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NANOMETROLOGY

The journal "Measurement Techniques" now contains a new section entitled "Nanometrology" devoted to the metrological problems that arise in the development of nanotechnologies. We open this section with a number of papers on the metrology of linear measurements, which are the basis of nanotechnologies.

METROLOGY AND STANDARDIZATION IN NANOTECHNOLOGIES AND THE NANOINDUSTRY

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The concept of ensuring unity of measurements in nanotechnologies is considered.
Key words*: nanotechnology, nanometrology, standardization, unity of measurements.*

The history of the development of science and technology is inseparably connected with the development of measurement systems, methods, and instruments. The transfer to nanotechnologies raises a number of new specific problems, due to the small dimensions of the components and structures with which it is necessary to deal in this area. Here, as nowhere else, the following thesis applies: "If you can't measure it accurately, you can't construct it." All countries venturing into nanotechnology recognize the need for an advanced development of metrology in this rapidly developing area of knowledge, since it is the level of accuracy and reliability of the measurements which will either stimulate the development of appropriate areas of the economy or will be a restraining factor. This is particularly underscored by the fact that, in nanotechnologies the instrument-analytical and technological components are operating at the limit of their possibilities, which increases the probability of error, all the more due to the human factor.

What is this metrology? On the one hand, it is the science of measurements, methods and instruments for achieving their universal unity and required accuracies. On the other hand, it is the institute for ensuring unity of measurements in the country, including the standardization of the units of physical quantities, their reproduction with the highest accuracy using State Standards and the transfer of the dimensions of the units of physical quantities in a hierarchical way downwards to all measuring instruments, which are allowed to be used in the country. The main problem of metrology is to ensure unity of measurements, i.e., the achievement of a state in which the results of measurements are expressed in legal units and the errors of the measurements are known with a specified probability.

The specific feature of nanotechnologies is their interdisciplinary nature, for which one and the same phenomenon, due to scale effects, can be used in different areas of the economic life of society such as information-telecommunications technology, medicine, pharmacology, the production of new materials and the study of materials, agriculture, the diagnosis of illnesses at the early stages, ecology, etc. This feature, and also the different terminology and different investigatory, technological and measurement approaches and methods, used in different branches of different scientific centers and laboratories, can lead to some misunderstanding, which makes it difficult to exchange technical information successfully.

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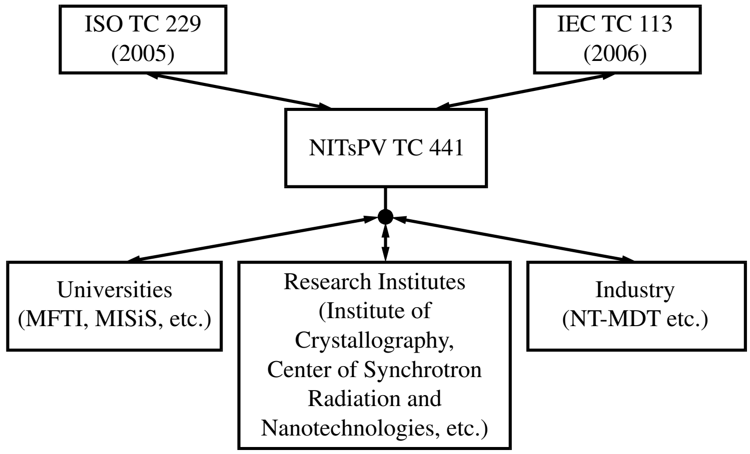

Fig. 1. Connection between the technical committee on standardization (TC 441) “Nanotechnologies and nanomaterials” with Russian and international organizations.

The interdisciplinary nature of nanotechnologies on an extensive scale initiated the setting up in 2005, within the framework of the International Organization on Standardization (ISO), of a technical committee (ISO/TC 229) on nanotechnologies. A year later, Technical Committee IEC/TC 113 was set up in the International Electrotechnical Commission called “Standardization in the area of nanotechnology for electrical and electronic components and systems.” The Russian side was represented on these committees by the national technical committee TC441 “Nanotechnologies and nanomaterials” (Fig. 1). It must be emphasized in particular that technical committees ISO/TC 229 and the IEC/TC 113 carry out their activities under conditions of equal partners, exchange information, hold joint meetings, consultations, forums, and form combined working groups on key problems of standardization.

