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PRESSURE GENERATION IN THE REACTION CELL USING SIX-PUNCH HIGH-PRESSURE PRESSES MANUFACTURED IN CHINA: A REVIEW

The review is devoted to the analysis of existing experimental work on the study of pressure generation characteristics in the reaction volume of Chinese-made six-punch high-pressure presses. It is shown that one of the main problems in the operation of HP presses is a decrease in the efficiency of pressure transmission inside the cell at pressures above 5.5 GPa, which leads to a significant loss of pressure in the reaction volume. The above problem can be overcome by increasing the efficiency of pressure generation in the reaction volume of a cubic press through the creation of hybrid high-pressure cell designs consisting of different modular materials, optimization of the geometric parameters of the reaction volume, and improvement of the physical and mechanical properties of carbide punches.

Key words: *HP press, high pressure, pyrophyllite, deformable seal, high-pressure cell*

Introduction

The study of changes in material properties under extreme conditions, particularly under high pressures and temperatures, is an important field in modern science. However, experiments in this area require appropriate equipment. The work of Nobel laureate (1946) Percy Williams Bridgman (1882–1961) in developing equipment for ultrahigh-pressure research was a fundamental contribution to the advancement of high-pressure physics and laid the foundations for modern methods of generating and measuring extreme pressures [1, 2]. A significant event that spurred developments in the field of devices capable of conducting experiments under high pressure and high temperature (HPHT) conditions was the synthesis of artificial diamond. The first synthetic diamond crystals were obtained under HPHT conditions on February 15, 1953, by Eric Lundblad, an employee of the Swedish company ASEA (Fig. 1), using a specially designed press apparatus by Baltzar von Platen [3].

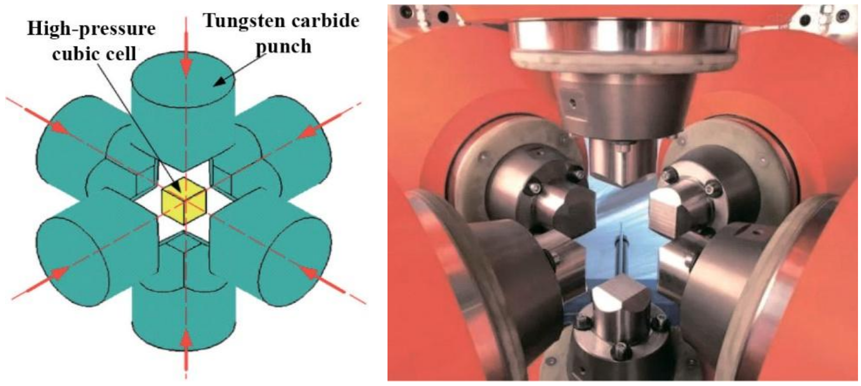
Fig. 1. Eric Lundblad at the Quintus press for diamond synthesis, 1953 (Quantis is the code name for ASEA's diamond synthesis technology development project, derived from the Latin word for «five», as diamonds have historically been considered the fifth element) [4]

In 1958, a paper by H. T. Hall was published, dedicated to the first multi-punch high-pressure apparatus – a tetrahedral device capable of achieving pressures of up to 10 GPa and temperatures up to 3000 °C simultaneously [5].

Since then, such multi-punch systems have been continuously improved and currently allow pressures close to 100 GPa to be achieved under high-temperature conditions [6].

According to the definition formulated by Tracy Hall and Eiji Ito, a multi-anvil apparatus is a high-pressure device with more than one loading axis and four or more panches compressing the sample [7]. This definition includes tetrahedral, cubic, and various octahedral devices, which are often interpreted as Kawai-type multi-punch apparatuses [8]. In terms of design, based on the classification provided in [9], multi-punch apparatuses can be divided into three groups: 1) all punches are driven by a single press; 2) each punch is driven only by a single press (single cylinder); 3) a hydrostatic drive for all punches.

Today, despite the complexity of its design, the six-punch press – a representative of high-precision hydraulic equipment designed to achieve high pressures in a reaction volume through the simultaneous application of uniform force from six sides – is widely used throughout the world (Fig. 2).



