of special equipment. In addition, these methods are not suitable for solving the problems of assessing the effect of binder destruction on adhesive strength. Given the existing potential of tools and engineering algorithms for the implementation of ultra-jet technologies developed at the department of SM-12 of BMSTU [8-9], of scientific and practical interest is the study of the possibility of their adaptation for the study of polymer composite material quality indicators (see Fig. 1).

Thus, the aim of the study is to develop a technique for ultra-jet diagnostics (UJD) of carbon plastics and assess its applicability for the diagnosis of material damage, including exposure to adverse environmental factors.
2. Experimental studies of ultra-jet diagnostics of a polymer composite material

The hypothesis of the study was that the action of an ultra-jet water on the surface of the sample witness will make it possible to obtain a hydrocavity on its surface, the parameters of which will be associated with a number of physical and mechanical characteristics of the material, its reinforcement pattern, the state of the binder (degradation), and the presence of a number of defects (damage), adhesive strength. A study of the fibers at the site of exposure to the ultrajet will allow us to evaluate the adhesive bonds in the binder-fiber structure by determining the ratio of the projection area of the binder particles to a plane parallel to the fiber axis perpendicular to the axis of the impact of the ultrasound to the surface of the sample.

To implement this technique, it is possible to effectively use the technological capabilities of the ultra-jet diagnostics, in particular by increasing the distance from the cut of the focusing tube to the surface of the sample, it is possible to provide a gentle effect on the material by the action of the spray skirt, and not the body of the ultrajet itself. All experiments on the ultra-jet diagnostics on a machine for waterjet cutting brand Flow Mach 3 3131b (USA).

Based on the experience of conducting experiments on the ultra-jet diagnostics of materials and coatings (see Fig. 1), an experimental scheme adapted to the stated task of polymer composite materials diagnostics was proposed, the experimental design shown in Fig. 2. The height h (from the cut of the focusing tube to the surface of the sample) varied from (recommended values for waterjet cutting of materials) 2–4 mm to 70–160 mm. Cutting head feed rate from 1000 to 7500 mm / min.
For the experiment, a carbon fiber sample was used, mounted on a table of a water-jet installation by means of a clamp using sheet equipment and clamps. The carbon fiber samples used in the experiments were made on the basis of a LU-P/0.1 carbon tape impregnated with the following characteristics: laying - \((0^\circ/45^\circ/135^\circ/90^\circ/90^\circ/135^\circ/45^\circ/0^\circ)\) n, modulus of tensile elasticity - 59 166 MPa, tensile strength - 263 MPa., thickness 3.6 mm.

As a result of test passes, the following diagnostic mode was chosen experimentally: feed of the cutting head 7500 mm / min, water pressure 130 MPa, the distance from the cut of the focusing tube to the surface of the sample 160 mm (hereinafter referred to as mode 1). When choosing these parameters, it was possible to ensure surface destruction of the binder without noticeable destruction of the fiber (Fig. 3a). An increase in pressure, a decrease in the distance h, and feed rate was accompanied by penetrating destruction of the material with rupture and damage to the fibers (Fig. 3b). To form a cavern in accordance with the scheme shown in Fig. 3 b the following diagnostic mode was selected: feed of the cutting head 6000 mm / min, water pressure 150 MPa, the distance from the cut of the focusing tube to the surface of the sample 80 mm (hereinafter referred to as mode 2).
Both of the presented variants of the results of the diagnostic effect of the ultrajet are of practical interest. In the first case (Fig. 3a), a spray of water (ultra-jet) is used to “bind” the binder (matrix) from the fibers, which, as described above, makes it possible to evaluate the adhesive properties of polymer composite material. In the second case (Fig. 3, b), the trunk of the ultra-jet of water carries out hydroerosive effect on the material on the local surface area of the sample. In this case, we can talk about the operation of the entire package. The higher cohesive strength of the binder and the adhesive strength of the binder-fiber structure will obviously prevent the formation of a cavern and, therefore, its geometrical parameters, especially (Fig. 3b) will be deliberately lower than in the case of poor adhesive strength, reduced in the result of a combination of adverse environmental factors leading to the destruction of the binder.

To enable a comparative analysis of the results of ultra-jet diagnostics to assess adhesion in the binder-fiber structure, part of the carbon fiber samples was exposed to x-ray radiation, and part was left in its original state. Samples were cut from a carbon fiber panel 80 mm long and 20 mm wide. X-ray irradiation of the samples was carried out in an industrial system of computed tomography and fluoroscopy XT N 450, manufactured by Nikon Metrology (Japan). The time and parameters of x-ray exposure are presented in table. 1. A large number of experiments and different exposure parameters are explained by the parallel conduct of other studies in which carbon fiber samples were placed in the camera of a computer tomograph.

| №  | Voltage | Current | Experiment time |
|----|---------|---------|-----------------|
| 1  | 360     | 185     | 109             |
| 2  | 300     | 100     | 135             |
| 3  | 360     | 100     | 74              |
| 4  | 350     | 145     | 105             |
| 5  | 375     | 185     | 74              |
| 6  | 325     | 120     | 57              |
| 7  | 380     | 200     | 26              |

At the next stage, the ultra-jet diagnostics procedure of both groups of samples was implemented, in accordance with the scheme presented in Fig. 2. To form a cavern, Fig 3b. Mode 2 was used.

