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High-pressure experiments in petrological researches by piston-cylinder device method

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Abstract:

The article describes the method used in experimental petrological studies of rock-forming mineral solid solutions. It describes in detail the concept of initial preparation of the materials. The article presents the description of the piston-cylinder type apparatus used in our experimental studies. Such equipment is suitable for the synthesis of high-pressure phases of the silicate systems with the pressure values up to 4.0 GPa and the temperature up to 1700 °C. The article also presents the details and describes special features of our method of conducting experiments with the piston-cylinder apparatus. To synthesis the phases the obtained homogeneous glasses are used which determine the equability of the substance distribution. It is revealed that the high-pressure cells made of sodium chloride are more suitable then the water-containing and silicate-containing ones usually used in similar experiments. The analytical methods applied to the obtained data analysis are also outlined.

Keywords: Petrological experiment; Piston-cylinder; Solid solutions; High-pressure cell; High temperature

1. Introduction

Numerous geological processes occurring in the Earth's crust depths and in upper mantle can be modeled only on the basis of experimental studies due to unavailability of these processes for direct observation. Rock-forming mineral solid solutions have their own original composition which depends on their formation conditions. The correlation between the phase composition and the pressure/temperature (P, T) parameters allows us to construct a polynomial dependence, phase diagrams, and to determine the minerals and rocks fields of stability. Studies of natural samples (Su et al., 2001; Janak et al., 2004; Katayama and Maruyama, 2009) don't doubt, however, it should be understood that the multicomponence of 'natural' rock and the results of corresponding experiments are reliable for a strictly specific deposit. The model systems allow us to build physics-chemical schemes and then to use them to interpret the genesis of deep rocks. In simplified systems, it is easier to control the kinetic processes and synergy of molecules during an experiment.

It is important both to prepare the starting material, to appropriate the equipment with accurate calibration and to develop the experiment methodology in order to obtain reliable results in experimental studies. The experiments on piston-cylinder equipment were carried out earlier and are still being provided (Boyd and England, 1963; Godovikov et al., 1971; Herrick et al., 1978; Milholland and Presnall, 1998; Bogaerts and Schmidt, 2006; Masotta et al., 2012; Banushkina et al., 2019; Condamine et al., 2022; Ezad et al., 2023; Ashrafi et al., 2024; Elmi et al., 2025). Some nuances have been noted over the many years of operating such devices. They allow us to obtain the most reliable results during the experiment. But, nevertheless, not all previously published works devoted to similar experiments mention them. Thus we hope that an article presenting the details and describing special features of the method of piston-cylinder experiments may be useful for experimentalists. There is a number of studies carried out in V.S. Sobolev Institute of Geology and Mineralogy SB RAS among others, for example (Godovikov et al., 1971; Surkov, 1983; Banushkina, 2021; Salehpour et al., 2025). The publications have a partial description of the device and the methods to use it. Therefore, the detailed description of our methodology to prepare starting materials and to carry out the high-pressure experiments would be useful for a wide range of researchers engaged in the field of experimental petrology.

Clinopyroxenes are found in almost all the types of the rocks and have the widest composition variation. This feature of the mineral is interesting to petrologists (Holloway and Wood, 1988). The process of clinopyroxene formation by the example of solid solution namely diopside Di ($CaMgSi_2O_6$) – calcium molecule Eskola CaEs ($Ca_{0.5}AlSi_2O_6$) can be investigated during synthesis of high-pressure phases of silicate systems. In the present article, this solid solution is mentioned only as an example when describing the method of conducting high-pressure experiments. The techniques themselves used in the experiments are presented below.

2. Methodology

2.1 Starting materials

The starting materials are oxides CaO, MgO, Al_2O_3, SiO_2 of 'extra pure substance' brand. CaO and MgO are calcined in platinum crucibles at the temperature 1300 °C during for 6 hours and Al_2O_3, SiO_2 are prepared the temperature 1000 °C during for 5 hours. The platinum is selected as a well-proven material for conducting experiments at high temperatures and pressures (Anzellini et al., 2019). So, the samples will be subjected to the extreme conditions in platinum ampoules. The bulk compositions with ideal stoichiometry corresponding to Di and CaEs were obtained by the weight method. It should be noted that in the study of silicate rock-forming minerals, it is highly important to keep the correct SiO_2 content in experimental products constant, that may be overestimated to use an agate mortar for grinding initial mixtures as, for example, in works (Zhao et al., 2011; Knapp et al., 2013; Schroeder-Frerkes et al., 2016). In our experiments for grinding the initial mixture a carbide mortar is used. The final grain size is ranging about $5-10$ µm. Mixture is calcined in a platinum crucible at the temperature from 1100 °C to 1500 °C depending on the material. Each time the selected value is about $20-30$ °C lower than the melting point of the mixture. This statement should be noted as very important while preparing the starting materials. The selected calcination temperature allows to obtain a relatively soft sample due to an active diffusion of the components near the melting temperatures. In case we try to calcinate at the melting point temperature it leads to the excessive high hardness of the sample.

