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DEVICE FOR INVESTIGATING THE PHYSICAL PROPERTIES OF SOLIDS AT HIGH PRESSURE (0-2500) MPA AND LOW TEMPERATURE (90-300) K

This pioneering study discusses the computations of the structure of the assembled piston-cylinder type chamber for plotting the ice diagram in P-T coordinate at high pressure (0-2500) MPa and low temperature (90-300) K and measuring the temperature, pressure of the sample, cracks in the chamber at a temperature of 100 K, a pressure of 3000 MPa, broken pictures of the piston are shown.

For the first time the dependence of the thermal, baric and thermal compressibility coefficients of pressure and temperature of the ice-twelve modification on the phase diagram obtained in practice was studied. We constructed the graphs of the dependence of the thermal coefficient $\frac{\partial K}{\partial T}$ of ice-twelve modification on pressure, the dependence of the partial derivatives $\frac{\partial K}{\partial p}$ of the ice-twelve modification on temperature, the dependence of the coefficients of thermal expansion of the ice-twelve modification on temperature. We also developed results that between the studied temperature and pressure, the values of $\frac{\partial K}{\partial T}$, $\frac{\partial K}{\partial p}$ do not change much as a function of temperature at constant pressure and pressure at constant temperature, the value of $\beta = \frac{1}{V}\left(\frac{\partial V}{\partial T}\right)_p$ decreases with increasing pressure of the ice modification under consideration at the same temperatures.

Keywords: high pressure, low temperature, heat treatment, armor, sensor, thermocouple, coefficients of thermal, baric compression.

Introduction

For the first time, Bridgman [1] showed that high-pressure steel tanks can withstand pressures up to 5000 MPa.

However, these vessels were not widely used in practice.

After all, armor-piercing chambers do not withstand pressures of 2000 MPa. Therefore, to increase the pressure inside the cylindrical chamber to 3000 MPa, multi-layer concentrated cylinders are tensioned and tightened together.

In recent years tungsten carbide or solid alloy core cylinders with a pressure of up to 5,000 MPa have been used.

The trapezoidal ring, which is tightened on the cylinder to which the sample is to be examined, creates a large mechanical stress on the walls inside the chamber. When there is no pressure in the chamber, the multi-layer chamber perceives inside the chamber as compressed compared to the outer one. The diameter of the inner layers begins to increase when the pressure generated by squeezing the sample in the cell inside the chamber is compared to the pressure that occurs when the outer layer is tensioned.

This method, proposed by Bridgman [2], is currently used by many researchers. The pistons are made of very strong armor or metallo-ceramic alloys as they are subjected to considerable compression during operation. Devices operating at very high pressures are of the following types [3]: piston-cylinder assembly, Bridging seams, the device is equipped with an external suspension, «Belt» type device, multiprocessor type cubic multiplier.

The special structures of these devices allow for low-temperature research [4].

Main part

The methods for producing high-pressure and low-temperature are divided into four groups. In the first case, the sample is cooled to room temperature [5] after being compressed to a certain pressure in the high-pressure chamber at room temperature. At low temperatures obtained with such devices, we can only experiment with one pressure. Therefore, the study of the dependence of any parameter on pressure can be time-consuming. In the second instance by determining the dependence of its elastic properties on the pressure by increasing or decreasing the pressure at a certain temperature of the sample. There are several ways to measure [6] pressure in these devices. Such systems are considered massive and ineffective due to the heat generated by the friction forces arising between the piston and the cylinder walls when changing the pressure at very low temperatures. In the third case, when Kan and Lazarev [7] cooled down some of the substances (water, bismuth, antimony, gallium) they used a method to increase their volume under pressure. The authors of a scientific article have shown that it is possible to increase the pressure to 200 MPa for liquid helium and raise it to 1000 MPa. In the fourth group of devices, the pressure is obtained through an externally controlled gas source from the cell chamber inside the crystal.

The stainless-steel cell [4] produces a pressure of 1000 MPa resulting from the diamond nickel cell. The pressure in the sample under study in the high-pressure chambers presented in the article is uneven. Only under certain additional conditions can the pressure in the ice, located in the cylinder-piston-type chamber, approach homogeneity.

At high pressure, parametric sensors depending on the displacement of the elements of electrical resistance [9], inductance [10] or displacement of the elements of electrical capacitance [11] are used to detect changes in the volume of the studied samples.

