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Method and means of researching the composition, structure and properties of materials

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The study guide was prepared in accordance with the program of the educational discipline "Methods and means of researching the composition, structure and properties of materials", which is included in the training plan of the Doctor of Philosophy in the specialty 132 "Materials Science".

The manual examines the most widely used methods in mechanical engineering and materials science for determining the chemical composition of materials, identifying the components of their structure, and the main practically significant properties.

The manual is designed for applicants and teachers of higher educational institutions conducting research in specialty "Materials Science".

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CONTENT

INTRODUCTION

Materials are the basis of the world, modern production and everyday life. The purpose, as well as the properties, of materials is directly related to the spheres of their use. If it concerns the production of machines and constructions, as well as their operation, then in this case the terms: service purpose, technical characteristics, technical task, durability, reliability, etc. have become widely used. These terms are supported by specific numerical indicators that must be ensured during the entire life cycle of the machine. Based on these indicators, a machine or other technical product is designed, manufactured, operated and disposed of. Indicators of environmental impact on the environment, cost-effectiveness of manufacturing, operation and disposal are of great importance. Without such a systematic approach, it is impossible to achieve success in the field of mechanical engineering.

A clear vision of the unity of the triad: composition - structure - properties in the vision of the essence of the material world has long been established in materials science. Research of existing materials, determination of the causes of accidents, loss of technical characteristics, appearance and other indicators usually begin with the determination of the composition of the material. At the same time, it is taken into account that the properties of the material depend not only on the main components, but also on micro-additives or impurities that got into the material during its production, processing or operation.

Historical experience and data of modern science indicate that even for materials with the same chemical composition, there can be a huge number of variants of their internal structure - structure. Grains, subgrains, crystals, crystallites, the type and arrangement of dislocations, grain boundaries, cohesion, surface properties and many other issues that significantly affect the properties of materials and products made from them.

In accordance with the issues that specialists solve in the process of manufacturing materials, products from them and their application, scientists and manufacturers have created a large number of devices and sophisticated modern equipment for research and control of the components of the triad composition - structure - properties . The basis for determining the characteristics of these components is an experiment.

1 METHODOLOGY OF THE EXPERIMENT

1.1 Basic Terms and Conditions

The most difficult and problematic part of scientific research is physical and mathematical experiments. They are one of the main ways of obtaining new knowledge. An experiment is a scientifically designed experiment or observation of processes, phenomena with the possibility of controlling them, for certain conditions that can be reproduced. When repeating pre-traces under similar conditions, the results should be reproduced. In contrast to ordinary observation during an experiment, the researcher actively influences the process or phenomenon.

During the experiment, patterns of processes or phenomena are revealed or theoretical postulates, hypotheses are tested, and the effects of new factors, conditions and external factors are taken into account. From an economic point of view, it is desirable to conduct the experiment in the shortest possible time with the lowest costs. It is important to get high-quality results in a short time while the topic remains relevant.

Experiments are classified into natural and artificial. Natural experiments are more often performed in the conditions of active production or operation of machines or technological processes, etc. An artificial experiment is used when solving physical and technical problems of applied sciences (primarily in technical ones). This makes it possible to avoid the influence of secondary factors that create various noises that are difficult to filter. In this case, it is possible to gradually complicate or simplify the research and identify all influencing factors.

Based on the results of the previous experiment, a complete research program is being developed.

A distinction is made between laboratory and industrial experimental research.

Laboratory studies are performed on special installations or stands using universal or created sensors, devices, recording devices, etc. The reliability and accuracy of such follow-ups allow to achieve the highest quality results. An important role is played by the mathematical planning of the experiment and the automatic fixation of the results in the aggregate using mathematical statistics.

To take into account production factors and conditions, production experimental studies are carried out.

Industrial experimental studies study processes under real conditions of influence of various random or unaccounted factors of production. They are carried out at factories under construction, operating plants, roads, buildings and structures. Due to the large volume of research, clear planning and organization of research is required.

It is advisable to organize a production experiment by collecting materials

in testing laboratories, organizations of the state statistical service. This information has been collected and accumulated for many years using the same methodology and using the same blocks of questions. This allows you to follow the dynamics of changes in certain phenomena and processes. These data can be processed statistically and trend equations can be obtained.

It is important to develop a technique for cleaning such materials from noise and errors caused by changing measurement conditions or changing measuring instruments.

In a number of cases, the result can be obtained using the questionnaire method.

Production experiments can be replaced by research on special stands. These stands are often organized by retrofitting industrial equipment with a special measuring and recording complex, which allows experiments to be carried out without interfering with the current technological process, which increases the reliability of the results and the efficiency of the use of equipment, machines and devices.

Depending on the problem being solved, the amount of research can vary significantly. Sometimes a laboratory experiment is enough to confirm the predictions, in an unfavorable case - it is necessary to perform a whole series of them: preliminary (research), laboratory, bench on production equipment.

Often, a lot of money and material resources, time is spent on the experiment without a positive result. The reason for this is hidden in insufficiently qualified tasks, when the issue has not been studied sufficiently in advance based on literary sources and patents, and production experience has not been taken into account. It is not good if the results of three experimental studies do not fully confirm the working hypothesis of the scientific research. Often this is also a consequence of careless preparation of the experiment, unsuccessful justification of the goal and tasks. First, before starting experimental research, their methodology is developed.

The methodology of the experiment includes general postulates, structure, setting and sequence of research. The methodology of the experiment should have the following main components: development of the plan-program of the experiment; assessment of measurement errors and selection of a complex of tools and devices for conducting an experiment; conducting an experiment; processing and analysis of results, determination of adequacy. This scheme of experimental research is traditional. The use of the mathematical theory of the experiment allows you to significantly increase accuracy with smaller volumes of research. For this case, the methodology includes the following stages: development of the plan-program of the experiment, assessment and selection of measurement tools for conducting the experiment, mathematical planning with the simultaneous conduct of experimental research, processing and analysis of the obtained results.

1.2 Development of the plan-program of the experiment

The research plan-program contains the name of the topic, the purpose of implementation, working hypothesis, experimental methodology, a list of necessary materials, devices, installations, a list of performers, a calendar plan of works. This also includes works on the design and manufacture of devices, apparatus, devices, their certification, as well as programs of experimental works at factories, etc.

The most important part of the program plan is the experiment method. The methodology systematizes techniques, methods, and sequence of experimental research, which ensures effective implementation of the goal and tasks of the experiment; selection of variable influencing factors; tools and equipment for measuring and processing results; organization and analysis of experiment results.

Analysis of information, hypotheses and existing fundamental laws and researches allow to substantiate and formulate the purpose and tasks of the experiment. It is necessary to limit the number of tasks for one follow-up to 5.

The selection of variable influencing factors and their ranking is extremely difficult and responsible. Sometimes the initial predictions have to be changed after the first preliminary experiments or even after the end of their series. In each specific case, approaches may be different. The use of methods of mathematical planning of the experiment for this purpose allows not only to increase their accuracy, but also to reduce the volume and time of work. This approach is especially effective in the presence of a significant number of factors affecting the phenomenon or process under study.

Reasonable selection and ranking of factors is very important to ensure the effectiveness of the experiment. Sometimes, a special, separately planned preliminary study is performed for this purpose.

To identify the priority (importance) of influencing factors, they are studied when all but one variable is fixed. This approach, as mentioned above, is advisable to use for studies with a small number of arguments.

The selection of a set of measuring instruments and devices is always multivariate. It is necessary to make this choice in the direction from universal to special, from simple to complex, from less accurate to ultra-accurate. Catalogs of this equipment are updated annually. It is better when this matter is handled by professionals – metrologists. After all, in order to recognize the results, certification of the devices and the complex as a whole is also required. During the development of new measuring devices, it is advisable to use ready-made tested blocks and units. This will not only make them cheaper, but also significantly speed up production. It is also effective to reconstruct or modernize existing stands, devices and other equipment.

During research, it is extremely difficult to ensure the stability of the conditions for conducting experiments. Therefore, repeated experiments give, as a rule, results that differ from the previous ones. That is, there are errors. Deviations are of different nature: inhomogeneity of materials, random factors

of influence, imperfection of devices and their accuracy class, characteristics of the experimenter, etc. The more random factors affecting the experiment, the greater the deviation of a specific measurement from the arithmetic mean value. A certain minimum number is required, which will ensure a stable average value of the measured parameters, which satisfies the purpose and objectives of the research.

In the research methodology, the technology of conducting experiments is developed in as much detail as possible. To begin with, the research route technology is thought out, and then individual operations of this route are worked out in detail. At the same time, load limits, devices, means and equipment are taken into account. Forms of registration of results and conditions of conducting experiments are of great importance, which is very important for their analysis and formulation of conclusions.

The choice of processing methods and the analysis of the results are the subject of special attention. They should be clear and understandable for the audience for which they are intended. Data should be displayed in tables, graphs, formulas, charts, available for quick understanding and analysis.

It is very important to show scientific results using the methods of mathematics and statistics using the appropriate widely used terms and definitions.

It is highly desirable to use the methods of mathematical planning of the experiment.

1.3 Statistical methods of measurement evaluation

Measurement is the main component of any experiment. The results of the experiment depend on the thoroughness of measurements and subsequent calculations. Therefore, every experimenter must know the regularities of measurement processes: be able to correctly measure the quantities being studied; estimate measurement errors; correctly, with the required accuracy, calculate the value of the quantities and the minimum number of measurements; to determine the best measurement conditions under which errors will be the smallest, and to develop a general analysis of the measurement results.

During measurements, special technical means are used, which are called measuring instruments and devices. Measurements are based on the process of comparing the measured value with a known value, which is called a standard.

If the quantity being measured does not change, then it is called constant, and the process is called static. Measurement of variables is called dynamic. Measurements can be made directly and indirectly (direct and indirect).

Three classes of measurements are defined.

Particularly accurate, high-precision, technical measurements. In mechanical engineering, technical and less often high-precision measurements are used. Absolute and relative measurements are also used.

The measurement error is the algebraic difference between the actual value of the measured quantity ad and the known measurement av. The value of ad is

such a value of the measured value, which is clearly more accurate than the one obtained during the measurement. With some assumptions, ad can be considered the true or exact value of a quantity

$$\varepsilon = ad - av.$$

The value of ε is called the absolute measurement error. Relative measurement error, %:

$$\Delta = (ad - av)/ad \cdot 100\%. \quad (1.1)$$

The accuracy of the measurement is the degree of approximation of the measurement to the actual value of the quantity.

The reliability of the measurement shows the degree of confidence in the measurement results, that is, the probability of deviations of the measurement from the actual values.

To increase the accuracy and reliability of measurements, it is necessary to reduce the error. Measurement errors occur due to a number of reasons:

- imperfection of methods and means of measurement;
- insufficiently thorough research;
– the effects of various external factors in the process of the experiment;
- subjective characteristics of the experimenter, etc.

These reasons are the result of many factors.

Errors are classified into systematic and random.

Systematic are such measurement errors that remain constant during repeated experiments (or change according to a known law). If the numerical values of these errors are known, they can be taken into account during repeated measurements.

Errors that occur randomly during repeated measurements are called random. These measurements cannot be ruled out as systematic. However, in the presence of multiple repetitions, statistical methods can be used to exclude random measurements that deviate significantly from the majority.

A type of random error is gross errors or omissions that significantly exceed systematic or random errors. Mistakes and gross errors are caused, as a rule, by mistakes of the experimenter. They are easy to find. These errors are not taken into account and are neglected when calculating xq. So, you can write down

$$\varepsilon = \varepsilon 1 + \varepsilon 2, \quad (1.2)$$

where ε1, ε2 are systematic and random measurement errors.

During the experiment, it is difficult to separate systematic and random errors. But with a careful and repeated experiment, it is still possible to exclude systematic errors (errors). The main task of measurements is to obtain, if possible, measurement results with smaller errors. The main principles and methods of eliminating systematic and random errors are discussed below.

Systematic errors can be divided into five groups. The first is instrumental errors that arise as a result of violations of measuring instruments, additional backlash or friction, inaccuracy of the graduated scale, wear and aging of nodes and parts of measuring instruments, etc.

The second is errors that arise due to incorrect setting of measurement devices.

The third is errors that arise as a result of the external environment: high air temperatures, magnetic and electric fields, atmospheric pressure and air humidity, vibration and oscillations from moving parts, etc.

The fourth – subjective errors, arising as a result of individual physiological, psychophysiological, anthropological properties of a person.

Fifth - errors of the method. They appear as a result of an unjustified measurement method (with various simplifications of schemes or functional dependencies, lack of theoretical justifications for the measurement method, a small number of repetitions, etc.).

Systematic errors can be constant or variable, increase or decrease during the experiment. They must be excluded. There are known cases when, due to the presence of systematic errors, incorrect scientific conclusions were drawn from the experiment. Systematic errors (errors) can be eliminated by the following methods.

It is often possible to get rid of systematic errors of all groups before the start of the experiment by adjusting or repairing the measuring devices, carefully checking the installation of the measuring devices, eliminating unwanted effects of the external environment. Special attention should be paid to the substantiation of the theory and methodology of measurements. One of the effective methods of eliminating systematic errors of groups 1-3 is to exclude them during the experiment. The main principle of this exclusion is repeated measurement of values.

The substitution method is also used. When measuring x, instead of the monitored object, a standard, measured in advance with high accuracy, is set. The difference in measurements will allow you to find the error of the measuring device.

If, after all, it is not possible to establish the value of systematic errors, then they are limited to the estimation of their limits.

Random errors. When conducting these or other experiments with the same thoroughness, the results of measurements of the same quantity (even taking into account the known law of systematic errors), as a rule, differ from each other. As emphasized above, this indicates the presence of random errors. Each experimenter, analyzing the results of measurements, must be able to correctly assess the random errors that inevitably occur. Accidental errors also include, as is already known, slips and gross errors.

The most typical causes of misses are errors during observations: incorrect reading on the scale of measuring devices, typos (errors) when recording measurement results, various manipulations with devices or their individual components (rearrangement, replacement of blocks, inspection, etc.). Gross

errors arise as a result of malfunctioning devices, as well as experimental conditions that have suddenly changed.

The analysis of random errors is based on the theory of random errors. This theory makes it possible to calculate the real value with a certain guarantee and to estimate possible errors, based on which a conclusion is drawn about the real value of the sought value.

The theory of random errors is based on the assumption that with a large number of measurements, random errors of the same magnitude but of different signs occur equally often; large errors occur less often than small ones, or the probability of the occurrence of an error decreases with an increase in its value, with an infinitely large number of measurements, the true value of the measured value is equal to the arithmetic mean value of all measurement results: the appearance of one or another measurement result as a random event is described normally by the small distribution law.

A general and a selective set of measurements are distinguished. The general population means the whole set of possible values of measurements xi or possible values of errors Δxi. For a sample set of measurements, n is limited and strictly defined in each specific case. It is generally believed that if $n > 30$, then the average value of a given collection of x measurements is sufficiently close to its true value.

The theory of random errors allows you to solve two main problems: to estimate the accuracy and reliability of the measurement for a given number of measurements; determine the minimum number of measurements that guarantees the required (given) accuracy and reliability of the measurement. Along with this, there is a need to exclude gross errors of the series, to determine the reliability of the obtained data, etc. These problems are solved with the involvement of metrologists.

2 MATERIALS AND METHODS OF RESEARCH OF TECHNOLOGY, ERRORS AND DEFECTS OF DEPOSITION OF COATINGS

Increasing the durability is impossible without ensuring the quality, i.e. operational characteristics, of parts during the production of blanks, their mechanical processing and during strengthening operations. In the case of production using worn parts for blanks with the increase of lost dimensions by coating, the technological process includes the following operations: washing, condition control (defecting), preparation of the part for coating (mechanical and thermal processing), coating, thermal treatments for internal stress relief and strengthening, finishing mechanical operations. The choice of methods and the composition of applied coatings and their strengthening are determined by the operating conditions, as well as the ratio of the properties of the conjugated parts.

This section does not consider in detail the known technological methods of controlling the state of the failed parts, as well as the immediate, preliminary processing of the parts. They were only reflected in the typical technological process of manufacturing (restoration).

Special attention is paid to the issue of finding rational technological schemes for the process of applying functional coatings by surfacing in the environment of protective gases. The application of welding in the environment of protective gases to compensate for the worn layer and create an allowance for mechanical processing of the surfaces of machine parts, the equipment used in this case, is described in sufficient detail in the literature [16-18]. However, in the vast majority, traditionally, the modes of conducting the process were determined according to typical recommendations. In cases where the surfaces of holes or surfaces with complex geometry are welded, the use of typical modes is impractical due to the influence of a number of unfavorable factors on the accuracy and quality of the surfaces. Therefore, it is suggested that the regimes and scheme of conducting the surfacing process be coordinated with the results of modeling and experimental studies for specific details.

2.1 Materials for research

2.1.1 Materials of parts to be coated and their characteristics In this section, high-quality and low-alloy steels widely used in machine building were considered: steel 41Cr4, 1045, 42Cr4, 1050, 1552. The choice of the specified materials is due to the fact that these materials are used for the manufacture of axles, shafts, rods, rods, trunnions, crankshafts and other parts that require increased strength. The durability of these parts is limited by the durability of the working surfaces of the base surfaces. The chemical composition and mechanical properties of the specified steels are given in Tables 2.1 and 2.2.