Both the calcination and the rubbing of the material should to be alternated just about every 12 hours. The process continues until the composition becomes homogeneous. Then the mixtures are melted in a high-temperature furnace in a platinum crucible. During this process the temperature is $1500-1620$ °C depending on the selected material. The melting occurs about 3 to 4 hours. The crucible is quenched in cold distilled water. As a result, the clear homogeneous glasses are formed inside it. This is an important point of experiment as these homogeneous glasses are determining the equability of the substance distribution. Furthermore, it has been shown in articles (Boyd and England, 1960; Presnall et al., 1978; Doroshev et al., 1982; Milholland and Presnall, 1998) that the recrystallization takes the minimal time to achieve the equilibrium in glass conditions. For example, the durations of our experiments are the following: At atmospheric pressure from 53 hours to 414 hours depending on temperature range; at 1.0 GPa the occurred time is 25 hours; at 1.2 GPa the time is 19 hours; at 1.3 GPa it is 16 hours; and at 1.4 GPa it holds 12 hours for the experiment. The long duration of the experiment allows to be confident that the equilibrium in samples is obtained.

2.2 The high-pressure apparatus of 'piston-cylinder' type

We use the piston-cylinder equipment to synthesize the silicate systems phases with up to 4.0 GPa pressure and up to 1700 °C temperature (Fig. 1). The experiments are carried out on the piston-cylinder by the quenching method. The design of the system is selected by simple and understandable principle of its operation, that allows not only to achieve necessary (P, T) conditions, but also to conduct an accurate calibration between the pressure of hydraulic oil and the actual pressure of the sample, thereby avoiding errors in measuring P and T parameters. The importance of the above-mentioned calibration is mentioned in the work (Moore et al., 2008).

The working volume of the device has cylindrical shape. There is a channel inside with the size 12.0 mm diameter and 50.0 mm length. The channel axis is located along the axis of carbide cylinder. There is a carbide disc in the upper part of the working volume that is required to apply the pressure to the inside (6, Fig. 1). The pressure is applied from the lower plunger to the top, transferring the force through a system of rods and pins (19, 16, 18, Fig. 1) on the carbide piston (14, Fig. 1). So, the upper cylinder is transferring the force of the press to the working volume. The carbide material of the tungsten carbide discs (6, 12, Fig. 1) (the melting point is 2870 °C) quietly withstands the operating conditions of the equipment, up to 1700 °C temperature.

The heating device - patent No.1762458 (Surkov, 1992) (Fig. 2) is located inside the working volume. A pressure transfer matter is a compressed calcined sodium chloride. It has high plasticity properties and the stable heat isolation. Due to that we can conduct long-term experiments at high temperatures. Besides, it allows to achieve hydrostatic pressure transfer, as in the case of apparatus developed in (Mirwald et al., 1975). The external shell of the device is greased with MoS_2 to reduce the friction.

To exclude the penetration of volatile components into the cell the sodium chloride should be used instead of a pyrophyllite ($Al_2O_34SiO_2H_2O$) or talc ($4SiO_23MgOH_2O$) used in the studies (Boyd and England, 1960, 1963; Presnall et al., 1978) while heating the device. A penetration of a foreign substance can lead to the mistakes in the experiment. Particularly, the penetration of volatile components such as H_2 and H_2O lowers the melting point of the system. So,