а *б*

Fig. 2. Schematic of a cubic press showing six opposing anvils and a cubic cell in the center (a), and an example of its practical implementation (b) [10]

Fig. 3. The first six-punch press in China, the «DS-023» model, designed for synthetic diamond production [11]

This type of press is used in various fields such as materials science, metallurgy, and tool manufacturing, and has, in particular, demonstrated high efficiency in the synthesis and sintering of superhard materials. In recent decades, the most popular has been the Chinese manufactured six-punch cubic press (China cubic press – CCP) with a plunger diameter of 560–950 mm.

The first six-punch press in China, the «DS-023» model, designed for synthetic diamond production and possessing fully independent Chinese intellectual property rights, was created at the Zhengzhou Sanmill (Three Abrasives & Grinding Institute) company in November 1965 (Fig. 3) [11].

Today, China is one of the world's leading manufacturers of six-punch presses intended for the synthesis and sintering of superhard materials. Among the leading research companies are Zhengzhou Abrasives Grinding Research Institute Co., Ltd. [12], while companies leading in China for manufacturing modern equipment for superhard material production include Henan Huanghe Whirlwind Co., Ltd. [13], Guilin Guiye Machinery Co., Ltd. [14], Zhengzhou Hanfa Prospecting Machinery Co., Ltd. [15], and others.

Modern six-punch presses from Chinese manufacturers provide working pressures of up to 6–8 GPa, are equipped with numerous automation systems, computer-controlled synthesis or sintering cycles, and feature improved energy efficiency. Developers continuously improve the layout, anvil materials, and heat and mass exchange systems to achieve optimal force geometry and pressure field uniformity required for high-quality diamond monocrystal growth [13]. It can be stated that Chinese press equipment today features a competitive balance between price, technology, and quality. This industry is rapidly developing and narrowing the technological gap with foreign counterparts, which traditionally remain the benchmark for high-tech and specialized applications.

However, it should be noted that on the path to narrowing the technological gap and enhancing competitiveness, Chinese developers of six-punch presses need to address a number of issues, as highlighted in [16]. Based on a comparative analysis of the designs and operational features of belt-type presses [17] and cubic presses during the HPHT synthesis and sintering of high-quality polycrystalline diamond (PCD) and large-diameter PCD and polycrystalline cubic boron nitride (PCBN), the following key problems need to be solved for the six-punch press [16]:

1. At pressures above 5.5 GPa, the efficiency of pressure transmission within the cell decreases, leading to a significant loss of pressure in the reaction volume.
2. The need to ensure precise creation and maintenance of the temperature field topology.
3. Ensuring thermal insulation and pressure maintenance during prolonged HPHT synthesis and sintering processes.
4. The problem of excessive deformation during the manufacturing of large-size PCD and PCBN plates.
5. With an increase in equipment power, issues arise regarding centering accuracy, punch deformation under high pressure, and excessive consumption of hard alloys.

Therefore, in developing high-performance technologies for manufacturing high-quality PCD and PCBN composites, including large-diameter plates, the development of control and management over pressure and temperature distribution in the HPHT reaction cell is an extremely relevant task. In particular, experimental studies of pressure distribution in a pyrophyllite cubic cell [18] have shown that at a pressure in the center of the cube of 5.5 GPa, its gradient along the cube's symmetry axis averages 0.05 GPa/mm. This means that a complex stress-strain state arises in the cell during loading, and the pressure distribution is not uniform. Consequently, a key to significantly improving PCD sintering efficiency is also studying the pressure generation process in HPHT apparatuses.

Analyzing the accumulated experimental and theoretical knowledge regarding the characteristics of pressure generation in the reaction volume, when using Chinese-made high-pressure six-punch presses, constitutes the aim of this review.

Methods for Determining Pressure in the HPHT Reaction Volume

As mentioned above, pressure and temperature are the most important technological parameters that determine the sintering process of superhard materials under HPHT conditions and all stages of the formation of their structure and properties.

