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Using Various Methods of Imaging and Visualization for Studying Heterogeneous Structures at Micro- and Nanoscales

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Abstract. This scientific work justifies imaging and visualization methods for analyzing heterogeneous PA-1 structures at micro- and nanoscales. It explores a key aspect of studying heterogeneous materials, namely the relationship between their microstructure and macroscopic behavior. Using Smart-EYE software, the microstructure and heterogeneous structure of PA-1 aluminum powders are justified through a range of factors. Among them, the extended functionality of the program allows for detailed analysis of particle sizes, shapes, and distribution, ensuring high accuracy and reliability of the analysis results. The capability for quick and efficient analysis of large volumes of data is also highlighted. Additionally, the software enables visualization of analysis results, simplifying their interpretation. Furthermore, the obtained results based on the histogram of particle size distribution, such as normal distribution, skewness, and modality, help avoid minor data defects and ensure proper interpretation.

Introduction

In the modern scientific world, understanding and controlling the structure of materials at micro- and nanoscales play a crucial role in addressing challenges related to the development of new technologies and materials [1]. Heterogeneous structures, characterized by diversity and complexity, require advanced visualization and analysis methods [2]. It should be noted that the use of various imaging and visualization methods for analyzing heterogeneous structures at micro- and nanoscales is an important yet undetermined issue [3]. Specifically, this applies to modern technologies such as scanning electron microscopy, atomic force microscopy, confocal microscopy, tomography, and other methods that allow obtaining detailed and objective images of material structures [4]. In particular:

1) Scanning Electron Microscopy (SEM): SEM enables high-resolution imaging of surfaces of heterogeneous structures using an electron beam. This method allows studying the microstructure of materials at very small scales and analyzing particle morphology and detecting major structural heterogeneities [5];

2) Atomic Force Microscopy (AFM): AFM uses a probe to scan the surface of a heterogeneous medium with atomic resolution. The use of this method enables high-resolution imaging of surfaces and measuring mechanical properties of heterogeneous structures and materials, such as stiffness and adhesion [6];

3) Confocal Microscopy: This imaging method is based on using a point light source and a special detector that focuses light only from a specific depth in the sample of the heterogeneous medium. Additionally, this method allows obtaining three-dimensional images of structures with high resolution [7];

4) Transmission Electron Microscopy (TEM): TEM allows detailed examination and description of material structures at nanoscales by passing electrons through thin slices of a sample of the heterogeneous medium. Additionally, transmission electron microscopy enables obtaining high-resolution images of heterogeneous structure and qualitatively analyzing micro- and nanoparticles [8];

5) Tomography: This method allows obtaining three-dimensional images of the internal structure of materials by comparing data obtained from different angles. To conduct quality tomographic research, it is necessary to use various computer algorithms to reconstruct the three-dimensional structure of the material and its medium with high accuracy [9].

It follows that the application of these visualization methods opens up broad opportunities for studying microscopic details and their structures, identifying heterogeneities, determining particle sizes and shapes, and analyzing other important parameters that affect the properties of both the heterogeneous medium and the finished materials as a whole [10]. Therefore, it is important to conduct a detailed review of current research in this area and assess their status to identify promising directions for further research aimed at expanding imaging and visualization methods for justifying heterogeneous structures. It is also important to note that such an approach is an indispensable tool for studying heterogeneous structures at micro- and nanoscales, the structural complexity of heterogeneous structures, heterogeneous media, and materials, as well as their impact on properties and behavior. It is also worth noting that heterogeneous structures are a key element in many important fields such as materials science, microelectronics, biology, medicine, and others. Understanding their nature and studying their properties pave the way for the development of new materials and technologies that meet the modern requirements of science and industry.

