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Projekt pt.: **„Zintegrowany Program UTHRad.”**, POWR.03.05.00-00-Z105/17
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Course title: Materials Engineering and Application

Lector: Dr. Prof. Edwin Gevorkyan

Ukraine State University of Railway Transport
Kharkov, Ukraine

LECTURE №3

Equipment and devices

The design of devices for hot pressing is determined by the method of heating and applying pressure, as well as by pressing temperatures, the need to use or not a protective gas environment or vacuum, and a number of other factors. In the hot pressing process, an electric current is usually used for heating, although the powder or the mold with the powder can be heated in any other way before they become pressured. The following methods are most common for electric heating of powders during hot pressing:

- direct electric heating of a conductive mold by passing current through a mold or sintered material;
- indirect electric heating of the mold placed inside the electric heater;
- direct induction heating of a conductive mold;
- indirect induction heating with a conductive pipe, inside of which a mold of non-conductive material is placed and heated.

The schemes of double-sided hot pressing devices in combination with the heating methods listed above, is shown in Fig. 3.1.

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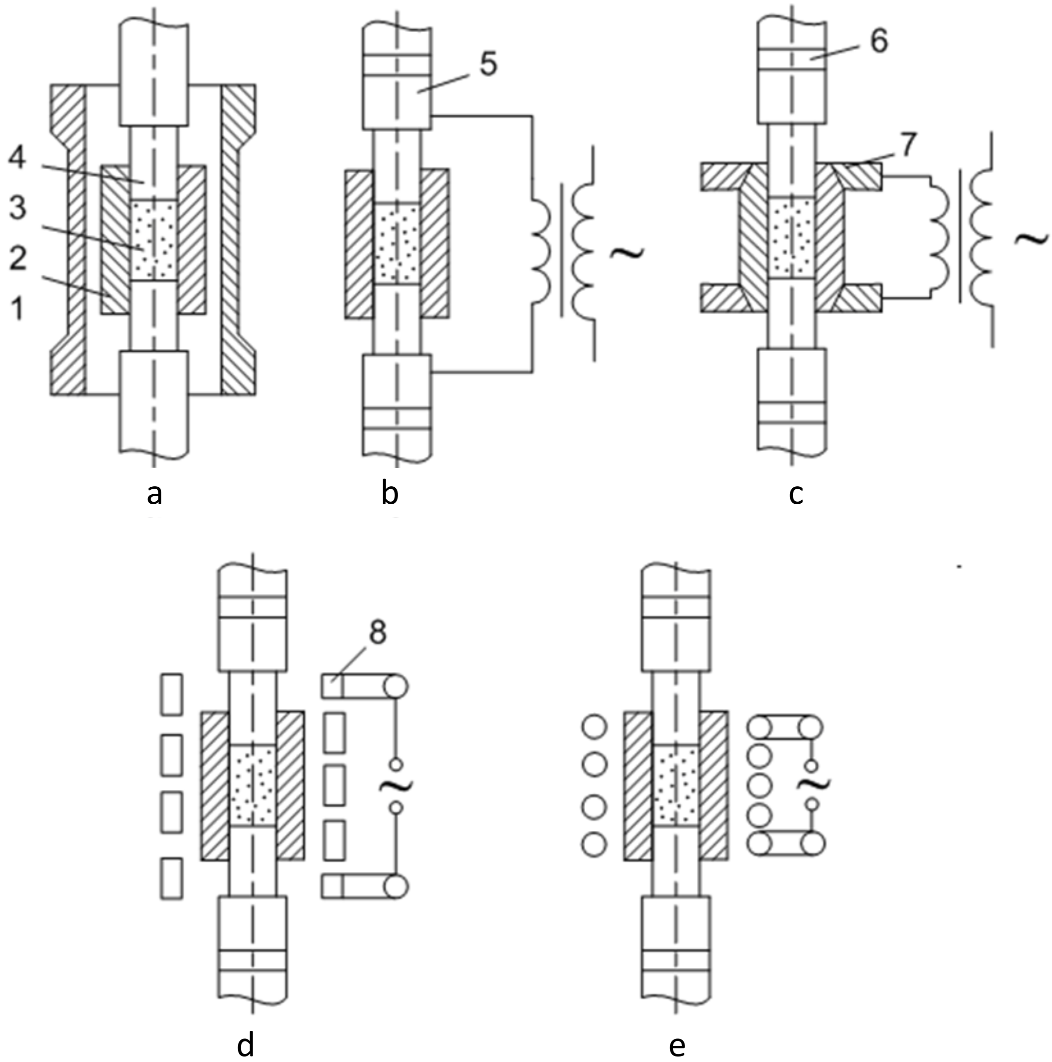