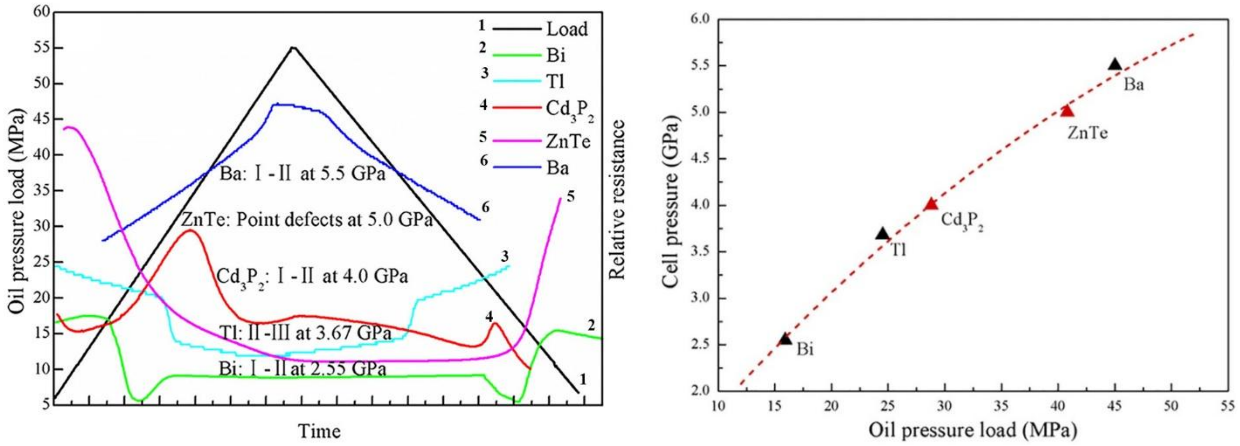
Typically, in mass production, the repeatability of pressure and temperature is maintained by accounting for and precisely controlling various technological parameters as completely as possible. Knowledge of the absolute values of pressure and temperature in each sintering cycle is not required. It is believed that meeting the above requirements ensures identical barothermal conditions, established during technology development, in each sintering cycle and, accordingly, the issuing of uniformly high-quality products. Since there are many types of apparatuses with solid media of varying configurations, which affect the degree of approximation to hydrostatic conditions, it is also considered that multi-punch apparatuses with multi-directional force application will create less shear deformation than uniaxial apparatuses, i.e., they will better to hydrostatic conditions.

In fundamental research on sintering processes under HPHT conditions, transferring superhard material (SHM) production technologies from one type of high-pressure apparatus to another, and in developing new SHM sintering technologies, knowledge of absolute pressure and temperature is important for determining phase equilibrium in the reaction volume, understanding the mechanism of SHM structure and property formation, and establishing the defining factors regulating these processes.

The pressure inside various types of HPHT apparatuses, used for SHM synthesis and sintering, cannot be calculated as it can be in simple piston-cylinder devices by measuring the compressive force and punch diameter. The relationship between these forces depends on the configuration of the matrix reaction cavity, the condition of the working surfaces, the cell material, the thickness of the sealing layers, etc., and is not amenable to precise calculation.

In most cases, pressure in HPHT apparatuses is not measured directly but by methods based on determining some physical phenomenon: a sharp change in specific volume, electrical resistance, etc. The main condition for measuring the dependence of a specific physical phenomenon on pressure is that the parameter to be measured must exhibit significant changes during the transition, so it can be reliably detected using a standard measurement procedure [19].

Typically, HPHT apparatus pressure calibration is performed according to a well-known methodology, most widely used in high-pressure technology, which is based on estimating the pressure in the reaction volume by recording known phase transitions (reference points) accompanied by abrupt changes in the electrical resistance of certain metals or compounds [2, 19, 20]. This technique is widely used for determining pressure in a high-pressure cell at room temperature. Fig. 4 shows the result of using this methodology for high-pressure calibration in a DS6×14 MN cubic press developed at Sichuan University, China [21].



a *b*

Fig 4. High pressure calibration example in a DS6×14 MH cubic press: a – the variation trend image of the relative resistance of Bi, Tl, Cd_3P_2, ZnTe, and Ba under high pressure; b – the fitting pressure curve (Bi, Tl, Cd_3P_2, ZnTe, and Ba) for pressure calibration [21]

