Analysis of metrological support of nano-measurements

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Abstract

The article analyzes the existing methods and means of measuring objects in the nanometer range and develops their classification based on the main principles of use. The main parameters on which each described method is based are considered and the conditions for their most effective application are determined. It is proved that the chemical and electrical sets of properties of the nanomaterial can change when the particle size decreases to the nanometer size, which requires the inclusion of additional chemical and electrical tests in existing methods. Based on the analysis, it was determined that the most functional and universal in solving a wide range of problems is the method of scanning probe microscopy. The classification of existing methods of scanning probe microscopy based on the nature of their applications is developed. The main information parameters on which each described method is based are considered, and the conditions of their most effective application are determined. To increase the accuracy of nanomeasurements, a methodology based on the principle of integration of information provided by different methods has been developed. The use of the differential-digital method is proposed, which includes the use of an additional information parameter in the mathematical model. An algorithm for including additional (a priory) information in the conditions for measuring the nanostructures has been developed, which leads the problem to the correct one according to the method of the control link, which characterizes the deviation of the parameters of measuring nanoobjects from their nominal values. It is proved that increasing the number of measurement methods used in the metrological analysis of nanoobjects will increase the reliability and accuracy of measurement results, and each method will provide additional information parameters to create a computerized method of calculating the control link. The main condition for correct comparison of the result is knowledge of the specific parameters on which each method is based.

Keywords

nanotechnology, nanomeasurement, metrology, methods and means of measurement, nanomaterials

1. Introduction

In recent years, the study of submicron, nano-, and cluster materials has developed rapidly in many fields of science and technology [1, 2, 3]. For several decades, almost all developed countries in various sectors of the economy have seen rapid progress in the field of nanotechnology.

Improving production efficiency and product quality, development of electronic equipment and biomedical devices, as well as the creation of new nanostructured materials with special properties are largely determined by the accuracy and precision of metrological support. It

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should be noted that research aimed at improving the instrumentation of nanotechnology has reached a new progressive level. Of course, large-scale development of the nanoindustry is impossible without appropriate metrological (MS) and software support.

Most of the theoretical and applied studies of new measurement methods with a minimum error have no practical application. The existing methods [4, 5] are unproductive, have low accuracy, noise immunity, reliability and cannot be used as part of flexible computerized measuring systems. These methods do not provide the required measurement accuracy for nanoobjects with complex, pronounced topography and do not meet modern requirements for the accuracy and speed of measurements.

In this regard, the problem of improving existing and developing new automated methods for measuring and evaluating the physical and mechanical properties of nanoobjects is relevant.

Therefore, the aim of the work is a thorough analysis of existing methods and measuring instruments in the nanometer range, their classification, and determination of the main characteristics. The analysis is carried out to create a new unified computer program for automatic correction of nano-measurements errors online.

2. Results

The essence of nano-measurements is to work at the molecular level, to study structures with fundamentally new properties. One of the main problems when working with nano-objects and nanostructures is associated with ultra-low signal levels. Another problem [6] is the wide range of behavior that objects and components can exhibit when measured, associated with the influence of destabilizing factors.

The essence of nano-measurements is to work at the molecular level, to study structures with fundamentally new properties. One of the main problems when working with nanoobjects and nanostructures is related to ultra-low signal levels. Another problem [7] is the wide range of behaviors that measuring objects and components can exhibit.

For example [8], measuring objects made of polymeric materials may have a resistance of more than one GM. However, being drawn into fibers with a diameter of less than 100 nm and doped with various nanoparticles, the polymer can be transformed from an excellent insulator into a high-conductivity wire. The result is an extremely wide range of test signals. High-sensitivity, high-resolution instruments are required to detect weak electrical signals at the bottom of the range.

Also, a prominent aspect is that DC measurements may require the characterization of some devices and structures by radio frequency signals. This requires a strict design of measuring instruments with reliable RF connections with low losses of the measuring head and a special electronic circuit for each signal path [9]. Otherwise, it will not be possible to achieve the resolution required for precision re-measurements. Therefore, measurement methods and tools should minimize noise and other sources of error that may interfere with the signal. No less important is the fact that metrological means of nano measurement should be easy to use and economical.

