Elemental boron and boron-rich compounds have been given a particular scientific and technological attention because of their specific physical properties: high melting temperatures, a wide energy band gap, high hardness, strong absorbance of neutrons, etc. The structures of these materials are based on icosahedral units as a common feature.

In 1965 R. H. Wentorf reported synthesis of a new boron modification at pressures above 10 GPa and temperatures above 1500 C. The existence of this material was not confirmed until recently, when Zarechnaya et al. (see, for example, ESRF Highlights 2009) reproduced synthesis of the high-pressure boron phase (further called γ-B_{28}) and solved its structure from single-crystal X-ray diffraction data. Moreover, based on the single crystal data collected in DAC at ID09a beam-line in He pressure transmitting medium we identified isostructural phase transformation in γ-B_{28} (see Experimental Report CH2821).

For years theoreticians predicted a transformation of low-pressure boron phases into α-Ga structure at 74 GPa (Haussermann et al., 2003) or about 90 GPa (Oganov et al., 2009; Zarechnaya et al., 2009). Room temperature compression of α- and β-boron does not result in any phase transitions in the crystalline boron phases (Sanz et al., 2002). In order to transform boron into a possible high-pressure phase heating required (Zarechnaya et al., 2009).

For our experiment HS3934 we got a beam time at ID09a, which so far is not equipped for laser-heating in DACs. To realize heating in our experiments we brought and tested at ID09a our portable laser-heating system (Dubrovnisky et al., 2009).

The system consists of two major components – the source of laser light (in our case it is 100 W Modulated High Power Fiber Laser SPI100, SPI Lasers UK Ltd., weight of 40 kg) and the universal laser-heating head (UniHead) (Fig. 1) (Dubrovinsky et al., 2009). The UniHead is based on the finite cutting laser head produced by Precitec KG (Germany). The functions of the UniHead are to focus incoming laser light on the sample within the DAC, to provide illumination by white light and observation, and to give access for optical spectroscopic measurements (multiwavelength spectroradiometry, ruby fluorescence measurements, Raman spectroscopy, etc.). For focusing of the 1064 nm laser radiation, the UniHead employs a bending mirror and a set of lenses with up to 80 mm working distance. The position of the mirror and the lenses can be adjusted in order to achieve an optimum (circular) beam shape and its centering with respect to the optical axis of the instrument. Illumination of the sample is achieved due to a built-in high-power LED. For observation of the sample in the DAC and for the process of laser-heating we use a high-resolution GiqE uEye™ (SUXGA, 2048x1536) digital camera.

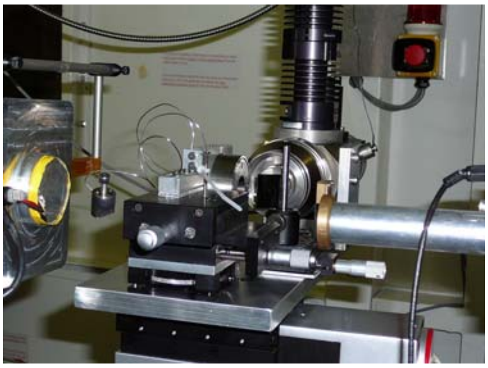

Fig. 1. The UniHead and DAC in "perpendicular" (90^o between the optical axes of the DAC and UniHead) geometry mounted on the goniometer at ID09a beam line.

Due to the modular construction, the portable laser heating system can be used in different modifications – for heating a sample in an alone standing DAC or in a cell coupled directly to UniHead with "normal" (optical axes of the DAC and the UniHead coincide) or "perpendicular" (90^{o} between the optical axes of the DAC and the UniHead, Fig. 1) geometries, with or without spectroscopic or radiospectrometric moduli. In experiments at ID09a beam line we employed UniHead in "perpendicular" geometry (Fig. 1) with a carbon mirror. Glassy carbon used as a substrate for silver coating does not introduce any features into X-ray diffraction patterns. In "perpendicular" geometry, a DAC is moving simultaneously with the UniHead (i.e. with the laser beam) and ω-scans (necessary for single crystal x-ray diffraction experiments) are naturally realised at high temperatures. In the present configuration we realised ω-scans in the range of -30 to 10 degrees. The total mass of the system is about 3 kg and the size of the UniHead with a DAC attached (Fig. 1) is about 25x35 cm. It is important to underline that assembling of the portable laser heating system from components and its full alignment are rather simple and can be made even by a non-experienced user within one hour.

In order to test application of the portable laser heating system for single crystal diffraction studies at ID09a we investigated high-P,T behavior of tungsten single crystals up to 40 GPa and 2500 K and silicate perovskite (Mg,Fe)(Si,Al)O3 at pressures over 75 GPa and 2700 K (Fig. 2).