The primary problems formulated by the countries participating in the ISO and the IEC, extremely interested in the development of this area, consist of standardization in nanotechnologies in the following areas: terms and definitions, metrology and testing and measurement methods, standard samples of composition and properties, modeling of processes, medicine and safety, and the effect on the environment. The solution of these primary problems provides a powerful impulse towards the development of nanotechnologies and their practical applications and introduction into different branches.

The area of activity of technical committees ISO/TC 229 and IEC/TC 113, as follows from their name, is standardization in nanotechnologies. The ISO/TC 229 understands nanotechnologies to means the following:

- the knowledge and control of processes, as a rule, on the nanometer scale (not excluding the scale of less than 100 nm) in one or more measurements, when the action of a dimensional effect (phenomenon) leads to the possibility of new applications;
- the use of the properties of objects and materials on the nanometer scale, which differ from the properties of free atoms or molecules, and also from the bulk properties of a material, consisting of these atoms or molecules, to produce more perfect materials, instruments and systems which realize these new properties.

One of the main problems is the standardization of the parameters and properties of materials, objects, components and structures of nanotechnology, which are to be measured. Because of the interbranch interdisciplinary nature of nanotechnologies, different terminologies and different research and measuring methods and procedures, this is not a simple problem that can be solved in sequence, having a unifying principle. Another problem is closely related to this, namely, the need to standardize terms and definitions in nanotechnology, directed towards solving the problems of commonality and mutual understanding between different groups of investigators not only inside one country but also within the framework of interdisciplinary exchange of information between countries. A consequence of this is the need for certified and standardized methods of making measurements, procedures for calibrating and checking measuring instruments, used in nanotechnology, and many other consequences, determined by the need to develop the infrastructure of the nanoindustry.

A particular aspect of standardization is the solution of problems of ensuring the health and safety of the operators of the technological processes, and the personnel who interact with the products of nanotechnology at all stages of their production, testing, investigation and use, and also the ecological safety of the environment.

What is meant by certification? This is nothing other than the confirmation that the parameters and properties of the objects, materials and structures, technological processes and also the instrumental and measuring systems correspond to the requirements of the technical rules, standards and other standard documents.

It logically follows that the "greatest statistical weight" must be given to metrology, since it is precisely this that is the quantitative basis of standardization and certification.

In Russia, similar problems are being solved within the framework of the Technical Committee on Standardization (TC 441) "Nanotechnologies and nanomaterials" of the Federal Agency on Technical Regulation and Metrology (Rostekhregulirovanie). The TC 441 secretariat manages the Research Center for Study of Surface and Vacuum Properties (NITsPV), the Kurchatov Institute, and the institutes of the Russian Academy of Sciences – Institute of Radio Engineering and Electronics, Institute of Crystallography, the Physico-Technological Institute, A. F. Ioffe Physico-Technical Institute, A. M. Prokhorov Institute of General Physics, the Photochemistry Center, Scientific-Technological Center of Unique Instrument Construction, Institute of Semiconductor Physics (Siberian Branch), Institute of Problems of the Technology of Microelectronics and Exceptionally Pure Materials, Technological Institute of Superhard and New Carbon Materials, State Research and Design Institute of the Rare Metal Industry (Giredmet), MT-MDT Company, Central Research Technological Institute (Tekhnomash), All-Russia Research Institute of the Metrological Service, All-Russia Research Institute of Optophysical Measurements, Moscow Institute of Steel and Alloys, Moscow Physicotechnical Institute, Elpa Company, Kontsern Nanoindustry Company, D. V. Efremov Research Institute of Electrophysical Apparatus, Research Institute of Technical Physics and Automation, All-Russia Research Institute of Aviation Materials, Central Research Institute of Construction Materials Prometei, Tsiklon-Test Scientific-Production Enterprise, S. A. Vekshinskii Research Institute of Vacuum Techniques, State University of Nature, Society and Man Dubna, Tambov State University, Institute of Nanotechnologies MFK, and Russian Research Institute Elektronstandart.

It is worth noting that the Research Center for the Study of Surface and Vacuum Properties (NITsPV) is also a member of the Semiconductor Equipment and Materials International (SEMI). The standards of the SEMI with regard to materials, equipment, the accompaniment of technological processes, etc., are very well known.

Within the framework of the International Organization for Euro-Asian Cooperation of State Metrological Institutions (COOMET), a project has been set up called the "Metrological Backup of Nanotechnologies," devoted to solving fundamental problems of metrology in nanotechnologies, in which Russia, Belarus, Ukraine, Slovakia, and Germany are participating. The coordinator of the project – the NITsPV – at the present time is developing the concept of metrological backup of nanotechnologies and technologies for transferring the dimensions of the units of physical quantities in the nanorange.

