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**NANOSTRUCTURES AND NANOCAPSULES**  
Methodical carts for independent work of students  
on discipline "Nanostructures and nanocapsules"  
for students of 163 "Biomedical Engineering"  
all forms of education



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## TOPIC 1

### The main methods of nanomaterials

#### 1.1 Terms

*1.1.1* Overview of the study of nanostructured materials The main element of the structure of consolidated material - grain or crystallites. In fact, this region of coherent scattering of X-rays. Therefore, by X-ray analysis (RSA) under conditions excluding the effect of instrumental factors

i mikrovkryvlen, get information about the size of grains in nanomaterials. This method is a single-phase material provides information on grain size up 2 - 3 nm.

Measuring the size of structural components made of nanomaterials and electronic-microscopic techniques using direct observation images and computer images from temnopolovyh'Books finish measurement results (correctly arrays, containing at least 1000 - 2000 crystallites).

When measuring the size of grains, and particle size, inclusions and pores taken to evaluate the following parameters: average diameter of about'Among the objects on their  $L_n$ , the average linear diameter  $L_l$ , the average diameter of the surface (or an equivalent circle diameter)  $L_s$  and the average diameter by about'emu (or equivalent sphere diameter)  $L_v$ :

$$\begin{aligned}L_n &= \sum_i (L_i n_i) / \sum_i n_i, \\L_l &= \sum_i (L_i^2 n_i) / \sum_i (L_i n_i), \\L_s &= \sum_i (L_i^3 n_i) / \sum_i (L_i^2 n_i), \\L_v &= \sum_i (L_i^4 n_i) / \sum_i (L_i^3 n_i),\end{aligned}\tag{1.1}$$

where n - number of grains or other objects of i-dimensional form.

Value (1.1) obtained for spherical elements.

Because of its methods of measuring electron microscopic analysis provides information about the average diameter of particles on their number ( $L_n$ ) or the average diameter of the surface ( $L_s$ ), and RSA provides information on the diameter of the sphere equivalent ( $L_v$ ). Among these parameters there are certain ratio and can see that  $L_v^3 = L_s^3 = L_l^3 = L_n$ .

To study the topography of the surface film breaks and using scanning electron microscope and an atomic force microscope.

Conductive nanostructures produced and examined using tunneling microscope that enables pull and move atoms from one place to another and use characteristics of the tunneling current, allowing identification of atoms of different elements.

Information about the size of grains (layers, inclusions, pores) and their distribution is very important, but it is often difficult to obtain objective. Therefore, it is desirable to use at least two independent methods. This increases the reliability of the information received.

### *1.1.2 The electronic microscope, variety and principles of*

Electronic image formed by electric and magnetic fields about the same as a light - optical lenses. Therefore, the electron microscope devices and focus the electron beam scattering called "electronic lens". There are many different types of electron microscopes, the most popular scanning electron microscope (SEM). In this micrograph of a thin beam of electrons (beam diameter of about 10 nm) runs (scans) the specimen on the horizontal line, point by point, and transmits the signal to the picture tube. The source of electrons is a metal (usually tungsten), which when heated as a result of thermionic emission, electrons are knocked out. With the passage of electrons through a sample of some of them scattered in collision with the nuclei of atoms of the sample, the other - after a collision with the electrons of the atoms of the sample, while others pass through it. Thus emitted secondary electrons and X-rays induced. All these processes are registered by special detectors in a modified form displayed on the screen, so get a larger picture of the object. The increase in this case refers to the ratio of the size of the screen

image to the size of the area that the electron beam passes the sample. In modern scanning electron microscope (SEM) increase could reach 10 million times, which corresponds to the resolution in units nm and allows to visualize individual atoms. To achieve said increase is possible because the wavelength of an electron by orders of magnitude smaller than photon, electron microscopy main drawback - the need to work at full vacuum

Neutron microscopes. Neutrons usually make up the atomic nuclei together with protons and with almost 2000 times more mass than the electron. According to the formula where Boyle-wavelength neutrons as many times smaller, that is picometre thousandths of a nanometer. Neutron Microscope has many advantages - particularly well reflect neutrons and hydrogen atoms easily penetrate thick layers of samples.

Scanning probe microscopy. How obstukuvannya probe surface underlying the so-called scanning probe microscopes that define small surface irregularities on it during superfine tip of the probe. Scanning probe microscopes provide atomic distinguished ability and work not only in a vacuum, but also gas and liquid environments. Today, they are a major analytical equipment nanotechnology.

Scanning tunneling microscope (STM) STM basis is very sharp needle, which slides over the surface under study, almost touching it (the gap between the needle and the surface is less than one nanometer) (Fig.1.1).

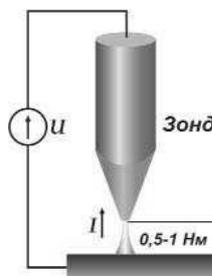


Figure 1.1 - Scheme atomic force microscope

However due to tunneling between the needle tip and the sample surface appears tunneling current. The strong dependence of the tunneling current distance (by changing the gap to one-tenth of a nanometer current changes 10 times) provides a highly sensitive microscope. Position the needle at such a small distance from the test surface is provided yezomanipulyatorom  $\phi$  n, who used to manage the measurement results of the tunneling current. Measure the height of control signals defining the study area and the movement of the needle along the surface of the sample surface profile is determined with an accuracy of individual atoms. Image

surface, constructed by measuring the tunneling current, provides information on the spatial distribution of the electron density of states near the surface.

However, the STM is a drawback - it can be used to study only materials that conduct electricity well. Therefore, to study using STM non-conductive substances, they should be covered with a thin metal film that is not always convenient.

Atomic force microscope free from this defect. His work scheme shown in Fig. 1.2, a schematic diagram and general form in Figure 1.3.



Figure 1.2 - Scheme of AFM

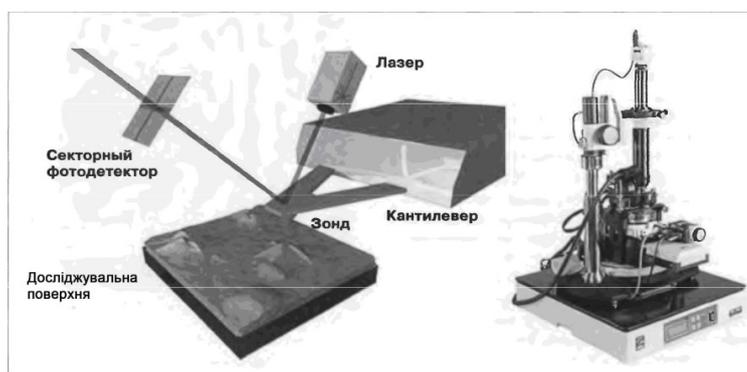


Figure 1.3 - Schematic diagram and a general view of an atomic force microscope

This microscope register changes attraction force of the needle to the surface. The needle is at the end of the cantilever that can flex under small interatomic forces that occur between the studied surface and the tip of the tip. The probe moves over the surface of the sample and touching it with some frequency, that in this way moves at a certain distance. Rejection of the cantilever detected by a laser beam reflected from its surface and gets feedback on the photodiode. By changing the current photodiode, whose value depends on the deflection of the cantilever, draw conclusions about the topography of the object. Atomic force microscopy has proved so effective that based on it created other specific techniques that allow you to get a picture not only topography but also many other parameters. In particular,

- magnetic force microscope (MFM) as a probe uses magnetized tip. His interaction with the surface of a sample allows you to record magnetic microfield and represent them as a map of the magnetization;
- electric force microscope (EFM) - it tip and the sample is considered as a capacitor and capacitance change is measured along the surface of the sample;
- thermal scanning microscope records the temperature distribution on the surface of the sample. His distinguished capacity is 50 nm;
- scanning frictional microscope "scratches" on the surface of the probe and thus creates a map of frictional forces;
- Magnetic microscope allows to obtain images of individual electron spins, track surface in response magnetic field is changing rapidly;
- Atomic force acoustic microscope is very accurate measure Young's modulus at each point in both soft and hard specimens.