Table 2.1 – Chemical composition of the studied materials

Brand off steel	Chemical composition, %							
	C	Si	Mn	Cu	Ni	*Cr*	P	S
41Cr4	0,36-0,44	0,17-0,37	0,5-0,8	0,3	0,3	0,8-1,1	0,035	0,035
1045	0,42-0,5	0,17-0,37	0,5-0,8	0,3	0,3	0,25	0,035	0,04
42Cr4	0,41-0,49	0,17-0,37	0,5-0,8	0,3	0,3	0,8-1,1	0,035	0,035
1050	0,47-0,55	0,17-0,37	0,5-0,8	0,3	0,3	0,25	0,035	0,04
1552	0,46-0,55	0,17-0,37	1,4-1,8	0,3	0,3	0,3	0,035	0,035

Table 2.2 – Physical and mechanical properties of the investigated materials

Brand off steel	Modulus of elasticity, 10^3, MPa	Tensile strength limit, MPa	Yield strength, MPa	HB	Thermal conducti-vity, Вт/м×°С	Specific heat capacity, Дж/кг×°С
41Cr4	214	980	785	217-552	43-46	
1045	200	610-800	360-650	220-229	47-48	469-481
42Cr4	206	850-1710	650-1490	229-500		
1050	216	640-830	380-600	170-240	48-48	561-787
1552	204	750-1320	430-1200	229		

2.1.2 Materials and characteristics of electrode wires used for welding (Sv-08, ER70S-6, 14331) Low-carbon welding wire Sv-08, ER70S-6, is produced in accordance with GOST 2246-70. The welding cold-rolled wire of the 14331 brand is manufactured in accordance with GOST 10543-82.

The chemical composition of the wires used for surfacing is given in Table 2.3.

Table 2.3 – Chemical composition of welding and surfacing wires

Brand Wire	Chemical composition, %							
	C	Si	Mn	Cu	Ni	*Cr*	P	S
welding wire Sv-08	0,1	0,03	0,35-0,6	0,2	0,25	0,12	0,03	0,03
ER70S-6	0,05-0,11	0,7-0,95	1,8-2,1	0,2	0,25	0,2	0,015	0,01
14331	0,25-0,35	0,8-1,2	0,8-1,2	0,4	0,4	0,8-1,2	0,015	0,01

2.1.3 Composition of deposited coatings When depositing with the above-mentioned wires in a carbon dioxide environment without additional alloying with carbon and other elements, the deposited metal will have a certain chemical composition (Table 2.4, 2.5) and mechanical properties (Table 2.6) [22].

Table 2.4 – Chemical composition of the deposited metal, %, when using Brand wire ER70S-6

Carbon	Silicium	Mangan	Chrom	Phosphorus	Sulfur
no more 0,30	1,0 – 1,50	1,0 – 1,50	2,50 – 3,20	no more	
				0,030	0,030

Таблиця 2.5 – Chemical composition of the deposited metal, %, when using Brand wire 14331

Carbon	Silicium	Mangan	Chrom	Phosphorus	Sulfur
no more 0,55	2,60 – 3,20	1,50 – 2,30	4,30– 4,90	no more	
				0,030	0,030

Table 2.6 – Mechanical properties of deposited metal

Brand wire	Research temperature, °C	Hardness of deposited metal, HRC
ER70S-6	20	42 – 46
14331	20	53 – 58

2.1.4 Carbon fabrics and threads used for carbonization

The structure formation of the deposited layer was ensured by combining welding wires Sv-08, ER70S-6, 14331 and carbon fabric of the brand UUT-2 ТК6-06 И 78-85, with densities of 150, 200, 250g/m2 and various fiber interweaving options (Fig. 2.1).

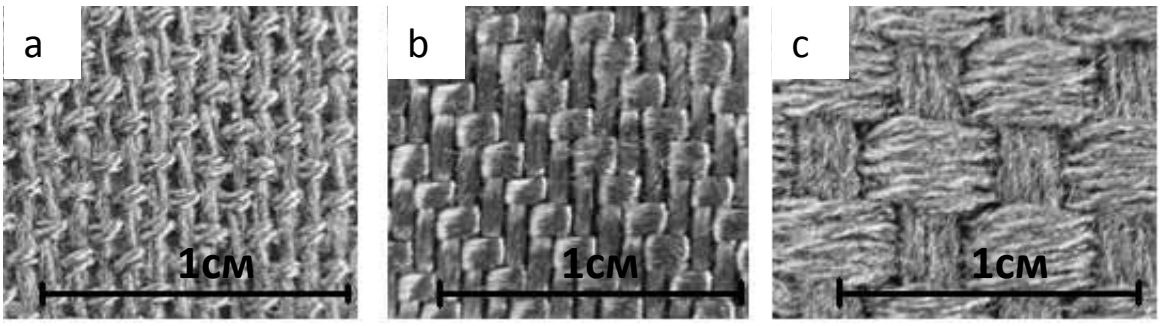


Figure 2.1 – UUT-2 brand carbon fabrics: density 150 g/m^2 (a), 200 g/m^2 (b), 250 g/m^2 (c)

2.2 Determination of the values and nature of the operation of the openings of the parts

The study of the nature and magnitude of failures was carried out on the knuckles of rotary trolleybuses that were undergoing major repairs. Cleaned, thoroughly washed and dried surfaces of the holes of rotary fists were to be

measured. Additional attention was paid to determining the state of factory technological bases and the possibility of their use during the technological process of manufacturing (restoration) of parts.

To measure the amount of wear of the holes of the fists, we used indicator gauges of normal accuracy verified by the metrology service. Before measurement, the indicator gauge was adjusted to the measuring size using a micrometer. Each hole was measured in six axial sections with a step of 30° and in five diametric sections (Fig. 2.2). Measurement data were recorded in measurement maps.

The number of planes for measurements is due to the nature of the operation of the holes of the rotary fists. In the process of their operation, under the action of loads, a significant ovality and saddle-like profile of the holes is formed.

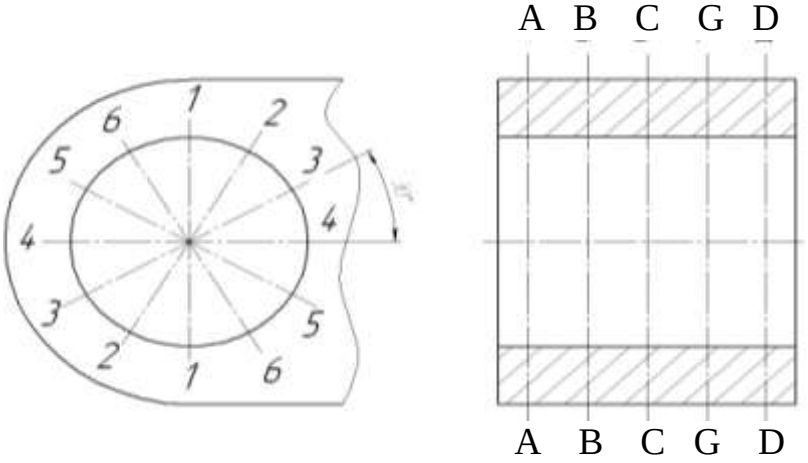


Figure 2.2 – Measurement scheme of the investigated holes of rotary fists: 1-1, 2-2, 3-3, 4-4, 5-5, 6-6 – measurement planes in axial sections; A-A, B-B, C-C, G-G, D-D – measuring planes in diametrical sections

2.3 Preparation of samples for experimental studies

The technology of applying functional coatings on the surface of the holes of the parts of the chassis of transport equipment consisted in the preliminary preparation of the surface of the hole; applying a coating layer with defined characteristics by electric arc welding in a carbon dioxide environment; mechanical processing of the applied coating to achieve the required surface geometry.

Two types of samples were used to conduct experimental studies in order to determine the influence of modes and operational parameters of the technological process of manufacturing parts with holes on their accuracy and the quality of the surface layer: parts that received major repairs and have permanent defects; specially made cylindrical blanks imitating characteristic elements of machine parts with holes.

2.3.1 Use of "defective" parts as samples For the purpose of studying the structure of the base metal, the transition zone, and the applied layer during the surfacing process, rotary fists were used that were sent for repair, but were rejected according to the results of defectoscopy, i.e. which have cracks, chips.

This made it possible to maximally take into account the process of heat removal from the deposition zone and the interaction of the base metal of the part with the deposited metal, which largely determines the structure, and therefore the physical and mechanical characteristics of the applied coating.

After the surfacing process and subsequent mechanical processing, in order to study the operational properties of the applied coating, templates were cut out. Cutting of large parts of welded blanks was carried out mechanically with thin cutting abrasive wheels with significant cooling, and samples for the production of microgrindings - with the help of an EDM machine.

2.3.2 Production of elements of details with holes In order to conduct studies to establish the effect of the amount of alloying elements, in particular carbon, on the quality of the applied coating, samples in the form of cylinders (Fig. 2.3) were made from the material steel 45. The choice of material was based on the previous analysis of machine parts with holes, which showed that this steel is the most widely used.

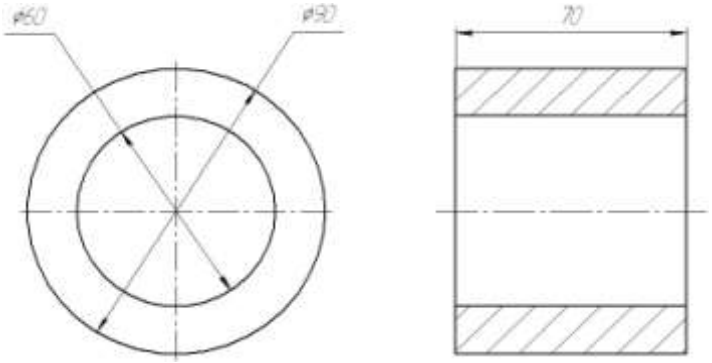
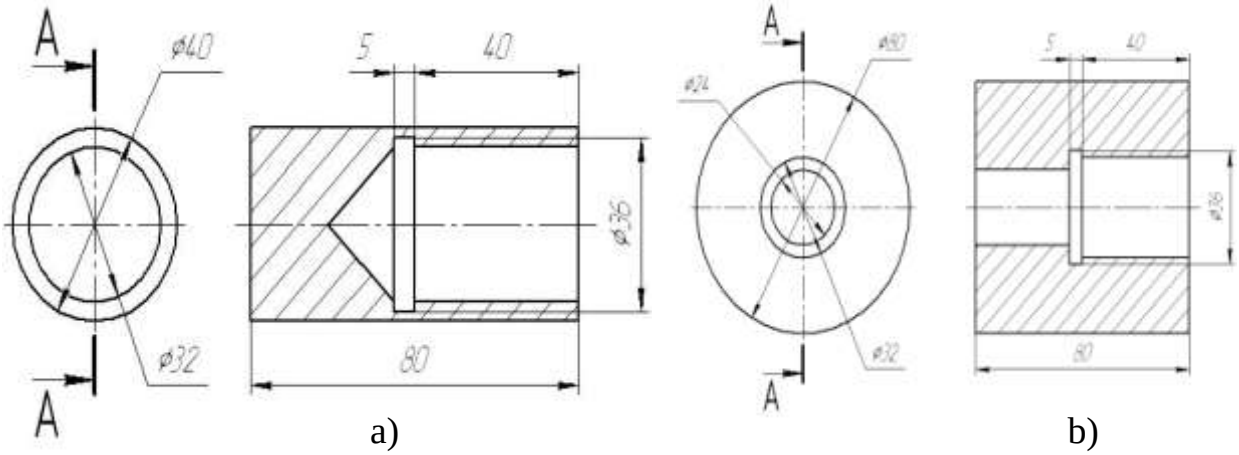


Figure 2.3 – Samples for surfacing operation studies

In order to check the adequacy of the models of the processes of thermal deformation of the tool and parts during the drilling of holes, samples were made, which are shown in fig. 2.4. They simulate thin-walled (a) and thick-walled (b) elements of parts with holes.



a) b)

Figure 2.4 – Samples for the study of thermal processes during hole boring

2.4 Determination of the structure, defects and defectoscpy

2.4.1 Ultrasonic defectoscopy (detection of porosity and other defects)

Ultrasonic control, as one of the methods of non-destructive control of deposited metal, was carried out according to recommendations described in a number of works.

The main method of control is the echo method using a combined inclined transducer of transverse waves. At the same time, an ultrasonic flaw detector UD-10UA was used. According to the recommendations, the following parameters of ultrasonic flaw detection were selected: wave frequency – 4 MHz; the size of the piezo plate – 5 mm; input angle 60^{o}; fixation level – 1 mm^2.

2.4.2 Measuring the hardness of the applied coating

The measurement of hardness, as one of the indicators of the quality of the applied coating, was carried out according to the Rockwell method [88]. At the same time, depending on the conditions, the following were used: dynamic compact TDM-1 hardness tester (Fig. 2.5, a); hardness tester TK-2M (Fig. 2.5, b).

a) b)

Figure 2.5 – Used hardness tester

2.4.3 Determination of the structure of the applied coating

The microstructure of the applied coating and the transition zone are studied using microslides. During the production of samples, they try to avoid the processes of heating and riveting. Electro-erosion machines were used for the production of micro-polishes. Preparation of the microsection was carried out according. The sample, placed in a metal mandrel, is filled with epoxy resin or Wood's alloy. The process of preparing a micro-sheet consisted of several

stages: preparation of a flat surface; grinding; polishing. Microsands to reveal the microstructure were subjected to chemical etching in a 4% solution of nitric acid in ethyl alcohol for 5-30 seconds, depending on the structure of the applied coating. Then it was washed with water, the surface was wiped with alcohol and dried. Samples of the prepared microslides are shown in fig. 2.6.

Microstructural analysis is carried out in parallel with the use of optical and electron microscopy. This makes it possible to analyze the microstructure of the coating in more detail and in full.

Optical microscopy is performed on a MIM-8M microscope (Fig. 2.7) with subsequent image capture using a Kodak C813 digital camera.

Figure 2.6 – Samples of microsands

Figure 2.7 – Microscope MIM-8M and examples of microstructures

Electron microscopy is performed on a REM-106M microscope (Fig. 2.8).

The microscope is intended for measuring the linear dimensions of topology elements and parameters of the surface microrelief of various objects in the solid phase and measuring the mass fraction of elements in the composition of objects by the method of X-ray microanalysis, for studying the

surface of non-conductive objects without special preparation in the mode of low vacuum

Technical characteristics:

The resolution of the microscope in secondary electrons in the high vacuum mode is no more than 4 nm.

The range of magnification changes is from 15 to 300,000 times.

The range of adjustment of the accelerating voltage is from 0.5...30 kV.

The limit residual pressure in the column of the microscope (in the area of the gun) is no more than 6.7 10^{-4} Pa.

The time to change the object is no more than 10 minutes.

The universal sample transfer mechanism provides:

- installation of a sample with a maximum diameter of 50 mm,
- movement along X, Y coordinates by ± 25 mm with a step of 0.5 microns,
- accuracy of positioning according to coordinates X, Y 1 μm,
- movement along the Z coordinate by 60 mm,
- rotation of the sample by 3600,
- platform slope from 0 to -200 and from 0 to 600,

The measurement range of linear dimensions is from 0.2 μm to 5000 μm.

Measurement error 4%.

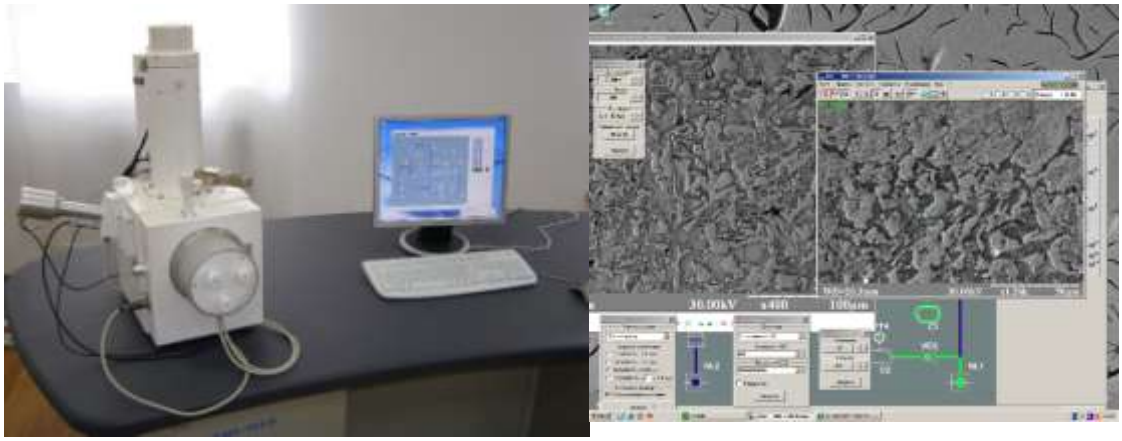


Figure 2.8 – REM-106Y electron microscope

Image formation was carried out using secondary electrons with an accelerating voltage of 18-20 kV. Photographs of micro-structures of coatings, after appropriate computer processing of the results of scanning with an electronic probe, are stored in the form of digital images directly in the PC memory.