It should be noted that the pressure determined at room temperature will differ from the actual pressures realized in the HPHT apparatus during synthesis or sintering. For instance, due to the thermal expansion of the HPHT apparatus components, an increase in pressure will occur; phase and structural changes in the materials within the high-pressure cell reaction volume, accompanied by volumetric effects, can lead to either an increase or decrease in pressure; and the outflow of cell material during the formation of the deformed seal also leads to a pressure decrease in the reaction volume. Analysis of a large volume of accumulated experimental data and estimation calculations shows that the resulting pressure value is most often higher than that obtained at room temperature. For example, H.M. Strong and F.P. Bundy [22] concluded that in high-pressure experiments using

NaCl as a pressure-transmitting medium at high temperature, the pressure increases by approximately 20%, while when using pyrophyllite cells, the pressure in them decreases quite rapidly at high temperatures and ultimately falls below the pressure calibrated at room temperature. Fig. 5 shows an example of the pressure-temperature dependence in the reaction volume of a DS6×14 MN cubic press [23].

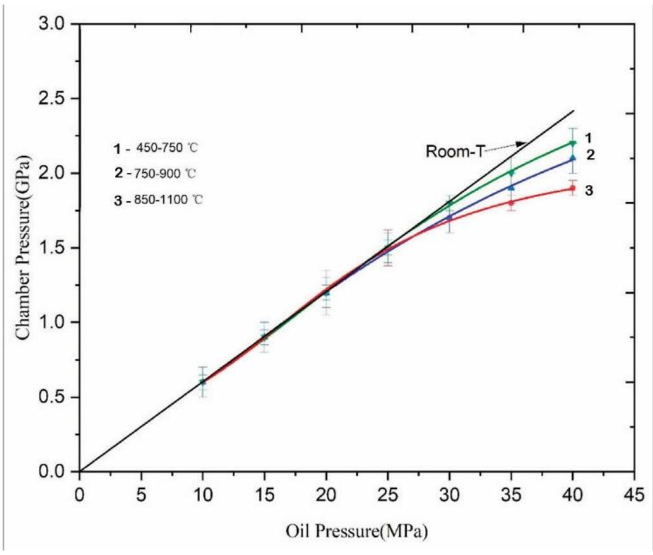


Fig. 5. Pressure calibration for the first-stage punch of the DS 6×14 MN 6–8 large volume press assembly [23]

One solution to the problem of simultaneous temperature and pressure estimation during SHM manufacturing is the methodology proposed by O. Fukunaga and colleagues [24]. This methodology is based on calibrating the high-pressure cell for temperature and pressure using results obtained from diamond synthesis using carbon solvent metals. The authors propose using the barothermal values based on the minimum p-T points of diamond formation in Ni, Co, and invar solvent systems as reference values for pressure and temperature. It was found that the calculated pressure and temperature agree well with independent experimental results.

Using the methodology described above, work [25] performed pressure and temperature calibration for the ZR800 6×50 MN cubic press [12] (Fig. 6, 7).

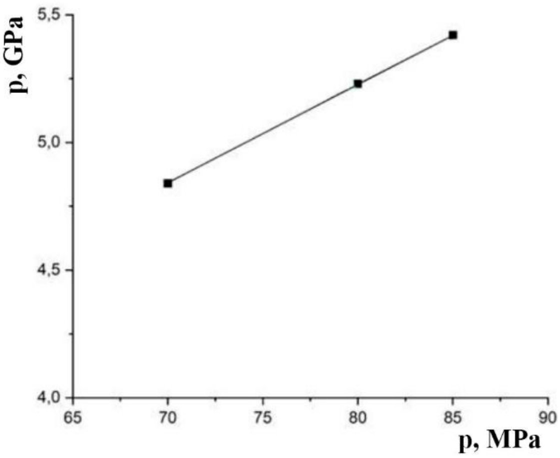


Fig. 6. Calibration plot of pressure in the HPHT apparatus during PCD sintering versus pressure in the press hydraulic system [25]