Fig. 3.1. The hot pressing double-sided pattern in molds in combination with heating methods:
a - indirect heating in a resistance furnace; b - heating by direct transmission of current through the punches; c - indirect heating while passing current through the matrix; d - induction heating of the conductive (graphite) matrix; e - induction heating of the powder in a non-conductive (ceramic) mold;

1 - heater; 2 - powder; 3 - sintered product; 4 - matrix; 5, 6 - punches; 7 - isolation; 8 - graphite or copper (water-cooled) contact

In the abovementioned heating methods, the outer layers of the samples reach the sintering temperature faster than the inner ones, since the sample is heated externally. To achieve thermal equilibrium, a long time is required. This leads to the capture of gases released from the colder inner zone, and thus prevent from obtaining high-quality products. The uniformity of powder heating can be achieved in ultrasound assisted process. Dissipation of ultrasonic energy in the powder not only leads to direct heating of the powder, but can also contribute to the removal of absorbed gases and better compaction. When pressing electrically conductive materials, this is ensured by direct transmission of current through the pressed powder using punches and a die where an insulating sleeve plays the role of electrodes (Fig. 3.2.)

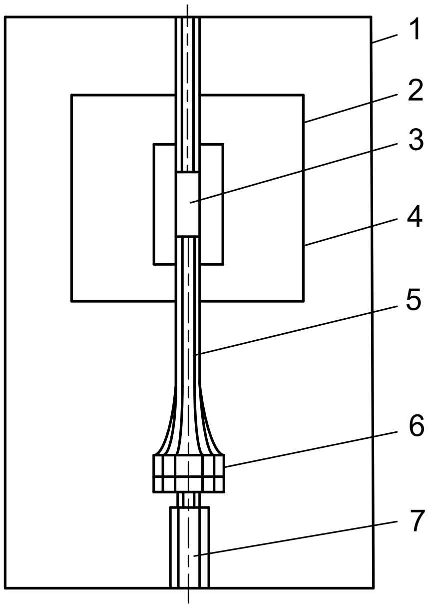

Fig. 3.2. Ultrasonic Hot Press device:

1 - press frame; 2 - top cover; 3 - powder; 4 - oven; 5 - connecting console; 6 - converter; 7 - hydraulic press

The design of the experimental unit PGP-2 is shown in Fig.3.3. It allows hot pressing in a protective and reducing medium, as well as in vacuum at temperatures up to 2500 °C and forces up to 20 kN with continuous recording of the kinetic curve of compaction. The mold is heated by a graphite heater or direct current transmission. Temperature is measured with a thermocouple or optical pyrometer. The hot junction of the thermocouple is placed in the immediate vicinity of the pressed sample, which makes it possible to measure the heating temperature with an accuracy of ±10 °C.

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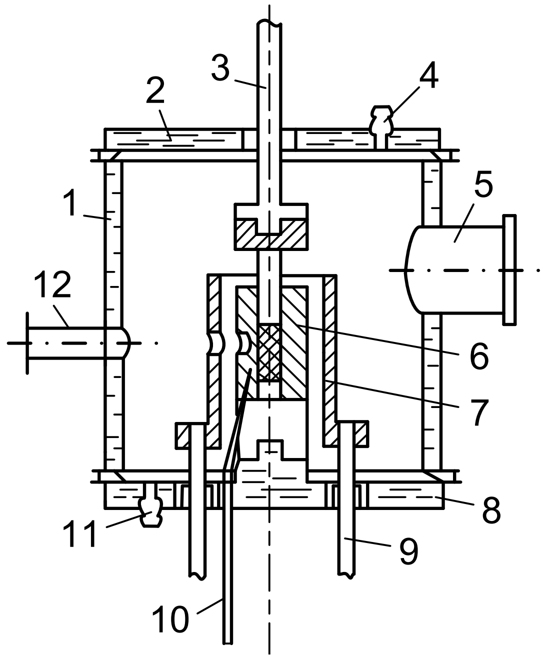

Fig. 3.3. Scheme of the working chamber of the experimental installation PGP-2: 1 - housing; 2 - top cover; 3 - powder; 4 - a fitting for supplying a protective gas; 5 - pipe for connecting a steam-oil pump; 6 - mold; 7 - heater; 8 - bottom cover; 9 - current leads; 10 - thermocouple; 11 - fitting for the discharge of protective gas; 12 - peephole

The loading system at the PGP-2 installation is made in two versions:

1) For hot pressing of samples with an area of up to 2 cm^2, a pneumatic cylinder operating on compressed air is used. At a maximum air pressure of 0.5 MPa, the press force is 6 kN.