Resistive, inductive and capacitive sensors [12] cannot measure large displacement with high accuracy. Therefore, the author [15] used a measuring range sensor (0-10 mm) that he invented.

The piston is a cylindrical chamber capable of withstanding a low pressure of 3000 MPa.

The article [13] summarized the design of a high-pressure piston - cylinder type chamber to study the phase diagram of twice-distilled water at pressures up to 3000 MPa and temperatures (90-300 K). The hardware is shown in Figure 1. All parts of the chamber are made of instrumental armor. We made it of three concentric cylindrical layers, which were heated and pressed together to increase the strength of the high-pressure tank.

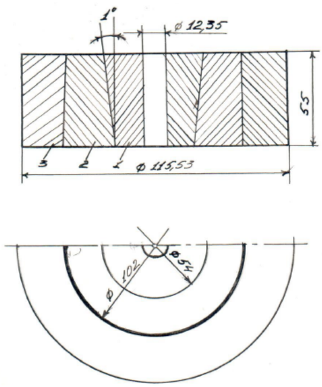


Figure 1 – The design of a multi-layer cylindrical high-pressure chamber

The inner (1) layer of the chamber is made of heat-treated (HRC = 58 ÷ 60) X12M; the second (2) P18 (HRC = 60 ÷ 62), and the third (3) X12M (HRC = 48 ÷ 50). The optimal dimensions of the high-pressure chamber and the maximum pressure inside [8] were calculated by the following formula:

$$P_0 = \left\{ \left[\sigma_{\beta_1}\right] + \sigma_S (N-1) \left[\left(1 - \frac{m_1}{m_0}\right)^{\frac{2}{N-2}} \right] \frac{m_1^2}{m_1^2 - 1} \right\} \frac{m_1^2 - 1}{m_1^2 (1 + \nu_{\beta 2})}$$

where σ_{β_1}, σ_{β_2} – the limit of the tensile and compressive strength of the material; σ_S – the limit of fluidity of the material when stretched and compressed; N – the number of layers in the chamber; $m_0 = \frac{z_1}{z_0}$ – the size of the high-pressure annular chamber; z_0, z_1 – internal and external radii of the high-pressure chamber; $m_0 = \frac{z_N}{z_0}$ – relative dimensions of the chamber; z_N – size of the n-th floor; $\nu_{\beta_1} = \frac{\sigma_{\beta_1}}{\sigma_{-\beta_1}}$ $\nu_{\beta_2} = \frac{\sigma_{\beta_2}}{\sigma_{-\beta_2}}$ – known values for materials [14].

The height of the chamber, which can withstand a pressure of 3000 MPa, made by us, is 55 mm, outer diameter 115,53 mm. The diameter of the inner cylinder, i.e. cell, which is a sample, is 12,35 mm. Fixed pistons, which compress the model in the cell (1) located in its lower part (2) made of steel P18 (HRC=60÷62) (Figure No. 2). The hole obtained in diameter between the pistons and the walls of the inner cylinder is 0.05 mm, the height of the upper piston is 50 mm, and the lower one is 20 mm. Lead (3), copper (4) and P18 protective rings (HRC=58÷60) were used for crimping the pistons in the high-pressure chamber.

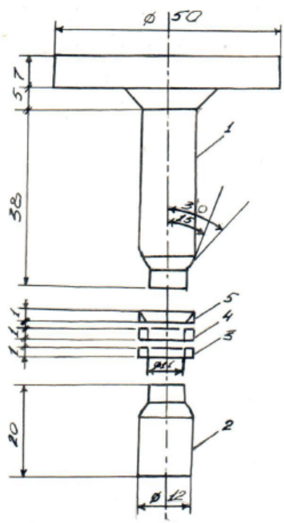


Figure 2 – Dimensions of sealing, protective rings
with high and low pistons of high pressure chamber

The angle between the piston axis and the cone components is 30° and 15°, since the pressure on the moving piston is 2500 MPa, the diameter of the upper part increases and creates large obstacles to movement in the cell, the diameter of the top is 12.15 mm, and the bottom is 12.35 mm.

The device of an automated installation for cooling the high-pressure chamber with nitrogen vapor, measuring temperature and pressure is given in the article [15].