One drawback is the inability AFM study the structure deep in the sample.

This problem can be solved by semblera, atomnozondovym device called a three-dimensional tomographic scanner that scans a small area, then removes a layer one atom thick and scans the area again, recording parameters of each new atomic layer. Modern tomography manage to remove the 20,000 atoms per second, that is 72 million atoms per hour.

Scanning near-field optical microscope. Special attention is given near-field optical microscope (SNOM). According to the principle of the tunneling microscope it resembles, just as the probe is applied very thin "transparent needle" with fiber and instead recorded tunneling current changes in the characteristics of the laser beam that slides in "transparent needle" (rys.1.4).

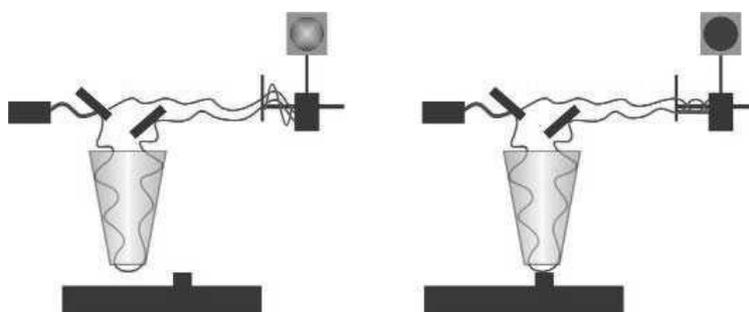


Figure 1.4 - Scheme of scanning optical microscope

Fiber-optic probe that tapers to a diameter smaller than the wavelength of light, rises close to the surface, which scan (a distance smaller than the wavelength), and he seemed to "feel" the surface.

"Feel" means that according to the laws of optics on border distribution of two media of different densities (glass / air) light beam is refracted and reflected from the end of the needle. In this light wave does not come from the waveguide over long distances, but only slightly "falls" from its tip. At the other end of the waveguide mounted receiver reflected on the free end of the world. The probe scans the sample needle like tunneling microscope, and if changing the distance between the investigated surface and the probe tip, the change and characteristics of the reflected light waves (amplitude and its phase). These changes are recorded receiver and used to construct an image of the surface relief. Distinguished ability, this method is 50 nm, which exceeds by orders given the conventional optical microscope. In addition,

### 1.1.3 The study of mechanical properties of nanomaterials

Nanoindentation method. To study the mechanical properties of various materials in the nanometer range is widely used method for determining the microhardness special substances - nanoindentation (from the English. "Indent" - form a recess) scheme is shown in Fig. 1.5.

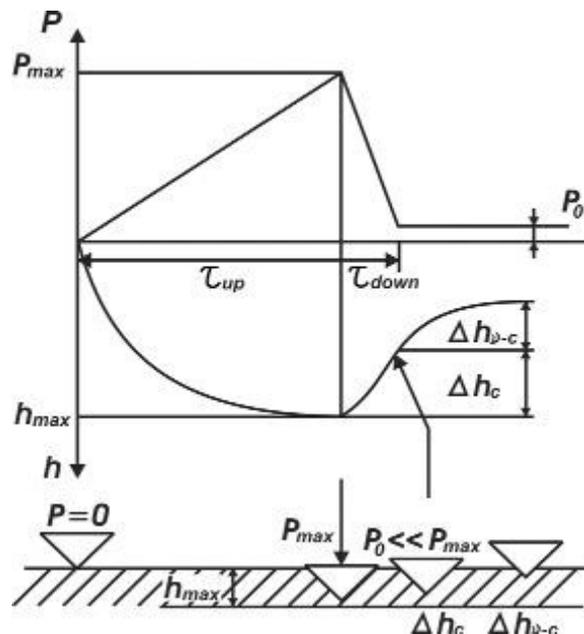


Figure 1.5 - The principle of operation nanoindentora

Nanoindentation is studied mechanical action on the surface and does not visualize its topography. The method is precision immersion probe in the sample surface to a depth of several nanometers and continuous registration of the applied force. Then this data is based diagram 'power load - diving depth ', which can identify dozens of parameters characterizing the material on the nanometer level. This method with minimum material allows comprehensive studies of its surface properties. Nanoindentation allows the investigation of dynamic processes in the nanoscale that is not available to other methods, including atomic power, electronic and optical microscopy.

In addition to direct scanning microscope supplied with them usually come even complex vibration, thermal and proofing installation. In addition to work at the atomic level, these minutes should be in a high vacuum and at very low temperatures.

This is directly reflected in their size and cost - microscope midrange take up much space and costs hundreds of thousands of dollars.

Scanning tunneling microscope "Umka" made concern "Nanoyndustryya", unlike foreign analogues, fits in a small carrying case, costs less than 9000 dollars and working indoors.

Created specifically for teaching nanotechnology This setup can be used for research and laboratory work to atomnomolekulyarnomu level in physics, chemistry, biology, medicine, genetics and other sciences. Complex "Umka" includes: tunneling microscope, vibration protection system, a set of test samples and instruments. The program is open source allows you to control experiments and record the results on the desktop. Its resolution of about 0.01 nm.

### **1.3 control questions**

1. Describe principles, which is based on the work of scanning electronic microscope. Their advantages and disadvantages compared with a light microscope.
2. Describe the purpose of reflected electron detector, ring detector and analyzer.
3. Describe by atomic-force microscopy (AFM) and examples of its use.
4. Describe tunneling microscopy method (STM) and examples of its use.
5. Describe a constant height mode and constant current.
6. What properties other than surface topography allows vizulizuvaty atomnosylova microscopy.
7. Describe application modes: contact, contactless and obstukuvannya the application of AFM.

8. Describe the essence of the method blyzkopolnoyi scanning optical microscopy BSOM. Compare its distinguished ability of conventional light microscopy.
9. Describe nanoindentation method.

## TOPIC 2

Study the possibility of using powder metallurgy methods for the production of nanomaterials

### 2.1 General Information

Powder metallurgy can be divided into two groups - methods of obtaining nanopowders compaction methods and products are. Several methods can be used to produce nanopowders and to form with them about intensive products.

#### 2.1.1 Methods for making powders

There are a number of general approaches, which are common to all methods of obtaining nanopowders, and that distinguish them from conventional methods for powders:

- high speed formation of centers of nucleation particles;
- low rate of growth of particles;
- received the largest amount of particles less than 100 nm;
- narrow range of particle size distribution;
- Stability reception given the size range of particles,
- reproducible chemical and phase composition of particles;
- increased demands on the control and management of process parameters to obtain powder.

A common feature of nanoparticle powders, received any-which method, is their tendency to about unity in aggregates and agglomerates. As a result, it is necessary to consider not only the size of individual nanoparticles, but also about their size'associations. There is no clear terminological distinction between aggregates and agglomerates can not hold, but considered, in units called'a stronger link between particles, and lower porosity. The next compaction to achieve the desired porosity of the material paclaged powders require higher temperature and pressure than neahrehatovanymy.