Main Part

In scientific research [15, 16] the main visualization methods for studying heterogeneous structures are presented. The work of these scientists has numerous advantages but also a range of imperfections, including limited resolution, inconsistency in method selection, data processing complexity, influence of external factors, etc. In the works [17] a technology for obtaining heterogeneous structures from a large amount of mathematical data is substantiated, and various imaging and visualization methods for studying heterogeneous environments are described in detail. It should be noted that such an approach may require complex data processing and analysis procedures, as well as high qualifications [18, 19]. Scientific groups [20] mainly describe imaging methods that are sensitive to external factors such as temperature, humidity, or loading, which can complicate obtaining stable and reliable research results. In the works [21, 22] primarily imaging methods with limited factors in studying heterogeneous structures are substantiated, primarily indicating the impossibility of applying increased resolution, which is extremely important for nano-scale research. Additionally, this can significantly complicate obtaining detailed information about structures [23, 24] that have a large number of nanoparticles or nanostructures. In the works [25] the technology of imaging methods is described, which often depends on specific properties of the investigated material, leading to inconsistency in the use of imaging and visualization methods and in comparing their results. Researchers [26, 27] largely overlook the qualitative indicators of the applied methods (including imaging and visualization methods) and their technologies for overcoming minor limitations in researching heterogeneous structures to obtain more accurate and reliable results in micro- and nano-scale structure studies. Scientific papers [28, 29] describe the theory of artifact formation or distortion, which can distort the image and complicate the correct interpretation of the obtained results. Scientific works [30, 31] specifically present the complexity of standardizing imaging and visualization methods, which complicates the comparison of results obtained by different researchers or in different laboratories. In articles [32, 33] a whole range of visualization and imaging methods are detailed, which are expensive and complex to use in studying heterogeneous structures and their environments, thereby restricting access to experimental data.

Analysis of literary sources shows that the use of various imaging and visualization methods for studying heterogeneous structures at micro- and nano-scales is an active and promising area of scientific research [34, 35, 36]. The variety of methods [37, 38, 39] such as scanning electron microscopy, transmission electron microscopy, tomography, atomic force microscopy, confocal microscopy, and others, allows obtaining detailed information about the structural features of materials at different levels of their organization. This, in turn, enables solving a wide range of tasks from fundamental research to the development of specific and new technologies and materials with high functional characteristics.

The main goal of this scientific research is to study imaging and visualization methods for analyzing heterogeneous PA-1 structures at micro- and nano-scales in order to obtain detailed information about their structure and properties, which is crucial for the further development of modern technologies. Additionally, to investigate the microstructure of PA-1 aluminum powders based on Smart-EYE software.

Materials. It should be noted that scanning electron microscopy (SEM) is a powerful tool primarily used for studying the surface of heterogeneous structures or materials at micro- and nanoscales. In scanning electron microscopy, the sample is illuminated with a small beam of electrons, and the image is formed using a detector that detects and records differences in radiation. One of the key advantages of the SEM method is the ability to obtain high-resolution images of the sample surface in a heterogeneous environment, allowing for detailed examination of its morphology, texture, and structure. When studying heterogeneous structures, the SEM method enables the detection of various defects, including cracks, pores, inclusions, and other anomalies. Moreover, the SEM method can be used to analyse the properties of the sample using additional modules such as energy-dispersive X-ray spectroscopy (EDS), which allows determining the chemical composition of elements on the sample surface.

Atomic force microscopy (AFM) is an innovative method mainly used for studying the surface of materials and their heterogeneous structures at any level, including micro- and nanoscales. With atomic force microscopy, the sample is scanned by a microscopic sharp lever, which in turn has a tiny tip consisting of a few atoms. The principle of this method involves the lever moving over the sample surface and its heterogeneous structure, measuring the interaction force between it and the sample. This force depends on the surface properties of the sample, such as hardness, texture, and chemical composition. Using the AFM method, high-resolution images can be obtained both on the sample surface and inside the internal structure of the heterogeneous environment, allowing even the smallest defects and anomalies to be detected. One of the main advantages of the AFM method is its ability to conduct research in non-destructive microscopy mode, meaning that the sample and its structure are not damaged during the experimental measurements. Additionally, the AFM method can be used to measure the mechanical and physical properties of materials (heterogeneous environments) such as hardness and elasticity.

Confocal microscopy is a visualization technique that allows obtaining high-resolution and high-contrast images at micro- and nanoscales. In confocal microscopy, the heterogeneous structure of the sample is mainly illuminated by a laser beam focused only on the area of interest. The microscope's ability to separate the specified pixels leads to the formation of a three-dimensional image. The main principle of confocal microscopy is that only light reflected from the focal point is transported to the detector, which in turn increases contrast and provides high-quality images. The main advantage of confocal microscopy is its ability to obtain three-dimensional images of heterogeneous sample structures with high resolution and penetration depth. It should be noted that this method also allows for the analysis of structures at different depths of the investigated materials using a series of optical converters.

