Device for Compressibility and Thermal Expansion Study up to 3 GPa

M. Krupski and A. Krupska*
Institute of Molecular Physics, Polish Academy of Sciences, M. Smoluchowskiego 17, 60-179 Poznań, Poland

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We present the realization of a project of a device utilizing a standard high-pressure chamber adapted to compressibility and thermal expansion measurements of solids at hydrostatic pressures up to 3 GPa. The device has been tested on the triglycine sulphate crystal (TGS).

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1. Introduction

High-pressures up to 3 GPa have been usually generated in piston-cylinder devices using the principle of unsupported area introduced by Bridgman [1]. More recently, the high-pressure range has been extended significantly towards pressures as high as 550 GPa with diamond anvil cell (DAC) systems [2]. However, the geometry of DACs usually imposes small sample volumes, which, depending on applications, might result in low accuracy of measurements. Clearly, a piston-cylinder device does not suffer from this limitation. Thus, it is then suitable for performing numerous practical high-pressure studies in materials and geophysical sciences [3]. Recently, a capacitive dilatometer, designed for high-resolution measurements of length changes of a material within the temperature range 1.4 K $\leq T \leq$ 300 K and hydrostatic pressures up to only $P \leq 250$ MPa has been described [4].

In this work, we present a custom-made piston-cylinder high-pressure apparatus for compressibility measurements up to 3 GPa. The construction of the device, designed around a high-pressure chamber, is a modified version of the apparatus applied previously in the dielectric studies (such as: electric permittivity ε, spontaneous polarization P_s and coercitive electric field E_c) in the TGS crystal under high hydrostatic pressure (see [5], Fig. 1, and [6]). In the following, a detailed description of the construction of the high-pressure setup is presented.

2. Device for compressibility and thermal expansion study up to 3 GPa

The herein presented device makes it possible, for the first time, to determine *in situ* both the compressibility and thermal expansion of solids over wide ranges of temperatures and pressures. The functioning of the high-pressure apparatus has been demonstrated on the example of TGS. The apparatus, schematically shown in Fig. 1, has been tested under pressures up to 3 GPa. Tungsten carbide (WC) and hardened steel were used for manufacturing of the piston (1) and the high-pressure chamber (2), respectively. The latter high-pressure chamber has been reinforced by autofrettage and by adding a massive steel jacket around (4). High-pressures were generated by acting on the piston with a hydraulic press. At the bottom, an immobile plug (9) made of hardened steel is applied. The conducting leads of 0.1 mm in diameter (five copper wires (green) and one constantan wire (red) are passing through the plug. The copper–constantan thermocouple (8) is used to measure the temperature inside the high pressure chamber. A pyrophyllite powder mixed with epoxy resin is applied as the isolation and sealing. Inside the pressure cell a Teflon container (7) (1.5 mm inner wall thickness) is placed. An unhardened Be–Cu ring (3) with a triangular cross-section prevents the Teflon and the pressure-transmitting liquid from leaking. The typical Bridgman sealing [1] between the plug and the cylinder is provided by a brass or beryllium bronze ring (11) and the rubber o-ring (12) (Fig. 1). The Teflon cell container (7) is placed in the piston-cylinder device. An induction sensor (15) is used to measure the shift of the piston relative to the chamber. This device may be used in studying solid crystals in the temperature range from 1.4 to 300 K under hydrostatic pressure up to $P = 3$ GPa. The constant temperature during pressure measurements is obtained by means of a heater (6) placed outside the chamber and connected to a temperature stabilizator through a heat exchanger (5).

Sealing between the high-pressure chamber and the moving plug is assured by means of the anti-extrusion ring (3) made of brass or beryllium bronze matched to the diameter of the chamber. The cross-section of the ring is of a triangular shape. The pressure of the liquid within the container causes a very small deformation of the ring between the chamber and piston, lower than 0.01 mm (Fig. 1).