Nowadays, raster electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), scanning tunneling microscopy (STM), microscopy, and focusing on

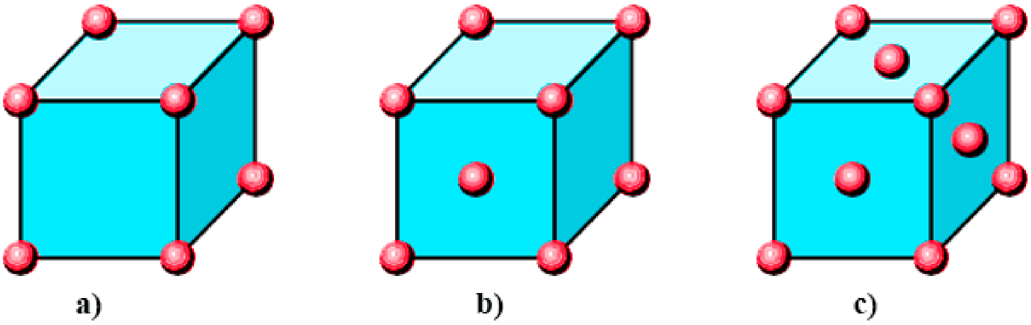


Figure 1: General crystalline atomic structures: (a) simple cubic structure, (b) volume-centered cubic structure, (c) bounded cubic structure

microscopy have become the most widespread. mass spectrometry, Auger spectroscopy, etc. From the point of view of research of the relief and physical properties of structures with high lateral resolution (less than 10 nm), one of the most perspectives is scanning probe microscopy (SPM).

The size and structure of the nanoobject have a great influence on the type of technique and the characteristics of the measurement methods used [10]. It is proved that optical microscopes are the most suitable for the study of macroscopic materials, for nanoscopic materials with a particle size less than 200 nanometers it is better to use STM, AFM, or combined methods of scanning probe microscopy.

SPM, STM, AFM methods are especially useful for the structures of crystalline nanoparticles. Figure 1 shows the atomic structures for some typical crystals of well-known shapes, such as a simple cube, a volume-centered cube, and a border-centered cube. Knowing the location of atoms in these structures helps to predict the properties of particles.

However [11, 12], on a nanoscopic scale, it is the particle size that radically changes the physics of its behavior and dictates the need to use other measurement methods.

Importantly, the chemical and electrical sets of properties of the nanomaterial can change as the particle size decreases to nanometer size. Therefore, in practice, additional chemical and electrical tests are required to determine the characteristics of most of these materials, which also affects the choice of the measurement method. Depending on the means of measurement, methods of analysis of nanomaterials can be divided into two main groups: discrete and ensemble methods of measurement.

Discrete methods of measuring nanoobjects typically use powerful microscopes, such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron microscopy (SEM), atomic force microscopy (AFM), and scanning probe microscopy (SPM). EM and SPM are fairly simple methods and reflect particle shapes, but are insufficient in terms of statistics [13, 14].

These methods have a resolution much higher than optical microscopy and can detect and measure discrete particles by scattering a high-energy electron beam (SEM and TEM), or by detecting through a probe attached to a slightly sprung console (AFM). In figure 2 shows a comparison of discrete methods of nano measurement concerning the indicators of the speed