Transmission electron microscopy (TEM) is a powerful tool used to study heterogeneous structures of materials at micro- and nanoscales. This type of TEM method allows obtaining high-resolution images of thin sections of samples. The main characteristic of the TEM method is that an electron beam passes through a thin area of the sample's heterogeneous structure, after which it is collected using an objective and other elements of the optical system. The image is formed on the detector and allows studying the sample structure at the atomic level with high resolution. The main advantage of the TEM method is its ability to obtain high-resolution and quality images of the internal heterogeneous structure of the sample, allowing for the investigation of atomic or molecular levels.

Tomography method in the study of heterogeneous structures at micro- and nanoscales uses principles of X-ray, electron, or atomic microscopy to obtain three-dimensional images of the object's internal structure. The tomography process involves rotating the object around its axis, resulting in a series of two-dimensional images (projections) focused at different angles. Mathematical algorithms are then applied to reconstruct the three-dimensional object based on these projections. It should be noted that the tomography procedure allows obtaining additional three-dimensional images of the object's internal structure, which in turn allows studying heterogeneity, microstructure of the heterogeneous environment, inclusions, and other properties at micro- and nanoscales.

Therefore, each of the methods discussed has its unique advantages and limitations when investigating heterogeneous structures at micro- and nanoscales. Specifically:

1) Scanning Electron Microscopy (SEM):

Advantages: SEM provides high-resolution images of the sample surface, allowing for the study of its morphology and texture. This method also enables the analysis of the chemical composition of the heterogeneous structure using EDS.

Limitations: SEM is not capable of investigating the internal structure of the sample.

2) Atomic Force Microscopy (AFM):

Advantages: AFM allows for obtaining high-resolution images of the surface of a heterogeneous structure and conducting experimental measurements of various mechanical and physical properties of materials.

Limitations: AFM also cannot experimentally investigate the internal structure of the sample.

3) Confocal Microscopy:

Advantages: Confocal microscopy allows for obtaining three-dimensional images with high resolution and penetration depth, making it useful for studying the internal structure of a heterogeneous sample.

Limitations: One of the limitations of confocal microscopy is its ability to investigate only thin layers of heterogeneous structures.

4) Transmission Electron Microscopy (TEM):

Advantages: TEM allows for obtaining detailed three-dimensional images of the internal structure of materials at the atomic level.

Limitations: Transmission electron microscopy requires thin samples, which can be challenging to experiment with.

5) Tomography Method:

Advantages: Tomography allows for obtaining high-quality three-dimensional images of the internal structure of an object, facilitating detailed investigation of heterogeneous structures at micro- and nanoscales.

Limitations: The tomography procedure requires specialized equipment and processing of experimental data.

Tests. In order to conduct research on visualization and imaging methods for heterogeneous structures, as well as to investigate the basic properties of heterogeneous media (materials) at the micro- and nanoscales at a qualitative level, we apply a sequence algorithm of experimental investigations. It is worth noting that the main essence of this algorithm of sequential experimental investigations when analyzing heterogeneous structures at micro- and nanoscales is as follows:

1) preliminary study of the sample structure (at the initial stage, it is necessary to analyze the main properties of the heterogeneous structure or sample);

2) optical microscopy (for conducting this study, it is necessary to use an optical microscope for a quick overview and assessment of the general morphology and structure of the environment);

3) scanning electron microscopy (at this stage, it is necessary to apply all possible imaging and visualization methods to obtain qualitative indicators of the heterogeneous structure, their surface, properties, analysis of morphology, texture, defects, as well as the basic relationships between these components;

4) application of atomic force microscopy (used to conduct comprehensive experimental studies to measure the mechanical and physical properties of the surface of the heterogeneous structure);

5) analysis of transmission electron microscopy (this type of analysis was applied for detailed examination of the internal structure of the sample, and based on transmission electron microscopy, the analysis of heterogeneous structures at the atomic level was carried out. That is, we conducted research on the chemical composition of the heterogeneous structure and properties based on atomic and molecular interactions. As a result, we were able to obtain a detailed analysis of the atomic structure, the arrangement of atoms and molecules in the heterogeneous environment (material), as well as the interaction between them;

6) confocal microscopy analysis (this analysis allowed us to focus on processing three-dimensional images of the heterogeneous structure of the sample with high resolution);

7) tomography method (used to obtain three-dimensional images of the internal structure of the heterogeneous sample);

8) analysis and interpretation of results (at the final stage, we analyzed the obtained experimental data and interpreted them in the context of our initial hypothesis of this research).