2.4.4 Study of composition and structure of thin films on solid surfaces

Raman spectroscopy is an effective, rapid, non-destructive and multi-informative method for diagnosing materials, oxide films and other secondary structures that arise during friction. This also applies to carbon materials, such as graphite, fullerenes, carbon nanotubes, carbine, diamond, graphene, etc. For each of the above-mentioned materials, characteristic bands appear in the

Raman spectra, and their shape, frequency, and intensity allow them to be quantitatively characterized.

When growing carbon nanostructures, the use of Raman spectroscopy for their diagnosis allows controlling the formation of one or another modification or their mixture and determining the influence of technological parameters on their properties. In the process of synthesis of carbon structures, Raman diagnostics is important for assessing their quality and determining the type of existing defects.

The use of Raman spectroscopy at various stages of development of the technology in growing graphene and ultra-thin graphene layers (≤10), as well as in the process of its production, is of great importance and opportunities. For this, a Raman spectrometer and a number of lasers (with the following wavelengths: helium-cadmium with λ=442 nm, helium-neon with λ=663 nm and solid-state with λ=457, 532 and 671 nm) are used to excite Raman spectra. It is also important to have an ultraviolet laser with a wavelength of 355 nm for the diagnosis of materials. Firstly, the high energy of the quanta of laser radiation makes it possible to excite radiation spectra in wide-band materials, secondly, in opaque materials, the penetration depth of ultraviolet radiation is quite small, which allows diagnosing only their near-surface layer. As for carbon materials, only the use of ultraviolet radiation to excite Raman spectra allows us to register the sp3 phase in them. As is known, only sp2 bonds are present in graphene, and both sp2 and sp3 bonds are present in multilayer structures.

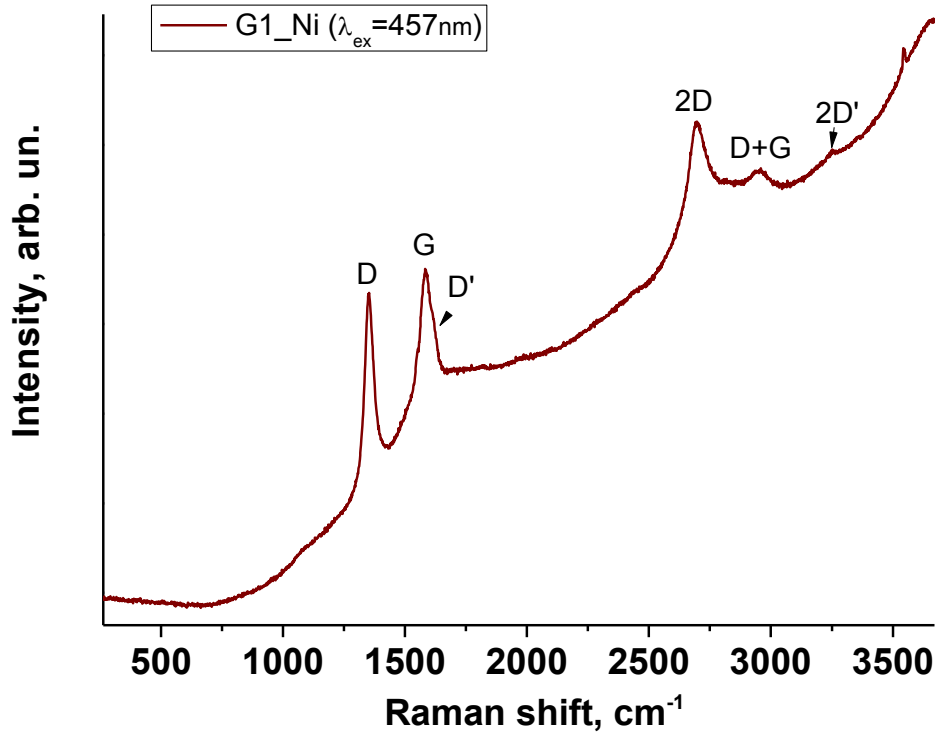
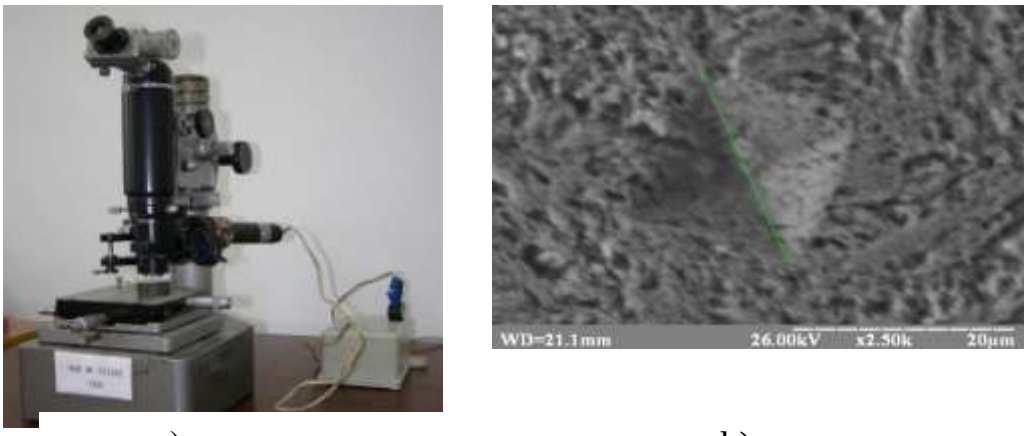


Figure 2.9 – An example of the Raman spectrum of thin carbon films

2.4.5 Measurement of microhardness of structural components

The micro-hardness of the structural components of the applied coating was measured in depth on metallographic sections using a micro-hardness tester PMT-3 (Fig. 2.10, a). Microsands were produced according to the technology described above. The load on the indenter was 0.5 and 1 N, depending on the structure being measured. As an indenter, a diamond pyramid with an angle of convergence of the faces of 45º was used. Further quantitative determination of microhardness value was carried out by measuring the diagonal of the print on a REM-106Y electron microscope, which made it possible to avoid the error of length determination with the optical measurement method, and recalculation of the obtained values according. An example of diagonal measurement is shown in Figure 2.10, b.



a) b)

Figure 2.10 – Measurement of microhardness

2.4.6 Determining the wear resistance of the applied coating during fretting

Often, the service life of individual nodes and connections is limited by premature wear of contacting parts as a result of fretting wear that occurs on mating surfaces at very small mutual movements due to various type of vibrations. As a result of intense wear of the contacting surfaces during the fretting process, the structural dimensions of the parts change dramatically and the operation of the joints is disrupted.

Experiments with fretting wear were carried out on the IFC-1 installation according to the "plane-plane" scheme (Fig. 2.11) with the reciprocating movement of the counter-sample relative to the stationary sample. The sample and counter sample (Fig. 2.12) were fixed in collet clamps and pressed against each other with their working surfaces with a force of P = 250 N, ensuring mutual fit with the help of a self-locking sample collet, after which the position of the sample collet was rigidly fixed.

The samples were touched to each other and a specified compressive load was applied, after which the drive was turned on with a specified amplitude and frequency of reciprocating movement. The experiments were carried out at a

constant amplitude and frequency of reciprocating motion (A = 80 μm, v = 20 Hz), the specific load P was changed step by step without interrupting the experiment in the range from 5 MPa to 55 MPa with a discreteness of 10 MPa. The duration of the entire experiment was $N = 2 \times 10^6$ cycles.

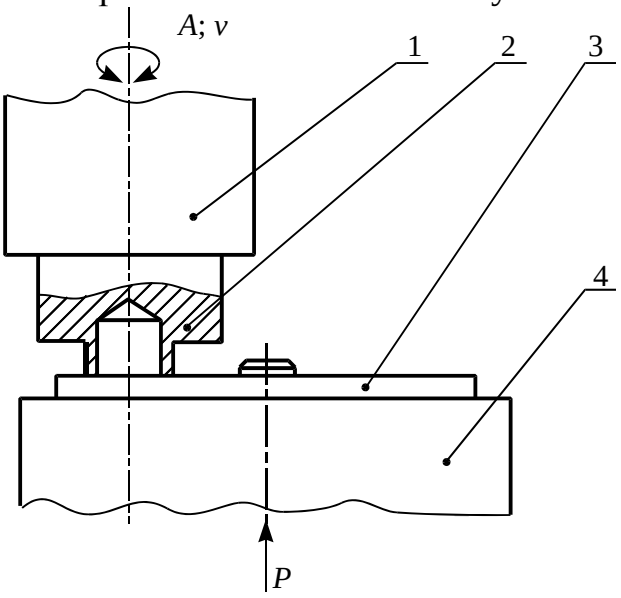
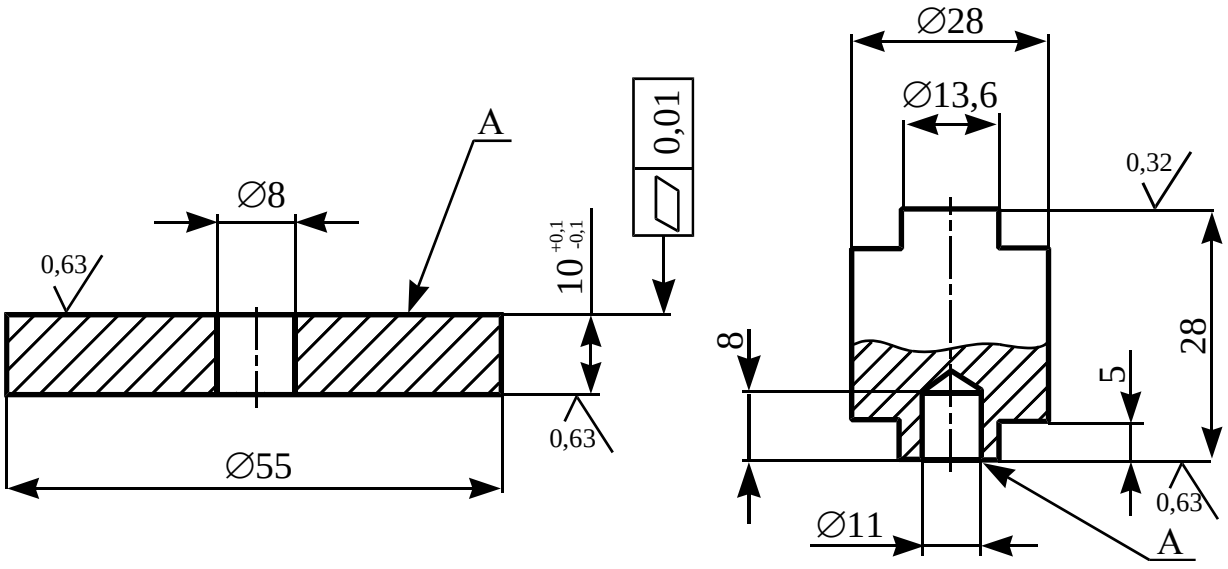


Figure 2.11 – Research scheme during fretting wear according to the plane-plane scheme: 1, 2 – moving and stationary samples; 3, 4 – fitting



a) b)

Figure 2.12 – Specimen (a) and counter-specimen (b) for fretting-wear studies according to the "plane-plane" scheme

The linear wear of each sample was measured using a profilograph-profilometer model 253. Profilograms were taken from several areas of the working surface of the sample in the radial direction. For the sections of the profilogram that corresponded to the unworn and working surfaces, the middle lines of the profile were drawn. The distance between the middle lines of the profiles is equal to the linear wear of the sample. Also, during the experiments,

the amount of linear wear of the friction pair was continuously recorded using strain gauges.

The samples were made by electric arc welding on steel 1045 coating using welding wire Sv-08, ER70S-6, 14331 wires. Counter samples were made of P – CuAl5 bronze. The research was carried out without lubrication. When testing for fretting corrosion, a salt solution was introduced into the contact area, thus creating a steel-electrolyte-bronze galvanic pair.

2.5 Method of measuring the temperature on the surface of the material

The temperature fields during the surfacing process were determined according to the scheme shown in Fig. 2.13. To do this, thermocouples were installed by drilling in the characteristic points of the test samples. The signal from which was sent to the PC through an analog-to-digital converter. Recording of the temperature change over time was recorded with the help of the developed software.

It is convenient to measure temperature remotely with the help of pyrometers.

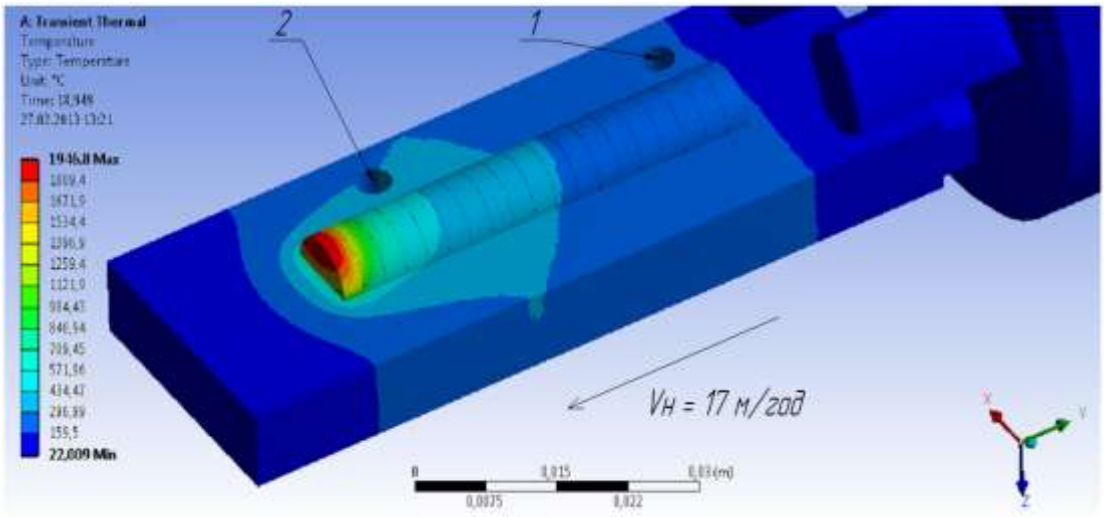


Figure 2.13 – Scheme of measuring the temperature of the part during melting (1 and 2 – places of installation of thermocouples)

2.6 Method of measuring residual deformations

To measure deformations of the workpiece, a coordinate grid was applied to the outer surfaces of the worn part before the start of surfacing, which was photographed using a digital camera. At the same time, the part and the digital camera were installed in special devices. Analogous photography was performed after the surfacing process. The obtained images were stored and processed using a package of applied graphic programs. After comparing the position of

the mesh nodes, the actual residual deformation of the workpiece was determined. Taking into account the significant inertia of the system, the measurement of the residual temperature deformations during surfacing was carried out after cooling the part to the initial temperature.

2.7 Methodology for experimental determination of hole errors caused by temperature deformations of the part and the tool during the boring process

In order to experimentally determine the influence of temperature deformations of the tool and the part during the process of drilling holes on their accuracy, thin-walled and thick-walled samples were made, which were divided into two groups, which were drilled according to the same scheme, with one difference. The first group of parts was bored with significant cooling of both the cutting zone and the outer surface of the part. The second group was bored without cooling. This approach made it possible to isolate the temperature component of the boring error.

Distortion of the hole profile after mechanical processing was determined by indicators of deviation from cylindricity: deviation from roundness in the cross section according to standard measurement methods, and deviation from straightness in the longitudinal section of the hole of the part.

The deviation of the profile in the longitudinal cross-section of the hole was studied on samples specially made and cut lengthwise on an electroerosion machine in distilled water, which ensures minimal distortion of the profile, according to two schemes:

- by direct measurement using ORIM-1 instrument microscope;
- photographing the profile with digital devices with high resolution (10 MP) and subsequent measurement of its parameters using a package of applied graphic programs AutoCad, Solid Works, etc.

The measurement data of the profilograms were subjected to statistical processing using the MathCAD mathematical package. According to the data of statistical processing, a statistically expected hole profile was built and temperature errors were determined.

3 METHODS OF STUDYING COMPOSITION AND PARAMETERS MATERIALS AND APPLIED COATINGS

When developing new composite materials, coatings, studying their structural-phase composition and its influence on wear mechanisms, as well as when studying the physical and mechanical properties of the coatings obtained from them, an important role is played by the choice of research methods. Obtaining reliable research results is ensured by the use of modern equipment and devices, checked and calibrated by metrological services, approved methods, careful preparation of samples and processing of experimental results, strict observance of the procedure for conducting the experiment.

To study the wetting of solid surfaces of materials with liquid alloys, polished plates obtained by hot pressing, cut and polished samples, which are accordingly cleaned of impurities and oxides, are used.

