

## HIGH-PRESSURE: GENERATION AND MEASUREMENT

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### Summary

The principles of investigation of physical properties of solids under pressure and their applications are considered. The pressure-induced polymorphism, for example, the graphite-to-diamond transformation, is described, as well as the properties of conduction electrons in solids and the electron transitions under pressure.

### 1. Introduction

High-pressure physics (HPP) is concerned with investigations under high pressures to study physical properties of various bodies (mainly solids), thus, is characterized by a general investigation technique. This is a particular feature of HPP distinguishing it from other areas of physics, for example, magnetism (general nature of phenomena) or physics of semiconductors (general matter of enquiry).

In the present chapter, we describe methods for production and application of high static pressure. Usually, books and surveys on high-pressure techniques are dedicated to attainment of record-breaking pressures and broadening the temperature range. Not disputing importance of their objectives revealing new laws, the problem of modern studies consists in applying such pressures to search for qualitative changes in properties of solids. Such a statement of the problem allows us to distinguish high-pressure physics as an independent field of solid state physics.

We consider some problems of modern HPP, in particular, the main problem of pressure-induced matter transformation. The graphite transformation yields artificial diamonds while production of metal hydrogen is an example of the insulator-to-metal transformation.

As particular advances of HPP, we describe the graphite-to-diamond transformation and anomalies in electronic characteristics of metals. We also give examples of the most prominent polymorphic transitions and show the role of pressure in the production of metal hydrogen and a drastic increase of the number of superconductors, variation of their properties, including the superconducting transition temperature.

## 2. Methods for Studying Physical Properties of Solids under Pressure

The SI unit of pressure is Pascal:  $1 \text{ Pa} = 1 \text{ N/m}^2$ ; however, kilobars are commonly accepted in physics:  $1 \text{ kbar} = 0.1 \text{ GPa}$ . The currently attainable pressure range is subdivided into two parts: up to 20-30 kbar (2-3 GPa) and above 30 kbar. In the former range, a pressure uniform on all sample sides is maintained by liquid. This is the so-called hydrostatic pressure. In the latter range, the liquid state cannot exist; therefore, solids as plastic as possible are used as media transferring pressure to studied samples. It becomes impossible to produce hydrostatic pressure; therefore, this range extending to a few hundred kilobars is referred to as quasihydrostatic. Only a few of laboratories of the world operate in the megabar range ( $1 \text{ Mbar} = 10^3 \text{ kbar} = 100 \text{ GPa}$ ).

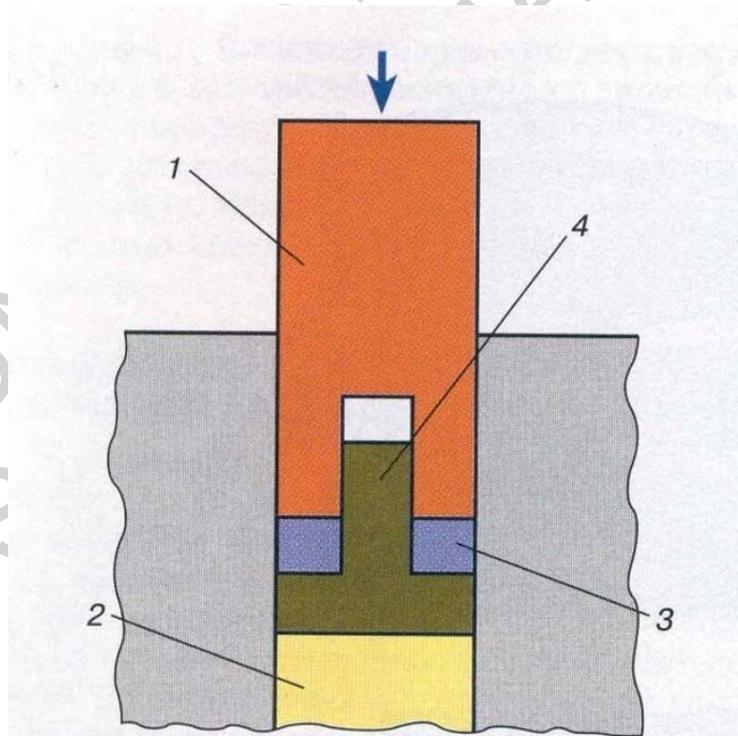


Figure 1. Cylinder—piston scheme with the Bridgman gasket: (1) piston, (2) medium in which pressure is built up, (3) gasket, and (4) mushroom, that is the bottom part of the piston

A basic method for producing the hydrostatic pressure is the use of a cylinder-piston system (see Fig.1). Piston 1 moved by an external force, for example, by a hydraulic press, decreases the medium 2 volume  $V$  and produces pressure in the cylindrical chamber. Since the volume  $V$  contains liquid, its leakage between the cylinder wall and the piston should be prevented. The liquid can be easily locked by an ingenious method proposed by the ancestor of modern HPP, American physicist P.W. Bridgman. The method essentially consists in the following. Solid gasket 3 (Fig.1) is subject inevitably to a higher pressure than the liquid due to a smaller support area than the piston diameter. Therefore, the piston is compound: its part 4 from the side of high pressure is mushroom-shaped. The mushroom leg length with a gasket is made shorter than the depth of a corresponding cylindrical hole in the piston basic part. Therefore, this method is referred to as the principle of uncompensated area.