The Research Center for the Study of Surface and Vacuum Properties in the framework of TC 441 "Nanotechnologies and Nanomaterials" has developed four pilot State Standards in the area of metrology in nanotechnologies, which were introduced from February 1, 2008:

- State Standard GOST R 8.628-2007 "State system for ensuring unity of measurements. Single-crystal silicon relief measures in the nanometer range. Requirements on the geometrical shapes, linear dimensions and the choice of material for manufacture;"
- State Standard GOST R 8.629-2007 "State system for ensuring unity of measurements. Relief measures in the nanometer range with a trapezoidal profile of the elements. Checking procedure;"
- State Standard GOST R 8.630-2007 "State system for ensuring unity of measurements. Scanning probe atomic force measuring microscopes. Checking procedure;"
- State Standard GOST R 8.631-2007 "State system for ensuring unity of measurements. Scanning electron measuring microscopes. Checking procedure."

In December 2007 in Singapore, a conference was held between two profile technical committees on standardization in nanotechnologies – ISO/TC 229 and IEC/TC 113. At this conference, the leader of the ISO/TC 229, Doctor Peter Hato in a plenary paper informed the participants of four Russian standards in nanotechnology. The Russian side, represented in the ISO/TC 229 and the IEC/TC 113 by Technical Committee TC 441, was commissioned by the conference to initiate, within the framework of the ISO and IEC, the development of international standards, based on the Russian standards.

The Research Center for the Study of Surface and Vacuum Properties together with the Moscow Physico-Technical Institute developed two more State Standards, which are to be introduced from August 1, 2008:

- State Standard GOST R 8.635-2007 "State system for ensuring unity of measurements. Scanning probe atomic-force microscopes. Calibration procedure;"
- State Standard GOST R 8.636-2007 "State system for ensuring unity of measurements. Scanning electron microscopes. Calibration procedure."

A dictionary with the working name "Nanotechnologies in Terms and Definitions" was also produced with terminology harmonized with the generally accepted terminology. This was distributed to the leading organizations operating in this area. This dictionary will be published after discussions are completed and a positive conclusion is reached by technical committee TC 441.

The specific features of nanotechnology have led to the development of a new area in metrology, namely, nanometrology, with which all the theoretical and practical aspects of the metrological backup for unity of measurements in nanotechnology are connected. First, these are standards of physical quantities and standard equipment, and also standard samples of composition, structure and properties to ensure the transfer of the dimension of the units of physical quantities in the nanoband. Second, there are certified or standardized procedures for measuring the physicochemical parameters and properties of nanotechnology objects, and also procedures for calibrating (checking) the measuring instruments used in nanotechnology. Third, there is the metrological accompaniment of the technological processes themselves for the production of materials, structures, objects, and other products of nanotechnology. Here it is pertinent to emphasize that the insufficient level of metrology in Russian microelectronics, including the associated technological processes, has had a negative effect on the remaining factors, resulting in its present state. And, finally, there are the measures taken by the State Metrological Service (Rostekhregulirovanie), the departmental metrological services, and the metrological services of various organizations to ensure the unity of measurements, including State testing with the aim of confirming the type of newly produced or imported measuring instruments, inspection of the state and use of measuring instruments being used, ensuring the traceability of the transfer of the dimensions of the units of physical quantities in the nanorange by all measuring instruments used, the metrological expertise of the standards and other standard documents, the organization of the standard reference data service, participation in the work of international metrological organizations, etc.

From the definition of nanotechnology itself, which operates with objects of nanometer dimensions, it naturally follows that the primary problem is to measure geometrical parameters of the object which, in turn, gives rise to the need to ensure the unity of linear measurements in the nanometer band. But this is not the only role played by nanometrology. It is also present in implicit form in the overwhelming majority of methods and instruments for ensuring the unity of measurements of the physicochemical parameters and properties of objects of nanotechnology such as mechanical, optical, electrical, magnetic, acoustic, and other parameters. It is often necessary to carry out precision spatial positioning of the probe of the measuring instrument at the point where the required measurement information is to be collected. Here the range of linear scanning along each coordinate may extend from a few nanometers to hundreds or more micrometers, and the required accuracy in determining the coordinate may amount to tenths of a nanometer.