3.1 Methodology for the study of contact interaction in "solid surface - metal melt" systems

Liquid spreading over the surface of a solid occurs due to a decrease in free energy when the area of contact between the liquid and solid phases increases. For an equilibrium system, Jung derived equation (3.1), which relates the final contact angle θ to the interfacial energy at the interface (σ_{SL}) and the surface tension of the solid (σ_{SG}) and liquid (σ_{LG}) phases (Fig. 3.1):

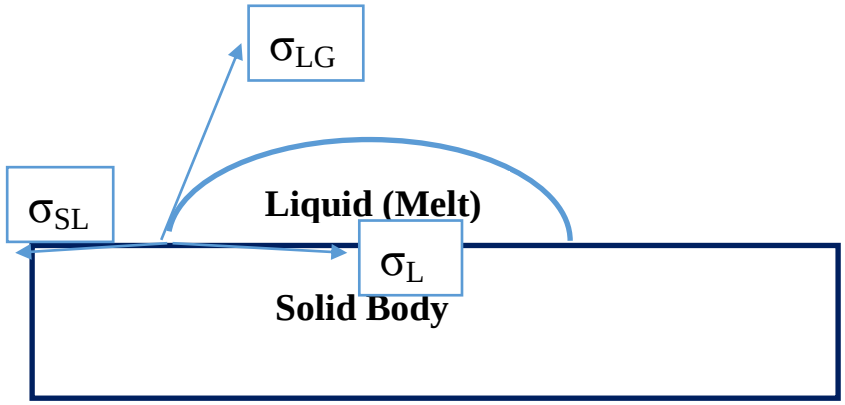


Figure 3.1 – Scheme of surface tension vectors σ_{SL}, σ_{LG} and σ_{LS} for the "solid body - liquid" system

Real spreading processes are non-equilibrium: the interphase energy changes over time. But this change occurs at a low speed and the liquid, de-forming, forms a contact angle of such magnitude that the vectors (σ_{SL}), (σ_{LG}) and (σ_{LS}) are balanced. Applying D'Alembert's principle for any instant of time, it is possible to write (for projections of vectors onto the horizontal axis):

$$\sigma_{SL} - \sigma_{LS} = \sigma_{LG} \cdot \cos(\theta). \quad (3.1)$$

The work of adhesion, that is, the energy of detachment of the liquid alloy from the surface of the solid phase, is determined from the ratio (3.2):

$$W_A = \sigma_{LG} \cdot (1 + \cos \theta). \quad (3.2)$$

he interphase tension$_2$ at the "solid – liquid" interface is determined from the ratio (3.3):

$$\sigma_{SL} = \sigma_{SG} - \sigma_{LG} \cdot \cos \theta. \quad (3.3)$$

The study of the wetting of the surfaces of metals and ceramics of different composition (TiS, TiB_2, ZrB and SgB_2) by the metal melt is carried out by the "lying" drop method. The advantages of this method are its simplicity and the possibility of determining the energy parameters of wetting (interfacial tension, work of adhesion). The study of wetting kinetics is carried out in a vacuum of 1.33 mPa at a temperature that is 50°C higher than the melting temperature of the alloy. The temperature in the furnace is controlled by a tungsten-rhenium thermocouple BP 5/20 and a micropyrometer OMP -- 43M. To further study the kinetics of wetting, we use the installation, the scheme of which is shown in fig. 3.2.

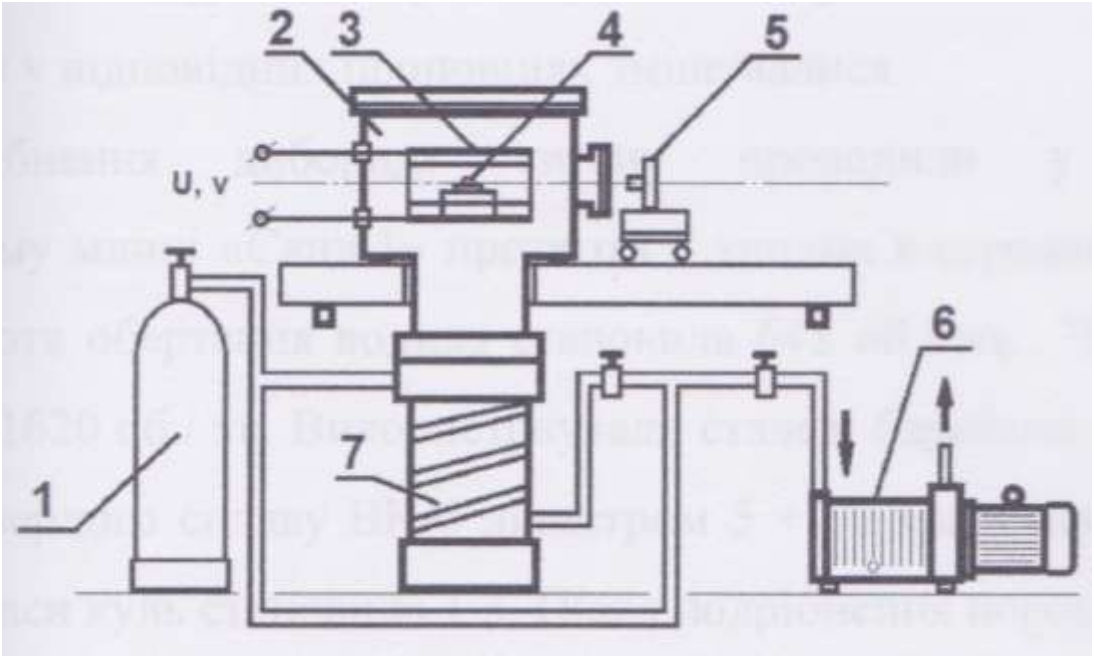


Figure 3.2 – Installation for determining the kinetics of spreading:
1 - tank for purified inert gas; 2 - vacuum chamber; 3 - furnace; 4 - sample; 5 - camera; 6 - pre-vacuum pump; 7- diffusive pump

The main part of the installation is a horizontal resistance furnace 3, which is a tungsten heater with molybdenum and nickel screens. The furnace is located in a vacuum chamber 2, which has windows for fixing with a camera 5 contact angles. The installation also includes pre-vacuum and diffusion pumps 6,7.

Before wetting, the samples are degreased, the oxide film is removed from the surface of the alloy, and the refractory substrates are polished. A sample of the alloy is placed on the flat surface of the investigated ceramic or metal and placed in the furnace 3, after which heating is carried out at the maximum possible speed. The process of spreading the alloy on a solid surface and the contact angles of wetting are determined using a digital camera 5. The process of wetting is continued for 30-40 minutes until a constant value of the contact angle θ is established.

3.2 Method of differential thermal analysis

Determining the temperatures of phase transformations in alloys and composite materials and studying the processes of phase formation in them are carried out by the method of differential thermal analysis (DTA) on the VDTA - 8M installation, created at the Institute of Physical Education named after I.M. Frantsevich of the National Academy of Sciences of Ukraine. The installation consists of a vacuum chamber with an oven, a device that regulates the heating rate, a thermocouple sensor, a recording device and a thermostat. The powder material under study is preliminarily compacted and weighed 0.25 - 0.35 grams, placed in an alund crucible. Experiments are carried out in the environment of "VCH" helium (high purity helium with a content of the main substance of more than 99.985%). Heating speed 20-80 degrees/min. Tungsten-rhenium thermocouples BP 5/20, connected according to the "combined thermocouple" scheme, are used to measure the temperature. The application of the DTA method to the studied composite materials makes it possible to determine the temperature regions of the flow of various stages of phase transformations.

3.3 Methods of studying the structure, phase and chemical composition of composite powders, cermets and gas-thermal coatings

For studies of the structure and phase composition of alloys, composite powders, cermets and gas-thermal coatings, as well as contact zones of the "alloy - substrate" interaction, metallographic, X-ray phase, micro-X-ray spectral analyzes are carried out.

Metallographic analysis of the studied materials is carried out on optical microscopes MMP-4, MIM-8M and raster electron microscopes of the type REM-106I.

X-ray studies are carried out in monochromatic Cu_{α}:

- radiation on diffractometers DRON – UMI, DRON-3. A graphite single crystal installed on a diffracted beam is used as a monochromator. Diffractograms are taken by step scanning in the range of angles 2 θ - 20-90°. The scan step is 0.05°, the exposure time at the point is 3 - 7 s. Data processing

of the diffractometric experiment is carried out using the program for full-profile analysis of X-ray spectra from a mixture of polycrystalline phase components PowderCell 2.4. The analysis of the diffraction profiles and the selection of the true physical broadening of the peaks are carried out by the method of approximations. Separation of the effects of broadening of diffraction maxima associated with the sizes of coherent scattering regions (COS) and second-order voltages is carried out in the Hall-Williamson approximation.

To study the microstructure, phase and chemical composition, as well as the distribution and content of elements in each of the phases in the studied materials, micro X-ray spectral analysis (MRSA) is used on Camebax SX-50 and JEOL JAMP 9500 microanalyzers. Quantitative MRSA in these devices is based on the excitation of the characteristic X-ray spectrum of the chemical elements contained in the surface layer of the studied area by electrons in the studied sample, and on the direct proportional relationship between the intensity of the lines of the characteristic spectrum and the number of atoms of the element in the irradiated electron volume.

To determine the porosity of coatings, as well as the number of different phases in composites and coatings, the linear method of Rosival (method of sections) is used, based on the fact that the ratio of the areas of the phases that make up the structure of the material under study can be used to determine their volume ratio.

3.4 Research methods of physical and mechanical properties of powders and coatings

Important characteristics of powder materials that determine their technological properties are fluidity and bulk density. The fluidity of the obtained powders is determined using a calibrated funnel (Hall device), and the bulk density using a Scott funnel and volume meter (for powders with low fluidity).

The microhardness of cermets and the obtained coatings is determined on the PMT-3 device by pressing a Vickers diamond pyramid into the polished surface of the sandpaper under a load of 0,05 – 0,1 N.

The adhesive bond of the coatings with the substrate is determined by the adhesive test. The type of the sample with the coating glued with the control sample is presented in fig. 3.3.

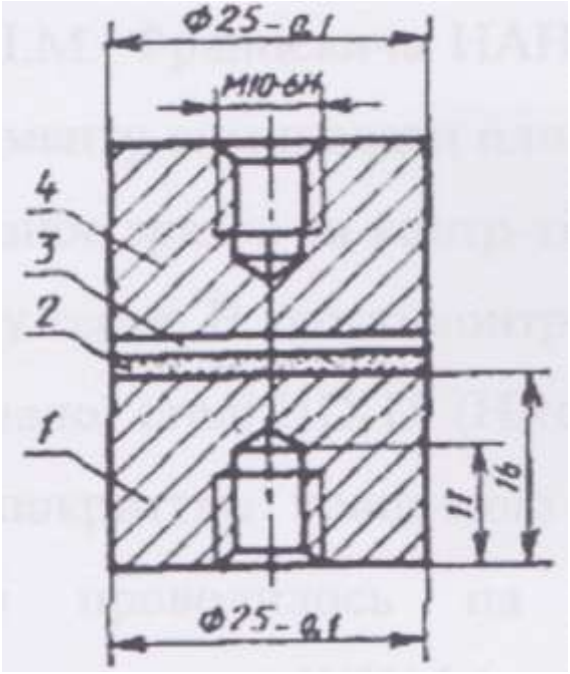


Figure 3.3 – Covered sample glued to the counterbody for adhesive strength tests: 1 – coated sample; 2 – coating; 3 – layer of glue; 4 – counterbody

The adhesion strength of the coating is determined by the formula (3.5):

$$\sigma = \frac{F}{S}, \tag{3.5}$$

where σ is the bond strength, MPa;

- pull-off force, N;
- cross-sectional area of the sample, m^2;

The adhesion strength of the tested coating is determined as the arithmetic mean of the results of five tested samples.

4 TRIBOTECHNICAL TESTING OF MATERIALS

The following characteristics are determined during tribotechnical tests of materials that are designed to work in conditions of sliding friction and wear:

- coefficients of rest friction depending on load conditions and environmental conditions;
- sliding friction coefficients depending on movement parameters, environmental conditions in the tribocontact zone;
- wear resistance for given or extreme working conditions.

4.1 Methodology and determination of friction coefficients at rest

The selection or synthesis of materials of tribotechnical pairs for use in drives of technological machines plays an important role not only from the point of view of minimizing energy losses and wear, but also as an important factor in ensuring stable movement without self-oscillations. This is especially relevant for mechanisms with a wide range of operating speeds, for starting under load and with high requirements for smoothness and accuracy of movements. For such mechanisms, it is important to have small friction coefficients with optimal characteristics. Particularly strict requirements are imposed on the friction characteristics of guides of metal-cutting machines, distribution pairs of hydraulic motors and other precise mechanisms operating in conditions of low movement speeds, where self-oscillations usually occur. A number of researchers have established that the cause of self-oscillations in mechanisms with sliding pairs is the nonlinearity of their friction characteristics. For different pairs of materials and modes of friction, parameters of the drive, very different laws of change of the coefficient of friction depending on the duration of stationary contact, speed of sliding, type and composition of lubricant, etc. have been revealed. As a rule, for each design and combination of friction pair materials, it is necessary to experimentally determine the possibility of implementing a variant of the design solution at the stage of experimental design and make the necessary corrections.

In this work, the method and results of experimental determination of the dependence of the friction force at rest on various factors are proposed. The static characteristics of friction (friction at rest) can be determined using a special setup (Fig. 4.1), which consists of an adjustable drive and a researched friction pair. The drive allows obtaining stable speeds in the range from $V=0.005\times10^{-2}$ m/s to $V=1\times10^{-2}$ m/s. The researched friction pair is made in the form of a thick-walled sleeve made of composite material and a steel cylindrical slider moving in it.

The experiments are carried out in the presence of a constant tension between the bushing and the slider of the order of 2 μm with surface roughness $Ra<0.03$ μm. The transmission of motion from the drive to the slider occurs through an elastic connection (spring) with a stiffness of C=640,000 N/m.

The tangential force applied to the slider along its axis was formed in the elastic connection C by its deformation by the drive at the speed V and is equal to:

$$P=C(Vt-X),$$

where X is the displacement of the slider,

t is an independent time.

The installation is equipped with a transducer D1 of the force P acting on the slider, displacement of the slider D2, its speed D3. Such a scheme with a friction pair – a thick-walled sleeve – a cylindrical slider is a mechanical system with one degree of freedom, where normal movements of the slider in relation to the frictional contact of the surfaces are unlikely. This makes it possible to interpret all the processes that appear during the loading of the slider, and then during its movement, as those caused by the properties of the processes of the appearance and change of the friction force in tribocontact, and not by kinematics, for example, by the separation of one body of friction from another in a pair due to their normal displacement.

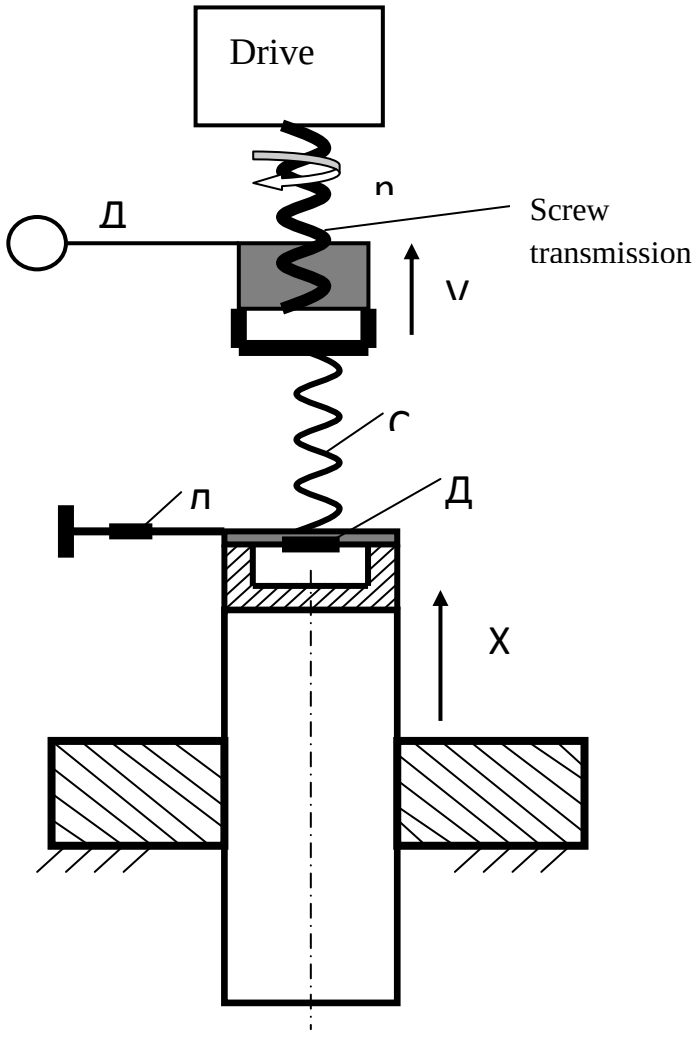


Figure 4.1 – Installation for experimental determination of friction coefficients at rest

Measurements of the resting friction force are performed under conditions of motion with self-induced relaxation oscillations. At the same time, the influence of the dP/dt derivative of the shifting force and the duration of the stationary contact before starting the slider on the resting friction force is studied.