The maximum pressure depends on the strength of chamber units, especially of the cylinder. Their strength can be increased by precompression, as well as by producing all the units (except for gaskets) from special steels and alloys with a high ultimate strength.

Such a chamber allows measurement of the volume compressibility—that is, a material volume change per unit pressure. To do that, it is necessary to know the piston displacement and pressure. The latter can be determined by the ratio between areas of the chamber and press pistons, knowing the pressure in the press cylinder, measured by an ordinary manometer. However, even special materials at pressures  $p > 1$  GPa are characterized by significant deformation, which causes friction between the cylinder walls and moving elements. An external force fraction is spent for overcoming the friction force; hence, a pressure measured by the ratio of areas is incorrect and manometers are used. The pressure gauge most commonly used in hydrostatics is a coil of Manganin (copper-manganese alloy) wire. The wire resistance depends on pressure – that is, proportional to its relative change.

To measure various physical parameters, some wires are introduced into the chamber; moreover, windows are made for inserting various radiations. The temperature in chambers of high hydrostatic pressure can be varied from  $+500^{\circ}\text{C}$  down to almost absolute zero. In the latter case, the hydrostatic pressure is achieved and fixed at room temperature and then is maintained on cooling.

There exist a few significantly different ways for producing pressure in the domain of quasi-hydrostatics. We briefly discuss three of them, received wide acceptance. One method (Hall H.T., 1960, USA) is based on a certain system similar to the piston—cylinder unit (see Fig.2b). It consists of two conic pistons 1 entering (on opposite sides) into ring (belt) 2 playing the role of cylinder. Plastic solid medium 3 for pressure transfer is placed into this cylinder together with the sample 4. To lock the medium between the belt and pistons, a special packing material is employed. All the basic parts of the chamber are at normal pressure in the state of strong elastic compression caused by exterior steel rings (supports). When the pistons are loaded, the rings, hence, belt 2 can expand almost up to a double limiting value and the maximum pressure grows strongly. The pistons (punches) are produced from hard alloys (for example, based on tungsten carbide) with a very high limit compression stress. The plastic medium should

be characterized by a high friction factor (for example, pyrophyllite mineral); then the medium supports the punch and ring, outflowing through the spacing between them, and gaskets the chamber.

As distinct from the belt (volume chamber), to produce quasihydrostatic pressures, Bridgman used earlier two truncated cones (anvils) from hard alloys with large angles at their vertices. Pressure arises in a thin medium layer between working cone faces. The medium and locking play the same role and have the same composition as the belt. A thin sample or film is placed between two medium plates.

The third chamber type has been developed by L.F. Vereshchagin in 1959. It is a volume chamber without belt, representing hard-alloy anvils with spherical hollows at working faces. The hollows are sufficiently deep to place at their center a studied sample surrounded by a solid medium for pressure transfer. The most commonly accepted material is silver chloride characterized by sufficient hydrostatic properties. The remaining hollow volume contains a locking medium (pyrophyllite) (Fig.2c). Such chambers produce pressures up to 10.0-20.0 GPa. An internal heater can be placed into volume quasihydrostatic chambers, therefore, these are employed for pressure-induced material transformations, for example, to synthesize artificial diamonds and other important materials (heating promotes the transformation). The force in such chambers is produced by hydraulic presses.