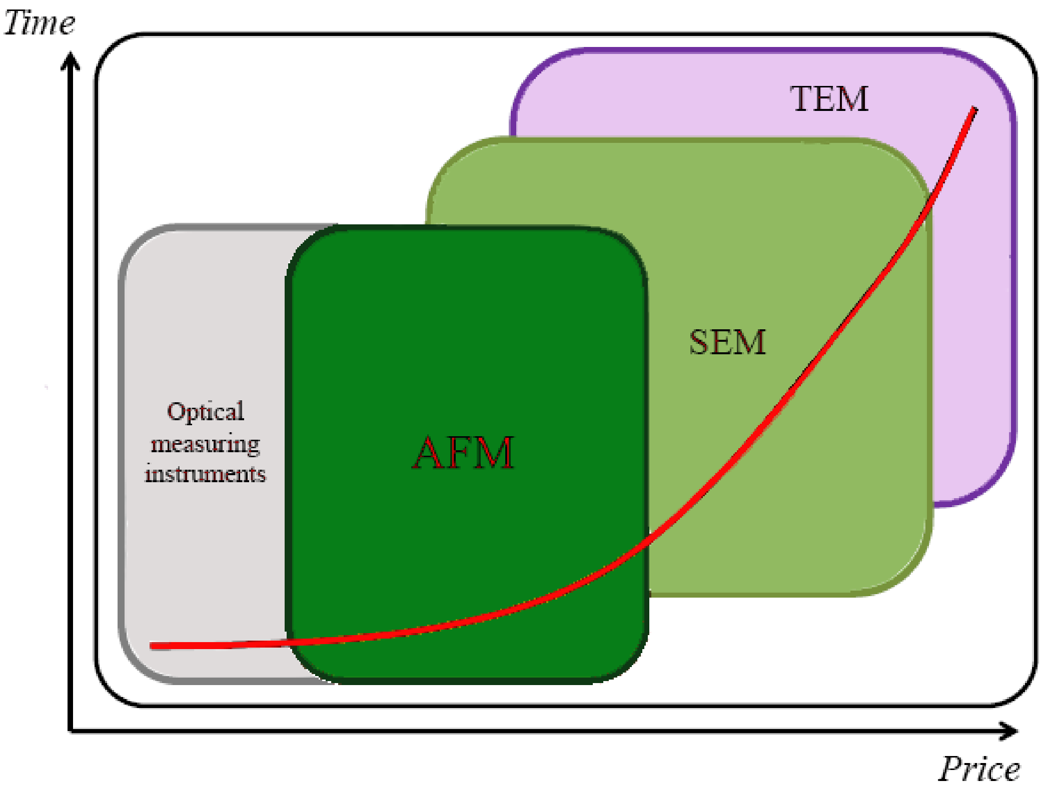


Figure 2: Comparison of discrete methods of nano measurements

of obtaining results and price.

Ensemble methods are usually indirect methods when information from many nanoparticles is obtained simultaneously. The methods of this group make it possible to establish the average size of nanoparticles as low-angle X-ray scattering (SAXS) and to detect photon interference.

One of the most frequently used and user-friendly methods for determining the size of nanoobjects in this group is the method of dynamic light scattering (DRS), which is based on the principles of coherence of light waves, and metrological characteristics are obtained by determining the phase difference of these waves after interaction with nanoparticles.

One method that potentially combines the advantages of a group of discrete methods and an ensemble approach is the nanoparticle tracking method, which involves determining the position of particles suspended in a liquid by detecting the light they scatter when irradiated by a laser source and viewing the suspension by using a camera with a charged connected device.

In figure 3 presents the ratio of the main methods of research of nanomaterials with different relief characteristics of nanoobjects.

Based on the analysis, it was determined that the most functional and universal in solving a wide range of problems is the method of scanning probe microscopy. SPM, in turn, covers