All groups of methods of obtaining nanopowders can be divided into two groups. The first group includes technologies, based on chemical processes, and the second - on physical processes. Accordingly closer look at the main methods of obtaining nanopowders.

Technology precipitation from solution. This is a group of technologies

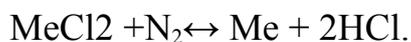
3 the most studied methods for producing nanopowders. A common feature of this group is carrying out chemical reactions in water solutions of salts. Are used several different methods.

Chemical deposition method is to prepare a solution of metal salts, adding substance - osadzhuvacha and deposition of metal powder in the form of metal oxide. The conditions governing precipitation by changing the pH, temperature, adding buffer solutions. As osadzhuvach most often use ammonia, ammonium carbonate, oxalic acid, ammonium oxalate, as well as substances usually use osazhdzhuyemi soluble nitrate salt. The result obtained oxide nanopowders. If necessary, by treating their environment in, restoring can be obtained metallic nanopowders. Method found quite widely used to produce multi-component powder, when precipitated multicomponent solutions from several'associations. The main disadvantage of this method is a significant amount of impurities in powders and a large variation of particle size.

Sol-gel process was developed specifically to produce oxide ceramics. The process consists of the following stages: preparation of solutions alkoxides, their catalytic interaction with subsequent hydrolysis, Condensation polymerization, further hydrolysis. As a product of the process is obtained oxide polymer (gel). He was subjected to aging, washed, drying and heat treatment. The disadvantage of this method is the complexity of hardware design, and benefit - high purity and uniformity synthesized'associations, and the possibility of obtaining nanopowders of various.

The method of liquid-phase recovery solutions used to produce nanopowders of metals with only low values of reducing capacity (copper, silver, nickel). He is preparing an organic metal salt solution followed by the addition of a strong reducing agent and separation of sludge metal nanopowders. Particle size of the resulting powder 20-40 nm particle size variation is very low. The advantages of this method include the possibility of obtaining nanopowders homogeneous, that represent a composite alloys. The present method are copper oxide powders, yttrium.

Use of recovery. The best known of this group is a hydrogen recovery method'metals associations. WITH'unity metals (hydroxide, chlorides, nitrates, carbonates) react recovery in the stream of hydrogen at a temperature of the order 500 ° WITH. Chemical reactions restoring the example of metal chloride can be written as:

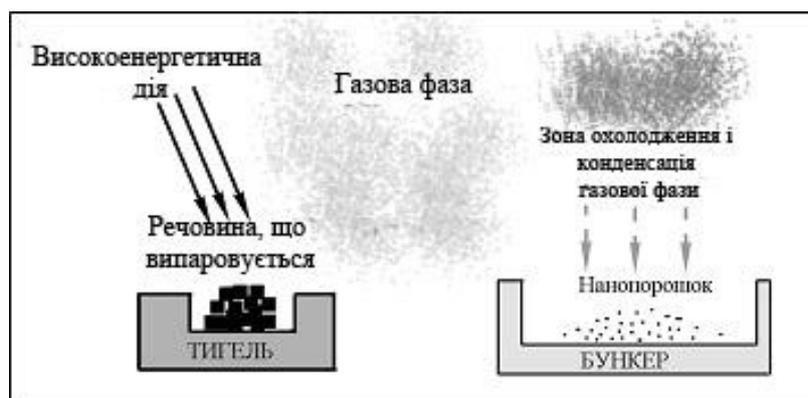


This method can obtain iron powder, tungsten, nickel, rhenium, molybdenum, copper, calcium. It is also possible to obtain powders and alloys steels. The

resulting metal nanopowders are low content of impurities and narrow size distribution.

This group may include chemical-metallurgical method. According to this method comprises a fusion reaction malovodnevyh hydroxide by gas phase interaction, and then heat treatment is performed hydroxide obtained in the recovery environment, for example in water. The result obtained nanopowders iron, nickel, cobalt, molybdenum, tungsten, copper. If the heat treatment is carried out in air, then get, example, nanoporoshok oxide  $Al_2O_3$  or composition. The advantages of this method is little variation in particle size nanopowders, low impurity content, relatively inexpensive process equipment, easy transition from one issue to issue another powder.

Thermal evaporation. With this method, heat conducting material, evaporating, in the crucible. This used different ways of heating (high-frequency induction, electron-ray, electric-, laser) usually, using high intensity sources of energy.. A typical schematic diagram of obtaining nanopowders this method is shown in Figure. 2.1.

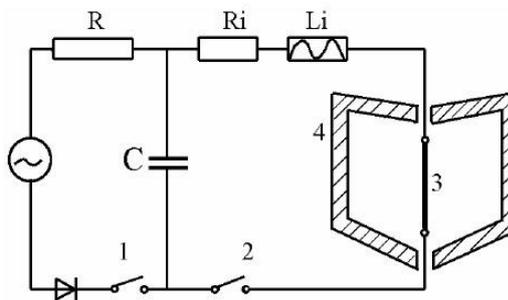


Drawing 2.1 - Schematic diagram of obtaining nanopowders by thermal evaporation and condensation of material vapor

Obtained by this method the powders have a spherical or faceted shape and can be, a metal, and represent an intermetallic or others with unity. Thermal evaporation electron beam oxides in an inert atmosphere obtained amorphous powder  $Al_2O_3$ ,  $Si_2O$  and crystalline  $Y_2O_3$ . The advantage of this method is to obtain pure powders with a narrow size distribution, and lack - low productivity process. This drawback is temporary and is not due to the technology process, A lack of large installations for the production of nanopowders at commercial scale.

The explosive evaporation. This method is based on the allocation of very large amounts of energy in a short period of time. This material evaporates, and then, due to the rapid increase of about' emu, cooled condensation of steam into particles small. In some cases the material may not have time to evaporate,

explosion melted and separated into liquid drops. An additional factor, contributing spray, allocation may be dissolved in the material gas. To supply the necessary amount of energy used powerful impulse electric current, arc discharge or laser pulse. The most common version of such technology received, in which the used wire diameter blast 0,1-1 mm under a short current pulse duration 10-5-10-6 c voltage 10-15 kV and current density 10<sup>4</sup>-10<sup>6</sup> AND/mm<sup>2</sup>. The scheme to obtain powder explosive evaporation of the wire shown in Fig. 2.2. In this case, the capacitor discharge current is created (WITH). Managing the size and structure of the particles is mainly due to changes in density and speed of energy fed. The method allows production of powders of high purity spherical particles with sizes 5 ... 10 nm, including metal with a high melting point and large chemical activity.

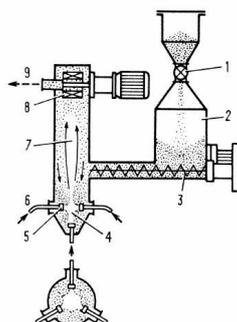


Drawing 2.2 - Apparatus for evaporation wire

The disadvantages of this method is significant energy consumption and, Consequently, Relative jewel obtained nanopowders, and difficulties in removing particles of micron size range, formed from molten droplets.

Mechanical crushing. Mechanical grinding particulate material (previously obtained powders, granules, grinded bars) is one of the most common methods of manufacturing powder. Especially easy to obtain powders from fragile materials. plastic powders, high and amorphous materials get harder. In this case, increases the risk of overheating the material and contamination of its products work wear parts processing equipment.

When using the method in psevdoridkomu grinding grinding powder layer is due to the collision between a. Schematic diagram of the process is shown in Figure. 2.3.