2) For hot pressing of larger products (up to 15 cm^2), a hydraulic cylinder operating from a hydraulic pump is used. In this case, the ultimate force transmitted to the punch is 25 kN. The continuous record of shrinkage kinetic curve during hot pressing is performed by an analog recording device, a diagram of which is shown in Fig. 3.4. The pressing rod 1 connected to a leash 2 rotates the two-lever floating arm 3, and the movable pen 4 is fixed to its end. The electric motor 5 rotates the drum 6 with special paper at a constant speed of 0.2 rev/min. The recording accuracy of absolute linear shrinkage is 0.1 mm. The PGP-2 unit is equipped with a fore-vacuum pump and an steam-oil pump, which provide a vacuum of 6.6 Pa.

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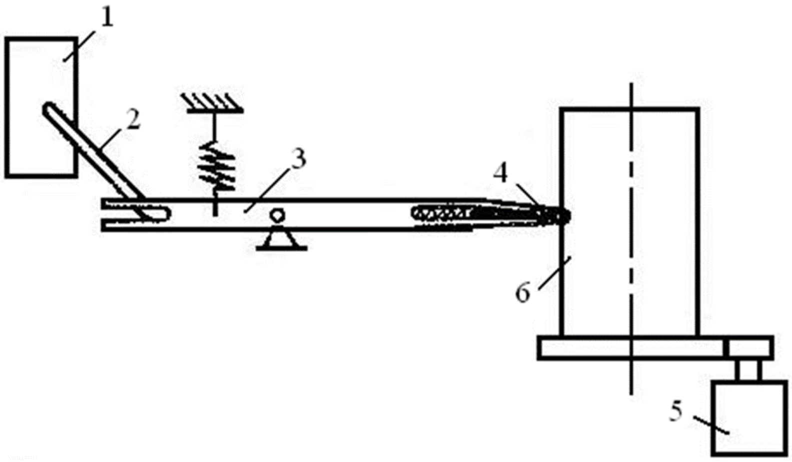

Fig. 3.4. The scheme of recording device

Among the materials used in the equipment for hot pressing, graphite is most widely used one. Graphite is relatively inexpensive, can be easily machined, it has a low density, low electrical resistance, high heat resistance and sufficient mechanical strength in a wide temperature range.
However, the graphite limited use for mold material for hot pressing is due to the low mechanical strength that does not allow the process to be carried out at high pressures and the ability to recover some materials, especially oxides. Graphite also reacts with transition metals, with their nitrides and silicides. Apart from graphite, the metal molds are most widely used, especially the ones made out of molybdenum-based alloys. They are used for pressing polycrystalline optical materials, such as difluoride and magnesium oxide, lead selenide, etc. The molds made of oxides and other ceramic materials are used much less frequently because they have low heat resistance, they are difficult to process and not always compatible with the pressed materials.
To prevent welding of the sintered material to the walls of the matrix and the ends of the punches, their working surfaces are coated with lubricants. Usually, a powder of flake silver graphite in the form of suspensions in glycerin or liquid glass, as well as colloidal graphite is used as a lubricant. Suspension of boron nitride in glycerol also has good lubricating properties.

Despite its great advantages, the hot pressing method is used relatively rarely. Its demerit is the presence of serious inner defects in the finished product in form of large internal stresses of thermal origin, sometimes reaching so high values that the cracks appear in the material during its storage. Thermal stresses are caused by the uneven distribution of temperature in the bulk material during hot pressing, and especially during cooling. The temperature field can be measured during the hot pressing process of hard alloys by simultaneously introducing several thermocouples at different points of the matrix and into the sintered product. In such a study, a significant temperature gradient was established. In the center of the mold with a diameter of 120 mm, the temperature can be 70 ÷ 100°C higher than that on the surface. The temperature difference along the height of the mold between the geometric center and the plane of contact with the cooled current leads is more than 1000 °C, while during sintering in a hydrogen furnace, the temperature difference in the sintering zone at the same distance does not exceed 10 °C. The appearance of thermal stresses is also promoted by the high heating and cooling rate. Among other disadvantages of the hot pressing method, it is worth noting the low productivity of the process, the high consumption of graphite and electricity. The hot pressing method in powder metallurgy in general, and for the production of composite materials in particular, is used only when it is impossible to obtain a dense product using the conventional pressing method with subsequent sintering.

Usually, the hot pressing method is designed to obtain bulk materials out of powders of refractory compounds (carbides, nitrides, etc.) or metal fibers. In this method, heating together the workpiece and the mold itself leads to its oxidation rapid destruction. Moreover, the energy required to heat the mold significantly exceeds the one required to heat the workpiece. However, it should be admitted that in a number of cases, this method is the only one that allows to obtain high-quality composite material.