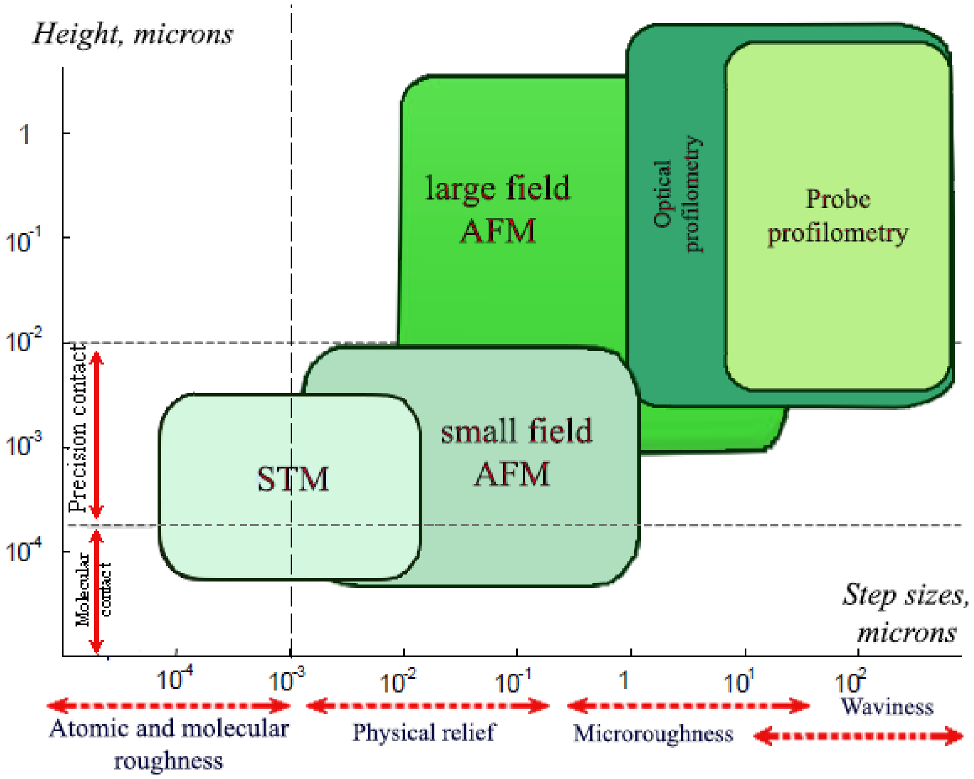


Figure 3: The ratio of the main methods of research of nanomaterials with different indicators of the relief of nanoobjects

several different experimental methods for studying the structure and properties of the surface, both at the micro-level and the level of individual molecules and atoms [4, 15].

The indisputable advantage of this method is the fact that with the help of SPM you can get information directly from a relatively large area of the surface, which allows you to use this method on-line. Therefore, it is not surprising that they are now widely used for the research, diagnosis, and modification of surfaces.

Common to all methods is the presence of a pointed probe as a tool for working with the surface of the samples. There are contact, semi-contact, and non-contact modes of operation, as well as various modes of operation, including tunnel mode, atomic force mode, spectroscopy mode, Kelvin probe method, electric power, magnetic force, near field, optical, confocal microscopy, etc.

With these methods, you can measure not only the topology of the structure but also many special properties, such as modulus of elasticity, distribution of various substances on the surface, the degree of surface roughness, static charge distribution, the orientation of magnetic domains, etc. [16].

Based on the nature of the applications of existing methods in SPM, they can be classified as

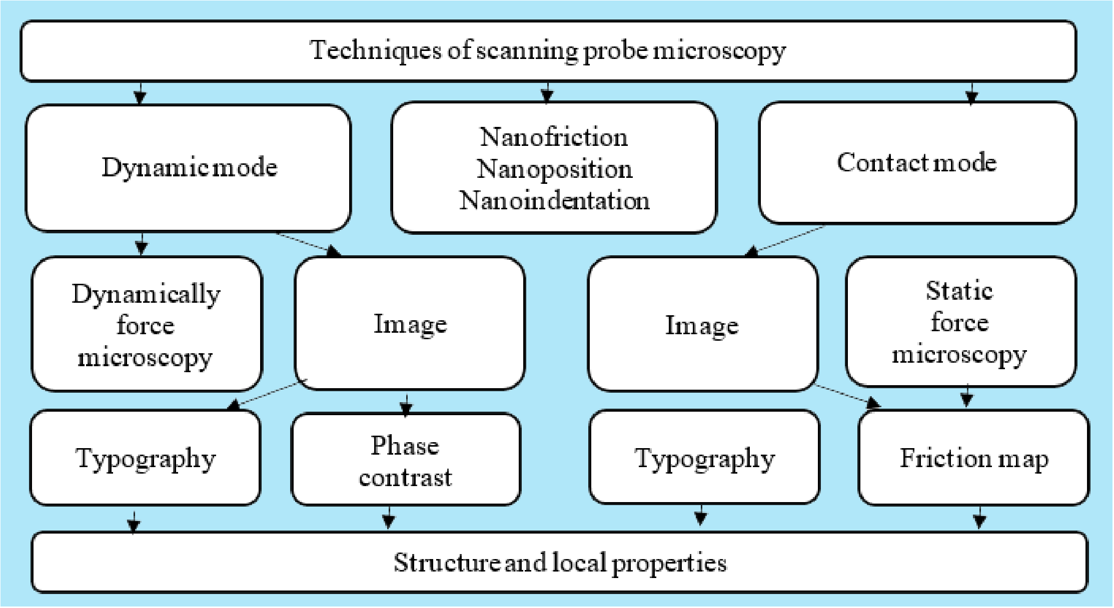


Figure 4: Methods of scanning probe microscopy at elastic and not the elastic effect of the probe

follows (figure 4).