Thus, the process of forming structures at micro- and nanoscales requires the execution of the following mandatory stages, including:

1) compression (at this stage, heterogeneous structures of the medium (material) are directed towards the pressing pressure. After that, large pores are filled without significant deformation. As a result of this process, the packing density of heterogeneous materials significantly increases, but the strength of the material remains low);

2) initial deformation (with further increase in extrusion pressure, particles of the material begin to deform (break down). Initially, small indentations occurred on the surface of particles of heterogeneous structures, followed by chipping (a process of soft deformation);

3) continued deformation (under the action of compression force, particles penetrate into the pores elastic and plastic deformation. It should be noted that this process increases the degree of deformation of particles of heterogeneous structures and the number of contacts between them);

4) change in the granulometric composition of the structure (with increasing compaction and pressure, the number of fine particles formed during grinding increases. It should also be noted that there was observed a jump in high stresses at contact points, as a result of which more fine particles were destroyed, changing the granulometric composition of the material). Figure 1 shows the heterogeneous structure of aluminum powders PA-1.

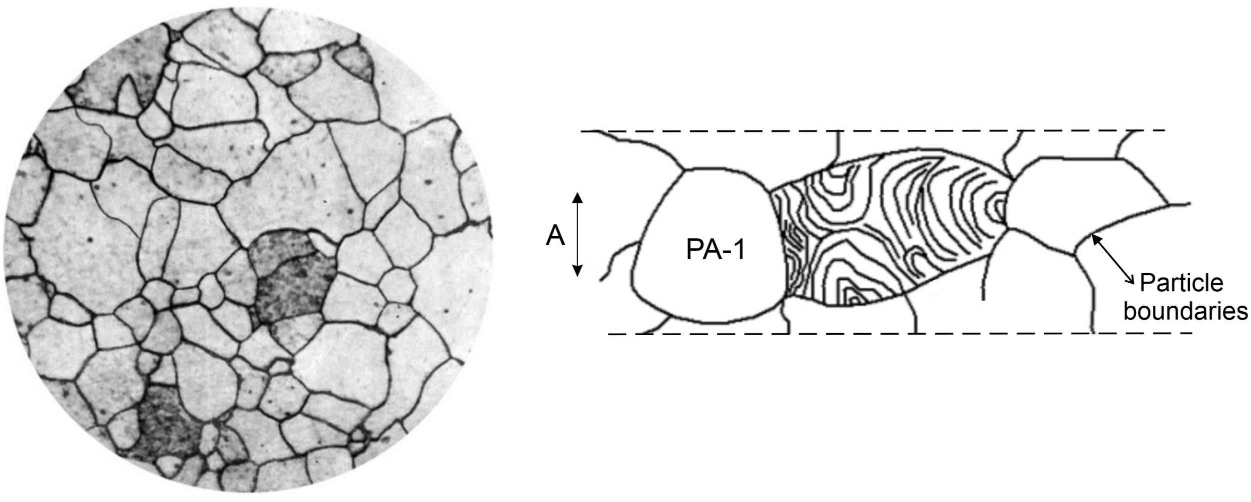

Fig. 1. General view of the heterogeneous structure of materials (in particular, aluminum powders PA-1).

Afterwards, the obtained images of the heterogeneous structures of aluminum powders PA-1 were processed and analyzed using specialized software Smart-EYE, which provided us with the ability to apply a wide range of tools to investigate the basic properties of the heterogeneous structures. An important aspect in this case was the analysis of particle sizes, assessment of their shapes, as well as analysis of the particle size distribution of aluminum powders PA-1. In the presented Fig. 2 illustrates the microstructure of the formed heterogeneous structure of PA-1 based on the Smart-EYE software.