The study of samples (hydro-caverns) after conducting an experiment on the ultra-jet diagnostics of polymer composite materials was carried out using a VHX-6000 digital microscope (Japan). In fig. Figures 4-5 show images of (a) hydro-caverns and a sample after the ultra-jet diagnostics (b).


Figure 4 - The results of the ultra-jet diagnostics of polymer composite materials sample after an X-ray exposure using a digital microscope (a - 3D profile of a cavern (1), a section with tracing (2), a profilogram; b - image of a sample with a cavern (3))

Figure 5 - The results of the ultra-jet diagnostics of polymer composite materials sample in its initial state using a digital microscope (a - 3D profile of a cavern (1), plot with tracing (2), profilogram; b - image of a sample with a cavern (3))

The results of determining the geometric parameters of the cavern after ultra-jet diagnostics of polymer composite materials in the initial state and after x-ray exposure are presented in table. 2.

| Sample | The depth of the cavity, microns | The width of the cavity, microns |
|--------|---------------------------------|---------------------------------|
|        |                                 |                                 |
After exposure
1538,12/1600,34*
initial state
1235,26/1165,33*

| After exposure | 1538,12/1600,34* | 1819,72/1910,12* |
|----------------|------------------|------------------|
| initial state  | 1235,26/1165,33*| 1447,12/1580,19*|

* The average value of 10 measurements of 3 samples

Further, mode 1 was used to form the hydro-cavern in Fig. 3a. As a result of the ultra-jet diagnostics, surfaces with traces of hydroerosion were obtained. For the study, an electron microscope manufactured by Phenom-World (Netherlands) was used.

Images of sections of carbon fiber samples after the ultra-jet diagnostics are shown in Fig. 6. It can be seen from the figure that in the case of the initial sample (Fig. 6a), the surface of the sample (zone 1) practically did not suffer from the action of the peripheral zone of the jet — the spray. This cannot be said about a sample exposed to radiation (Fig. 6b), where there are practically no traces of a binder on the surface. In the fiber images (Figs. 6c and 6d) obtained at 2000x magnification when focusing in the zone of the cavern (zone 2), one can notice a significantly larger amount of binder on the fiber surface for the initial sample (Fig. 6c). Obviously, the causes of this state of the surface of the samples and fibers are associated with the destruction of the binder, its embrittlement and removal of the products of destruction of the ultra-fluid from the zone of the ultra-jet diagnostics.

![Figure 6](image-url)

**Figure 6.** - Sites of surface hydroerosion of samples after the ultra-jet diagnostics (mode 1) at 600x magnification: a - initial sample, b - sample subject to x-ray irradiation; at 2000x magnification: c - initial sample, g - sample subject to x-ray irradiation.

Studies have shown that the ultra-jet diagnostics method demonstrates the presence of various adhesive bonds in polymer composite materials, which may be the basis for its refinement, the creation of an engineering diagnostic mechanism, the formation of hydraulic fracture databases for various composite materials, and the introduction at the stage of technological preparation of production or for selective control of batches details or samples of witnesses. The possibility of varying the technological parameters of the ultra-jet diagnostics will allow a diagnostic effect on the test object to be given at a given depth or number of fiber layers, which expands the potential capabilities of the method, for example, if it is necessary to study veils or tapes embedded in the package and intended to change the specified characteristics and properties of the polymer science materials, in particular, thermal conductivity, electrical conductivity, etc.

**Conclusions**
In conclusion, it should reflect the capabilities, features and advantages of the developed method of the ultra-jet diagnostics of polymer composite material:

- a simulated local wave and shock-force hydraulic action of an ultrajet of a liquid appears on the material;
- the implementation of the ultra-jet diagnostics method is possible with a combination of external forces and factors, such as preloading, for example, tensile or bending of the sample, temperature effect (complex or local);
- The ultra-jet diagnostics does not provide for the production of special samples for testing, and any products made from polymer composite material can be examined; the possibility of both qualitative and quantitative assessment of the quality of polymer composite material and its adhesive properties is provided, in particular by analyzing the area of the binder remaining after the ultra-jet diagnostics on the fiber surface;
- it seems possible to evaluate both the binder-fiber structure and the polymer composite material components separately, in particular, the cohesive properties of the binder;
- for the implementation of the method does not require the creation of special technological equipment or instruments, research is carried out on standard measuring equipment and installation for waterjet cutting of materials;
- a diagnostic effect can be exerted in several sections of the sample at a given depth and area, taking into account the direction of reinforcement, winding, laying, etc.

Thus, there is the possibility of a fairly widespread implementation of the ultra-jet diagnostics of polymer composite material method, first of all, at the stage of testing the technological preparation of production, diagnostics and control of witness samples. The method is of particular importance in assessing damage to polymer composite material, which is exposed to various factors of influence, such as temperature, thermal cycling, climatic, X-ray, ultraviolet, etc.

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