Despite the variety of types and applications of modern scanning microscopes, their work is based on similar principles, and their designs differ little from each other.

Another common feature that unites not only sounding instruments, but also other measuring equipment presented in the article for measuring in the nanomaterial range is their increased sensitivity to external destabilizing factors, such as the composition and properties of the environment, lighting, potential difference, and magnetic field, temperature, etc. Because the properties of certain nanomaterials are different from conventional ones, the effect of destabilizing factors on the measurement results can be completely unpredictable.

Therefore, when choosing an existing or developing a new measurement method, the features of the kinetics and mechanism of the interaction of the measuring nanoobject with the environment should be thoroughly studied, the properties of nanostructures, process parameters, and the model of their mathematical description should be determined.

A necessary condition for the choice of measurement conditions, such assessments, and subsequent interpretation of the data is the reproducibility of the results and the invariance of the sample for a set of properties.

When changing the size can change not only the values of certain values but also the nature of their dependencies on properties, environments, and influences.

The main directions of creation and use of methods of measurement of micro- and nanosystems are defined:

- creation of model representations of connections of topological characteristics and reaction of the object to external influences

- establishing links between the properties of the element or system and the topology of the composition, structure, charges, and fields
- establishing links between technological factors, the kinetics of formation, and the properties of nanostructures
- creation of models that link the functional parameters of devices with the properties of structures that are determined during their manufacture

Therefore, to obtain more reliable results, the method of nano measurements should take into account the effects of external destabilizing factors on the nanoobject and ensure the possibility of correcting the deviations caused by them.

3. Discussion

Based on the analysis, a method for measuring nanoobjects in static and dynamic modes is proposed. This method will take into account the influence of destabilizing factors for nanostructures of different types. The method consists of the continuous determination of the coordinates of the points on the surface of the nanoobject, their processing, and presented in the form of a three-dimensional image. A cluster is introduced to determine the compensating link that corresponds to the tolerance field according to the spectrum of the color image.

The color value of the discrete points of the digital image forms the area, which is represented as isolines. Mathematical software includes geometric parameters of the reference nanostructure. As a result, a region of the color image is formed, which corresponds to the deviations from the shape and location of the surfaces and differs from each other in color (figure 5).