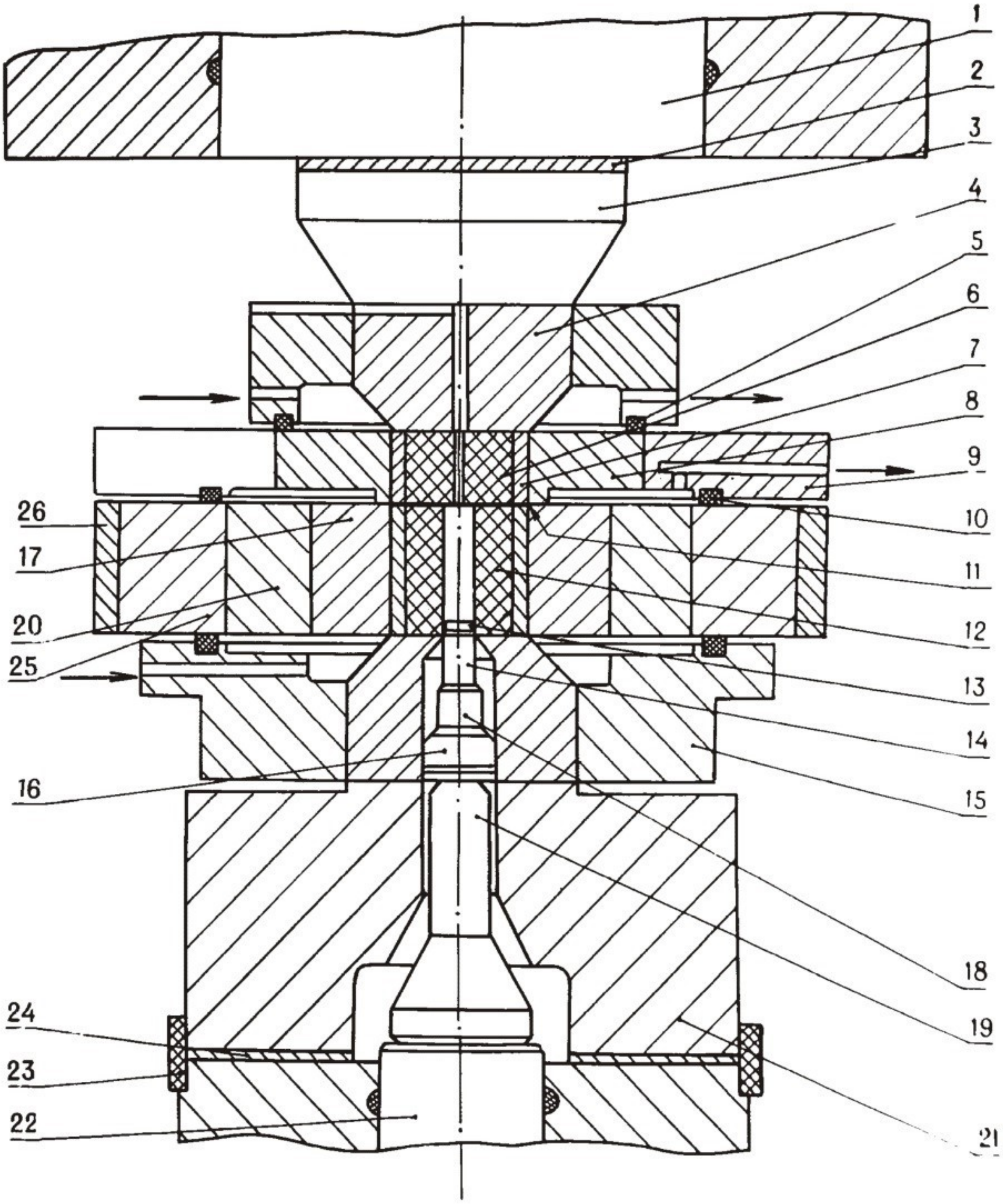

Figure 1. Piston-cylinder scheme for conducting high-pressure experiments.
1-upper plunger, 2-upper current-carrying gum, 3-nozzle, 4-intermediate block, 5-rubber seal, 6-carbide disc, 7-intermediate ring, 8-inner ring, 9-outer ring, 10-seal, 11-electrical insulation gasket (mica 0.1 – 0.3 mm), 12-carbide disc, 13-seal ring, 14-working rod, 15-stand, 16-steel plate, 17-split ring, 18-carbide heel, 19-intermediate rod, 20- pressing ring, 21-base, 22-lower plunger, 23-locking ring, 24-lower current-carrying gum, 25-bandage ring, 26-protective ring. An accuracy of maintaining pressure is ±0.03 GPa. Temperature deviation from the set value does not exceed ±1 °C.

the material through which the pressure is transmitted is important. After all, when minerals such as talc or pyrophyllite are heated, they will release the water. The water interacts with a graphite heater, creating additional hydrogen pressure. Hydrogen penetrates through the walls of Pt capsule. As a result, a melt can occur at the temperature about 300 °C lower than in the case of a pure mixture. The use of NaCl as a pressure transmitting medium excludes the penetration of the volatile components (H_2 and H_2O) into the working cell. Our conclusions about the use of NaCl in a high-pressure cell as a medium transmitting pressure and its advantages over talc and pyrophyllite are confirmed by the data (Boettcher et al., 1981).