1-feeding device, 2- hopper with raw material particles, 3- supply system particles in the grinding chamber, 4- pseudoridkyy layer, 5- the ink supply gas, 6- pipeline gas supply pressure, 7- camera for grinding, 8 separator, 9 collector output of gas particles

The process of mutual collision of particles, scattered to the high velocity gas jet, place in the middle layer pseudoridkoho, these particles formed. Only a very small number of particles facing the chamber walls, in which the process of grinding.

IN the bottom of the working chamber is a system of nozzles, of which high-pressure gas goes. Gas jets, formed, meet each other in the center of the bottom of the camera, loosen substance, that crushed, and forming a layer pseudoridkyy. This layer is milled particles, that mixed with high speeds from the edges to the center of the chamber. With the grinding zone a stream of particles carried by the gas jets in the upper part of the installation, which is a separator to separate particles by size. Particles, that smaller right size, sweep of the gas flow in filter, which are separated from the gas stream and fall into the storage bin. Large particle separator sends back to the grinding zone.

Powders, obtained by this method, meet high requirements on purity, have high homogeneity and contain particles of about the same size. Intensive gas flow significantly reduces the heating particles during grinding. It can handle amorphous and nanocrystalline powders. The main disadvantage - the complexity and high cost of process equipment in the preparation of powders with nanoscale particles.

### 2.1.2 Methods of forming articles from nanopowders

Compact is a process, which resulted from the powder receiving finished parts. The process is usually carried out in two stages: forming and sintering. Several methods provided by a combination of stage one. To get about'intensive nanomaterials powder formation is mainly used at room temperature, followed by sintering.

Methods of forming. For pressing of nanopowders most widespread technology uniaxial pressing.

We use techniques such as static compaction ( pressing the press-or punching forms) dynamic compaction (magnetically-impulsive and explosive) and vibratory compaction (Ultrasonic).

To obtain a high density using paving pressing, conditions in which compression of the material close to the full. This technology was called isostatic pressing. There are several variants: hydrostatic, hazostatychna, Kvazihidrostatychna. The resulting molding differ almost homogeneous density (although sometimes forming internal volume is less) and have a pronounced anisotropy of properties. The downside is sufficient complexity and high precious equipment and difficulty of obtaining accurate dimensions forming.

Hydrostatic extrusion is carried out using rubber or other elastic membrane thickness 0,1-2 mm, which placed powder. Shell powder is placed in the cooking chamber and fed back hydrostatic fluid (oil, water, glycerin, etc.). High pressure (from 100 to 1200 MPa). The resulting molding can have complex shape.

Hazostatychna pressing conducted using metallic shell (capsules) of aluminum or plastic steel. The form membranes - simple as close to the shape and size of the finished products. Often subjected to pressing hazostatychnomu has previously received other methods workpiece. Metallic capsule placed in the chamber hazostata, which create pressure up to 200 ... 300 MPa.

Kvazihidrostatychna pressing is a simplified version of hydrostatic compression. The powder is placed in a flexible membrane pressing are carried out at unilateral or bilateral additional compression in a conventional stamping .. shell material (rubber or epoxy resin weight) should behave like pressure fluid have a certain elasticity and does not stick together with the powder.

Methods sintering. Sintering paving of nanopowders sintering compared to conventional paving with limited powder inability to use high temperature. Increasing the sintering temperature leads to a decrease in porosity, but on the other hand leads to the growth of grain. This problem is solved by a number of methods of activation that provide a low porosity products at a relatively low temperature sintering:

- using high microwave heating (with increasing heating rate of 10 to 300 deg / min required TiO<sub>2</sub> nanopowders sintering temperature is reduced from 1050 to 975 ° C
- stupinchate controlled sintering;
- plazmoaktyvovane sintering;
- of sintering in a vacuum or recovery media (for metal powders).

Methods sintering under pressure. The combination of forming and sintering process of sintering or pressure can achieve higher density values, including those close to the theoretical value when using lower temperatures compared to conventional sintering.

The easiest way - sintering under uniaxial additional pressure. In conducting sintering iron nanopowder under pressure to 400 MPa sintering temperature, where no porosity, declining 700 to 350 ° WITH, and the size of grain products decreases 1.2 μm to 80 nm. Apparatus, developed in YMET Sciences, nanopowders for sintering under pressure is shown in Fig. 2.4. In the case of metallic nanopowders activation process is carried out in a vacuum or an atmosphere of renewable.

A method progressive combination of processes is forming and sintering hot isostatic pressing. By hot isostatic pressing (ISU) using a universal hazostativ. In modern installations can be achieved to pressure 300 MPa and temperature to 2000 ° WITH. Welded steel capsule powders produced in this case, carbon steel or korroziynostiyykoyi. For simple products use only steel capsule, and for products rather complex form metal-made special form.

The quality of products is influenced not only technological parameters ISU, but the quality of manufacturing capsules and powder filling process their. When filling powder capsules frequently used vibrouschilnennyya powder and conduct thorough degassing and sealing caps. Nanopowders have a large surface area and very exposed to the gas saturation, because of drainage is especially important.

More economical option, instead of using the GUI, is the so-called non-directional fast compaction. This process is used thick-walled cylindrical press-form, after filling the powder is heated and undergoes uniaxial compression at high pressure (to 900 MPa). The external wall of the press-forms tight to a metal cylinder with a size, which prevents deformation of the press-shape. As a result, full powder inside about' Capacity, which has the shape and dimensions of the details of the future, under quasi-static pressure. The method allows spresuvaty metal powder almost 100% the density of a few minutes.

High gas extrusion method is to obtain paving hydrostatic method at room temperature, its heat treatment in a hydrogen atmosphere at a relatively low temperature and subsequent extrusion at elevated temperatures. This method allows for compaction of nanopowders at short-term temperature effects.

Ejecting molding method based on the addition of a special powder called'bandages (example, thermoplastic polymers such polypropylene, polyethylene or polystyrene) providing a low'powder mixture viscosity. The powder mixture is then subjected to injection molding (similar to injection molding of metals and plastics). After receiving metallic conducting firing thermoplastic materials, and then to carry out sintering. Disadvantages of the method floor'related to the complexity of the removal process called'yazuvalnyh substances (change shape details, interaction processes of decomposition products of thermoplastic material powder substances, ensure free removal of the mixture during firing). This method is used to produce limited small special parts of

complex shape with a wall thickness of less 10 mm. A simple variant of this method is the use of an aqueous suspension of powder. The suspension was cast into shape, and then frozen. Drying the workpiece is in frozen form, and then conducting sintering process.

#### control questions

1. Describe the features of obtaining nanopowders, that distinguish them from conventional methods for powders.
2. Describe predisposition to about'unity in aggregates and agglomerates nanopowders.
3. Describe the nature of technologies of nanopowders methods of deposition solutions.
4. Describe the nature and use of appropriate examples sol-gel processes.
5. Describe the nature and examples using appropriate methods of liquid-recovery phase.
6. Describe the nature and use of appropriate examples of the use of recovery processes.