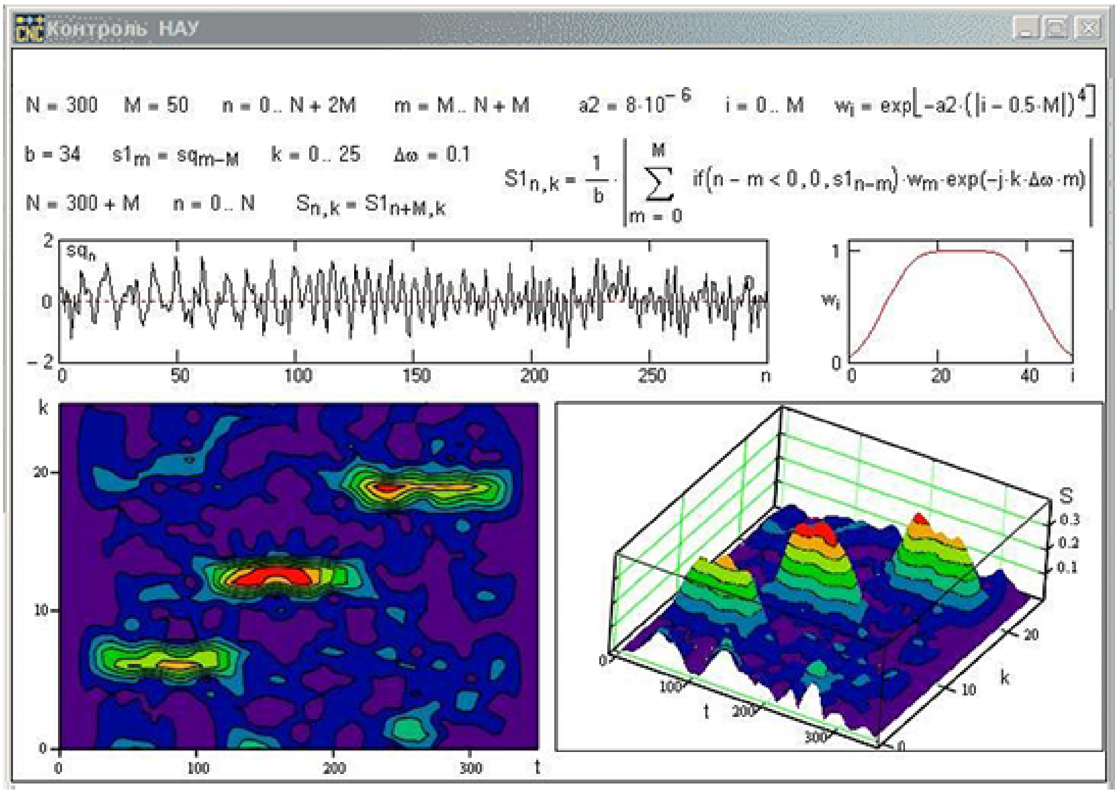
In addition to measuring flat nanostructures, a big task is to determine the topographic features of the surface and the angle of rotation of the plane of the nanoobject. This dependence is formed in the form of a three-dimensional array, in which one column occupies the angle of inclination of the plane, the second – the topographic features of the surface, the third – the coordinate. When scanning the surface of the part, a function based on a mathematical model of the process of traversing the measuring object is used.

The obtained values of the angle of inclination of the plane and topographic features, as well as the values of the coordinates, are compared with the tolerance field, which is presented in the color image (figure 6).

The differential-digital method makes it possible to study errors, determine the parameters of the nanoobject and obtain cross-sections using the developed methods of algorithmic error compensation, which provides zero offsets for the operating conditions of the measurement after the training procedure to obtain a statistical result. The development of the differential-digital method involves the use of an additional information parameter in the mathematical model in the form of a compensating link. The introduction of additional (a priori) information leads the problem to the correct according to the method of regularization for the functional

$$\Omega(x, \lambda) = |Ax - b|^2 + \lambda |x - x_0|,$$

where$\lambda \longrightarrow \infty$, x_0 – is the a priori solution vector that coincides with the regularization coefficient.



$N = 300$ $M = 50$ $n = 0..N + 2M$ $m = M..N + M$ $a2 = 8 \cdot 10^{-6}$ $i = 0..M$ $w_i = \exp\left[-a2 \cdot (|i - 0.5 \cdot M|)^4\right]$

$b = 34$ $s1_m = sq_{m-M}$ $k = 0..25$ $\Delta\omega = 0.1$

$$S1_{n,k} = \frac{1}{b} \cdot \left| \sum_{m=0}^{M} \mathrm{if}(n - m < 0, 0, s1_{n-m}) \cdot w_m \cdot \exp(-j \cdot k \cdot \Delta\omega \cdot m) \right|$$

$N = 300 + M$ $n = 0..N$ $S_{n,k} = S1_{n+M,k}$

Figure 5: The result of measuring the nanostructure using the differential-digital method