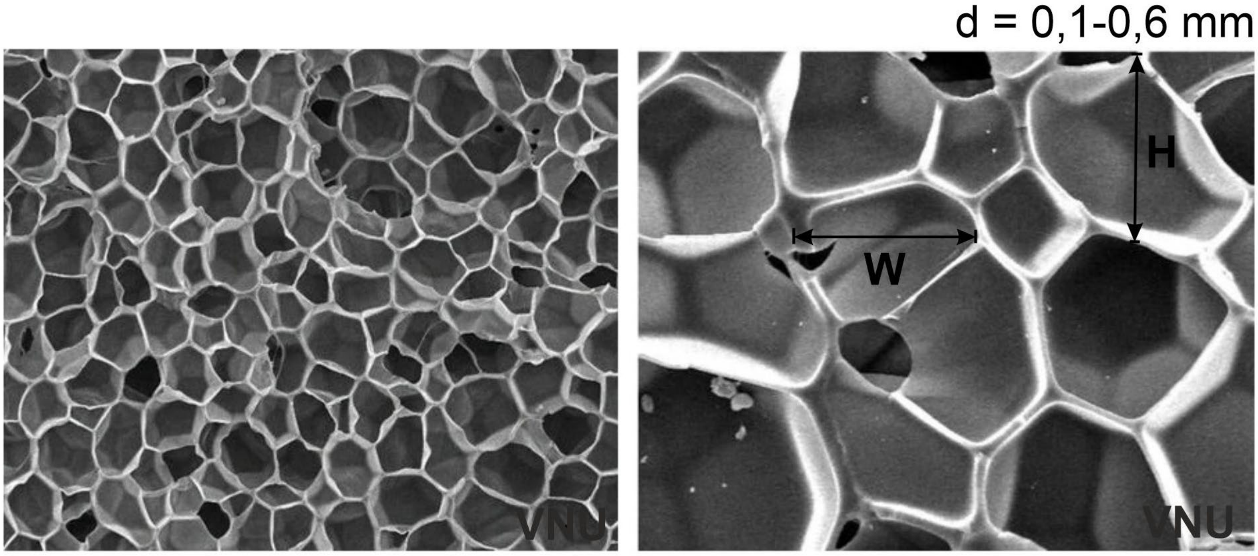

Fig. 2. Image of the microstructure of PA-1 based on the Smart-EYE software application.

The use of applied software allowed us to observe the following:

1) measurement of particle sizes: using the Smart-EYE program, we automated the measurement of each particle size within the heterogeneous structure on the image. The size (diameter) ranged from 0,01 to 0,1 mm. The area was 68 892 mcm², and the perimeter was calculated according to the formula: $P = 2\pi r = \pi d$ (where: r – radius, d – diameter).

2) evaluation of particle shape: based on the software, we also conducted several different methods of shape assessment, such as aspect ratio diameter, length/width ratio, length-to-width ratio, etc (data presented in Table 1).

3) analysis of size distribution: using the software, we processed the data based on micrographs of PA-1 aluminum powder materials to determine the size distribution of particles within the heterogeneous structure. The histogram of sizes is presented in Figure 3.

4) additional characteristics: the Smart-EYE software application provided us with the ability to determine textural features, identify different phases within the formed structure, and quantitatively assess agglomerations.

In the Table 1 presents the experimental results of the study of the most pronounced areas (regions) of heterogeneous structures based on micrographs of PA-1 aluminum powder materials.

Table 1. Experimental results of the investigation of areas (segments) of heterogeneous structures of PA-1 aluminum powder based on micrographs.