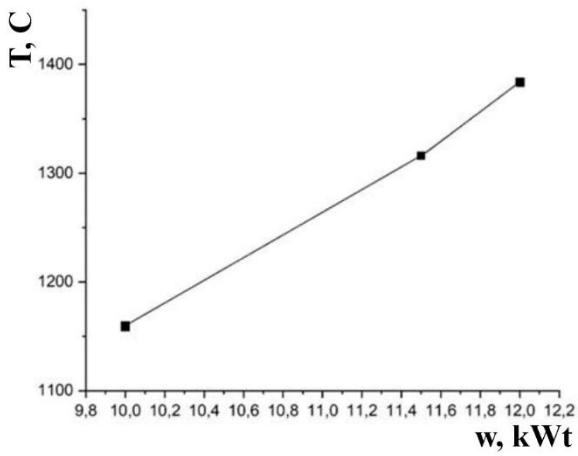


Fig. 7. Calibration plot of temperature in the HPHT apparatus during PCD sintering versus applied electrical power [25]

Work [26] reports on the development of a differential method for measuring high quasihydrostatic pressures in six-punch press installations by constructing a load characteristic $p = f(Q)$, where p is the pressure value in the quasihydrostatic high-pressure cell, and Q is the press force. The pressure in the cell is determined using measurements of the difference in the polymorphic transition temperatures in Co(α→β) and Fe(α→γ), and the melting of Ag and Cu. The proposed methodology allows pressure determination in the reaction cell at temperatures up to 1400 °C.

Research on Pressure Generation Efficiency in the Reaction Volume of a Cubic Press

As noted earlier, during press operation, a complex stress-strain state arises in the cell, and the pressure distribution is non-uniform. It should also be noted that the level of generated pressure in the HPHT apparatus is determined by both the dimensional parameters of the punches and cubic cell, and the physical-mechanical properties of the punch material. For instance, work [27] performed computer finite element simulation of the compression of a cubic pyrophyllite cell with an edge length from 56 to 60 mm. It was established that increasing the cell edge length at a stable axial displacement of the punches leads to a decrease in the pressure at its center from 5.89 to 5.07 GPa, and the strength assessment of the HPHT punches revealed three possible regions of their failure under load, namely: at constant pressure in the cell center, increasing its edge length leads to a noticeable increase in the level of equivalent stresses on the edges of the working side surfaces of the punches.

Efficient pressure generation in the reaction volume and its maintenance throughout the synthesis or sintering cycle depend significantly on the pressure ratio inside the high-pressure cell and within the deformed seal.

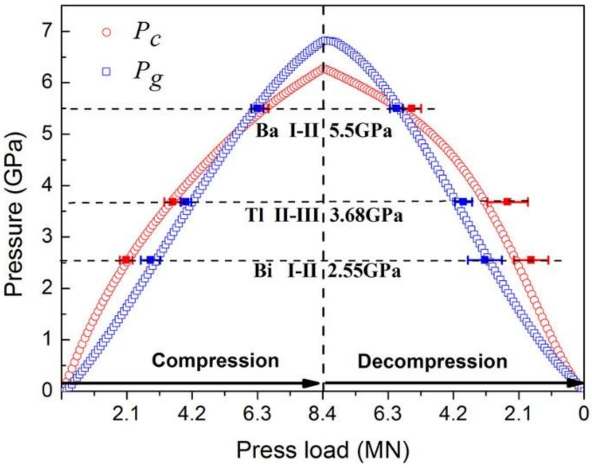


Fig. 8. «Load-pressure» dependencies obtained during calibration with manganin in the compression and unloading process, and calibration results obtained using in situ measurements of electrical resistance of Bi, Tl, and Ba under high pressure [29]

In work [28], two approaches are proposed for evaluating the possibility of pressure generation in the working volume of the Chinese-made ZR 800 six-punch press. The first method determines pressure as the ratio of the force acting normally to the area of the cube and the deformed seal; the second method involves simulating the compression of a pyrophyllite cube considering the dependence of the volumetric compression coefficient on pressure. Calculations showed that at the maximum press force (330 MN) at room temperature, the pressure in the working volume can reach 4.69 GPa.