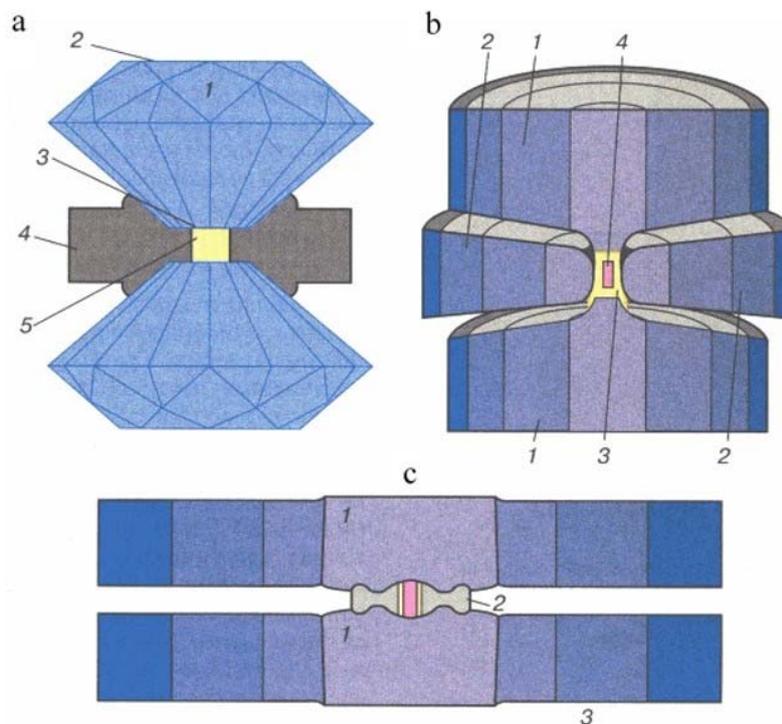


Figure 2. High-pressure quasihydrostatic chambers. (a) The diamond chamber: (1) diamond anvils with a plane area (culet), (2) anvil base, (3) calette, (4) metal gasket, and (5) space for sample; (b) the “belt” setup: (1) pistons (punches) with supports, (2) ring with supports, (3) medium in which pressure is produced, and (4) sample; (c) the Vereshchagin chamber with a toroid: (1) punches, (2) toroidal hollow with a sample in the center, and (3) supports

Chambers of all the three types are applied to measure resistance and magnetic susceptibility, as well as to input and output various radiations. Bridgman and Vereshchagin microchambers are cooled to  $T = 4.2$  K.

To measure the pressure in quasihydrostatic chambers, the following approach is most commonly accepted. Sample resistance jumps are indicated, caused by polymorphic transformations (see below). Metals are most frequently used as references (see Fig.3). Pressures at which transformations proceed are known in advance. These are determined by special methods. Simultaneously with fixing a transformation, a force applied to the chamber is measured by a pressure gage. Then the dependence of the force on the chamber pressure is constructed.

Pressures above 100 GPa are produced in diamond chambers developed by American physicist Mao (1976). He deserves the credit for the record pressures of 270-280 GPa.

The chamber shown in Fig.2a employs diamonds 1-2 of jeweler faceting with plane area 3. To avoid cleaving of diamonds, metal gasket 4 with cylindrical hole 5 is used. Such chambers can be used to measure any radiation and resistance in a wide temperature range. The effort is produced by levers or nuts. Figure 2a displays the main chamber part. The pressure is measured by a ruby luminescence spectrum.

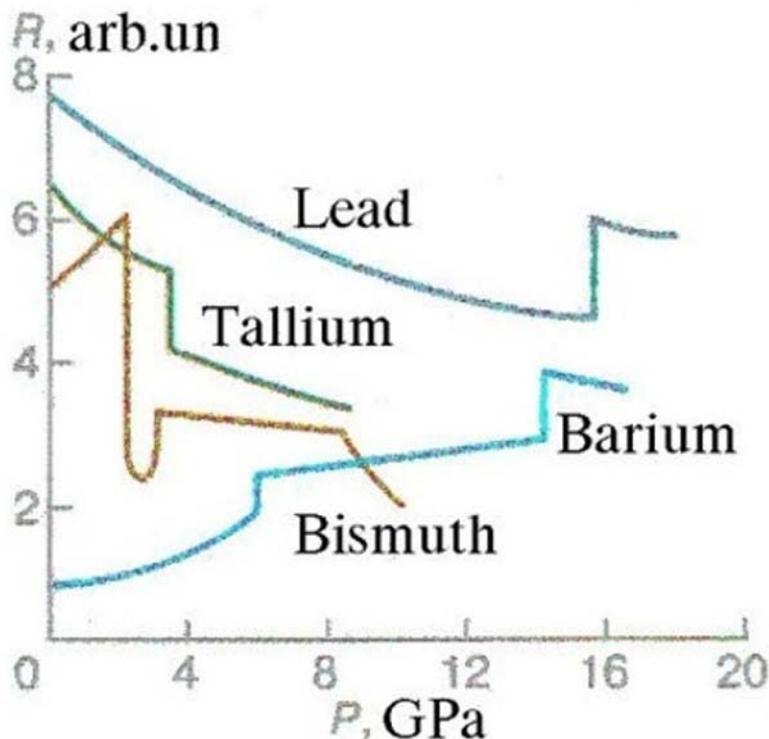


Figure 3. Dependence of the resistance of some metals on pressure. The pressures corresponding to the resistance jumps are used as reference points

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### Biographical Sketch

**Efim Solomonovich Itskevich**, is Honored Soros professor, doctor of physico-mathematical sciences, head scientist of the Institute of High-Pressure Physics, Russian Academy of Sciences (Troitsk, Moscow Region), and Honored scientist of the Russian Federation. He is a specialist in the field of low-temperature and high-pressure physics. He authored more than 200 articles and of the discovery of electron phase transitions of the 2.5 kind in metals. He graduated from the Physical department of the Lomonosov State University in 1951. His scientific interests: low-temperature and high-pressure physics.