## TOPIC 3

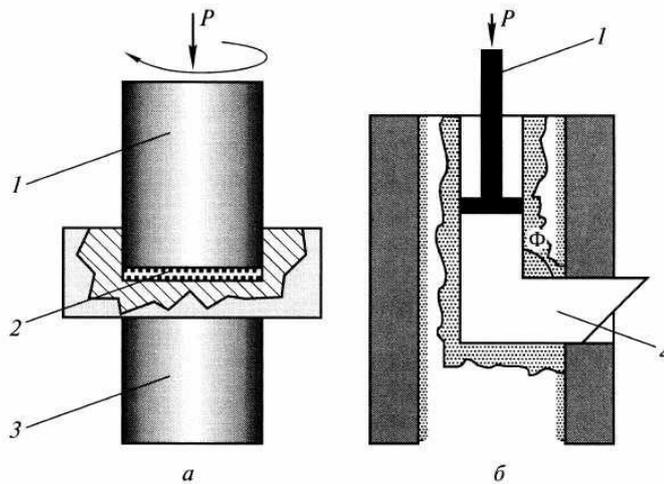
The study of the structure and properties of nanomaterials obtained by methods exactly-channel angular pressing and torsion under pressure

### 3.1 General Information

Group methods EQUAL CHANNEL ANGLE pressing and torsion pressure based on the conduct of plastic deformation with a high degree of deformation at high pressure applied at relatively low temperatures. Under such conditions there is a strong deformation of grinding microstructures in metals and alloys to nanosized range. In developing these methods, a number of these requirements: ultradribnozernystyh preferential formation of structures with grain boundaries velykokutovymy (in this case, there are qualitative changes in properties) the need to ensure the stability of homogeneous material properties due to the formation of nanostructures around about' volume of material, no mechanical damage and cracks, even under severe plastic deformation of the material.

This group of methods will get about'succinct bezporysti metallic nanomaterials. It should be noted, that grain size range of materials, The method proceeds, usually, is still more 100 nm. Structure, received the intensive plastic deformation, different nerivnovazhnisty strong due to the low density of dislocations and preferably free velykokutovym nature of grain boundaries. Therefore processed products used additional heat treatment or additional plastic deformation at high temperatures with a high degree of deformation. Currently most spent two methods (Fig. 3.1) -Method torsion under high pressure and method exactly channel angular pressing.

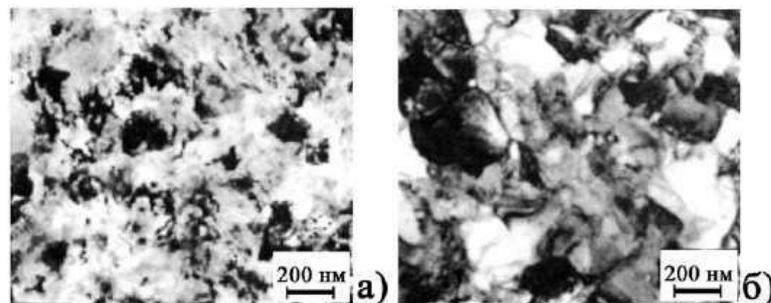
Method torsion under high pressure based on the principle nakovalen Bridgman, in which the sample is contained between Boyko and compressed by applying pressure in a few GPa, and then deformed with more (10 and more). The lower rotating firing pin, and the forces of surface friction force to deform the sample shift. The sample has a geometric shape as the disc diameter 10 ... 20 mm thick 0.2 ... 0.5 mm, providing conditions for the main hydrostatic compression against'volume of material and the condition of the sample neruynuvannya. The structure of the material begins podribnyuvatysya strain after half a turn pattern. Formation ultradribnozernystoyi structure achieved after several revolutions the deformation of the sample. The average grain size can reach 100-200 nm (Fig 3.2and) and defined deformation conditions - pressure, temperatures, deformation rate and type of the material.



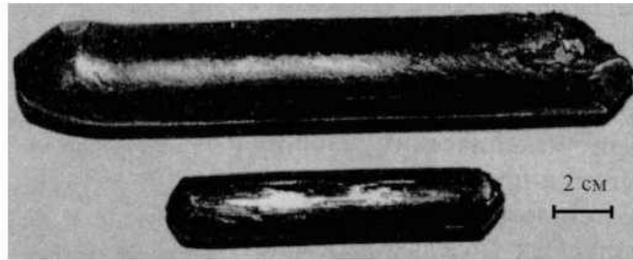
Drawing 3.1 - Scheme severe plastic deformation methods: a- Method torsion under high pressure, b- EQUAL CHANNEL ANGLE pressing method.

EQUAL CHANNEL ANGLE method gives a pressing parts with a diameter up to 60 mm and length 200 mm (Fig. 3.3). This method is also based on the use of strain shift. For this billet is forced to repeatedly equipping special two intersecting channels with identical cross sections. Often used between channels equal angle  $90^\circ$ , where in one push material provided the true degree of deformation  $\approx 1$ . Process temperature depending on the material being processed is selected room or slightly elevated. An important issue is to maintain the integrity of the samples obtained for maloplastychnyh and hard deformable materials. The method allows to form ultradribnozernystu structure with an average grain size in the range of 200 to 500 nm (Fig 3.2b).

Developed other methods of severe plastic deformation, example, comprehensive forging and rolling special.



Drawing 3.2 - Nanostructures copper, obtained by different methods: and- by torsion under high pressure, b- by pressing EQUAL CHANNEL ANGLE.



Drawing 3.3 - About'succinct piece of nanostructured titanium.

### 3.3 control questions

1. In what ways carry IPA?
2. Describe requirements, that apply when using IPA for crushing structure.
3. Describe a range of sizes and materials disorientation grains, IPA derived methods.
4. Why the material structure, received by IPA is characterized by a strong nerivnovazhnisty? How will reduce the specified structure nerivnovazhnist?
5. Describe the opportunities and examples of application methods SDI industry.

## TOPIC 4

Stability studies the structure and properties of nanomaterials consolidated at high temperatures

### 4.1 Overview

IN creating new materials are increasingly using nanostructured approach based on the use of nanomaterials benefits. Features of the structure of nanomaterials (small grain size and inclusion, high surface separation boundary, the presence of nonequilibrium phase Segregation and residual stress) causing a high level of physical and mechanical and physico-chemical properties. These features determine nerivnovazhnist vsyh nanomaterials in nature. It is therefore necessary, with special attention to treat the problem of stability. From general considerations it is obvious that thermal, deformation and radiation effects, leading to recrystallization, homogenization, relaxation and so on. phenomena resulting nanostructures can be modified (and sometimes its annihilation) and therefore reducing properties. But, to nanostructures characterized by factors that retard recrystallization (presence of a large number of triple junctions, nonmonotonic dependence of the free energy of the size of crystallites during formation nanovklyuchen spinodal decomposition, the presence of insoluble precipitates in dyspersno- and dispersion-strengthened alloy). Since multicomponent alloys difficult to clearly assess the contribution of different factors that contribute to the preservation of the nanostructures or vice versa, contributing to its elimination is studying the stability of nanostructured multicomponent alloys mainly conducted experimentally. the presence of insoluble precipitates in dyspersno- and variance-hardened alloys). Since multicomponent alloys difficult to clearly assess the contribution of different factors that contribute to the preservation of the nanostructures or vice versa, contributing to its elimination is studying the stability of nanostructured multicomponent alloys mainly conducted experimentally. the presence of insoluble precipitates in dyspersno- and variance-hardened alloys). Since multicomponent alloys difficult to clearly assess the contribution of different factors that contribute to the preservation of the nanostructures or vice versa, contributing to its elimination is studying the stability of nanostructured multicomponent alloys mainly conducted experimentally.