In fig. 4.2 shows an example of a typical motion oscillogram with relaxation oscillations. The oscillogram clearly shows periods of relative rest when the speed of movement dx/dt=0 and periods of movement (jump) where the speed almost instantly increases from zero to a maximum and then decreases to zero. During the period of rest, the driving force P grows in direct proportion to the speed V of spring stretching, which is set by the drive, and the friction force is manifested as a reaction to this force. During this period, the actual strength of the formed friction bonds is greater than the frictional force fixed by the donor. And only at the moment of displacement of the slider from its place, the driving force is equal to the force of friction, which, of course, is called the force of friction at rest.

Observing the movement of the slider at such low speeds allows a deeper understanding of the physics of the processes of formation of friction pair interaction in tribocontact and the postulate that the friction force F manifests itself only as a reaction to external influences.

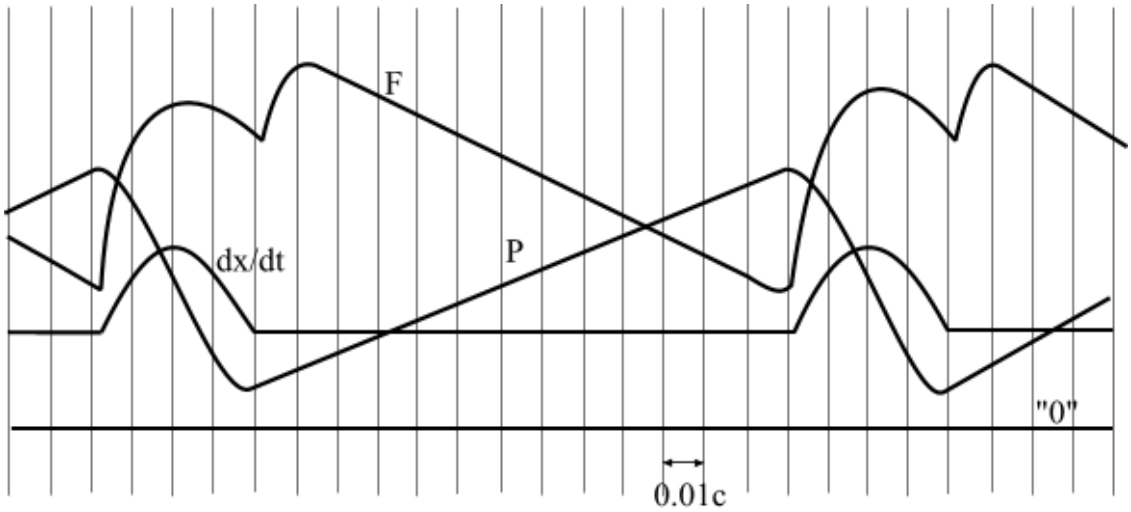


Figure 4.2 – Example of a typical motion oscillogram with relaxation oscillations

Fig. 4.3 shows examples of oscillograms of the movement of the slider with different speeds of growth of the driving force. The condition for starting the movement of the slider is the equality of the driving force P and the friction force F. Therefore, the value of the force that displaces the slider from its place determines the friction force at rest. To study the factors affecting the magnitude of the force F, oscillography of the change in the driving force P was performed in the mode of relaxation oscillations (in this series, the readings of the speed sensor dx/dt, sensors of the friction force F and displacement were not displayed on the oscilloscope). The occurrence in a fairly wide range of speeds of stable

relaxation oscillations of the slider movement, obtained on this experimental setup, and their identity with the relaxation oscillations of the slider movement in a system with two or more degrees of freedom, allows us to assert their single nature.

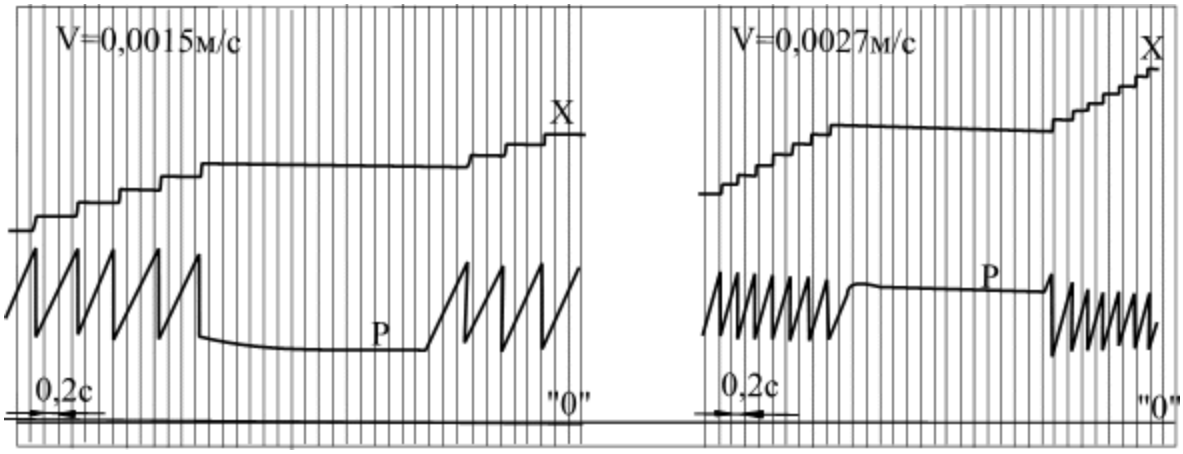


Figure 4.3 – Examples of oscillograms of slider movement with different speeds of growth of driving force

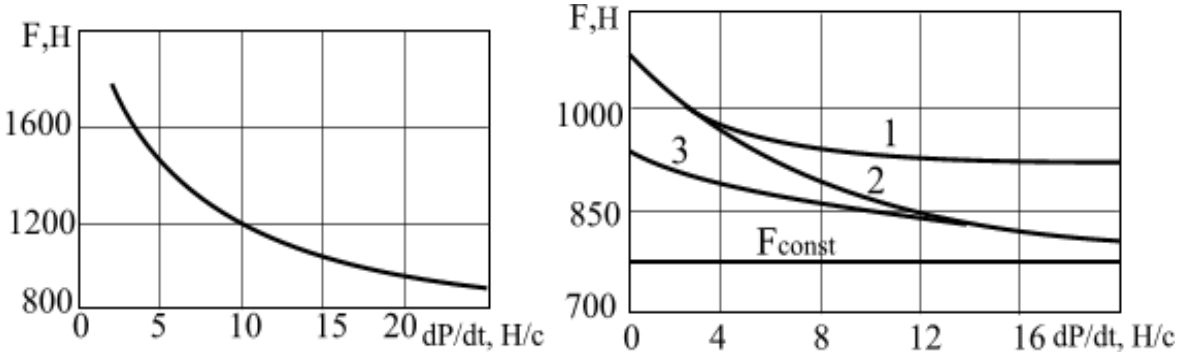
In order to determine the dependence of the friction force at rest on various factors, experiments are carried out to determine the influence of the driving force during the endurance of friction surfaces in stationary contact, on the force of friction at rest in the 1st jump, the influence of the direction of movement before endurance, as well as the nature of the slider's setting movements before the start of the experiment -ryments It was established that the factors listed above do not have a significant effect on the formation of frictional bonds and the magnitude of the force of friction at rest. The exception is cases when the frictional contact was loaded with a driving force that almost ensured displacement and was kept in this state for a certain time. In this case, the friction at rest has a value that exceeds its value under other conditions. It should be noted that the degree of proximity of the driving force to the friction force at rest has a significant effect on the force of rest friction. It is obvious that the increase in the strength of friction bonds is a consequence of their arrangement in the process of the previous microshift, which is characteristic of similar pairs of friction.

In order to rule out the influence of the position of the slider in the sleeve and possible non-uniform wear of the friction surfaces, the experiment was carried out for each speed several times along the entire length of the slider. The stability of the obtained results in all sections of the slider was the basis of their reliability. The methods of probability theory and mathematical statistics were widely used to process the results. It was established that all the parameters of the characteristics of the friction forces in the batch of experiments quite well fall under the laws of normal distribution.

A series of experiments with dry friction obtained the characteristics of friction at rest depending on the load speed parameters for a pair of steel - composite cast iron (Fig. 4.4). The influence of the duration of the stationary contact on the friction at rest was not revealed in this series of experiments.

Experimental studies of sliding processes with lubrication were carried out using oils I-20, Tkp-22, AMG-10. There was no qualitative difference between the friction characteristics when lubricated with different oils. Fig. 4.3 shows examples of oscillograms of slow movements when lubricating with Tkp-22 oil for the regime with spring stretching speed V = 0.004 m/s. The first jump on the oscillogram occurs at a driving force that significantly exceeds its value in subsequent jumps. After several jumps, the force of rest friction gradually decreases, and then stabilizes and remains constant. The oscillograms showed the dependence of the difference between the friction force at rest in the 1st jump and in constant oscillations, as well as the dependence of the friction force at rest in constant oscillations from the load growth rate V.

This is a consequence of the dependence of the force of rest friction on two main factors: the duration of stationary contact (the force of rest friction in the 1st jump is greater than in the following ones) and the speed of the tangential load (the force of rest friction in constant oscillations is not the same for different speeds) spring stretching V).



a b

Figure 4.4 – Influence of the rate of increase of the shear force on the resting friction force (sliding without lubrication)

A special technique is used to divide the force of rest friction into two components: as a function of the duration of stationary contact F(t) and as a function of the speed of tangential loading F(dP/dt). At the same time, set the specified speed V of the drive and perform oscillography of the change in driving force after exposure of the friction pair in a stationary state with different fixed durations t. Because at the moment of displacement, the driving force and the force of friction are equal, the force of rest friction is determined from the driving force. The dependence F(t) of the friction force at rest is constructed as the difference of its value in the 1st jump and in constant oscillations. It was found that an increase in the duration of stationary frictional contact to 2 – 3 seconds leads to a corresponding increase in frictional forces at rest, provided there is lubricant. A further increase in the duration of stationary frictional contact has no effect on the friction force at rest. Fig. 4.5, b) shows the change in the resting friction force (curve-2) F(t) as a function of the duration of stationary contact.

Fig. 4.5, a) shows the component of the friction force, which depends on the speed of the tangential load F(dP/dt), which can be obtained from the values

of the friction force at rest in the 1st jump after a long stop by calculating from it the maximum the value of the friction force component F(t).

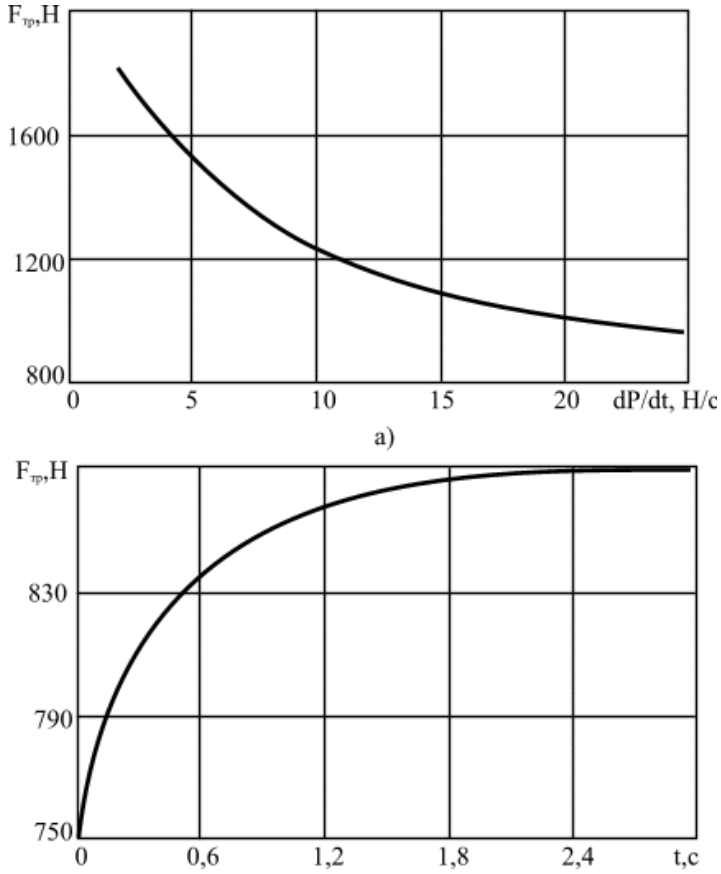


Figure 4.5 – Static characteristics of friction (tribocontact with grease):

a) dependence of the resting friction force on the rate of growth of the shear force;

b) dependence of the force of rest friction on the duration of stationary contact

CONCLUSIONS

1. In couples with sliding friction with lubrication at low speeds, the resting friction force should be considered as consisting of the following most significant parts: constant component - curve, Fig. 4.4, b); component of friction, which depends on the duration of stationary contact F(t) – Fig. 4.5, b); component friction, which depends on the speed of the tangential load F(dP/dt) – Fig. 4.5, a).

2. In pairs with sliding friction without lubrication at low speeds, the force of rest friction should be considered as including a constant component and dependent on the speed of the tangential load F(dP/dt) – Fig. 4.4.

b)

4.2 Methodology and determination of wear resistance of metal carbide materials and coatings

Most of the working surfaces of machine parts work under conditions of abrasive wear. This is due to a high level of contamination with abrasive particles (dust) coming from the external environment and products of wear of sliding surfaces. The working bodies of agricultural, mining, and road machines are designed to function in an abrasive environment.

Methods of laboratory testing of materials for wear under the influence of abrasive particles are known. The abrasive can be fixed (for example, emery cloth) or unfixed - for example, quartz sand in a liquid medium. The microhardness of sand is usually estimated at 10.0 – 13.0 GPa. According to the results of the experiment (see section 5), the microhardness of the reinforcing carbide structural components of the obtained coatings is close to this value, and the microhardness of the matrix is two to three times smaller, but still comparable to it. Therefore, it was planned to conduct two stages of testing. The first involved wear of samples on a grinding cloth with an abrasive made of carborundum, and the second - wear on an abrasive layer of quartz sand in a liquid medium. The hardness of carborundum is much higher (it is about 20.0 GPa) and the first stage of testing makes it possible to evaluate the ultimate characteristics of the strength of the coating, and the second – simulates operating conditions that are as close as possible to real working conditions.

Reference samples for determination of relative wear resistance were made of 40KHN steel (a common material for cutting bodies of earthmoving and drilling machines), which were subjected to heat treatment (hardening at 820 °C while cooling in oil and tempering at 300-350 °C). The hardness of such samples was HRC 53-56.

4.3 Design of the experimental setup

To evaluate the tribotechnical properties of coatings, an experimental setup was used for testing working surfaces for wear on an abrasive layer. The schematic diagram of the installation is shown in fig. 6.6. According to the conditions of wear, it is similar to the "fixed ring" type machine [24], but three samples fixed at an angle of 120°, one of which is the standard, are subject to wear at the same time.

The wear resistance was evaluated as absolute (by linear wear) and in relation to the reference sample (made of 3135 steel). The use of three support points ensures the flatness of the trajectory of the samples along the abrasive layer. Structurally, the installation can be used in two modifications that meet the conditions of abrasive wear on fixed and non-fixed abrasives.

During the first version of the tests, the sandpaper is fixed on a stationary disc. The samples and the standard were fixed on disk 3, which is installed on the vertical spindle of the installation. The disc with fixed samples rotates from

the drive with a rotation frequency n. The samples are worn by describing circular trajectories of close radii, and the standard is on the average circle.

Test conditions: sample effort – 150 N; shaft rotation frequency with fixed samples – 1.0; the friction path of the sample for one test interval – 100 m (10 min.). The dimensions of the samples are 5×15 mm^2. The area of the sample on which the friction process takes place is 75 mm, the pressure is 2.0 MPa.

According to the second option, all three samples were placed in a container filled with sand and water and went through the same friction path. The shaft rotation frequency is 3.0 revolutions per second.