To insulate and seal the conductors introduced into the chamber, we used cotton impregnated with an epoxy resin other than the Amata method [16]. The conductor, insulated with silk, and with a diameter of 0.10 mm, is pre-Packed in cotton with an epoxy resin impregnation and pressed into the chamber through the conical hole of the lower piston. The angle of the conical ceiling is cut-out 12°, the diameter of the foot is 1.5 mm and 1 mm. The advantage of the method used from others is the simplicity and ease of extracting them from the conical gaps after practicing. It also allows multiple measurements at low temperatures up to 3000 MPa. To bring the pressure in the sample closer to the hydrostatic pressure, polycrystalline 1h ice from twice-distilled water was crystallized and compressed in a cell made of fluoroplastic. The volume of ice at the initial atmospheric pressure was compressed to a pressure of 3000 MPa, and the equilibrium volumes were observed when the pressure returned to atmospheric pressure.

Figure 3 shows the changes in the relative volumes of fluoroplastic isobaric (2150 MPa) cooling and heating and isothermal and isothermal (90 K, 173 K, 253 K) processes, increasing and decreasing the pressure to 2300 MPa. We can see from the diagram that there are no phase transitions at the studied temperatures and pressures.

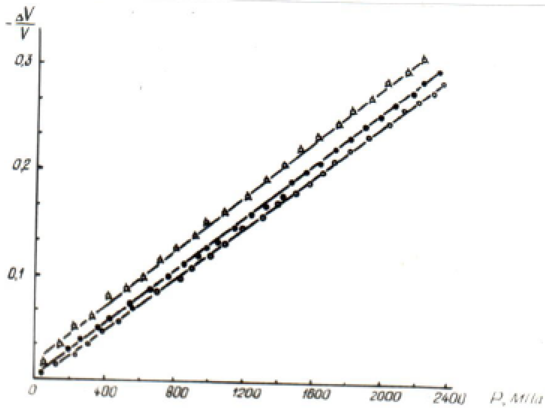


Figure 3 – Isotherms of fluoroplastics at temperatures of 250K, ● 113K, ◦ 90K

The dependence of the pressure formed in the sample on the press pressure up to 137A was determined at temperatures of 250 K and 300 K through the transition points 1h→III. VI→VII to ice types [17]. In addition, the calibration was performed by measuring the electrical resistances of the tin II→I and Galia I→II passages located inside the model [18]. The dependence of tin and halium on pressure in isothermal processes and the phase transitions obtained in our practice are indicated in Figure No. 4.

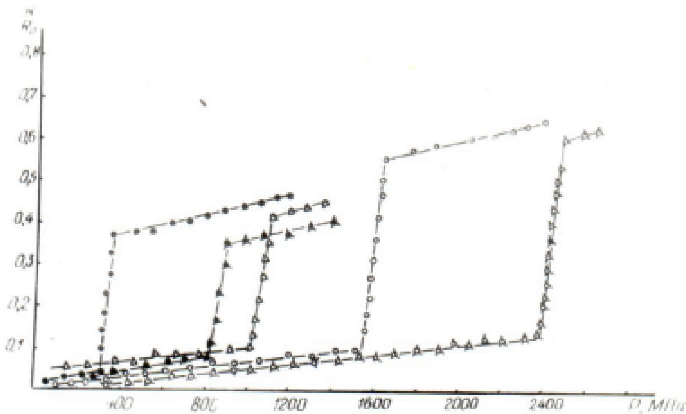
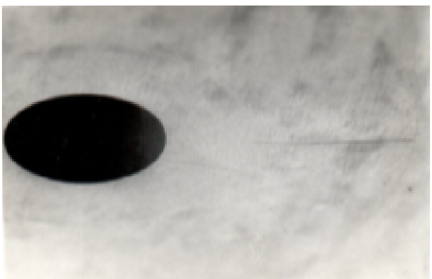


Figure 4 – Sn, Ga Pressure dependence of electrical resistance at low temperatures.

● Sn – 250 K, ▲ Sn – 173 K, Δ Sn – 90 K, ◦ Ga – 250 K, ▲ Ga – 173 K

The breakdowns of the high-pressure chamber and the moving piston at a temperature of 100 K and a pressure of 3000 MPa are shown in Pictures 1, 2.