Particle	X	Y	Width	Height	d_{min}	Convexity	Perimeter	Area	Porosity, %
1	0.01	0.05	0.8	1.2	3.50	0.20	20.01	7.2	0.01
2	0.02	0.05	0.8	1.2	3.32	0.20	20.04	8.03	0.02
3	0.03	0.46	0.9	1.3	3.06	0.31	20.82	8.11	0.01
4	0.031	0.45	0.17	1.4	3.73	0.31	29.41	8.34	0.16
5	0.032	0.46	0.50	1.5	3.66	0.15	29.81	8.60	0.21
6	0.031	0.47	0.3	1.3	3.38	0.32	30.69	8.85	0.49
7	0.034	0.52	0.6	1.6	3.83	0.33	30.73	8.40	0.50
8	0.011	0.53	0.8	1.8	3.53	0.36	30.34	8.85	0.55
9	0.012	0.53	0.81	1.81	3.50	0.31	30.35	8.79	0.56
10	0.013	0.51	0.83	1.83	3.53	0.42	32.46	8.56	0.67
11	0.01	0.05	1.0	1.88	3.54	0.44	35.87	9.92	0.67
12	0.02	0.07	1.07	1.78	3.36	0.53	36.83	9.04	0.65
13	0.031	0.06	1.3	1.83	3.80	0.53	37.45	9.01	0.62
14	0.04	0.41	1.50	1.70	3.47	0.52	37.55	9.11	0.63
15	0.011	0.42	1.33	1.83	3.38	0.33	37.50	9.37	0.62
16	0.022	0.04	1.3	1.93	3.33	0.53	37.15	9.22	0.7
17	0.032	0.42	1.4	1.94	2.83	0.06	37.70	9.26	0.7
18	0.02	0.31	1.3	1.93	2.30	0.06	38.00	9.77	0.71
19	0.02	0.32	1.17	1.97	2.91	0.03	38.02	9.87	0.73
20	0.033	0.31	1.83	1.93	2.58	0.06	38.12	9.99	0.72
21	0.033	0.36	1.00	1.92	2.40	0.51	38.81	9.99	0.75
22	0.02	0.37	1.72	1.98	3.19	0.52	38.65	9.91	0.77
23	0.011	0.46	1.50	1.98	3.90	0.51	36.40	10.31	0.78
24	0.014	0.45	1.60	1.91	2.93	0.62	38.88	10.10	0.79
25	0.012	0.46	1.7	1.97	2.66	0.70	39.36	10.67	0.78
26	0.014	0.4	1.72	1.88	3.78	0.72	39.50	10.33	0.75
27	0.015	0.41	1.73	1.98	3.84	0.73	39.15	10.34	0.73
28	0.014	0.42	1.7	1.96	3.84	0.73	39.70	10.65	0.74
29	0.026	0.44	1.8	1.96	3.85	0.72	39.00	10.66	0.79
30	0.033	0.45	1.82	1.97	3.86	0.72	39.02	10.67	0.8
31	0.043	0,41	1,82	1,96	3.87	0.73	39.92	10.77	0.8
32	0.013	0.42	1.80	1.97	3.87	0.69	39.81	10.70	0.8
33	0.042	0.43	1.80	1.98	2.90	0,61	39,95	10,70	0,83
34	0.042	0.46	1.81	1.98	2.88	0.72	39.99	10.77	0.82
35	0.014	0.47	1.84	1.99	2.20	0.71	39.98	10.81	0.85
36	0.043	0.46	1.83	1.97	2.39	0.72	39.96	10.82	0.87
37	0.42	0.47	1.87	1.98	2.70	0.70	39.90	10.87	0,9
38	0.04	0.48	1.88	1.97	2.63	0.72	39.99	10.81	0.9
39	0.05	0.48	1.87	1.97	2.76	0.73	39.99	10.00	0.8
40	0.05	0.49	1.82	1.96	3.01	0.83	39.00	10.91	0.81
41	0.51	0.47	1.80	1.97	3.04	0.86	39.02	10.91	0.82
42	0.054	0.47	1.80	1.99	3.4	0.8	39.12	10.92	0.85
43	0.051	0.48	1.87	1.99	3.8	0.81	40.01	10.93	0.87
44	0.052	0.41	1.9	2.01	3.3	0.8	40.65	10.93	0.88
45	0.053	0.42	1.91	2.02	2.3	0.81	40.40	10.94	0.9
46	0.054	0.41	1.92	2.03	2.0	0.82	40.01	10.97	0.9
47	0.054	0.46	1.93	2.02	2.1	0.81	40.06	10.99	0.91
48	0.053	0.47	1.94	2.2	2.91	0.82	40.00	10.99	0.92
49	0.054	0.46	1.94	2.03	2.96	0.80	40.115	10.99	0.95
50	0.051	0.48	1.95	2.04	3.93	0.82	40.80	11.01	0.98

From the obtained results, it is evident that the macroscopic behavior of PA-1 heterogeneous structures deeply depends on their microstructure. This connection between macroscopic behavior and microstructure is crucial for understanding and predicting the physical-mechanical properties and parameters of heterogeneous materials. Research on microstructure and its influence on the properties of material heterogeneous structures in quantitative terms opens up possibilities for developing simple and effective quality control methods at a qualitative level. Specifically: 1) quantitative characteristics of microstructure (measurement of particle size, shape, and distribution or phases in the material, as well as evaluation of porosity and its distribution); 2) mechanical properties based on microstructure (establishment of relationships between geometric characteristics of microstructure and mechanical properties, such as strength, stiffness, deformation behavior, etc.); 3) prediction of product quality (development of models based on the relationships between microstructure and macroscopic properties to predict the quality of the manufactured product); 4) development of quality control methods (establishment of acceptability criteria and development of control methods based on microstructural parameters).

The construction of a histogram of particle sizes of PA-1 aluminum powders and their heterogeneous structure is presented in Fig. 3.