#### 4.1.1 Examples of nanomaterials and their production variants

As advanced refractory materials currently considered niobium silicide alloys with strengthening and reinforced ceramic composites based on silicon carbide. In

addition to high long-term strength (MPa at hundreds of aging at 100 or more hours) these materials pre-are a number of other requirements: high resistance to thermal cycling and creep, heat resistance, high performance in viscosity destruction, termomitsnosti and adaptability, low density, etc.. If niobiyevyh alloys (example, Nb-16Si-15W-5Mo-5Hf-5C (atm%)) expected, what high strength stohodynna (150 MPa) at 1500 ° C is achieved due to doping niobiyevoyi matrix of tungsten and molybdenum, and the resulting dispersion strengthening silicide Nb<sub>5</sub>Si<sub>3</sub> and complex carbide (Nb, Hf, Mo) CX. The required corrosion resistance is achieved by using a three-layer termobar' yerno cover.

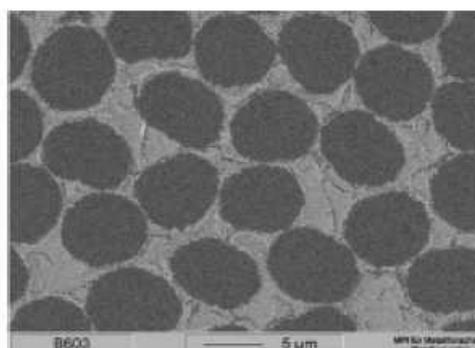
Production of nanomaterials with high heat resistance can be realized using mechanical-chemical synthesis components using high-grinding, followed by consolidation of nanopowders of membranes evacuated using high-temperature extrusion.

Example, in the alloy MA957 (that alloy Cr-0.9% weight., Ti-0,3%weight., Mo-0.25% weight). Size strengthening nanovklyuchen (complex oxides Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>) in the initial samples was 2,1 ± 0,4 nm after annealing for 3000 h. at 900 ° WITH, 950 ° C and 1000 ° With varied slightly: 2,1 ± 0,4; 2,6 ± 0,5 and 3,1 ± 0,9 nm. in accordance. The value of initial microhardness and annealed samples were similar and were 3330 ± 80, 3320 ± 80, 3230 ± 70 and 3300 ± 60 MPa.

With developments in ceramic composites are promising materials based on amorphous matrix Si-BCN, strengthened nanovklyuchennyamy Si and graphite fibers.

Fig. 4.1 illustrates the microstructure of the composite, produced multiple impregnation pyrolysis liquid precursor Si-B-CN powder preparations based powder α-SiC and graphite fibers.

Relative density after seven cycles of impregnation and pyrolysis was more 90%; weight loss when heated to 2100 ° C was about 1.8%. The value of flexural strength at initial samples 1500 ° C was at ~ 210 MPa. After a ten-aging temperature 1700 ° With strength at room temperature of the material is preserved in 96% and the destruction was of no fragile nature; creep deformation under constant bending over 60 hours (T = 1400 ° WITH, tension 100 MPa) was only 0.25%.



Drawing 4.1 - The microstructure of nanocomposites based amorphous matrix Si-B-CN, strengthened nanovklyuchennyamy Si and graphite fibers

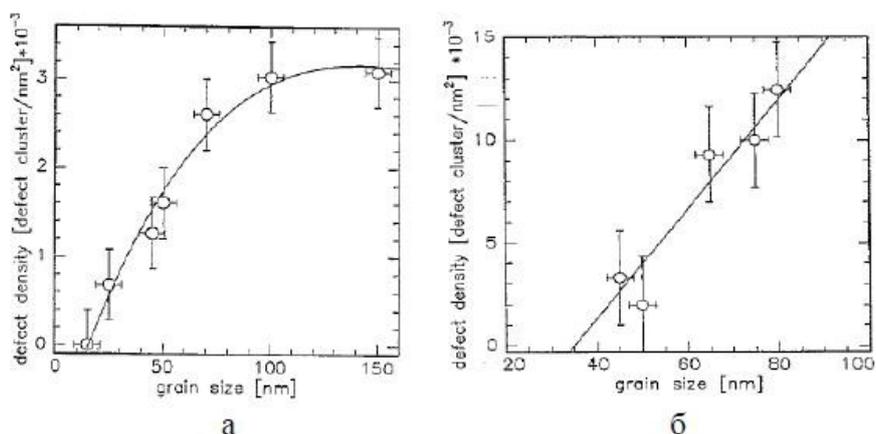
High thermal stability parameters also have Nanocomposites SiCfiller / Si-CNmatrix. Flexural strength of samples of these nanocomposites at 20 ° C in the initial state and after a ten-annealing temperature 1700 ° C, 1900 ° C and 2000 ° C was  $357 \pm 45$ ;  $373 \pm 27$ ;  $323 \pm 33$  and  $314 \pm 40$  MPa respectively.

The value in'viscosity destruction ( $K_{IC}$ ) is known, is the weak point of ceramic materials. cOMPOSITE SiCfiller / Si-CNmatrix K value<sub>IC</sub> as low ( $2.7 \text{ MPa}\cdot\text{m}^{0.5}$ ). However, the transition to nanostuktur may be accompanied by a significant increase in resistance to crack propagation. So, for tetragonal zirconia modification, alloy 3% Y2O3 (3YTZP), a decrease in grain size 10 time (with 850 nm 85 nm) meaning  $K_{IC}$  increased approximately 2.5 fold (with  $5,9 \pm 0,2 \text{ MPa}\cdot\text{m}^{0.5}$  to  $15,5 \pm 7,9 \text{ MPa}\cdot\text{m}^{0.5}$  ). mention, that in'viscosity of about destruction steel 40  $\text{MPa}\cdot\text{m}^{0.5}$ .

#### 4.1.2 Effect of irradiation on structure and properties of nanomaterials

Results of general considerations suggest that exposure to radiation defects or nanostructures can amorfizuvatys or, on the contrary, facilitate their removal due to the presence of numerous separation surfaces that may function as drains. Fig. 4.2 shows the dependence of the density of radiation defects fixed by deformation prosvichuvalnomu contrast electronic microscope, the size of the grains in the samples irradiated ZrO2 and Pd. As seen from these data, the grain size of less than 15 nm (ZrO2) and 30 nm (Pd),

defects are not detected, that they go to Intergrain beyond the borders of samples and / or recovered at grain boundaries.



Drawing 4.2 - Effect size nanocrystals on the concentration of radiation defects in the oxide of zirconium (a) and palladium (b) irradiated with ions Kr

The difference in the size of defect-free grains ZrO<sub>2</sub> and Pd, probably, caused by a high diffusion mobility of point defects in palladium compared to zirconium oxide.

The positive impact on reducing nanostructure particles amorphization structure, Radiation hardening and swelling observed for nanostructured ferritic, ferrite-martensitic and austenitic steels.

However, in the case of isolated nanocrystals ZrO<sub>2</sub> and Cu observed their amorphization, reflecting competition radiation-induced amorphization process and removal of radiation defects, which may occur in nanomaterials. Developed net division surfaces, on the one hand, promotes excess free energy and reduces energy barrier for amorphization, On the other hand, helps to remove radiation defects and prevent amorphization. Based on the energy approach, You can select preferred dimensional zone of influence of a factor, However, quantitative evaluation and comparison of the experimental data so far hampered.

Thus, nanomaterials exhibit higher radiation resistance compared to their counterparts Coarse and nanostructures is rather helpful to reduce the negative effects of radiation. It makes nanomaterials based on chromium steels, silicon carbide and tungsten promising candidates for use, for example, new models of reactors.