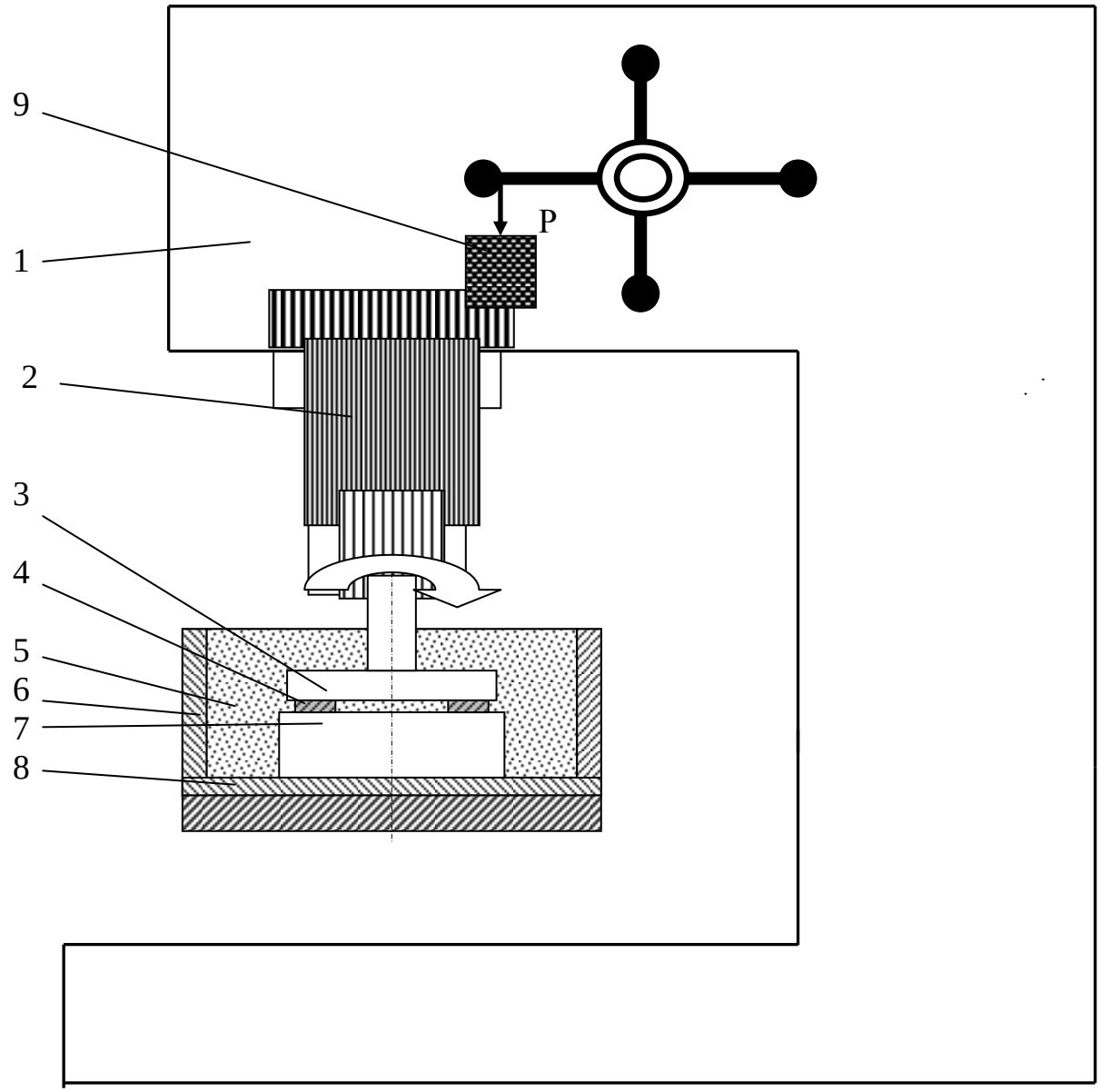


Figure 4.6 – Scheme of the experimental setup for tests on abrasive wear resistance:
1 – bed; 2 – spindle; 3 – drive disk with samples to be examined;
4 – samples; 5 – friction process environment; 6 – capacity;
7 – counterbody; 8 – installation table;
9 – load P

4.4 Results of research on wear resistance

The results of the experiment are shown in tables 4.1 – 4.3.

The tables describe the structure of the coatings. The carbide phase with a size of more than 0.5 – 1 μm was considered as excess, structurally free, although by origin they are eutectic carbides that grew in size during the time of exposure of the samples during the formation of the coating.

During tests on the fixed abrasive, the wear resistance of the obtained coatings is 2 – 2.4 times higher than that of 3135 steel. It depends on the volume ratio between the matrix and the strengthening carbide phase, their microhardness. The influence of the microhardness of the matrix on the wear resistance of the coating is more pronounced than that of the carbide phase. This can be explained by the fact that the hardness of carbides and abrasive is closer. An increase in the hardness and content of the structurally free carbide phase with a simultaneous decrease in the hardness of the matrix does not lead to an increase in wear resistance.

Table 4.1 – Relative wear resistance of composite coatings during tests on fixed abrasive

№	Coating thickness, mm	Coating structure	Microhardness, GPa		Relative wear resistance
			Carbides	Matrices	
1	1.6	carbide phase - 10%(0.5- 1,5 μm)	10,0	6,0	2.3
2	1,6	carbide phase - 20% (~1 μm)	10,0	8,0	2, 6
3	1.8	carbide phase -30% (~ 3 μm) dendrites of a solid solution -25%	12,0	6,0	1.8
4	1.4	carbide phase -20% (1-2 μm)	10,0	8,0-9,0	2.5
5	1.5	carbide phase 30% (5 - 10 μm)	13,0	8,0	2,4
6	1.6	carbide phase - 10% (2 μm)	11,0	8,0	2,2
7	1.5	carbide phase -30% (15-20 μm)	11,0	7,0	2.5
8	1.4	carbide phase - 25% (25-40 μm), eutectic	14,0	8,0	2.4
9	1.5	carbide phase -25% (40-50 μm), eutectic	13,0	7,0	2.35
10	1.5	carbide phase - 10% (5-15 μm)	12,0	8,0	2.2
11	1.5	carbide phase -30% (до 100 μm), solid solution	10,0- 13,0	7,0	2.0

Table 4.2 – Relative wear resistance of composite coatings when worn in a hydroabrasive environment

Coating No	Coating thickness, mm	Relative wear resistance
1	1.5	1.9
2	1.5	2.5
3	1.5	2.6
4	1.5	2.7
5	1.5	2.3

During tests in a water-abrasive environment, the wear resistance of all the analyzed coatings is higher, which is explained by the lower microhardness of the abrasive sand particles than in the abrasive cloth, and the wear is less intense.

Table 4.3 – Linear wear of composite coatings based on carbides of refractory metals during friction on a fixed abrasive

System / mode	Structural components	Ratio between structural components	Microhard-ness, GPa	Linear wear, mm/100m
Cr + C / 1	carbide phase + solid solution	1 : 3	11,0 - 5,0	0.1
Cr + C / 2	carbide phase + eutectic	1 : 4	12,5 - 6,0	0.05
V + C / 1	carbide phase + solid solution	1 : 3	11,0 – 5,0	0.1
V + C / 2	carbide phase + евтектика + solid solution	1 : 2 : 2	13,0 – 7,0	0.02
W + C / 1	carbide phase + solid solution	1 : 2	14,0 - 5,5	0.01

The analysis of the results shows that the wear resistance of the coatings is determined by the amount and size of the carbide phase, the microhardness of the matrix and especially increases in the presence of eutectics. The wear resistance is reduced by the presence and unfavorable orientation of solid solution dendrites, which have a relatively low microhardness. Increasing the size of carbides does not have a significant effect on wear resistance.

The composition of the mixture, the characteristics of the components, the thermal regime of the coating formation process determine its structure and, as a result, its wear resistance. Therefore, in order to develop the coating formation

technology, it is first necessary to determine the interdependence between the parameters of the technological process, structure and wear resistance.

4.5 Study of wear resistance in the process of abrasive wear of composite coatings using a multifactorial experiment

In order to determine the technological factors of the formation of coatings on parts that most affect wear resistance, the nature and degree of their influence on it, i.e. to establish direct connections between the mode of application of coatings and their properties, a study was conducted using methods multivariate experiment. For all samples, coating was carried out using furnace heating, according to the layering scheme "part - metal powder - carbon fiber material". The tests were carried out on the above-described installation with the specified parameters. Mass wear was studied, which was determined on analytical balances with a value of 0.1 mg division every 10 minutes of testing.

Based on the conducted experiments, the following factors were selected for the study:

- external heating temperature;
- duration of external heating;
- content in the mixture of carbon material (calculated from its surface density, density and thickness of the powder layer).

It was assumed on the basis of preliminary data that all these parameters, as factors of the wear resistance research experiment, meet the conditions of their controllability, independence and compatibility with each other. The controllability of the factors was considered as the possibility of giving them an arbitrary level in the area of definition and fixing them constant throughout the experiment, independence - as the absence of a correlation between them, and compatibility - as the possibility of fixing each of them at any level regardless of the values of the levels other factors.

Conducting a full multifactorial experiment is planned in the range of permissible values of these three factors, which are determined by preliminary calculations and experimental studies. The method of developing a mathematical model of wear resistance of coatings was chosen, which allows to present it in the form of a polynomial of the first degree:

$$Y= b_0 + b_1X_1+b_2X_2+ b_3X_3 +b_{12}X_1X_2+ b_{13}X_1X_3+ b_{23}X_2X_3+ +b_{123}X_1X_2X_3 . \quad (4.1)$$

The selected factors are marked with X_1, X_2, X_3. Coded variables were introduced to simplify calculations. Levels of factors, experiment and change intervals in natural and coded values are given in table 4.4.

Table 4.4 – Levels of experimental factors and intervals of change in natural and coded values

Levels of factors	Найменування факторів та їх натуральні значення		
	Temperature heating, °C X_1	Duration heating, min, X_2	Carbon content in mix%, wt., X_3
Base Level (0)	1200	40	15
Variation interval (I)	100	20	5
Top level (+1)	1300	60	20
Lower level (-1)	1100	20	10

Table 4.5 – Planning matrix and results of the wear resistance experiment

№ of the experiment	X_0	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	$X_1X_2X_3$	У
1	+	+	+	+	+	+	+	+	0.55
2	+	-	+	+	-	-	+	-	0.49
3	+	+	-	+	-	+	-	-	0.55
4	+	-	-	+	+	-	-	+	0.7
5	+	+	+	-	+	-	-	-	0.6
6	+	-	+	-	-	+	-	+	0.68
7	+	+	-	-	-	-	+	+	0.7
8	+	-	-	-	+	+	+	-	0,75

Table 4.5 shows the coding of the values of factors +1 and –1. To simplify notation, the unit is usually omitted.

A column of the fictitious variable X0 is entered into the matrix, which in all cases takes only the value "+" and serves to calculate the free term b0. Columns of double and triple interaction are obtained by multiplying columns X_1, X_2, X_3 and are used to calculate the corresponding coefficients of equation (4.1). For setting up each of the eight experiments, factors X_1, X_2, X_3 are set in accordance with the planning matrix (table 4.5 on the upper (+) or lower (-) level.

To ensure the required accuracy of the experiment, three independent experiments with unchanged modes were conducted. The homogeneity of the dispersion of parallel experiments was assessed using the following method.

The average arithmetic value of the optimization parameter for each strip of the matrix was determined by the formula

$$\bar{y}_j = \sum_{u}^{r} \frac{y_{ju}}{r},$$

where r is the number of parallel experiments;

u – parallel experiment number;

y_{ju} – is the value of the response function in the u-th parallel experiment of the j-th row of the matrix.

To estimate its deviations from the average value, the variance of parallel experiments was calculated

$$S_j^2 = \sum_{u=1}^{r} \frac{(y_{ju} - \bar{y}_j)^2}{(r-1)}.$$

The homogeneity of the variance of parallel experiments was checked by the Cochrane criterion, which is the ratio of the maximum variance to the sum of all variances

$$G_p = \frac{S_{j\max}^2}{\sum_{j=1}^{N} S_j^2}.$$

The hypothesis of the homogeneity of variances is confirmed if the calculated values of the criterion do not exceed the tabulated values (Table 4.6).

Table 4.6 – Cochrane G-criterion value (α=0.05)

$f_2=N$	$f_1=r-1$				
	1	2	3	4	5
4	0,9065	0,767	0,684	0,628	0,589
6	0,7808	0,616	0,582	0,486	0,444
8	0,6798	0,505	0,437	0,391	0,359
10	0,6020	0,445	0,373	0,331	0,302

The level of significance of all considered criteria was assumed to be α=0.05. At the same time, the probability P of a true experiment is

P=1-0.05=0.95 or 95%.

The calculated value of the criterion is compared with the table value for the degrees of freedom of the numerator f_1=r-1=3-1=2 and the denominator f_2=N=8. If Gtable >0, then the hypothesis of homogeneity of parallel experiments is accepted.

From where the repeatability variance will be equal to

$$S^2(y) = \sum_{j=1}^{N} \frac{S_j^2}{N}.$$

The error of the experiment is

$$S(y) = \sqrt{S^2(y)}.$$

To carry out calculations, use programs in one of the computer programming languages. Below are the results of the program.

Table 4.7 – Value of dispersion of parallel experiments

№ exper.	1	2	3	4	5	6	7	8
Variance of parallel experiments	0.0045	0.001	0.0014	0.0053	0.0022	0.0032	0.0021	0.0017

The calculated value of Cochren's G-criterion is 0.3231, the table value (at f_1 = 2 and f_2 = 8) is 0.5057, that is, the hypothesis of homogeneity of parallel experiments is accepted. The repeatability variance is 0.00218, and the experimental error is 0.0467.

The coefficients of the regression equation (4.1) were calculated according to the following sequence. The free term b_0 is calculated by the formula

$$b_0 = \sum_{j=1}^{N} \frac{\overline{y_j}}{N}.$$

The coefficients characterizing the linear effects of the model (b_1,b_2,b_3) are determined from the expressio

$$b_u = \sum_{j=1}^{N} \frac{X_u \overline{y_j}}{N}.$$

Coefficients of the interaction effect (X_1X_2, X_2X_3, X_1X_3, $X_1X_2X_3$) are calculated from the dependence

$$b_{12} = \sum_{j=1}^{N} \frac{X_{1j} X_{2j} \overline{y_j}}{N}.$$

Based on the results of the research, the regression equation was obtained:

$$Y = 0.63 - 0.028X_1 - 0.047 X_2 - 0.055 X_3 + 0.022 X_1 X_2 + 0.005 X_1 X_3 - 0.006 X_2X_3 + 0.03 X_1X_2X_3.$$

The statistical significance of the coefficients was checked using the Student's t-test. For a full-factor experiment, the errors of all coefficients are equal to each other and are determined by the formula

$$S(b_i) = \frac{S(y)}{\sqrt{Nr}}.$$

Next, a confidence interval of length $2\Delta b_i$ is determined:

$$\Delta_{b_i} = \pm t_{кр} S(bi).$$

The critical value t_{kr} = 2,12 was taken according to the table. 4.8 for the number of degrees of freedom N(r-1)= 16.

Table 4.8 – Student's t-test value

f(r-1)	5	6	7	8	9	10	11	12	13	14	15	16
The value of the t-test	22,571	22,447	22,365	22,306	22,262	22,228	22,201	22,179	22,160	22,145	22,131	22,12

The coefficient is significant if $|b_i| \geq \Delta b_i$. Statistically insignificant elements are excluded from the equation. The coefficients in the equation will indicate the strength of the influence of the factors. The larger the coefficient, the more impact it has.

If the factor has a "+" sign, then the optimization parameter increases as the factor value increases.

The "-" sign at the coefficient shows that if this factor increases, the influence of the optimization parameter decreases.

The check showed that two coefficients (b13 and b23) should be excluded from the regression equation (confidence interval was 0.2). After correcting the

regression equation, a mathematical model of the dependence of the wear resistance of the coating on the parameters of its application was obtained:

$$Y = 0.63 - 0.028X_1 - 0.047\ X_2 - 0.055\ X_3 + 0.022\ X_1\ X_2 + 0.03\ X_1X_2X_3.$$

Adequacy was tested using Fisher's test

$$F_p = \frac{S^2_{ad}}{S^2(н)}.$$

Here is the adequacy variance

$$S^2_{ad} = r \sum_{j=1}^{N} \frac{(\bar{y}_j - \hat{y}_j)^2}{N - \lambda},$$

where λ is the number of significant coefficients of the equation;

$\bar{y}_j$ – the average arithmetic value of the optimization parameter in the j-th trace;

$\hat{y}_j$ – the value of the optimization parameter, which is calculated by the model for the condition of the j_{th} experiment.

Since the condition F_p <. F_{tabl} is fulfilled, the developed model is adequate.

The analysis of the obtained model showed that all the selected factors, with an increase in the studied interval, affect the wear resistance of the coatings in the direction of its increase, since the coefficients in the regression equation, which models the wear process, are negative. But quantitatively, the influence of the time of exposure of the heated system and the carbon content is more significant than that of such a factor as the heating temperature, and the coefficients that take into account the effects of the interaction of this factor with others turned out to be insignificant. This means that increasing the heating temperature above 1100°C does not have the same effect of increasing wear resistance as the intensification of other technological parameters.

This can be explained by the fact that the heating temperature has less effect on the amount of carbide phase that is formed, and the excessive growth of carbides above the optimal size can even negatively affect the wear resistance (Fig. 4.7).

Therefore, in order to ensure high wear resistance, it is more profitable to organize the coating process with a longer exposure time at the minimum required heating temperatures and to increase the carbon content in the initial mixture. This conclusion is also supported by the fact that exposure to high temperatures leads to deterioration of the substrate metal structure.