The platinum crucible (wall thickness is 0.5 mm, initial height is 5 mm, final height is about 2 – 3 mm) with a sample is calcined at 1000 °C during for 6 hours and then it is sealed with an electric welding apparatus. Then it is placed into the middle of a tubular graphite heater (7, 11, Fig. 2). The heater has the following dimensions: Its outer diameter is 8.5 mm, the inner diameter is 6.0 mm, the initial length is 46 mm and the finite length ranges from 36 mm to 40 mm. The platinum capsule (11, Fig. 2) is separated from the graphite heater by the ceramic ring (1, Fig. 2). The pressed sodium chloride fixes the capsule from the below.

A platinum-rhodium thermocouple PtRh6-PtRh30 is installed on top of the crucible and it is separated by a ceramic plate (the thickness is from 0.3 mm to 0.4 mm). The thermocouple is isolated by means of a two-channel high-alumina ceramics straw with the diameter 1.6 mm (Al_2O_3 + Al_2O*SiO_2). The thermocouple is placed into the pipe of a ceramic column through a hole (6, Fig. 1). The high-precision temperature controller regulates the temperature. The temperature maintaining accuracy is of 1 °C. With the platinum crucible height and the heater length given above the temperature difference over the whole sample should not exceed 10 °C (Boyd and England, 1963; Cohen et al., 1966; Surkov, 1983), that is, the accuracy of temperature measurement in the experiment is also 10 °C. Thus, the pressure influence to electromotive force of the thermocouple was not considered.

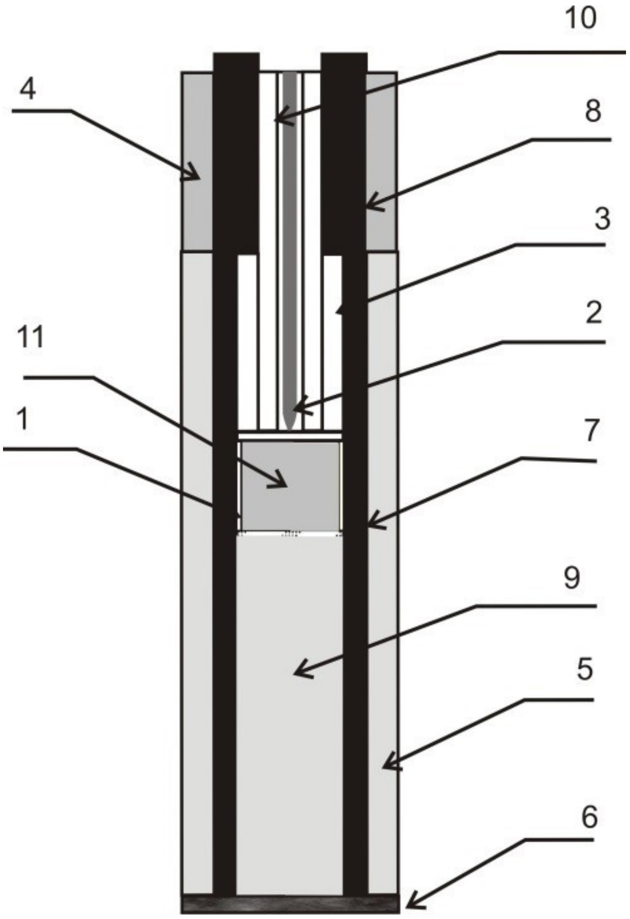

Figure 2. Heating device scheme (patent No. 1762458 (Surkov, 1992). 1-tube ceramic, 2-thermocouple pipe, 3-cylinder ceramic, 4-cylinder talc, 5-cylinder NaCl, 6-graphite tablet, 7-graphite heater, 8-graphite cylinder, 9-NaCl capsule stand, 10-tube ceramic, 11-sample in a Pt capsule. The use of such heating device allows long-term experiments at high temperatures, also this excludes the penetration of H2 and H2O into the cell.

The pressure is measured by the force immersion. The pressure fluctuations are 0.1 – 0.25 GPa. This was obtained by the difference between the force calculated and the one required for bismuth transition (from I-II to II-III valence) at 2.54 GPa and 20 – 25 °C. Bismuth wire was placed into a silver chloride tablet and then on the working rod. Wire size is 0.2 by 5 mm. The calibrations repeated many times have shown that the correction value remains constant regardless the number of the performed experiments. The calibration at high temperatures was carried out using the quartz-coesite transition (Malinovskiy and Doroshev, 1974). The pressure measurement accuracy is of 0.03 GPa, that is close to the values of measurement accuracy in research (McDade et al., 2002).