### 4.3 Quiz

1. Describe the particular structure of nanomaterials.
2. What factors caused by high physical and mechanical properties of nanomaterials?
3. What caused nanocrystalline structure of nanomaterials?
4. The reasons for the possible instability of the structure and properties of nanomaterials.
5. What factors can slow recrystallization in nanomaterials?
6. What methods is preferably carried out stability study of nanomaterials?
7. What are pre 'are heat resistant to nanomaterials?

8. Give examples of promising technologies for heat resistant nanomaterials.
9. What are the reasons nanomaterials can be stable at elevated temperatures.
10. Give examples of the impact of radiation defects on the nanostructure of nanomaterials.
11. Describe how promising nanomaterials and high-temperature nuclear.

## TOPIC 5

Studying the properties of structural steel ultradribnozernystyh, obtained during deformation nanostructurization

### 5.1 General Information

At a certain stage of deformation as traditional methods, so i severe plastic deformation methods (IPA) structural changes in metal materials is largely similar. By the plastic deformation in the coarse-grained metallic materials leads to dislocation slip schilnoupakovanyh crystal lattice planes. Vysokokutovi boundaries impede dislocation transition from one grain to another, which leads to their accumulation in grains. When the degree of deformation is  $\geq 0.2-0.3$  accumulation of dislocations sealed, and form hard-interlaced spatial grid imperfect boundaries, separating the grain into many small cells. The peculiarity of the cellular structure is relatively low dislocation density inside the cells compared to their borders and between small disorientation, that is a small fraction degrees. Such disorientation cells not prevent dislocation shift, However, its development requires continuous improvement of external stresses, because stress field, created imperfect cell borders, opposing dislocation motion. In addition to increasing their stress shear stress leads growth, necessary to generate dislocations in the lattice of small cells. With the recent formation of the further evolution of structures in normal and intensive strains differ.

Unlike traditional methods in IPA after deformation with a degree  $f \sim 3-4$  many materials is not usually Way, and rivnoosna zerenna structure and subzerenna. Some areas are also small cell. With further increase of deformation of cells disappear altogether, and the proportion of severely crushed grains in a mixed structure grows and becomes overwhelming.

There are several ideas about the mechanism of boundary velykokutovyh, leading to the formation of structure in the IPA ultradribnozernystoyi. One of them is based on the passage of the low-temperature recrystallization, also known as

"cold" or recrystallization in place. Due to diffusion nature recrystallization, its passage at high degrees of cold deformation floor'yazuyut accumulation of metals in unusually high concentration of point defects and a significant gradient elastic stresses, created dislocation clusters. In favor of the formation mechanism of rekrystalizovanoho velykokutovyh boundaries also known pattern suggests lowering the temperature of beginning of recrystallization in metals up to a temperature higher degree of turnover accumulated strain.

However, it should be noted, that when the temperature circulation usually refers to the temperature, where dislocations in imperfect cells redistributed boundaries and form a perfect malokutovi border cells-blocks (subzeren). In clusters dislocation loops focused segments of the same sign and the same type, known as charge dislocation. spontaneously, Ambient heating, these borders (congestion charge dislocation) not improving and are not velykokutovymy, what, and confirmed the presence of cold material in imperfect cells. The required thermal activation is provided with deformation temperature increases in the areas of shear localization. At the big shifts briefly raising the temperature can reach hundreds of degrees, which is sufficient to rebuild the grain-boundary structure, especially pure metals, which are characterized by high mobility of dislocations. However, the total improvement need common borders heating material, at least, to a temperature of turnover, and to form velykokutovyh borders while also requires considerable inflow of lattice dislocations in the boundary cells.

Considering, IPA that when strain is nonmonotone (multidirectional) nature, for the formation of large-angle boundaries not necessary accumulation of material deformed extremely high concentration of point defects. The role of the low-temperature diffusion in this case plays rotary (turbulent) type strain, arising under the influence of external stress points.

IN vyschevidznacheni whole idea considering the transformation step are formed during deformation imperfect boundary cells in velykokutovi. By the later stages of formation velykokutovyh boundaries of small grains include model, based on the process of twinning. This process is activated, When the field

razoriyentatsiyi, emerging during deformation (cell, subzerna, grains) become so small sizes, that the generation of lattice dislocations in them becomes impossible.

The main role in the formation of small grains or owned by other types of linear defects, without which neither the active material deformation, It formations arbitrary boundaries velykokutovyh, including by converting them in twin boundaries.

There are two ideas about the formation of small grains, due to changes in linear defects during deformation. One of them is considering the formation of grain boundaries and subzeren as a result of more or less imperfect saturation boundary cell lattice dislocations. Another idea is based on the formation and movement at an early stage of deformation at  $e \geq 0.2-0.3$  collective forms of strongly interacting dislocations - partial dysklinatsiy.

The main factor, which determines the size of the grains formed, are the minimum size of cells. These errors without much comparable grain size and subzeren, obtained both by direct saturation boundary cells dislocations, and because passing through them partial dysklinatsiy, because in the latter case, the size of the cells determines the distance between the boundaries of bands and frequency of crossings.

Themselves cell sizes become the principle of community strain. For the first time this law was clearly demonstrated P. Kahn an example of structure bending zinc plate. In this experiment, bending plate, followed termoaktyvatsiyeyu it the return of a small heating, led to the formation of blocks (subzeren) a trapezoidal shape, which has more bases located in an area stretching, and less - in the area of compression plates. The principle of community strain is reflected not only in acquiring the specified form subzernamy, but in the, Corner razoriyentatsiyi that their side boundaries (sides trapezoids) amounting answered corner bend plate.

So, in connection'connection with the prospect of the application process for obtaining strain nanostructurization structural materials with complex high mechanical properties, one of the important issues is a comprehensive study of the evolution of properties of structural steel grades ultradribnozernystyh 20 and 45 using destructive and non-destructive methods of control.

5.1.1 The study of mechanical properties ultradribnozernystyh material non-destructive method of control

At present none of the technological process for responsible products are not introduced into the industry without a proper system of NDT measurements with various informative parameters. Among the most common are the following informative parameters: coercive force, magnetization, induction (residual induction) permeability, Barkhausen effect, which is related to the physical-mechanical properties of a material. Example, strength and magnetic parameters.

When the magnetic control physical-Mechanical properties against a material used, the relationship between strength and magnetic parameters. This relationship is called the Jiles-Ashby relationship. It occurs when, if the processes of structure and phase composition of ferromagnetic steel forming their magnetic properties. One example of such a correlation is called the relationship between hardness and low-carbon steels and their coercive force.

### 5.3 control questions

1. By what mechanism of plastic deformation occurs coarse-grained metals?
2. Why the development of plastic deformation requires constant raising tensions?
3. Above what extent changes in the structure in the usual plastic deformation and different IPA. What is the difference?
4. Describe the understanding of the mechanism of UDS.
5. What is the role of low rotational deformation mode, arising under the influence of external stress points.
6. Calling factors, determining the size of grains formed in the IPA.
7. To prove the need to use non-destructive methods of control was nanostructures.
8. What is the basis of the relationship between strength and magnetic parameters.