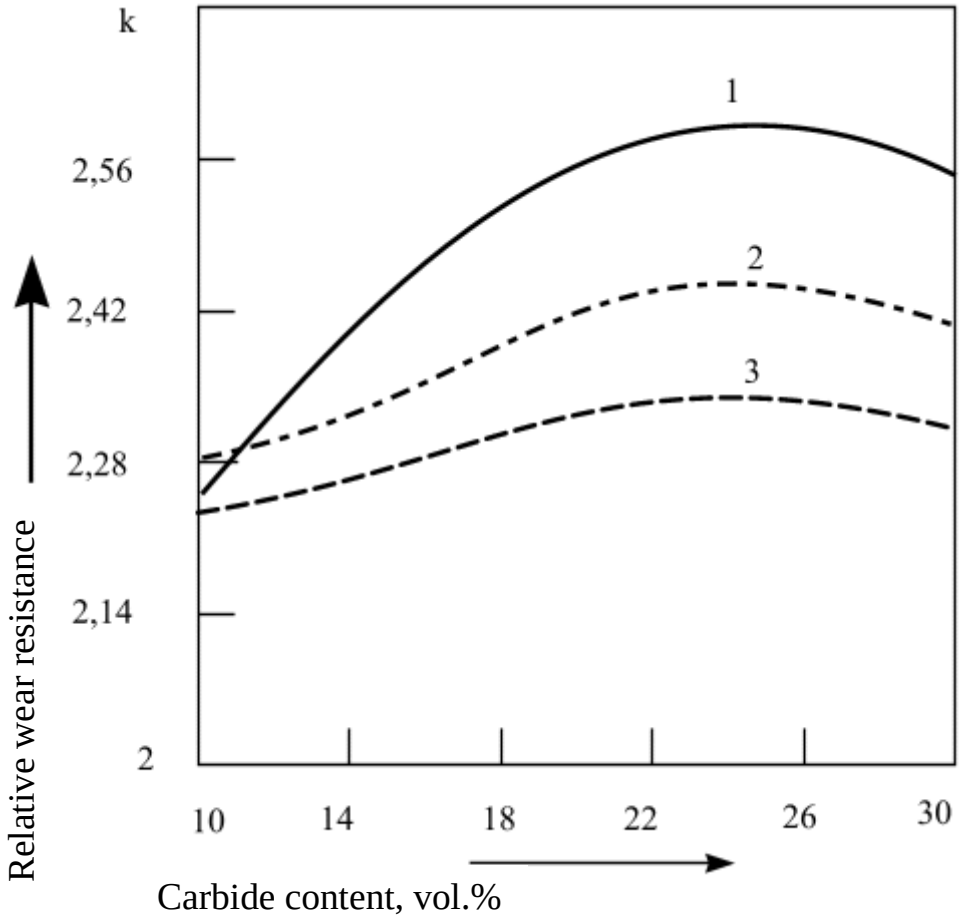


Figure 4.7 – Dependence of wear resistance of composite coatings on the content of carbides with dimensions: 1) 2 – 5 μm; 2) 5 – 15 μm; 3) more than 15 μm

4.6 Methodology for studying the tribotechnical properties of gas-thermal coatings

The study of the wear process of materials and coatings made of alloys and composite coatings is carried out using two methods.

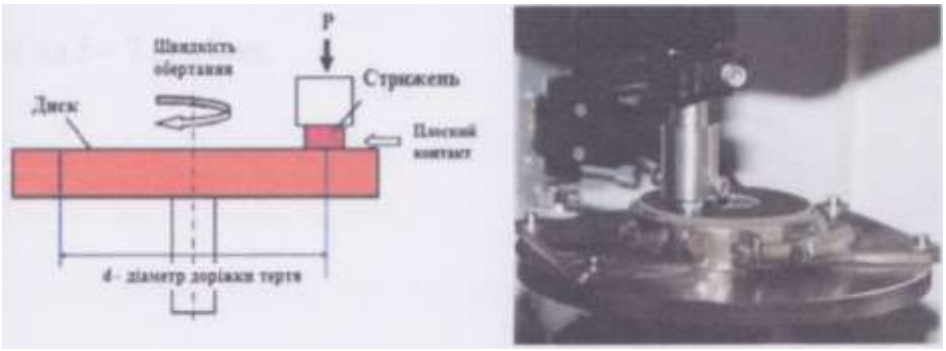
According to the first method, the tests were carried out under conditions of sliding friction without lubrication according to the “shaft - partial liner” scheme on friction machines, the design of which allows the linear wear of the pair and the friction coefficient to be measured simultaneously during the experiment. The weight wear of the sample and the counterbody is determined by weighing after each kilometer of the friction path. As a counterbody (shaft), disks with a diameter of 40 mm from hardened steel 100Cr6 (HRC 55), 41Cr4 are used. As a sample (liner), a rod with a diameter of 5 mm with a coating thickness of 0.5 - 0.7 mm or a prismatic sample is used. Preliminary running-in of the coating is carried out on a diamond wheel, which is installed instead of a disk made of steel 100Cr6 and has the same dimensions. After the sample is worn in to the

diamond disc, a counter body is installed and the coating is finally worn in on it. To obtain reliable comparative results, tests of all materials and coatings are carried out under the same conditions. The load value is P = 2 kg (diameter 5 mm, pressure rpit = 1 MPa), the length of the friction path L = 5 km, the sliding speed V = 0.5; 1.0; 3.0 and 6.0 (m/s) or others in accordance with the operating conditions.

According to another method, tests are also carried out under sliding friction conditions without lubrication according to the “finger - disc” scheme on the Bruker UMT Multi-Specimen Test System tribotester (Fig. 4.8 and 4.9).

Figure 4.8 – Bruker UMT Multi-Specimen Test System tribotester

This device allows for automatic measurement of the main parameters of the friction process in a short time over a wide range of speeds and loads, with their registration on a computer.



а б

Figure 4.9 – Test scheme (a) and view of the friction unit (b) on the Bruker UMT Multi-Specimen Test System tribotester

A cylindrical rod with a diameter of 5 mm and a height of 15 mm is used as a sample, and a steel cylindrical disk with a diameter of 40 mm and a height of 10 mm is used as a counterbody. The surfaces of the disk and rod are ground and then polished to a roughness Ra of 0.5 μm. Immediately before the tests, the samples are cleaned ultrasonically in an acetone environment for 2 minutes to remove possible contaminants.

To ensure tight contact between the surfaces of the disk and rod, the friction pair is pre-hardened at a load of 800 g and a rotation speed of 60 rpm. Hardening is performed in several stages until the contacting surfaces are completely adjoined, using abrasive paper with silicon carbide with an average grain size of 12 μm and 4 μm. After running-in, the surface of the disk and rod is treated with alcohol to remove wear products and abrasive particles.

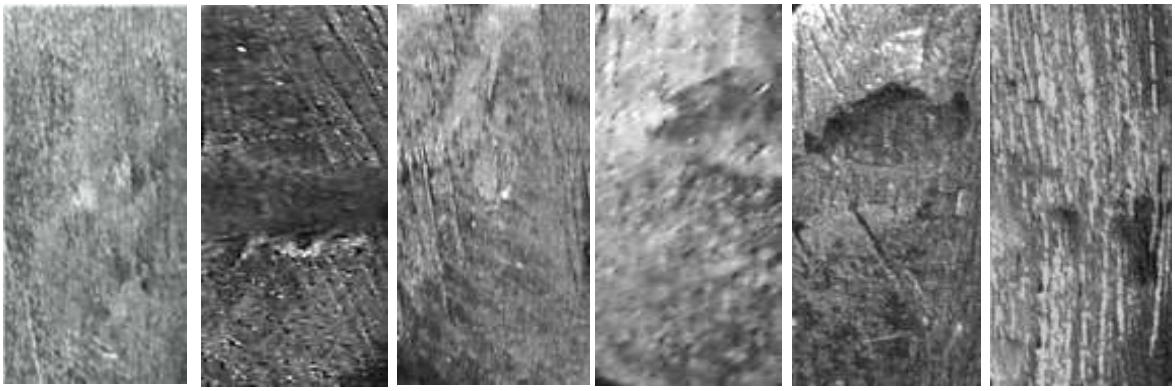
The load value is P = 0.4 – 0.8 kg (with a diameter of 5 mm, rpit = 0.2 – 0.4 MPa), the length of the friction path L = 680 – 1800 m, the sliding speed V = 0.5; 1.0; and 1.5 (m / s).

During the tests, the friction coefficient and vertical movement of the finger are recorded. After the tests are completed, the total wear of each friction pair is determined and these values are brought to 1 km of the path.

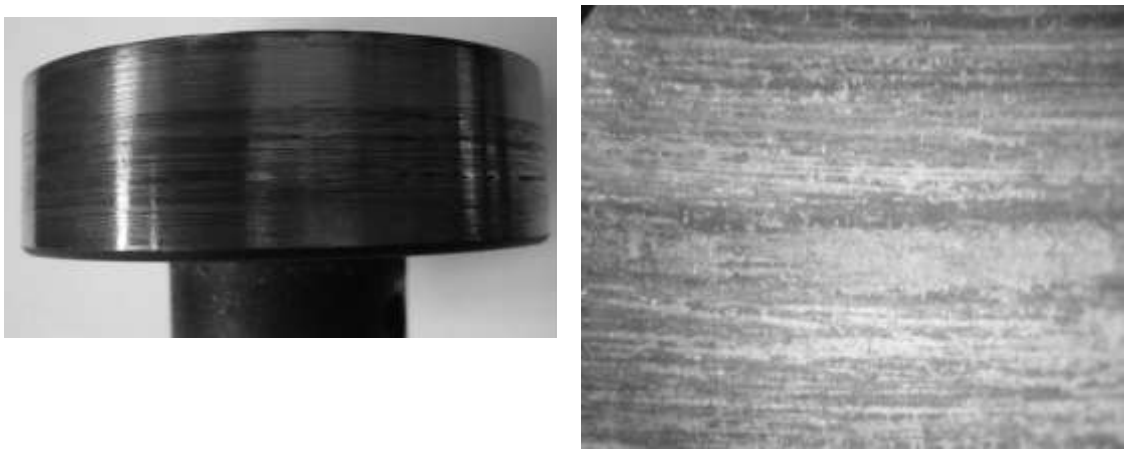
In both methods, the main parameters are determined after the completion of the work as the arithmetic mean value from measurements carried out on 5 - 7 samples.

4.7 Wear resistance of high-carbon layers deposited by the method of electric arc welding using carbon fiber materials

The friction surfaces of the deposited high-carbon coatings and the counter body made of hardened steel 45 after working in conjunction on a path of about 1800 meters are shown in Fig. 4.10.



a)



b)

Figure 4.10 – General view of the surfaces of the friction pair of samples (a) and the counterbody (b) after wear under dry friction conditions

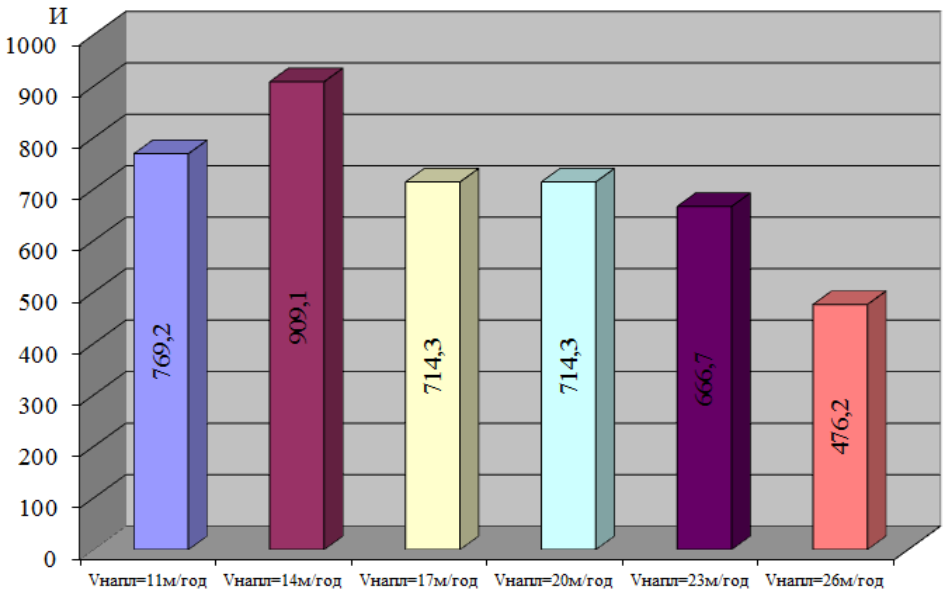


Figure 4.11 – Wear resistance of a high-carbon coating when changing the deposition rate

Table 4.9 – Main parameters of the friction pairs: high-carbon coating – steel C45-1, with different coating deposition rates

Coating and counterbody	Hardness, HRC	Mass, г		Weight wear, g
		To friction	After friction	
1	2	3	4	5
	coating deposition rate 11 m/h			
Sample 1	50	34,8498	34,8483	0,0013
Sample 2	52	34,8512	34,8495	
Sample 3	54	34,8365	34,8358	
Контртіло	54-56	182,6150	180,3210	2,294
	coating deposition rate 14 m/h			
Sample 1	52	36,8115	36,8103	0,0011
Sample 2	53	36,9527	36,9517	
Sample 3	51	36,8017	36,8006	
Counterbody	54-56	183,9090	181,62	2,289
	coating deposition rate 17 m/h			
Sample 1	48	32,4627	32,4612	0,0014
Sample 2	50	32,2158	32,2141	
Sample 3	46	32,9035	32,9025	
Counterbody	54-56	182,3260	180,4070	1,919
	coating deposition rate 20 m/h			
Sample 1	44	37,1072	37,1054	0,0014
Sample 2	47	37,5612	37,5597	
Sample 3	41	36,9873	36,9864	
Counterbody	54-56	185,7921	184,4733	1,3188
	coating deposition rate 23 m/h			
Sample 1	44	33,0835	33,0823	0,0015
Sample 2	45	33,1642	33,1626	
Sample 3	43	33,5316	33,5299	
Counterbody	54-56	189,6928	188,3895	1,3033
	coating deposition rate 26 m/h			
Sample 1	40	34,3149	34,3127	0,0021
Sample 2	42	34,7359	34,7335	
Sample 3	38	34,2951	34,2934	
Counterbody	54-56	187,6951	186,3492	1,3459

The weight wear of the coatings Δm obtained at different deposition rates is determined by the change in mass loss. As can be seen from (Fig. 4.12), the weight wear of high-carbon coatings at a friction path of 1756 meters varies on average within 0.0011 – 0.0021 g. However, the mass loss of the counterbody varies within 1,3033 – 2,294 g.

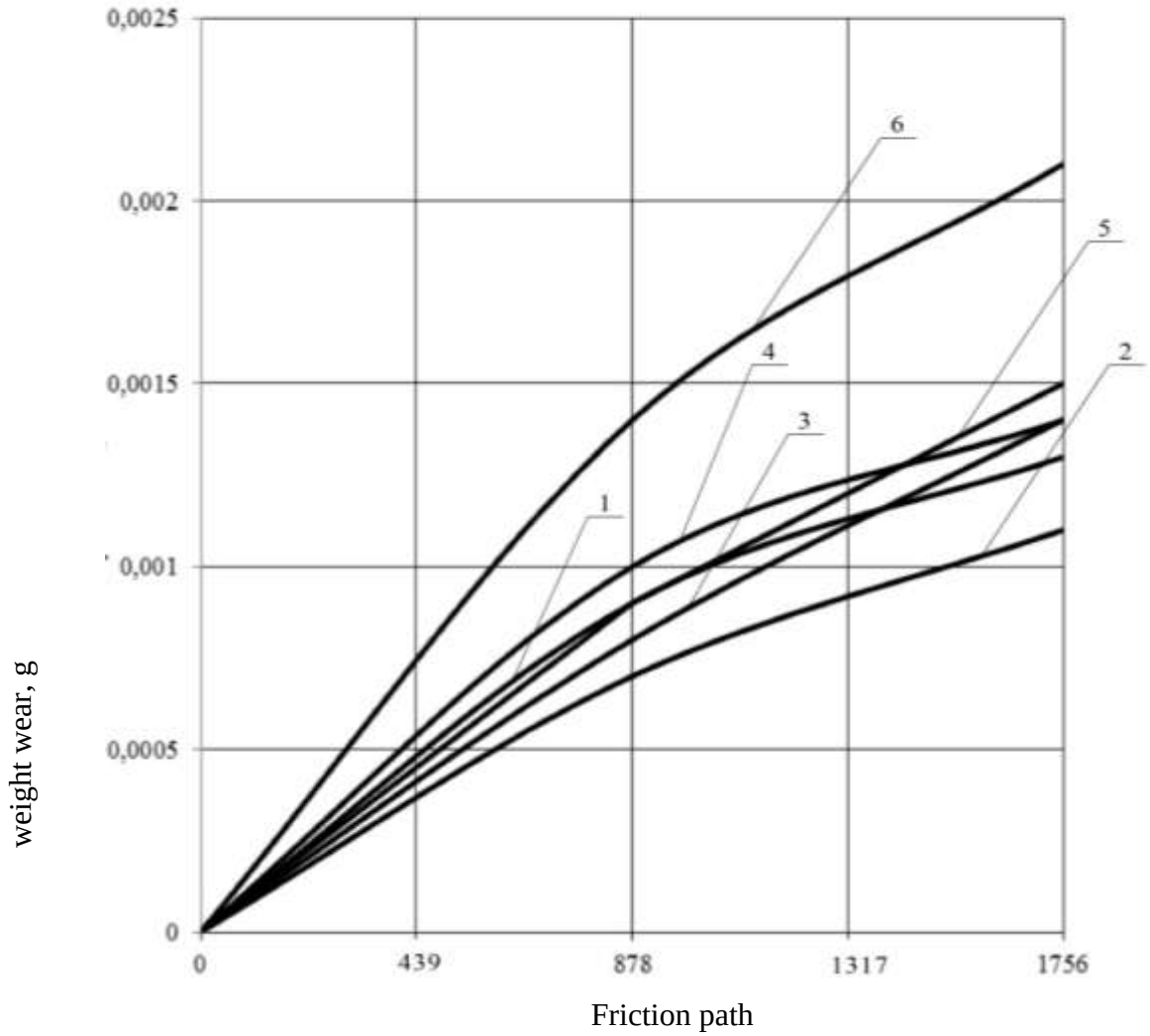


Figure 4.12 - Wear kinetics of high-carbon coatings deposited at speeds:
1) Vsurface=11m/h; 2) Vsurface=14m/h; 3) Vsurface=17m/h;
4) Vsurface=20m/h; 5) Vsurface=23m/h; 6) Vsurface=26m/h.

he wear resistance of the surface I is quantitatively determined as the reciprocal of the weight wear (g^{-1}):

$$I=\Delta m-1,$$

where Δm – weight wear, g;

I – sample wear in g-1.