In [29], an original methodology was proposed for determining the pressure inside the high-pressure cell (P_c) and in the deformed seal (P_g) during press loading-unloading using in situ measurements of the electrical resistance of bismuth, thallium, barium, and manganin. The obtained dependencies are shown in Fig. 8.

All pressure calibration experiments in the mentioned work were conducted at the same press loading speed (2.31 kN/s) in a 6×14 MN cubic press at room temperature.

The obtained experimental data indicate that the pressure in the reaction volume (P_c) exceeds the pressure in the deformed seal (P_g) when increasing the press load until a pressure of approximately 5 GPa is reached. The forces arising from the pressure difference ($\Delta P = P_c - P_g$) are balanced by reaction forces, which are closely related to the following two factors: first, the maximum static friction force between the deformed seal and the punch surface, and second, the internal friction of the pyrophyllite deformed seal itself.

At a pressure of about 5 GPa, P_g begins to exceed P_c, meaning a significant portion of the press load is expended on compressing the region around the deformed seal, resulting in the limitation of the P_c value generated in the large-volume press by the rapid growth of P_g above ~5 GPa. Therefore, to obtain higher pressure in the high-pressure cell reaction volume, the working pressure in the press hydraulic system has to be increased, but this leads to a higher risk of punch failure.

Furthermore, as emphasized in [29], given the constancy of the maximum static friction force and the shear strength of the compressed pyrophyllite seal, a larger ΔP value is more likely to cause a loss of seal in the high-pressure cell (the so-called spontaneous «depressurization» of the reaction volume). Fig. 9 shows the dependence of ΔP on P_c during press loading and unloading, obtained in [29].

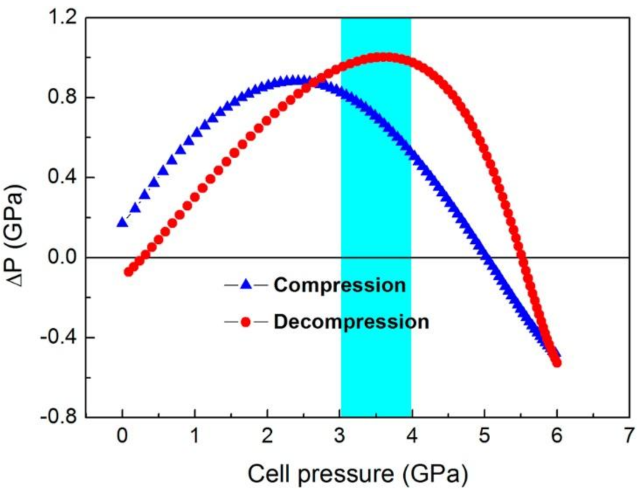


Fig. 9. Dependence of ΔP on P_c during press loading increase and decrease [29]

The maximum ΔP value during press loading increase is less than during its unloading. ΔP reaches a maximum of approximately 1 GPa when P_c decreases to ~3.5 GPa. Observation statistics by the authors of [29] indicates that at ΔP > 0.9 GPa, there is a high probability of seal failure in the high-pressure cell.

Based on an analysis of the processes occurring during six-punch press loading, [30] identifies three main stages of its operation.

First Stage. During press loading, the six punchs compress the pressure-transmitting medium (pyrophyllite cell), and part of the medium is distributed between the punches, forming a sealing zone (deformed seal). At this stage, both the pressure-transmitting medium and the sealing zone area undergo mainly plastic deformation. The pressure in the cavity at the end of this process can reach approximately 2 GPa.

Second Stage. Upon further press loading, the deformed seal is compressed and, accompanied by minor flow, thins out. Simultaneously, the volume of the high-pressure cell decreases. At this stage, only a small amount of the pressure-transmitting medium (pyrophyllite) flows into the inter-e space, replenishing the volume of the deformed seal. The area of the pressure-transmitting medium in the cavity undergoes mainly elastic deformation, while both elastic and plastic deformation are present in the sealing zone area. The pressure in the cell at the end of this process can reach approximately 5 GPa.

Third Stage. After the completion of the second stage, the material flow of the deformed seal terminates, and both the pressure-transmitting medium and the deformed seal deform predominantly elastically. Since the deformed seal is already very thin and practically does not flow, further increases in external load practically do not affect punch movement to increase pressure in the cell area. The pressure in it at the end of this stage can reach a maximum of about 6 GPa.