## TOPIC 6

The study of the structure and properties of nanomaterials, obtained by ion surface treatment

### 6.1 General Information

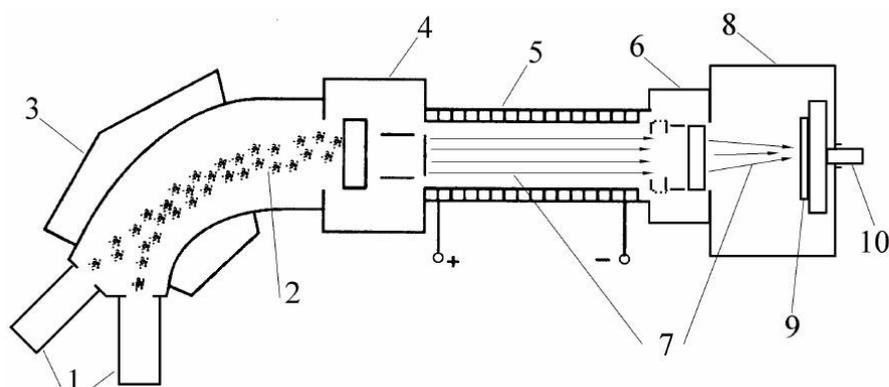
Preparation of nanocrystalline structure about'yemi product is difficult even using this method as deformation hidroekstruziya (TSE). Much easier to carry out surface nanostructurization. With this treatment there is healing defects, products formed on the surface of nanocrystalline layer thickness of about 20 nm, which behaves under load as superplastic. As a result, increases border fluidity without reducing ductility, allowing the use of surface nanostructurization to enhance structural strength products. Its advantages are very high ductility without reducing strengthening, which does not provide any of the known methods.

One way to obtain nanocrystalline surface layer is ion-plasma treatment, which has long been successfully used to increase tool life, improving the tribological characteristics of surfaces of machine parts, products provide corrosion resistance. This type of treatment is not only changing the surface properties, but also greatly strengthens the entire product, that affect its "atb'succinct" mechanical properties.

Different types of treatment - ion implantation, ion-radial mixing, etc..

By ion implantation

This method involves the introduction of high-energy ions into the surface material. The process is carried out in a vacuum procedure  $10^{-4} \dots 10^{-3}$  Pa using ion-beam accelerators (implanteriv). These settings (Fig. 6.1) include one or more ion sources, which converts the material into an ionized state - plasma. ions, implanted, can be created by electric arc, thermal evaporation techniques, etc.. ions, formed, coming into the analysis and separation, which are separated from the main beam of ions unwanted impurities. After separation using ion beam focusing lens focuses a beam, which falls into the accelerator, where they are accelerated to high energy in an electric field. To further stabilize the ion beam and scanning its implementation is the system of electric lenses and deflecting plates.



1- ion source, ions flow 2, 3 system analysis and separation of ions focusing system 4-, 5- ion accelerator, 6- Stability and scanning ion beam, ion beam 7-, 8- vacuum chamber, the material 9- that the modified 10-piece fastening material that is modified

Figure 6.1 - Scheme ion surface treatment method.

In the event the material is modified, the ions are introduced into it to a depth 5-500nm depending on their energy. Conditionally emit low-energy ion implantation of 2-10 keV ion energy and high-energy ion implantation energy of 10-400 keV. Depending on the design implantera spot diameter of the ion beam on the surface of the material can range from 10 to 200 mm, and the average value of the ion current of 1-20 mA.

When bombarded with ions of the surface layers of the material that is modified, there is a range of complex processes. Besides implantation (penetration) ions in the surface material occur, including processes such as sputtering surface cascade (ballistic) mixing of atoms in the surface layer of the material, radiation-induced diffusion, the formation of metastable phases, radiation-induced segregation (redistribution of atoms in the surface material layer), preferential sputtering, adsorption Gibbs (change the surface structure by reducing the free energy), etc. The relationship between these processes depends on the type of ions implanted, and material that is modified and technological mode of treatment.

The main advantages of ion implantation method for creating a modified surface nanolayers are:

- possibility to get almost any combination of materials in the surface nanolayers;
- independent of the solubility limits of the components in the solid phase (that can receive such alloys that are impossible under normal conditions due to thermodynamic constraints);

- low temperature material that is modified, no significant changes in the size, structure and properties of the basic material, no obvious boundaries of separation and adhesion problems;

- kontrolyuyemist depth processing;

- high stability and reproducibility of the process;

- high purity process in a vacuum;

- opportunity through high-precision scanning ion beam on the surface of the treated surface to create complex nanostructures;

- simultaneous or sequential implantation of various materials.

The disadvantages of the method include:

- the possibility of surface treatment materials only in the line of action of the ion beam;

- low penetration of ions in the material (especially at low energies);

- processes of spraying the surface;

- the high cost and complexity of the equipment and processing;

- the complexity, lack of knowledge and difficulty controlling all the complex processes that occur during ion implantation.

**Ion-beam mixing**

The process leading to a vacuum of  $10^{-3} \dots 10^{-2}$  Pa. As the inert gas argon is used often or xenon. Use ions with higher atomic numbers results in improving the efficiency of mixing processes.

### **6.3 control questions**

1. In what ways exercise DCI?
2. Describe requirements, that apply when using DCI.
3. Describe the grain size range of materials, DCI obtained by methods.
4. Why the material structure, received by DCI characterized by a strong nerivnovazhnisty?
5. Describe the opportunities and examples of application methods DCI industry.

## Appendix A Safety Regulations in the performance laboratory work

1. Begin to perform work only with the permission of the teacher.
2. To work with equipment allowed persons, who studied design features and technical description of concrete testing machine, and tested knowledge of electrical safety.
3. In the mechanical laboratory equipment can operate person, who received safety training and have tolerance for working on equipment laboratory.
4. In the heat and preparates laboratories person can work, who have been instructed on safety and design features identified furnaces and exhaust cabinets. All electric conductive parts must be isolated and fenced, and the rest of nestrumoprovidni - firmly grounded.

### Safety requirements before starting work

1. Study the instructions of, performing.
2. Check availability of the necessary equipment and tools.
3. Report to the possibility of teacher performance.
4. Students do not have to get to work, until you get safety training.
5. Working near electrical appliances must be carried out with the use of insulating means (insulating mats, insulating supports).
6. Check serviceability of devices and equipment ovens, prepare the work tool, vantazhnozahvatni devices and ensure proper operation.
7. Loading and unloading of items performed using special tools pickup.
8. If installed furnace malfunction, should inform the teacher.
9. Repair equipment independently by students is prohibited.

### Safety requirements during performance

1. Zaboronyayetsya students to turn on and off electrical appliances and electric without permission of the laboratory work.
2. Let all repairs, establishing electrical electrician must perform, serving lab.
3. Do not work alone in a laboratory. Mandatory' compulsory presence of the head of laboratory work. Switching, change the mode of installation, of measurements and off settings to prevent malfunctions, must be performed only with the permission of a teacher in strict accordance with the provisions of, provided in the technical description of.
4. Persons, which are not fixed by this installation, perform any-repairs are prohibited.

### Safety requirements in heat and laboratories preparates

1. Switching on and off electrical equipment must be carried out only with the permission of the teacher, conducting classes.
2. If you find all kinds of faults should immediately stop.

3. It is prohibited to repair equipment to students and persons, with no qualifying group on TV.

4. Loading and unloading kilns permitted only off.

5. Work with faulty grounding pad is prohibited without dielectric.

#### Safety requirements after work

After testing a mechanical laboratory must: disable all sections of electrical installations and disable devices.

B heat and prepares the laboratory after work you must turn off the oven, off ventilation, electrical switch, remove jobs

#### Safety requirements in emergency situations

In case of fire or fire must:

1. Call the fire department by phone 01.
2. Off the grid, ventilation, equipment.
3. Notify lab manager and teacher.
4. Immediately leave the space lab.