The obtained calculations are shown in diagram 4.11.

4.8 Statistical dependence "wear resistance - hardness" of the coating based on a sample of experimental data

The dependence of mass wear of coated samples on hardness after changing the deposition rate is investigated. Hardness is determined on a

Rockwell instrument, wear – under conditions of sliding of the sample on the surface of a counterbody made of steel 45, hardened and tempered. The amount of wear is determined by the loss of mass of the sample by weighing on an analytical balance. The values of hardness and wear (HRC, Δm) are determined as the arithmetic mean of three measurements. The obtained values are summarized in the table (Table 4.11).

Table 4.11 – Initial data

Sample No.	Deposit rate, m/h	Hardness, HRC (X)	Wear Δm, g	Wear resistance, g^{-1} (Y)
1	2	3	4	5
1	11	54	0,0007	1428,57
2	11	50	0,0017	588,24
3	11	52	0,0015	666,66
4	14	53	0,0012	833,33
5	14	52	0,0010	1000
6	14	51	0,0011	909,09
7	17	46	0,0015	666,66
1	2	3	4	5
8	17	48	0,0017	588,23
9	17	50	0,0010	1000
10	20	47	0,0018	555,5
11	20	44	0,0015	666,66
12	20	41	0,0009	1111,11
13	23	45	0,0012	833,33
14	23	44	0,0016	625
15	23	43	0,0017	588,23
16	26	40	0,0022	476,2
17	26	38	0,0024	416,66
18	26	42	0,0017	588,23

The tasks of regression analysis are:

1. Obtain the regression equation I = I(HRC) for each sample and for the entire sample (n = 18).

2. Check the presence of the correlation relationship I = I(HRC) in each case.

3. Analyze the results obtained.

4.8.1 Calculation of sample characteristics

1. Arithmetic mean measurement X

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i \; ; \; \bar{Y} = \frac{1}{n}\sum_{i=1}^{n} Y_i \tag{4.2}$$

2. Empirical variance S^2 (standard deviation)

$$S_X^2 = \frac{1}{n-1}\left[\sum X_i^2 - \frac{(\sum X_i)^2}{n}\right]$$
$$S_X^2 = \frac{1}{n-1}\left[\sum Y_i^2 - \frac{(\sum Y_i)^2}{n}\right] \tag{4.3}$$

3. Empirical standard deviation

$$S = \sqrt{S^2}$$

4. Empirical covariance

$$S_{XY} = \frac{1}{n-1}\sum_{i=1}^{n}(X_i - \bar{X})\cdot(Y_i - \bar{Y}) \tag{4.4}$$

5. Correlation coefficient

$$r_{XY} = \frac{S_{XY}}{\sqrt{S_X^2 \cdot S_Y^2}} \tag{4.5}$$

6. Linear regression coefficients

$$b = \frac{S_{XY}}{S_X^2} \; ; \; a = \bar{Y} - b\bar{X} \tag{4.6}$$

7. When working with small samples (n <30), it is necessary to determine the presence of a correlation between the measured values Xi, Yi from the ratio

$$r_{XY} \geq r_{XY\,\min} \tag{4.7}$$

The regression equation has the form

$$Y = a + bX$$

Table 4.12 – Calculation table for building a statistical model

I	X_i	Y_i	$(X_i)^2$	$(Y_i)^2$	$X_i Y_i$
1	54	1428,57	2916	2040812,245	77142,78
2	50	588,24	2500	346026,2976	29412
3	52	666,66	2704	444435,5556	34666,32
4	53	833,33	2809	694438,8889	44166,49
5	52	1000	2704	1000000	52000
6	51	909,09	2601	826444,6281	46363,59
7	46	666,66	2116	444435,5556	30666,36
8	48	588,23	2304	346014,5329	28235,04
9	50	1000	2500	1000000	50000
10	47	555,5	2209	308580,25	26108,5
11	44	666,66	1936	444435,5556	29333,04
12	41	1111,11	1681	1234565,432	45555,51
13	45	833,33	2025	694438,8889	37499,85
14	44	625	1936	390625	27500
15	43	588,23	1849	346014,5329	25293,89
16	40	476,2	1600	226766,44	19048
17	38	416,66	1444	173605,5556	15833,08
18	42	588,23	1764	346014,5329	24705,66
Сума	840	13541,7	39598	11 307 653,89	643530,1

The calculation is carried out according to formulas 4.2 – 4.7.

Table 4.13 – Calculation result

Sample	Regression equation	Correlation coefficient
Sample 1 (3 points)	Y=-10029,8+210,082X	0,905
Sample 2 (3 points)	Y=2883,9-37,88X	-0,454
Sample 3 (3 points)	Y=-3249,08+83,335X	0,763
Sample 4 (3 points)	Y=4852,232-92,602X	-0,945
Sample 5 (3 points)	Y=-4710,01+122,55X	0,927
Sample 6 (3 points)	Y=-1221,9+42,89X	0,985
All samples (18 points)	Y=-601,883+29,019X	0,547

For the entire sample, the regression equation has the form

$$Y=-601,883+29,019X. \quad (4.8)$$

Correlation coefficient

$$r_{xy}= 0,547$$

Let's check the presence of a correlation for the data sample for all samples. Let's determine the acceptable value of the correlation coefficient.

For n=18 and α=0.90, we have the Student's coefficient t=1.717

$$r_{XY\,\min} = \sqrt{\frac{t_{\alpha,n}}{t_{\alpha,n}+n-2}},$$

$$r_{xymin}= \sqrt{\frac{1,717}{1,717+18-2}} = 0,3113.$$

Condition (4.7) is fulfilled.

4.8.1 Construction of wear resistance series of deposited samples

We will rank the samples by wear resistance using experimental data (table 4.11). In order to analyze the proposed dependencies, we calculate wear resistance using the regression equation (4.8). The results are entered in table 4.14.

Table 4.14 – Experimental I_E and calculated I_c wear resistance series

Depositing speed, m/h	I_E	I_C
11	1428,57	965,143
11	588,24	849,067
11	666,66	907,105
14	833,33	936,124
14	1000	907,105
14	909,09	878,086
17	666,66	732,991
17	588,23	791,029
17	1000	849,067
20	555,5	762,01
20	666,66	674,953
20	1111,11	587,896
23	833,33	703,972
23	625	674,953
23	588,23	645,934
26	476,2	558,877
26	416,66	500,839
26	588,23	616,915

Based on the results obtained, appropriate graphs or diagrams are constructed to simplify the analysis of the influence of various factors on the wear resistance of samples, determined experimentally (by I_E) and calculated (by I_C).

Based on the results obtained, optimal coating application modes are determined.

5 EXPERIMENTAL STUDIES OF PROCESSES FORMATION OF METAL CARBIDE MATERIALS USING EXOTHERMIC MIXTURES

5.1 Justification of the main research methods

The theoretical analysis of the synthesis of metal carbide materials in exothermic mixtures revealed the influence of the parameters of the initial system and thermodynamic conditions on the probability of phase formation and their quantitative ratio. Quantitative determination of the degree of this influence and development of methods for forming coatings required a series of experiments:

- study of the regularities of the synthesis processes of composite materials in exothermic powder and powder-fibrous mixtures based on refractory metals and carbon;
- development of the composition of mixtures for obtaining materials with a heterogeneous structure containing hard and soft structural components;
- study of the nature and degree of influence of the characteristics of the initial ingredients, the composition of the mixture, its density, the temperature of external heating and the kinetics of the process on the structure of materials and their antifriction properties;
- development of the technology for the synthesis of metal carbide materials.

To obtain the necessary information about the properties of the initial mixture and its components and the obtained experimental samples, it is necessary to use the following analysis methods;

- determination of the exothermic effect when the processes of synthesis of metal carbide materials in the studied powder mixtures are carried out by the method of their thermal analysis using electric heating;
- metallographic and microdurometric methods for determining the structure of the formed coatings and the properties of individual components;
- X-ray phase method for determining the composition of materials;
- testing of samples for wear resistance.

The factors whose influence on the synthesis processes was studied were selected on the basis of theoretical analysis.

Dispersion of powders.

The processes occurring in the mixture are heterogeneous, their first stage occurs on the surface of powder and fibrous components and the mechanism and kinetics of such a reaction depend on the total surface area and its properties.

Surface energy of powders. This characteristic, in turn, depends on many factors: dispersion, surface tension, surface defects, shape and morphology of powders. It can be significantly increased by pre-treatment - surface activation of powders. Therefore, it can be specified as a value such as an increase in the reactive activity of powders after a certain pre-treatment.

Density of the mixture.

This factor is noted as influential in numerous works, but it is emphasized that its nature is somewhat contradictory. For mixtures, there are certain optimal values, when going beyond their limits, the speed of processes decreases. This is sometimes explained by the existence of optimal conditions for the movement of gases that are released during combustion and provide high thermal conductivity in the system. The theoretical analysis showed that the density of the powder mixture under experimental conditions determines the amount of the gas phase and the carbon potential of the gas phase and the direction of the main processes of carburization of the metal components of the system.

External heating temperature.

According to preliminary calculations, it is 900 - 1100°C for chromium-based mixtures.

5.2 Methodology and results of determining the characteristics of powder components

Granulometric analysis of powders and identification of their structure and morphology were carried out by quantitative metallography methods.

Quantitative measurement of powders and analysis of their size distribution were performed according to standards, determination of the shape of powder particles. The specific surface was determined by methods of linear analysis and statistical processing of results from metallographic sections.

Table 5.1 – Characteristics of powder components

Characteristics of powders	Powders				
	Cr	W	Mo	V	C(soot)
Bulk density, g/cm^3	3,57	4,1	2,86	2.7	0,15
Granulometric composition: d_m, мкм:					
300 - 400	-	-	-	23	-
200 - 300	5	-	50	7	-
100 - 200	60	-	40	40	-
50 - 100	25	-	10	10	-
20 - 50	10	-	-	9	-
5 - 20	8	-	-	5	-
1 - 5	1,5	60	-	1	-
< 1,0	0,5	40	-	5	100
Powder purity, %	98,5	99,0	96,0	97,5	not recog-nized
Specific surface area, m^2/g	0,008	0,04	0,005	0,007	~ 10,0
Structure and morphology	Compact crystals	Crystals of geometrically regular shape	Crystals with unde-veloped porosity	Polycrystals with developed open porosity	Spherical particles

5.3 Methodology for thermal analysis of powder mixture samples

Thermal analysis of mixtures was carried out on the installation, the schematic diagram of which is shown in Fig. 5.1. Samples of mixtures were pressed into a steel shell and an alternating electric current of constant value was passed through it. The temperature was controlled by a platinum - platinum rhodium thermocouple, which was welded to the steel shell, and recorded on a thermogram. After heating to a temperature of about 1100°C and isothermal holding for 1…5 minutes, an exothermic peak appears on the thermogram – an increase in temperature by 80 – 400 °C, which indicates the passage of an exothermic reaction.

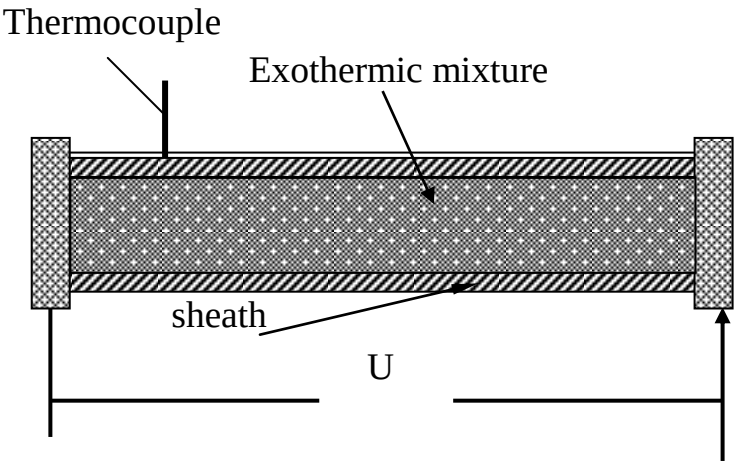


Figure 5.1 – Scheme of the installation for the synthesis of metal carbide material with electrical heating and thermal research

Studies were conducted on the mode and mechanism of reactions between carbide-forming materials Cr, V, W and carbon in various forms. Mixtures of Me and C (powder, fiber) were filled into steel shells under a certain pressure so that their density varied from 0.4 to 0.7. The ends of the shells were also pressed or left free for gases to escape.

5.4 Results of studies of the results of exothermic processes

The nature of the influence of the main factors on the structure of materials synthesized in exothermic mixtures.

The obtained structures were analyzed by metallographic and X-ray phase methods.

Experimental data show that 2 processes are possible in the system.

The first is the formation of chromium carbides, which is accompanied by an exothermic effect and the formation of a liquid phase, the composition of which is Cr and chromium carbide, which appears as a result of the melting of chromium particles, which gradually dissolve in it. Such a process takes place at temperatures of to 1100 °C. Calculations have shown its possibility at

temperatures above 1000 °C, but this discrepancy is a consequence of the difficulties associated with thermal calculations, which contain, as a component, heat losses. The iron shell is not carburized, but is covered with a molten carbide-containing phase over the entire inner surface with a thickness of up to 0.01 mm.

A competing process is carburization of the steel shell. It partially occurs in mixtures. In a mixture where the density is the highest (0.7), and heating occurs to a temperature of 1200 °C, the microstructure of the resulting alloy is a hypoeutectic white cast iron, the cementite in which is alloyed with chromium.

Chromium carbides were not detected in it. Carburization of iron in this mixture occurred before the formation of the Fe-C eutectic and eutectic melting.

These same mixtures were subjected to furnace heating. Samples with mixtures were placed in a chamber furnace at temperatures of 800, 900, 1000, 1100, 1200 °C, heated together with the furnace to a temperature of 900 – 1200 °C and kept isothermally for 1,5-2,0 hours. In all cases, when heated from T=800-900°C, oxidation of chromium powder occurred and the reduction processes did not have time to occur. When heated from 900°C to 1000 – 1100 °C, the chromium powder did not react, but was not oxidized either. When heated from 1000 to 1100 °C, the resulting structures were similar to those described above. When heated to 1200 °C, the formation of an iron-chromium matrix is observed, the amount of which depends on the porosity. At a porosity of 30-50%, the carbide framework is surrounded by a ferrochrome-type matrix with different Fe and Cr contents. A typical structure is shown in Fig. 2.5. Such experiments were carried out for 3 forms of carbon: graphite, activated carbon, VBM. Processing time – 0,5 h. The proportion of metal powder that did not react was determined and on this basis - the activity of the carbon source as a supplier of carbon monoxide CO. According to this feature, they were evaluated as follows: VBM-graphite-activated carbon.

Based on the experiments conducted, it was concluded that 2 reaction modes are possible - solid-phase and gas-phase, but the process mainly has a stage of carburization of metals through the gas phase with a transition to the eutectic plan. This is evidenced, first of all, by the nature of the dependence of the process speed on the factors that determine the volume and carburizing capacity of the gas phase - porosity, pressure, fractional composition, form of carbon. The amount of carbon in this case is the factor that affects the porosity. At a carbon potential of the gas phase close to equilibrium, active carburization of the iron substrate occurs with the formation of a cemented layer, and at high temperatures - ledeburite, and at T>1147 °C eutectic melting occurs. The solid-phase mode in the system is realized with direct contact of the component particles, but further, as evidenced by numerous works, carbon diffusion occurs along the surface of the metal particles, forming a surface carburized layer. Therefore, both reaction modes lead to the same results. The formation of the same ledeburite, on the contrary, is a competing process, because it is accompanied by heat absorption, lowers the temperature in the mixture and delays the exothermic reaction of carbide formation. Its result is the formation of

alloyed ledeburite. To verify this model, experimental work was carried out using layered Me-C compositions that were applied to the surface of a steel plate. Heating was carried out at t=1250 °C. The results obtained show that for the formation of a carbide-eutectic type structure, it is necessary to organize the interaction process so that at the first stage the exothermic reaction of carbide formation mainly takes place.

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