A composite materials obtained from nano- and submicron powders by hot pressing assisted with direct current

Traditional powder metallurgy operates with powder sizes of more than 1 μm. However, it is known that, for example, to obtain structural ceramics with high hardness and strength, it is necessary to use the finest powder. This reduces the sintering temperature, allows to get a fine structure, which ultimately improves the physico-mechanical properties of the obtained products. Difficult and important task is to ensure a uniform distribution in the mixture of nanodispersed powders and to keep it in compacts of even uncomplicated shape. It is necessary to preserve the nanostructure in compacts to ensure formation of nanosized grains during sintering, i.e., to create conditions for inhibiting grain growth (to prevent

recrystallization) and for sintering high-quality nanoceramic products with specified desired functional properties. It is also important to ensure chemical purity and the required phase composition of the final product. Obviously, the extreme activity of small particles in powders and appearance of plasm in chemical synthesis enable to decrease the sintering temperature and time. Thus, more economical temperature conditions can led to the same desirable results.
In this context, several types of materials can be distinguished: nanoconductors, nanoporous structures, nanopolymers, numerous nanotubular objects, nanobiomaterials, catalysts, and supermolecular structures. Common feature of these materials is that the size of the main structural components (crystallites, phases, pores, particles, molecular ensembles) usually does not exceed 100 nm (0.1 μm). The main methods of manufacturing of consolidated nanomaterials are given in table 3.1.

Table 3.1.

The main methods of obtaining consolidated nanomaterials

Method of production	Main variants	An object
Powder technology	Glater's method (gas-phase deposition and compaction). Electric discharge sintering. Hot forming	Elements, alloys, connections
Intense plastic deformation	High static and dynamic pressures. Controlled heating rate sintering	Metals and Alloys
Crystallization from amorphous state	Equal angular pressing. High pressure torsion strain	Amorphous substances
Film technology	Pressure treatment of multilayer composites. Phase hardening. Normal and high pressure. Chemical precipitation. Physical precipitation. Electrodeposition Sol-gel technology	Elements, alloys, connections

The interconnections of various properties with the characteristic sizes of objects were reported in different publications. In particular, the following patterns have been established:

- with a decrease in the size of structural components, the role of the particles interface increases significantly;

- the properties of the interface in the nanometer range may be different from those for ordinary large-crystalline objects;

- decrease in the size of crystallites, particles, and so on, their size can be commensurate with the characteristic size of some physical phenomena (for example, the mean free path of carriers in transport phenomena).

In order to successfully solve the problems of fabrication the bulk structures from nanodispersed powders, it is necessary to have an appropriate technological level and instrumentation. In the study of structure of nanomaterials, transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM) are mainly used. A high resolution of electron microscopy methods is necessary to identify features such as the presence of very small particles, pores and grains of size less than 10 ÷ 20 nm. It is a good instrument for observation dislocations and establishing the structure of interfaces (grain boundaries and phases), as well as the nature of the destruction of nanostructures. The area important for research, is the particle size range from 100 to 1000 nm. In some cases, the size of the initial nanograins in the material does not always remain at the nanometer level due to grain growth during sintering.
In that case, the submicron size particles comprise only 20% of the total mass of the powders, which, of course, does not allow to draw conclusions about the properties of the powders of the submicron level, with grain size up to 1 μm. For instance, attritor grinding gives higher percentage of submicron powders, but the presence of a large number of particles with a size of more than 1 μm does not allow us to draw general conclusions regarding the consolidation of particles up to 1 μm in size. Nevertheless, this size range is little studied, especially when it comes to refractory materials.
Currently, the main global manufacturers of nanopowders of refractory compounds are Infarmat (USA), A.L.M.T. (Japan), Wolfram (Austria), and Spark (Germany).
At present, two types of thin refractory ceramics are used as structural materials: oxide and oxygen-free ones. Oxide ceramics serve well at high temperatures, they are ductile, sinter well, but they are not resistant to sudden heating and cooling.

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Oxygen-free ceramics are materials that are made on the basis of powders of oxygen-free refractory compounds: they are characterized by high heat resistance, resistance to corrosion and wear. To increase the crack resistance of ceramic materials, thin additives are introduced into their composition, alloyed with ceramics, reinforced with whiskers of the highest strength, or carbon fibers, etc.

It is obvious that the use of nanopowder technology allows scientific and technical development the following areas:

- structural materials with high mechanical properties;

- materials for direct energy conversion - semiconductors, emission and heat pipe materials, as well as La-Ba-Cu-O superconductivity materials;

- catalysts for the deep processing of oil and the production of high-quality gasoline and gas;

- electrical insulating, heat-shielding and anti-corrosion coatings;

- materials for information recording systems;

- materials for hydrogen energy;

- composite materials made up of plastics and polymers with fillers from ultrafine powders;

- biologically active environments for agriculture, biology and medicine;

- porous structures for fine filters, soldering, welding and other applications.