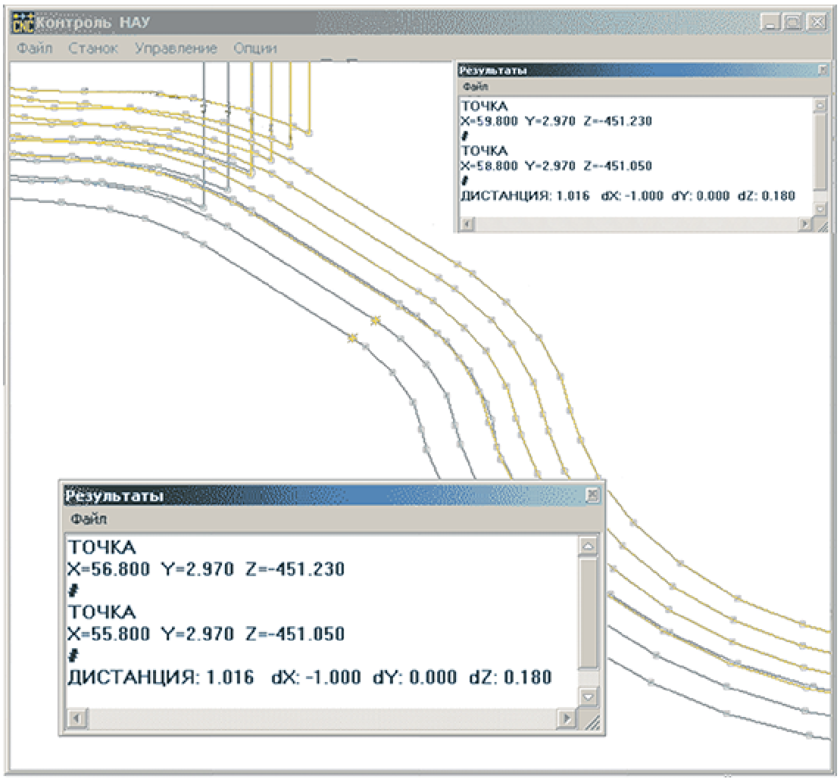
Using this approach to the problem of determining the geometric parameters of the aviation part of a complex spatial surface in small segments, using Delaunay triangulation, using the operation of minimizing the sum of the squares of the deviation, we obtain the following expression:

$$\Phi(\alpha) = \sum_{(i=1)}^{n} \beta_i^2 + reg(a, w),$$

where $\sum_{(i=1)}^{n} \beta_i^2$ – the sum of the squares of the deviation of n measured points, then from the constructed surface; $a = a_0, a_1, \ldots, a_i, \ldots, a_k$ – the required geometric parameters of the aviation part; $reg(a, w)$ - regulatory member, which includes information about the nominal value of the geometric parameter and the regularization coefficient w:

$$reg(a, w) = w \sum_{(i=1)}^{k} (a_i - a_{i_{yjv}})^2.$$

Thus, we obtain the problem of the regularizing link, which characterizes the deviation of the parameters of nanoobjects from their nominal values, we can obtain a clear point of minimum in the minimization function. The obtained results were stable and satisfied the value of the tolerance field of the measuring part.



```
ТОЧКА
X=59.800 Y=2.970 Z=-451.230
#
ТОЧКА
X=58.800 Y=2.970 Z=-451.050
#
ДИСТАНЦИЯ: 1.016  dX: -1.000  dY: 0.000  dZ: 0.180
```

```
ТОЧКА
X=56.800 Y=2.970 Z=-451.230
#
ТОЧКА
X=55.800 Y=2.970 Z=-451.050
#
ДИСТАНЦИЯ: 1.016  dX: -1.000  dY: 0.000  dZ: 0.180
```

Figure 6: Program window with the results of scanning the surface of the nanoobject

4. Conclusions

There are many advantages and disadvantages in choosing a specific measurement method to measure the topography of non-objects. There is no best general method. The use of integration of information provided by different methods is proposed, but the results of different methods may be contradictory in some cases, namely:

1. Each method is based on the need to study the different properties of nanoobjects
2. The steps of sample preparation can modify the results by further unintentional movements of the particles in the matrix, changing the average diameter detected by the SPM
3. Uncertainty that is not taken into account may also affect the final measurements
4. The use of different weighing in determining the average diameter of the size distribution. Comparing the results of different measurements of the same methodology will be less problematic in this sense because the existing error is constant

Comparing the results of different measurements of the same methodology will be less problematic in this sense because the existing error is constant. Therefore, to test the method, the error must be accurately measured, and the results must be consistent with other methods. Therefore, increasing the number of measurement methods used in the metrological analysis of nanoobjects will increase the reliability and accuracy of measurement results and each method will provide additional information. The main condition for correct comparison of the result is knowledge of the specific parameters on which each method is based. The analysis was conducted to obtain the most complete information for the development of a computer program based on a discrete-digital measurement method, which would take into account the measurement information obtained by different measurement methods, taking into account the impact of destabilizing factors.

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