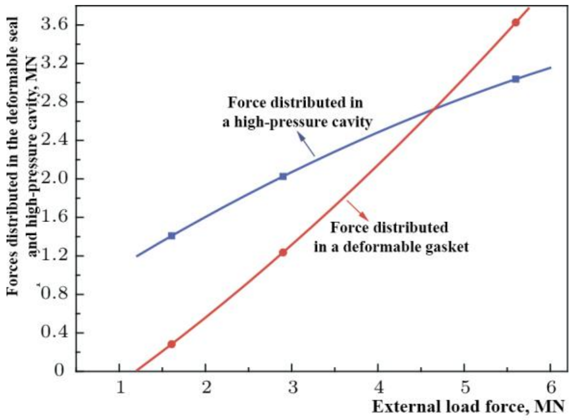


Fig. 10. Dependence of the forces borne by the cubic cell and the sealing zones on the external load [30]

Fig. 10 shows the dependence of the forces borne by the cubic cell and the sealing zones on the external load.

Analysis of experimental data shows that with increasing external load, its proportion expended on the sealing zones of the cubic cell abruptly increases (the slope of the curve increases), while the proportion expended on the cell zone itself increases slowly (the slope of the curve decreases) [30]. After the pressure in the cell reaches 6 GPa, most of the additional external load is expended on the deformed seal areas, leading to a slow increase in pressure within the cell itself. Further increases in external load can reach the strength limit of the hard alloy, leading to punch failure.

The experiments published in [29, 30] explain the reason why the pressure in the cubic cell of a six-punch press barely exceeds 6 GPa.

Pathways for Improving Pressure Generation Efficiency in the Reaction Volume of a Cubic Press

Today, in mechanical engineering and industries related to mineral extraction, there is a great demand for superhard material tools with enhanced strength, crack resistance, and wear resistance. An important component of technologies, enabling the production of materials with such operational properties, is the availability of equipment that ensures the sintering of SHMs at pressures above 7 GPa.

At the same time, from the above analysis of literature sources, it follows that one of the main problems in the effective operation of six-punch presses is generating pressures above 6 GPa in the reaction volume. Do pathways exist to overcome this limit?

In [30], based on studying the distribution, magnitude, and direction of forces acting on the cell during press loading, and quantitative pressure measurement within it, the following possible pathways for increasing pressure in the reaction volume were proposed. First, to increase the pressure increment in the cell, it is necessary to use materials with a relatively high bulk modulus as the pressure-transmitting medium. Second, materials with a relatively low bulk modulus can be simultaneously used for the sealing zone to reduce the pressure increment within it. Third, since punch hardness and the geometric dimensions of the high-pressure cell are also key factors affecting the pressure in the reaction volume, the pressure can be increased by selecting punches with enhanced physical-mechanical properties and designing a more optimal reaction volume configuration.

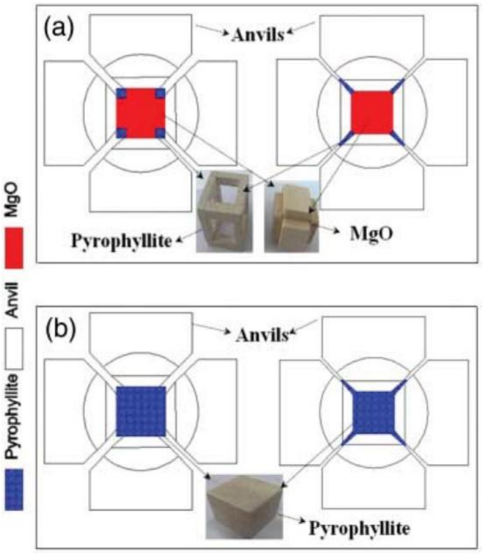


Fig. 11. High-pressure cells: a – hybrid cell of pyrophyllite and magnesium oxide at the beginning (left) and end (right) of press loading; b – traditional pyrophyllite cell at the beginning (left) and end (right) of press loading [32]

[31, 32] present the results of research on the effectiveness of using a combined assembly of a cell made from pyrophyllite and magnesium oxide (Fig. 11, 12), as well as a system for pre-strengthening punches, which allowed increasing the maximum pressure in the reaction volume from 6 to ~9 GPa.