Picture 1 – Explosion of a high-pressure chamber at a temperature of 100 K and a pressure of 3000 MPa

Picture 2 – Destruction of a moving upper piston at a temperature of 100 K and a pressure of 3000 MPa

The temperature inside the high-pressure chamber was measured to an accuracy of ± 1 °C using a copper-constant thermocouple with a calibration of 0.10 mm diameter located in the test sample [19, 20, 21]. The correction to needs to be made in a copper-constant thermocouple at a pressure of 40 kbar and a temperature of 100 °C ± 2 °C. Moreover, when the temperature drops, the correction decreases. Therefore, in our experiment, no correction was made for the copper constant thermocouple. Cooling of the chamber was carried out by passing nitrogen vapors through a spiral jacket placed on the outside. The flow rate of nitrogen vapor was monitored by means of a heater located inside a 40-liter dew. The copper tube that delivers the nitrogen vapor to the chamber jacket is wrapped with nichrome wire to change the temperature of the sample and connected to a thermocouple inside the test ice via a specially assembled electronic temperature controller.

Pressure dependence of the products of the compression modules of the XII ice modification and the coefficients of thermal expansion

In the phase diagram of ice in the P-T coordinate [13], the equilibrium state of the XII ice modification is located at pressures above 1200 MPa and below 130 K.

The author of the article [22] showed that the structures of all ice modifications that occur at low temperatures and high pressures are preserved and do not change at low temperatures when the pressure is released back into the atmosphere. There is also speculation that it is semiconductor. Therefore, the study of the physical properties of ice modifications is of great theoretical and practical importance. Determination of compressive and thermal coefficients of solids – provides information about its energy state.

Experimental measurement of known laws and formulas in physics [23] and the dependence of the change in the volume of the sample under study on temperature and pressure allowed us to estimate the coefficients of thermal volumetric expansion in ice-twelve modification.

Table 1 shows the pressure and temperature dependences of the independent derivatives of the compression modules of the ice-twelve modification on temperature and pressure (Figures 5, 6, 7). Figures 6, 7 show the graphs of the pressure and the temperature dependence of the coefficients of thermal expansion of ice-twelve modification, calculated on the pressure and temperature curves of the sample volume.

$-\frac{\partial K}{\partial T} \times 10^{-3}\,\frac{MPa}{K}$ *P, MPa*

Table 1

Phase	T, K	P, MPa	$-\frac{\partial K}{\partial T}$	Phase	P, MPa	T, K	$-\frac{\partial K}{\partial p}$
XII	100	1600	0,493	XII	1900	103	0,075
		1700	0,504			98	0,073
		1800	0,511			93	0,070
		1900	0,513			90	0,066
		2000	0,516				
		2100	0,518				

Table 2

Phase	P, MPa	T, K	$\beta \times 10^{-4}, \frac{1}{K}$
XII	● 2150	115	0,59
		110	0,58
		105	0,57
	◦ 2300	120	0,55
		115	0,53
		110	0,50

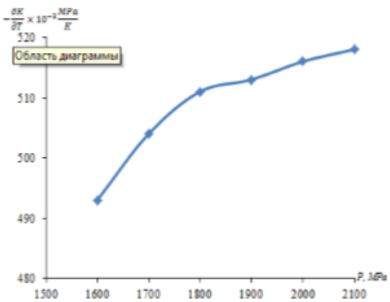


Figure 5 – The dependence of the thermal coefficient of ice-twelve modification on pressure at the temperature of 100 K

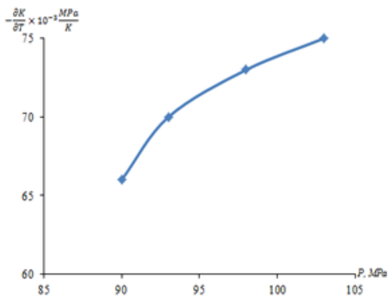


Figure 6 – The dependence of the partial derivatives of the ice-twelve modification on temperature at a pressure of 1900 MPa

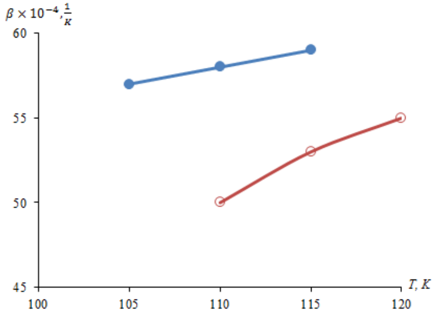


Figure 7 – The dependence of the coefficients of thermal expansion of the ice-twelve modification on temperature at pressures ● 2150 MPa, ◦ 2300 MPa

Conclusion

We see that between the studied temperature and pressure, the values of $\frac{\partial K}{\partial T}$, $\frac{\partial K}{\partial p}$ do not change much as a function of temperature at constant pressure and pressure at constant temperature.