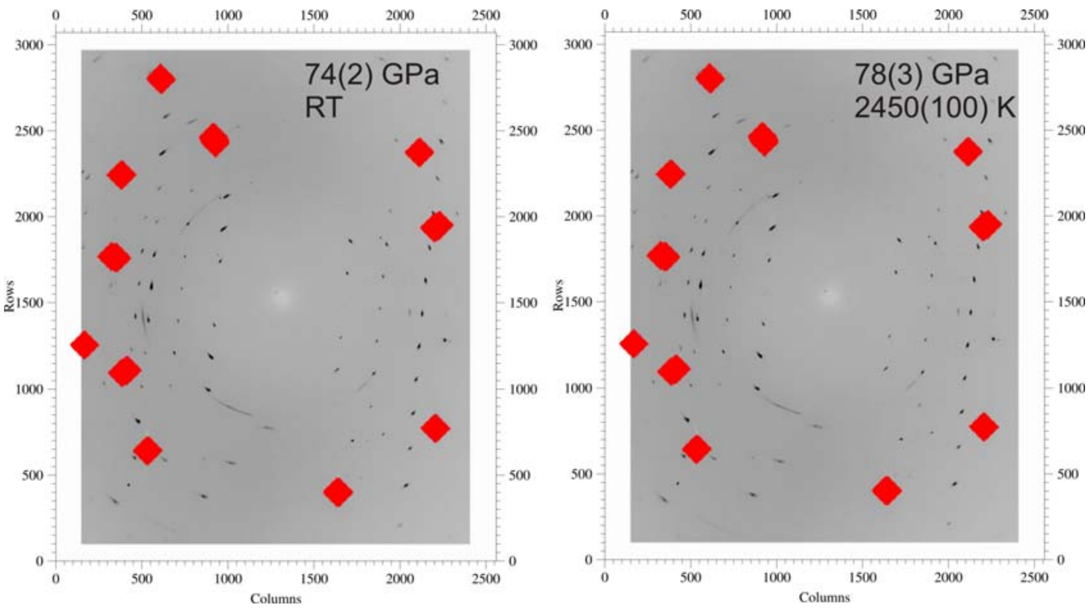

Fig. 2. Example of rotational (40 degree) X-ray diffraction images of silicate perovskite compressed in Ne pressure medium (arcs) collected before (left) and during (right) laser-heating in DAC (diamond peaks are masked).

We conducted a series of experiments on γ-B_{28} at pressures over 120 GPa in laser-heated DACs. We found that at ambient conditions γ-B_{28} is stable up to highest pressures reached in this study. Upon heating at pressures above 95 GPa we observed drastic changes in the diffraction patterns and tentatively assigned them to the appearance of the new phase with the α-Ga structure (Fig. 3). Further experiments are required in order (a) to refine the structure of high-P,T phase; (b) to reveal phase relations with the γ-B_{28} phase; (c) to determine its thermoelastic properties.

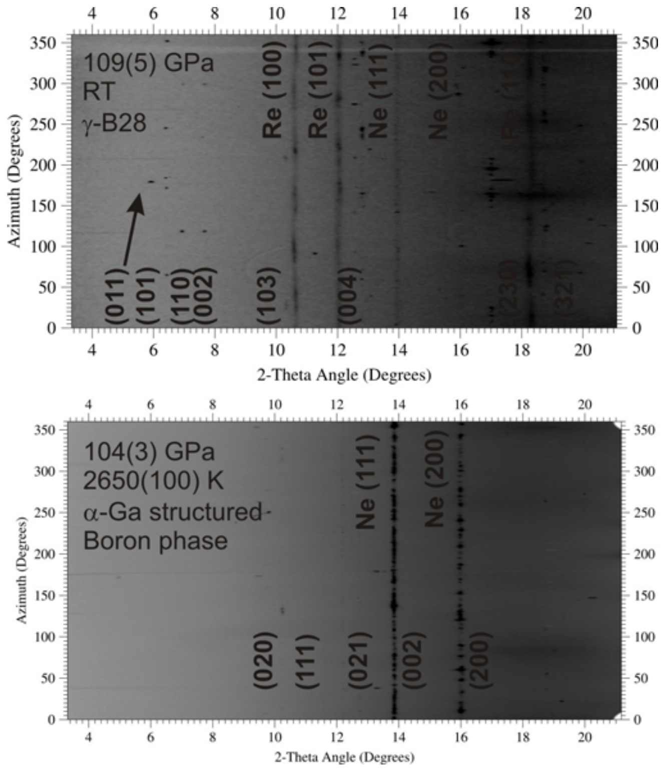

Fig. 3. Examples of diffraction patterns collected in framework of the experiment HS3934.