The results of the test measurements, performed at room temperature, are shown in Fig. 2. The arrows indicate the direction of measurement, between 0 and 2.3 GPa. Under pressure of 2.3 GPa, the changes in the

*corresponding author; e-mail: krupska@ifmpan.poznan.pl

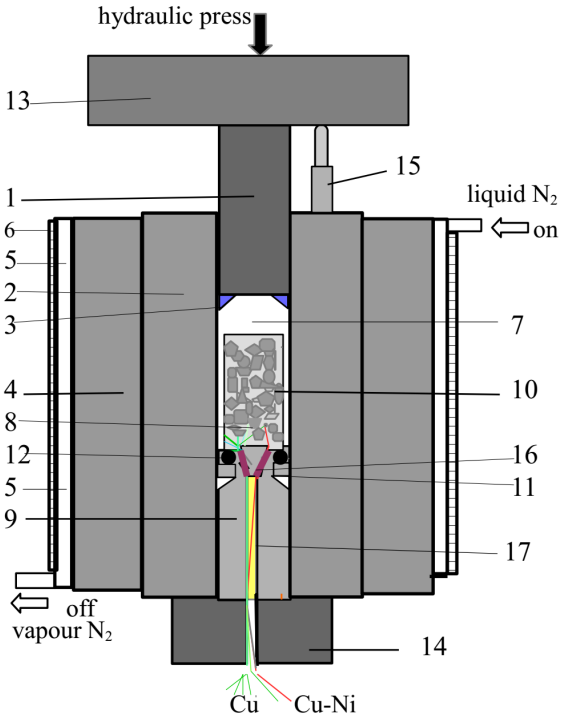

Fig. 1. The cross-section of the high-pressure chamber adapted for the compressibility and thermal expansion measurements. 1 — piston WC, 2 — high-pressure chamber, 3 — anti-extrusive ring, 4 — massive hardened steel jacket, 5 — heat exchanger, 6 — electric heater, 7 — Teflon container, 8 — copper–constantan thermocouple, 9 — hardened steel plug, 10 — TGS sample in the pressure-transmitting liquid, 11 — brass or beryllium bronze ring, 12 — rubber O-ring, 13 and 14 — hardened steel supports, 15 — induction sensor, 16 — pyrophyllite powder (violet), 17 — epoxy resin (yellow).

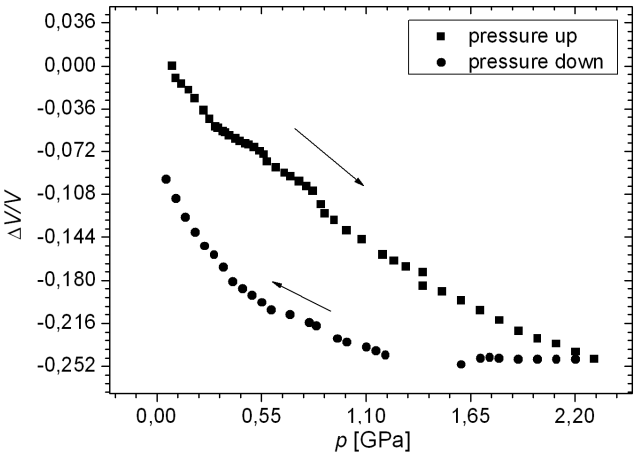

Fig. 2. Compressibility $\Delta V/V$ as a function of hydrostatic pressure P of an exemplary TGS crystal at room temperature measured using the device described herein (see Fig. 1).

volume $\Delta V/V$ was determined at the range 2.025. Upon releasing pressure from 2.3 GPa down to 0.1 MPa (atmospheric pressure), we noticed that the starting values were not reached. We assume that the observed discrepancy was caused by the friction of the piston sealing during the movement.

3. Discussion and conclusions

It can be concluded that the compressibility measurements at high pressures up to 3 GPa can help to understand many physical phenomena in solids, e.g., phase transitions. The high-pressure device described in this work enables one to measure in a wide range of temperatures, from about 80 to 400 K. Clearly, the device can also be used for the measurement of thermal expansion at constant pressure.

The pressure measurement error in the set of equipment is significant. This is due to the position measurement error caused by the friction of the sealing during the movement of the piston relative to the chamber. An alternative method for pressure measurement is the use of the managing coil placed inside the chamber. However, this method is not convenient and does not lead to good results. Additional four copper wires are free. They may be used for electrical connections to a pressure resistive sensor and to measure the dielectric permittivity of, for example, a TGS cystal [5] or other properties of solids dependent on high-pressure in a wide temperature range.

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