A heating element is placed in the center of the working volume. To supply the current to the heating element, two holes are made to install the electric leads. The electric power leads are made in the form of a flanged copper sleeve, inside of which there is a mushroom-shaped rod made of high-strength material. A disc spring is installed on the cylindrical surface of the sleeve from the outside of the vessel, and an electric contact is installed on the conical surface of the sleeve shank.

The two-stage compression method is applied. At first, the pressure is about 0.5 – 0.6 GPa lower than required. After that the heating to the required temperature is carried out. Then at the second stage, the pressure raises to the required value. This method makes it possible to avoid the influence of heating device's thermal expansion. The quenching is provided by turning off the voltage on the heater. Temperature droppes at a rate of about 450 °C/s. The room temperature is reached after 5 – 6 seconds.

2.3 Methods of analysis of synthesized samples

The obtained samples are getting in epoxy and polished with diamond pastes with the grain size indicators of 10/7, 5/3, 2/1. Then the double-sided polished sections with orientation along the vertical axis are made. Petrographic polarization microscope POLAM L-211 and Olympus BX51 with camera adapter are used for studying phase relationships. The compositions of finished samples are determined with the electronic microprobe Comebax-Micro microanalyzer and the scanning electron microscope MIRA 3 LMU (Tescan Orsay Holding), the latter equipped the INCA Energy 450 X-Max-80 microanalysis system (Oxford Instruments Nanoanalysis Ltd.) and it has a high accuracy of analysis. Energy-dispersive X-ray spectra are typed in the scanning mode. A small raster 5 – 50 µm is used for minerals, 50 – 500 µm for a quenched melt. The accelerating voltage is of 20 kV, the electron beam current is of 1.5 nA and the counting time is of 20 s (Lavrent'ev et al., 2015). The additional phase diagnostics is performed with the help of RAMAN spectrometer (Horiba Jobin YVON LabRam HR800) with 532 µm Nd solid-state laser. The spectra are obtained at ambient conditions in backscattering geometry with a laser power of about 1 mW and a spectral resolution

2 cm^{-1}. The wavenumbers (wavelengths) are calibrated by the emission lines of Ne lamp (585.25 µm and 540.06 µm). The phase spectrum standards are taken from the Database of Raman spectroscopy, X-ray diffraction and chemistry of minerals (Richet et al., 1998) (http://rruff.info/).

3. Results

As an example of the use of a cylindrical piston device, the experiments were conducted with compositions of a solid solution of clinopyroxene. Some results of the analysis of the obtained substance are shown in Table 1 and Table 2.
A detailed overview of the obtained data allows us to highlight some significant results. For example, according to the data the phase composition of Di is characterized by several features: 1) the predominance of the magnesian component over the calcium one, 2) the absence of aluminum oxide Al^{3+}, 3) the amount of silica SiO$_2$ is close to ideal, 4) the amount of the enstatite component is up to 5 mol.%.
Such analyses can make it possible to understand the processes occurring at various depths of the earth's crust and mantle without leaving the laboratory conditions. A detailed presentation and analysis of the results of studies of the solid solution diopside Di (CaMgSi$_2$O$_6$) – calcium molecule Eskola CaEs (Ca$_{0.5}$AlSi$_2$O$_6$), carried out on the

Table 1. Some results of phase Di compositions obtained by the scanning electron microscope.

Exp. Num.		A-145	A-144	A-100	A-99	A-113	P-487	P-488	P-565
Component.wt. %	CaO	24.84	24.58	24.6	24.35	25.34	25.45	25.42	24.96
	MgO	18.95	19.47	18.98	18.99	17.71	19.09	19.02	18.84
	Al$_2$O$_3$	0.51	0.64	1.21	1.8	1.87	0	0	1.1
	SiO$_2$	55.71	56.2	54.79	54.79	54.83	55.47	56.16	55.62
	total	100.01	100.89	99.58	99.93	99.75	100.01	100.6	100.52
Formula coefficients	Ca	0.955	0.936	0.951	0.936	0.978	0.982	0.973	0.955
	Mg	1.014	1.031	1.02	1.016	0.951	1.025	1.013	1.003
	Al	0.022	0.027	0.051	0.076	0.079	0	0	0.046
	Si	1.999	1.997	1.976	1.967	1.976	1.997	2.007	1.986
	total	3.99	3.99	3.998	3.995	3.985	4.003	3.993	3.991
Minal	Di	94.8	93.6	92.4	90.2	93.9	98.3	98.2	94.1
	En	3.2	5.1	4.4	5.5	0.6	2	1.9	3.3
	total	98.1	98.7	96.9	95.7	94.5	100.3	100	97.4

Table 2. Some selected compositions of clinopyroxene solid solutions Cpx(ss) obtained by the scanning electron microscope.