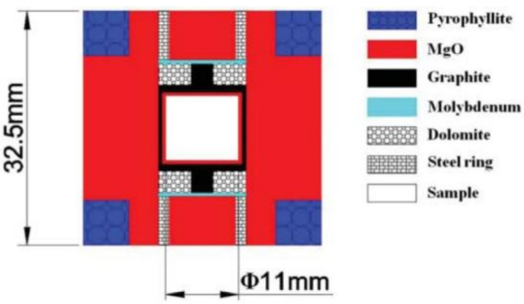


Fig. 12. Example of assembly of a hybrid cell from pyrophyllite and magnesium oxide [32]

The hybrid high-pressure cell made from pyrophyllite and magnesium oxide, which can be used in large-volume cubic presses, consists of a cubic frame (made of pyrophyllite with square holes located on the cube faces) from which the deformed seal is formed, and heteromorphic magnesium oxide, which functions as the pressure-transmitting medium. The results of experiments [32] showed that the pressure generation efficiency using the hybrid cell was increased by approximately 40% (compared to the traditional pyrophyllite cell) without reducing punch size or losing sample volume.

The experimental results presented in [31] showed that pressure gradients in the pressure-transmitting media (both pyrophyllite and MgO) increase with rising pressure in the cell. The pressure gradient in the pyrophyllite medium was approximately 50 MPa/mm when the cell pressure reached 5.5 GPa, while in the MgO medium, when the cell pressure reached 7.7 GPa, the pressure gradient was approximately 140 MPa/mm.

The cell developers emphasize that samples with a volume of 1000 mm³ can be compressed in it to ~8 GPa, which significantly exceeds the value achievable using a traditional pyrophyllite high-pressure cell. It is also noted that such a high-pressure cell design is not only suitable for cubic apparatuses but also serves as a reference for other types of multi-punch presses. The hybrid high-pressure cell of pyrophyllite and magnesium oxide is also applicable in two-stage high-pressure apparatuses.

Conclusions

1. The six-punch press (CCP), despite its complex design, is widely used throughout the world and is employed in various fields such as materials science, metallurgy, tool manufacturing, and the synthesis and sintering of superhard materials.

2. One of the main operational problems of CCP presses is the decrease in pressure transmission efficiency within the cell at pressures above 5.5 GPa, leading to a significant loss of pressure in the reaction volume.

3. Overcoming the abovementioned problem is possible by enhancing pressure generation efficiency in the reaction volume of the cubic press through the creation of hybrid high-pressure cell designs consisting of materials with different moduli, optimization of the reaction volume geometric parameters, and improvement of the physical-mechanical properties of the hard alloy punches.

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ГЕНЕРАЦІЯ ТИСКУ В РЕАКЦІЙНОМУ ОБ'ЄМІ ПРИ ВИКОРИСТАННІ ШЕСТИПУАНСОННИХ ПРЕСІВ ВИСОКОГО ТИСКУ КИТАЙСЬКОГО ВИРОБНИЦТВА. ОГЛЯД

Огляд присвячений аналізу наявного експериментального доробку по дослідженню особливостей генерації тиску в реакційному об'ємі шестипуансонних пресів високого тиску китайського виробництва. Показано, що однією з головних проблем експлуатації CCP-пресів є зниження ефективності передачі тиску всередині комірки при тиску вище 5,5 ГПа, що веде до серйозної втрати тиску в реакційному об'ємі. Подолати означену вище проблему можливо за рахунок підвищення ефективності генерації тиску в реакційному об'ємі кубічного преса шляхом створення гібридних конструкцій комірки високого тиску, що складається з різномодульних матеріалів, оптимізації геометричних параметрів реакційного об'єму, покращення фізико-механічних властивостей твердосплавних пуансонів.

***Ключові слова**: CCP-прес, високий тиск, пірофіліт, деформуюче ущільнення, комірка високого тиску*

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