Why is so much attention devoted to the problem of creating a linear scale in the nanometer and adjacent ranges in nanometrology? Firstly, because the solution of the primary problem of metrology in nanotechnology is ensuring unity of measurements of the geometrical parameters of a nano object, it rests on the metrology of linear measurements. Secondly, as mentioned above, the measurements of mechanical, electrical, magnetic, optical, and many other parameters and properties of nano objects are connected with the need to position the probe of the measuring instrument at a specified point with the highest accuracy [1, 2].

To ensure the unity of measurements of the physicochemical parameters and properties of an object, it is necessary to tie in the corresponding measuring instruments to the standard which reproduces the unit of the given physical quantity (for example, the conductivity is tied to the standard of resistance), and in nanotechnology in the majority of cases it is also necessarily tied into the basic standard of the unit of length (Fig. 2) for accurate aiming. The uniqueness of the basic stan-

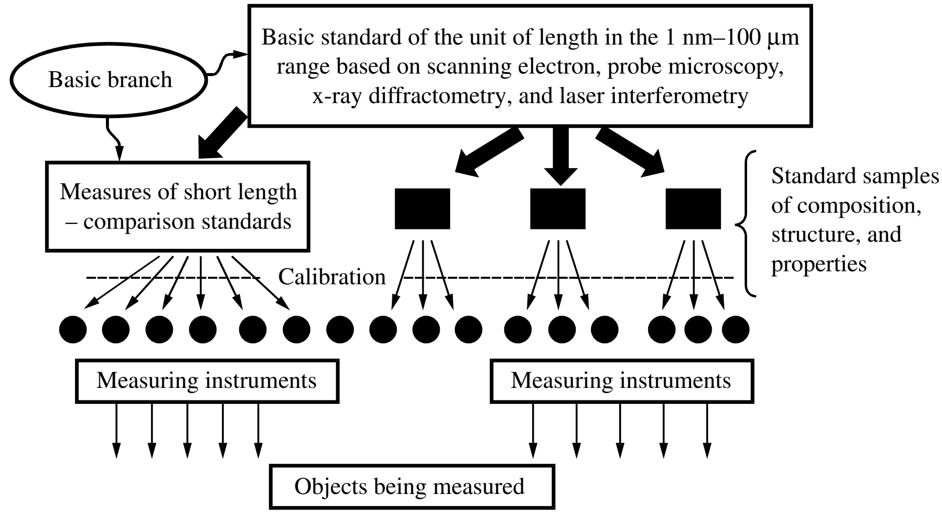

Fig. 2. A standard system and the metrological backup of the nano industry.

dard is not limited by this dualism. If we turn our attention to the parameters, it can be seen that the range of measurements of length from a few nanometers to hundreds or more micrometers covers more than five orders of magnitude of the measured quantity with a measurement accuracy of tenths to a few nanometers over the whole range.

The range of objects in nanotechnology and particularly in the nano industry is extremely wide, and it extends from ultradispersed media to nanostructured multilayer materials and crystals, including quantum dimensional structures with dimensions of localization: so-called quantum wells (superthin layers) are one-dimensional, quantum wires or filaments are two-dimensional, and quantum points are three-dimensional. The manifestational features of physical effects and the processes thereby occurring, including optical, luminescent, electrical, magnetic, mechanical, and many others, are determined by the characteristic dimension, and, in one and the same material, different effects, connected with the dimensions, may manifest themselves differently, for example, the particular features of the optical properties of a material in the ultradispersed form may manifest themselves for some dimensions of nanoparticles while thermophysical ones may manifest themselves for other dimensions.

The majority of methods of investigating and measuring the properties of nano objects – transmission and scanning electron microscopy, scanning probe microscopy, ion-field microscopy, photoemission and x-ray spectrometry and x-ray diffractometry, etc., widely used when measuring the properties of materials and products of the nano industry, require calibration of the measuring instruments using standard samples of composition, structure and properties with known dimensional (i.e., geometrical) characteristics.

For example, one of the well-known methods of determining the dimensions of ultradispersed particles involves investigating the light they scatter. The scattering depends on the relation between the size of the particles, the wavelength of the incident radiation, and the polarization. When determining the dimensions of particles, as a rule, laser radiation is employed, but to calibrate such measuring instruments it is necessary to have a set of ultradispersed particles with a discrete series of dimensions.