Exp. Num.		P48	P56	P429	P438	P439	P441	P442	P444
Component.wt. %	CaO	24.57	22.18	24.46	23.88	25	23.51	24.79	24.54
	MgO	13.07	4.21	16.03	14.89	15.99	14.6	14.6	13.71
	Al$_2$O$_3$	10.16	28.53	4.98	9.33	6.72	9.67	6.98	11.66
	SiO$_2$	52.45	44.98	54.73	51.45	52.17	51.97	53.36	49.84
	total	100.25	99.92	100.2	99.54	99.87	99.75	99.73	99.75
Formula coefficients	Ca	0.936	0.837	0.934	0.918	0.963	0.9	0.952	0.945
	Mg	0.693	0.221	0.852	0.797	0.857	0.777	0.78	0.734
	Al	0.426	1.184	0.209	0.394	0.285	0.407	0.295	0.494
	Si	1.866	1.583	1.95	1.847	1.876	1.856	1.913	1.79
	total	3.921	3.825	3.945	3.956	3.981	3.94	3.94	3.963
Minal	Di	68.3	21.83	82.94	72.09	82.07	69.62	77.07	69.77
	En			1.11	3.79	1.82	4.06		1.82
	CaTs	13.24	41.17	4.96	15.33	12.39	14.38	8.62	20.96
	CaEs	15.51	34.65	10.99	8.78	3.73	11.94	11.88	7.44
	Wol	2.96	2.35					2.427	

above-described piston-cylinder setup, are the subject of a separate article.

4. Conclusion

With the help of suggested equipment, it becomes possible to model the geological processes occurring in the Earth's crust and in upper mantle due to experiments in petrology. It allows to interpret the genesis of rocks from a physico-chemical point of view by the result of the experiments held in the laboratory. The piston-cylinder device is an integral part of high-pressure and high-temperature research due to its simple and reliable construction, rapid quenching of samples, reasonably simple maintenance, clear operating concept and the ability to control changes in conditions during the experiment. This type of equipment is suitable for the synthesis of high-pressure phases of silicate systems at pressure up to 4.0 GPa and temperature up to 1700 °C. It is revealed that the use of a carbide mortar for mechanical grinding of silicate samples is preferable to agate, since it avoids increased silicon content in obtained products. While the penetration of volatile components (H_2O and H_2) through the walls of the Pt capsule can reduce the melting point of the system, the use of sodium chloride as a pressure transfer material excludes the penetration of the volatile components into the working cell. Thus the use of NaCl as the pressure medium ensures the undistorted phase relations in samples obtained from high-pressure experiments.

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Authors contributions
Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials
The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Conflict of interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

Anzellini S., Monteseguro V., Bandiello E., Dewaele A., Burakovsky L., Errandonea D. (2019) In situ characterization of the high pressure – high temperature melting curve of platinum. *Scientific Reports* 9:13034. DOI: https://doi.org/10.1038/s41598-019-49676-y.

Ashrafi N., Dabiri R., Jahangiri A. (2024) Some chemical variations in biotite, phlogopite, and muscovite, considering their tectonic setting. *Geopersia* 14 (2): 307–332.
DOI: https://doi.org/10.22059/geope.2024.373882.648749.

Banushkina S. V. (2021) Phase relationships and melting features in the quartz-normative area of the compositions of CaO-MgO-Al_2O_3–SiO_2 (CMAS) system based on an experimental study of diopside – calcium molecule Eskola cross section. PhD Thesis, Institute of the Earth's Crust SB RAS, Irkutsk, Russia

Banushkina S. V., Surkov N. V., Golitsyna Z. F. (2019) Melting features of phases in the diopside Calcium molecule of Escola section in the pressure range 1 kg/sm^2 – 20 kbar. *Transbaikal State University Journal* 25 (7): 6–17.
DOI: https://doi.org/10.21209/2227-9245-2019-25-7-6-17.

Boettcher A. L., Windom K. E., Bohlen S. R., Luth R. W. (1981) Low-friction, anhydrous, low- to high-temperature furnace sample assembly for piston-cylinder apparatus. *Review of Scientific Instruments* 52:1903–1904. DOI: https://doi.org/10.1063/1.1136545.

Bogaerts M., Schmidt M. W. (2006) Experiments on silicate melt immiscibility in the system Fe_2SiO_4–$KAlSi_3O_8$–SiO_2–CaO–MgO–TiO_2–P_2O_5 and implications for natural magmas. *Contributions to Mineralogy and Petrology* 152:257–274.
DOI: https://doi.org/10.1007/s00410-006-0111-6.