The values of the coefficients of thermal increase in the studied temperature and pressure zone of the ice-twelve modification are positive. From the diagram 7 we see that the value of $\beta = \frac{1}{V}\left(\frac{\partial V}{\partial T}\right)_p$ decreases with increasing pressure of the ice modification under consideration at the same temperatures.

The research paper presents a method for calculating the design of a piston-cylindrical chamber assembled for the purpose of studying phase diagrams of high-pressure and low ice temperatures. The thermal $\frac{\partial K}{\partial T}$, baric $\frac{\partial K}{\partial p}$ universal compression coefficients of ice-twelve modification, as well as the coefficients of volumetric thermal expansion at different temperatures and pressures are studied.

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ЖОҒАРЫ ҚЫСЫМ (0-2500) МПА ЖӘНЕ ТӨМЕНГІ ТЕМПЕРАТУРАДА (90-300) К ҚАТТЫ ДЕНЕЛЕРДІҢ ФИЗИКАЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕЙТІН ҚОНДЫРҒЫ

Бұл мақалада мұздың жоғары (0-3000) МПа қысымдағы және төменгі (90-300) К температурадағы Р-Т координатасындағы диаграммасын тұрғызу үшін жинастырылған поршень-цилиндр типтес камераның құрылысын есептеу және үлгідегі қысымды, температураны өлшеу әдістері талқыланып, 100 К температурадағы, 3000 МПа қысымдағы камераның жарылған сызаттары, поршеннің сынған суреттері келтірілген.

Тәжірибе жүзінде алынған фазалық диаграммасындағы XII мұз модификациясының термиялық, барлық және жылулық сығылғыштық коэффициенттерінің қысым мен температураға тәуелділіктері алғаш рет зерттелген. XII мұз модификациясының жылулық коэффициентінің $\frac{\partial K}{\partial T}$ *қысымға тәуелділігі, дербес туындыларының* $\frac{\partial K}{\partial p}$ *температураға тәуелділігі, жылулық ұлғаю коэффициенттерінің температураға тәуелділігі графиктерін құрдық. Сонымен қатар, алынған нәтижелерге сүйенсек, зерттелген температура мен қысым* *аралығында $\frac{\partial K}{\partial T}$, $\frac{\partial K}{\partial p}$ шамалары тұрақты қысымда температураға, тұрақты температурада қысымға тәуелділіктері көп өзгермейтінін, қарастырып отырған мұз модификациясының қысымды арттырғанда $\beta = \frac{1}{V}\left(\frac{\partial V}{\partial T}\right)_p$ шамасының кемитіндігін көруге болады.*

Кілтті сөздер: жогары қысым, төменгі температура, термоөңдеу, құрыштар, датчик, терможұп, термиялық, барлық коэффициенттері.

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УСТРОЙСТВО ДЛЯ ИССЛЕДОВАНИЯ ФИЗИЧЕСКИХ СВОЙСТВ ТВЕРДЫХ ТЕЛ ПРИ ВЫСОКОМ ДАВЛЕНИИ (0-2500) МПА И НИЗКОЙ ТЕМПЕРАТУРЕ (90-300)

В этом исследовании обсуждаются расчеты конструкции камеры поршнево-цилиндрового типа в сборе для построения ледяной диаграммы в координате Р-Т при высоком давлении(0-2500) МПа и низкой температуре (90-300) К и измерения температуры, давления образца, трещин в камере при температуре 100 К, давление 3000 МПа, показаны поломки поршня.

Впервые исследована зависимость термических, барических коэффициентов и коэффициентов сжимаемости давления и температуры модификации льда XII от полученной на практике фазовой диаграммы. Построены графики зависимости теплового коэффициента $\frac{\partial K}{\partial T}$ от давления, зависимости частных производных $\frac{\partial K}{\partial p}$ от температуры, зависимости коэффициентов теплового расширения модификации льда XII от температуры. Мы также разработали результаты, согласно которым между исследуемой температурой и давлением значения $\frac{\partial K}{\partial T}$, $\frac{\partial K}{\partial p}$ не сильно меняются в зависимости от температуры при постоянном давлении и давления при постоянной температуре, значение $\beta = \frac{1}{V}\left(\frac{\partial V}{\partial T}\right)_p$ уменьшается с увеличением давления рассматриваемой модификации льда при тех же температурах.

Ключевые слова: высокое давление, низкая температура, термообработка, броня, датчик, термопара, коэффициенты термического, барического сжатия.

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