When reducing wideband semiconductor compounds of group A_2B_6 to the ultradispersed state, a "blue shift" of the luminescence band occurs, from which one can judge the sizes of the ultradispersed particles of the scintillator. But in each specific case of the semiconductor material used for the calibration, it is necessary to select standard samples of the same material with a whole series of dimensions.

When monitoring technological processes to produce multilayered thin-film structures, including multilayer heterostructures, it is necessary to employ x-ray diagnostic methods of monitoring the hidden layers and, correspondingly, to have multilayer standard samples of the composition and structure in order to calibrate the corresponding measuring instruments.

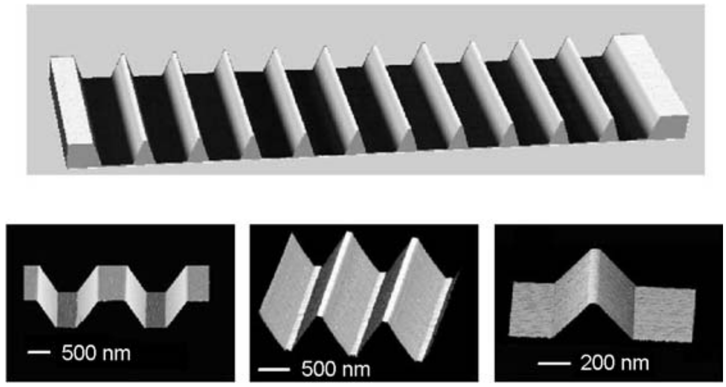

Fig. 3. Image of a standard of comparison in the atomic force microscope.

Fundamental investigations, connected with the direct measurements of the physicochemical parameters of the substances and materials of nanotechnology, and the components and instruments of nanotechniques, require a knowledge of the interaction between the measuring instrument and the object being measured. The problems of the metrology and standardization of such measurements, metrological backup, and the transfer of the dimensions of the units of a physical quantity in the nanometer band [3], characterizing specific features, acquire particular importance.

There are good reasons why the leading countries of the world, participating in the nanotechnology race, devoting considerable attention and contributing considerable means to the instrumental, analytical, and technological infrastructure of nanotechnology, and to the paramount problem of the advancing development of nanometrology, recognize the need to realize nanoscales in the nanometer and adjacent ranges. It is precisely to this paramount problem of nanometrology that many foreign conferences and numerous publications are devoted. Here we cannot ignore the considerable contribution of Russia to solving this fundamental measurement problem. The achievement of the limits in measurements of length in the nanometer band involves the use of high-resolution methods of scanning electron and scanning probe microscopy in conjunction with laser interferometry and x-ray diffractometry while preserving the absolute connection to the primary standard of the meter.

As a result of long investigations in Russia with universal priority, the problem of providing a basis for the metrological backup of measurements of length in the 1–1000 nm range has been conceptually solved. The following have been developed:

- methodology for ensuring the unity of measurements in the 1–1000 nm length range, based on the principles of probe microscopy and laser interferometry and x-ray diffractometry;
- standard measuring instruments, which enable the dimension of the unit of length to be reproduced and transferred in the 1–1000 nm range using real measures of length with an error of 0.5 nm;
- a new generation of measures of short lengths for calibrating measuring instruments in the 1–1000 nm range, including measures of surface nanorelief;
- methodology and algorithms for measuring the parameters of the profile of microstructure and nanostructure components and computer software packages for automating these measurements.

An important stage in the solution of problems of the metrological backup of linear measurements in the nanometer range was the development of *real carriers of dimensions* – measures with programmable surface nanorelief, which enable measuring instruments to be calibrated with the highest accuracy (Fig. 3).

It is precisely these *three-dimensional measures* of short length, or standards of comparison, – material carriers of dimensions, which enable complex calibration and monitoring of the fundamental parameters of scanning electron and scanning probe microscopes to be carried out. These microscopes are designed to transfer these complex instruments from a class of devices for *visualizing* an object into a class of *measuring instruments*, i.e., into a class of *instruments* for measuring the linear dimensions of objects being investigated, thereby ensuring that the measured quantities in the nanometer range are tied to the primary standard of the unit of length, namely, the meter.

The standards of comparison are certified using a standard three-dimensional interferrometric system of measuring nanodisplacements, presented above. The step of the measure and the dimensions of the upper and lower bases of the protuberances and grooves (the line width) are certified, as well as the height (depth) of the relief. Note that this certification of the line width is the first in the world to be achieved. In one and the same step of a structure, it is possible to manufacture standards of comparison with a line width in the 10–1500 nm range and a height of the relief of 100–1500 nm [4].