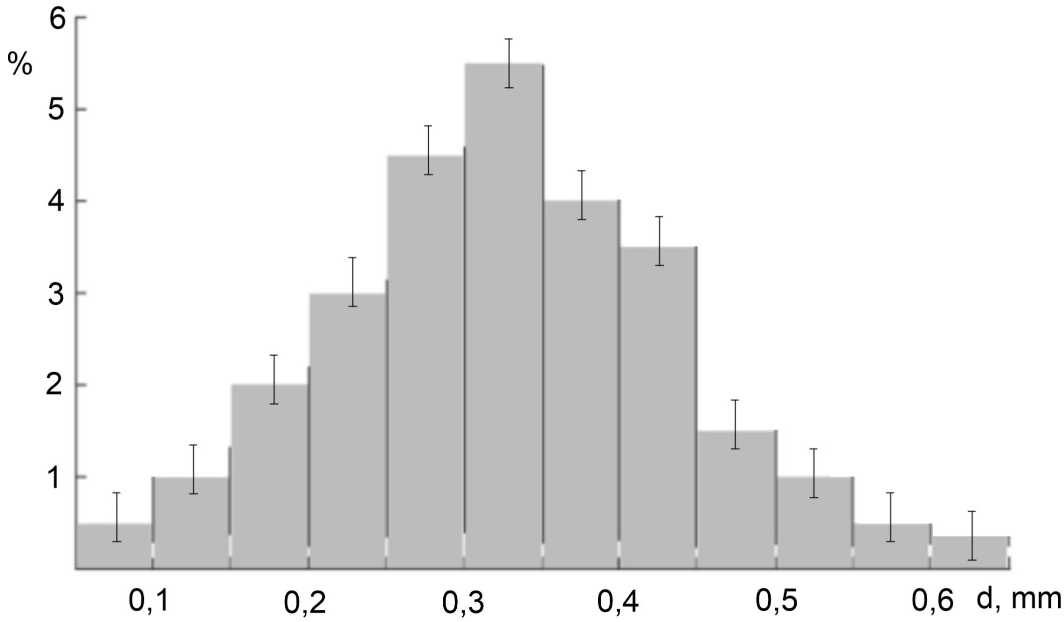

Fig. 3. Histogram of particle size distribution of the heterogeneous structure of aluminum powders PA-1.

It should be noted that on the abscissa axis of this distribution histogram (horizontal axis), we have presented the values of the sizes of aluminum particles PA-1, in millimeters (mm). On the ordinate axis of the distribution histogram (vertical axis), the fraction (percentage) of aluminum particles PA-1 that fall into each size interval is represented. The constructed histogram primarily indicates a qualitative distribution of the sizes of aluminum powder particles PA-1. This is because the following factors were taken into account when constructing this distribution histogram of the heterogeneous structure PA-1:

1) normal (Gaussian) distribution: when particles have an average size around which they cluster;

2) skewed right or left: if most particles of the heterogeneous structure are concentrated in one direction relative to the mean value, while the other end of the histogram is far from the center;

3) modal: when there are several distinct peaks (regions) indicating the presence of different subgroups of particles with different sizes.

Conclusion

This scientific study reflects a key aspect of studying heterogeneous materials: the relationship between their microstructure and macroscopic behavior. Based on the specialized software Smart-EYE, the microstructure of aluminum powder PA-1 and its heterogeneous structure were justified through a series of factors:

1) enhanced functionality of the program: Smart-EYE provides a wide range of tools for image analysis and processing, allowing for a detailed analysis of particle sizes, their shapes, and distribution.

2) accuracy and reliability of results: the use of specialized software ensures high accuracy and reliability of analysis results, as the program is developed to meet the specific needs of analyzing such structures.

3) efficiency: using Smart-EYE allows for the efficient analysis of large volumes of data and quickly obtaining results, which is important in measuring and analyzing material properties.

4) visualization of results: the software enables the visualization of analysis results, simplifying their interpretation and aiding in understanding the basic properties of heterogeneous structures.

The use of Smart-EYE is justified in terms of ensuring accuracy, efficiency, and the ability to conduct comprehensive analysis of heterogeneous structures of aluminum powder PA-1. Additionally, the specified factors obtained based on the constructed particle size distribution histogram, such as normal distribution, skewness, and modality, help avoid minor data defects and ensure their correct interpretation.

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