Boyd F. R., England J. L. (1960) Apparatus for phase-equilibrium measurements at pressures up to 50 kilobars and temperatures up to 1750 °C. *Journal of Geophysical Research* 65 (2): 741–748.
DOI: https://doi.org/10.1029/JZ065i002p00741.

——— (1963) Effect of pressure on the melting of diopside, $CaMgSi_2O_6$, and albite, $NaA1Si_3O_8$, in the range up to 50 kilobars. *Journal of Geophysical Research* 68 (1): 311–323.
DOI: https://doi.org/10.1029/jz068i001p00311.

Cohen L. H., Klement W., Kennedy G. C. (1966) Investigation of phase transformations at elevated temperatures and pressures by differential thermal analysis in piston-cylinder apparatus. *Journal of Physics and Chemistry of Solids* 27:179–186.
DOI: https://doi.org/10.1016/0022-3697(66)90179-X.

Condamine P., Tournier S., Charlier B., Médard E., Triantafyllou A., Dalou C., Tissandier L., et al. (2022) Influence of intensive parameters and assemblies on friction evolution during piston-cylinder experiments. *American Mineralogist* 107:1575–1581.
DOI: https://doi.org/10.2138/am-2022-7958.

Doroshev A. M., Malinovskiy I. Y., Surkov N. V., Kalinin A. A. (1982) Features of the process of achieving equilibrium between garnets and clinopyroxenes in CaO-MgO-Al_2O_3-SiO_2 system. In: Problems of experiment in solid-phase and hydrothermal high-pressure equipment: Collection of scientific papers. Nauka, Moscow, Russia:97–111.

Elmi R., Arian M. A., Ashja Ardalan A., Yazdi A. (2025) Petrology of volcanism in the Alasht-Haraz road of the Alborz mountain range, south of Amol (north of Iran). *Iranian Journal of Earth Sciences* 17 (3) DOI: https://doi.org/10.57647/j.ijes.2025.16800.

Ezad I. S., Shcheka S. S., Buhre S., Buhre A., Gorojovsky L. R., Shea J. J., Forster M. W., Foley S. F. (2023) Rapid quench piston cylinder apparatus: An improved design for the recovery of volatile-rich geological glasses from experiments at 0.5 – 2.5 GPa. *Review of Scientific Instruments* 94:055107.
DOI: https://doi.org/10.1063/5.0129417.

Godovikov A. A., Smirnov S. A., Malinovskiy I. Y., Ran E. N., Pankov M. S., Rosinskiy G. A., Tokmin B. P. (1971) Device for creating a pressure of up to 40 kbar at temperatures of up to 1700 degrees C. *Instruments and Experimental Techniques-USSR* 6:159–160.

Herrick C. C., Bustamante S. J., Charles R. W., Cowan R. E., Hakkila E. A., Hull D. E., Olinger B. W., Roof R. B., Sheinberg H., Herrick G. C. (1978) Preparation of synthetic standard minerals. *LA-7072-MS Informal Report. Los Alamos Scientific Laboratory, UC, Los Alamos, NM, USA,* DOI: https://doi.org/10.2172/5142664.

Holloway J. R., Wood B. J. (1988) Simulating the Earth: Experimental geochemistry. Springer, Dordrecht, Netherlands
DOI: https://doi.org/10.1007/978-94-011-6496-2.

Janak M., Froitzheim N., Luptak B., Vrabec M., Ravna E. (2004) First evidence for ultrahigh-pressure metamorphism of eclogites in Pohorje, Slovenia: Tracing deep continental subduction in the Eastern Alps. *Tectonics* 23 (TC5014): 1–10.
DOI: https://doi.org/10.1029/2004TC001641.

Katayama I., Maruyama S. (2009) Inclusion study in zircon from ultrahigh-pressure metamorphic rocks in the Kokchetav massif: an excellent tracer of metamorphic history. *Journal of the Geological Society* 166:783–796. DOI: https://doi.org/10.1144/0016-76492008-019.

Knapp N., Woodland A. B., Klimm K. (2013) Experimental constraints in the CMAS system on the Ca-Eskola content of eclogitic clinopyroxene. *European Journal of Mineralogy* 25 (4): 579–596.
DOI: https://doi.org/10.1127/0935-1221/2013/0025-2326.

Lavrent'ev Y. G., Karmanov N. S., Usova L. V. (2015) Electron probe microanalysis of minerals: microanalyzer or scanning electron microscope? *Russian Geology and Geophysics* 56 (8): 1154–1161.
DOI: https://doi.org/10.1016/j.rgg.2015.07.006.