Measures enable one, using a single image of it in the scanning electron microscope (even using a single signal), which is very important for monitoring technological processes, to calibrate the microscope (to determine the magnification of the microscope, the linearity of its scale, and the diameter of the electron probe) [5].

Moreover, when it is necessary to confirm the correctness of measurements one can monitor the parameters of the scanning electron microscope directly while measuring the dimensions of the object being investigated, which is an additional guarantee of high measurement quality. A measure enables linear measurements to be easily automated and enables automated measuring systems to be designed based on scanning electron microscopes. A number of such systems already exist. In particular, at the Research Center for Study of Surface and Vacuum Properties an automated system has been constructed for linear measurements in the range of dimensions from 10 nm to 100 μm based on the JSM-6460LV scanning electron microscope.

In a similar way, using specified parameters of the measure, one can calibrate and monitor such characteristics of atomic force microscopes as the value of a scale division and the linearity of these scales with respect to three coordinates, the orthogonality of scanning systems, the radius of the tip of the probe (the cantilever), and adjust the parameters and output of the microscope under operating conditions [6].

Systems for calibrating and certifying atomic force microscopes are being successfully introduced into enterprises specializing in constructing equipment for nanotechnology. The development of nanotechnology imposes requirements on measuring systems, the measurement errors of which must be comparable with interatomic distances. All this requires a serious attitude to the problem of ensuring the unity of linear measurements in the nanometer range.

The scanning electron and scanning probe microscopes can only be regarded as measuring instruments when their parameters are certified, calibrated, and monitored in an appropriate way, the latter being carried out directly during the measurements. Three-dimensional measures or standards of comparison – material carriers of dimension – a unique bridge between the object being measured and the standard of the meter, are an ideal means of carrying out these operations.

One thing is unalterable: the culture of measurements requires that any scanning electron or scanning probe microscope, irrespective of where it is, whether in the science laboratory or the industrial laboratory, an educational institution or part of a technological process, must be completed by measures which ensure the calibration and monitoring of the parameters of this complex instrument. Only then can measurements carried out with it be regarded as reliable.

Moreover, the use of methods and instruments for calibrating and certifying scanning electron and scanning probe microscopes by the manufacturers of the corresponding instruments enables them to design new instruments with better characteristics, which, in turn, enables us to advance further along the road of nanotechnology development.

To provide the standard base of nanometrology, as indicated above, four pilot Russian standards have been developed and introduced, namely, GOST R 8.628-2007, GOST R 8.629-2007, GOST R 8.630-2007, and GOST R 8.631-2007. Two others – GOST R 8.635-2007 and GOST R 8.636-2007 – will be introduced from August 1, 2008. These standards are recommended by Russia as a basis for producing interstate standards (CIS). It is proposed to recommend them as a basis for developing standards within the framework of the technical committees ISO/TC 229 and IEC/TC 113.

To solve the scientific-technical problems of providing the unity of measurements in nanotechnology, it is necessary to take a number of scientific-procedural, technical and organizational measures. Firstly, this involves producing new structural systems for transferring the dimensions of the units of physical quantities from the primary standards to the working measuring instruments, eliminating the multistep nature of the transfer (see Fig. 2). The following measures also play a part in this:

- fundamental investigations of the *mechanisms* by which the probes of the measuring systems interact with object being measured;
- the development of new *measurement algorithms* and the corresponding software, taking into account the effect of the interaction of the working measuring instruments with the object being measured;

- the development of *new measures* – the material carriers of the dimensions, possessing properties similar to the properties of the secondary standard and the object being measured;
- the development and construction of *standard samples* of the composition, structure and relief of a surface, and *standardized procedures* for measurements in nanometers, which enable the transfer of the dimensions of the unit of a physical quantity to be tracked from the standard to the working measuring instruments in the nanometer range without any appreciable loss of accuracy for certifying, calibrating, and checking measuring instruments.

It is quite feasible to achieve this aim, since the foundation for solving this problem is based on the concept of a basic standard (see Fig. 2), in which the nanoscale is realized. This standard is the basis for transferring the units of physical quantities into the nanometer range. It does not require very much – just a harmonized system of standard samples of composition, structure, and properties, which serve the needs of nanotechnology.

All this provides the prerequisites and lays the foundations for the accelerated development of high technologies in Russia, and particularly the main one of these – nanotechnology.

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