Malinovskiy I. Y., Doroshev A. M. (1974) MgO-Al_2O_3-Cr_2O_3-SiO_2 system at 1200 °C and 30 kbar. In: Experimental studies in mineralogy (1972-1973): Collection of scientific papers. Institute of Geology and Geophysics, Siberian Branch, USSR Academy of Sciences, Novosibirsk, Russia:62–69.

Masotta M., Freda C., Paul T. A., Moore G. M., Gaeta M., Scarlato P., Troll V. R. (2012) Low pressure experiments in piston cylinder apparatus: Calibration of newly designed 25 mm furnace assemblies to P = 150 MPa. *Chemical Geology,* 312–313:74–79.
DOI: https://doi.org/10.1016/j.chemgeo.2012.04.011.

McDade P., Wood B. J., Van Westrenen W., Brooker R., Gudmundsson G., Soulard H., Najorka J., Blundy J. (2002) Pressure corrections for a selection of piston-cylinder cell assemblies. *Mineralogical Magazine* 66:1021–1028. DOI: https://doi.org/10.1180/0026461026660074.

Milholland C. S., Presnall D. C. (1998) Liquidus phase relations in the CaO-MgO-Al_2O_3-SiO_2 system at 3.0 GPa: The aluminous pyroxene thermal divide and high-pressure fractionation of picritic and komatiitic magmas. *Journal of Petrology* 39 (1): 3–27.
DOI: https://doi.org/10.1093/petroj/39.1.3.

Mirwald P. W., Getting I. C., Kennedy G. C. (1975) Low-friction cell for piston-cylinder high-pressure apparatus. *Journal of Geophysical Research* 80 (11): 1519–1525.
DOI: https://doi.org/10.1029/JB080i011p01519.

Moore G., Roggensack K., Klonowski S. (2008) A low-presure-high-temperature technique for the piston-cylinder. *American Mineralogist* 93:48–52. DOI: https://doi.org/10.2138/am.2008.2618.

Presnall D. C., Dixon S. A., Dixon J. R., O'Donnell T. H., Brenner N. L., Schrock R. L., Dycus D. W. (1978) Liquidus phase relations on join diopside-forsterite-anorthite from 1 atm to 20 kbar: Their beaming on the generation and crystallization of basaltic magma. *Contributions to Mineralogy and Petrology* 66 (2): 203–220.
DOI: https://doi.org/10.1007/BF00372159.

Richet P., Mysen B., Ingrin J. (1998) High-temperature X-ray diffraction and Raman spectroscopy of diopside and pseudowollastonite. *Physics and Chemistry of Minerals* 25 (6): 401–414.
DOI: https://doi.org/10.1007/s002690050130.

Salehpour S., Arian M. A., Rad A. J., Zarei Sahamieh R., Yazdi A. (2025) Geochemistry and technomagmatic environment of Eocene volcanic rocks in Yuzbashi Chay region, west of Qazvin (Iran). *Iranian Journal of Earth Sciences* 17 (1): 1–13.
DOI: https://doi.org/10.57647/j.ijes.2025.1701.04.

Schroeder-Frerkes F., Woodland A. B., Uenver-Thiele L., Klimm K., Knapp N. (2016) Ca-Eskola incorporation in clinopyroxene: limitations and petrological implications for eclogites and related rocks. *Contributions to Mineralogy and Petrology* 171:100–118.
DOI: https://doi.org/10.1007/s00410-016-1311-3.

Su W., You Z., Wang R., Liu X. (2001) Quartz and clinoenstatite exsolutions in clinopyroxene of garnet-pyroxenolite from the North Dabie Mountains, eastern China. *Chinese Science Bulletin* 46 (17): 1482–1484. DOI: https://doi.org/10.1007/BF03187037.

Surkov N. V. (1983) Experimental study of the stability of clinopyroxenes of CaO-MgO-Al_2O_3-SiO_2 system in connection with the petrology of the upper mantle. PhD Thesis, Institute of Geology and Geophysics SB USSR Academy of Sciences, Novosibirsk, Russia

Surkov N.V. (1992) Cell of a device for creating high temperature at ultrahigh pressure. Inventor's Certificate No. 1762458, Byull. Izobret., No. 34

Zhao S., Nee P., Green H. W., Dobrzhinetskaya L. F. (2011) Ca-Eskola component in clinopyroxene: Experimental studies at high pressures and high temperatures in multianvil apparatus. *Earth and Planetary Science Letters* 307 (3-4): 517–524.
DOI: https://doi.org/10.1016/j.epsl.2011.05.026.