ESRF	**Experiment title:** High resolution X-ray phase contrast microscopy for in situ study of mechanical stresses in a diamond anvil and failure mechanism under ultra high pressures.	**Experiment number**: MI-1278
Beamline: ID15B	**Date of experiment**: from: 27.10.2017 to: 30.10.2017	**Date of report**: 28.02.2018
Shifts: 6	**Local contact(s)**: Gaston Garbarino	*Received at ESRF:*

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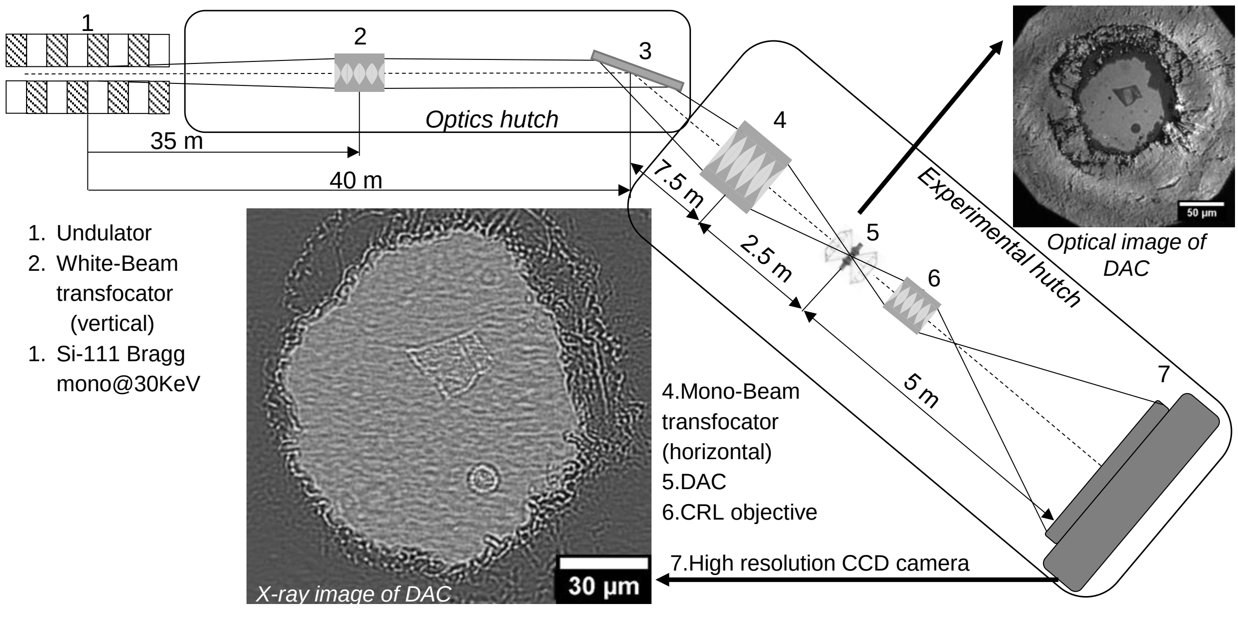
Report:

To studies crystal by X-ray high resolution microscopy at high pressures in the diamond anvils cells the X-ray optics of the ID15 ESRF station was calibrated in order to obtain the standard image with maximum sharpness. The values of the experimental increase, obtained by means of a beryllium composite refractive lens (hereinafter referred to as CRL - Compound Refractive Lenses) was estimated.



The main characteristics of a high-resolution x-ray microscope are:
the energy of the x-ray radiation is 30 keV, (0.41328 Å);
the spatial resolution is 0.306 µm;
a block of X-ray optics consisting of 97 beryllium lenses with a radius of curvature of 50 µm and a physical aperture of 450 µm.
Spatial resolution is **300 nm @ 30 KeV** with X-ray magnification factor x4.8

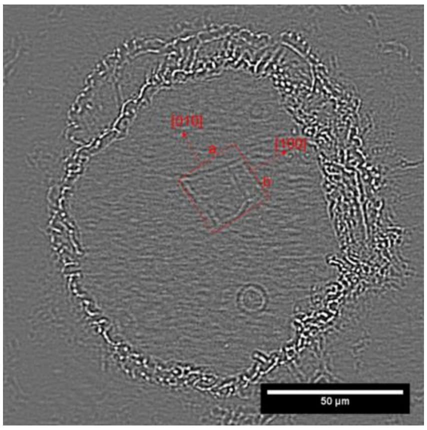
The first test sample is GdFe3(BO3)4

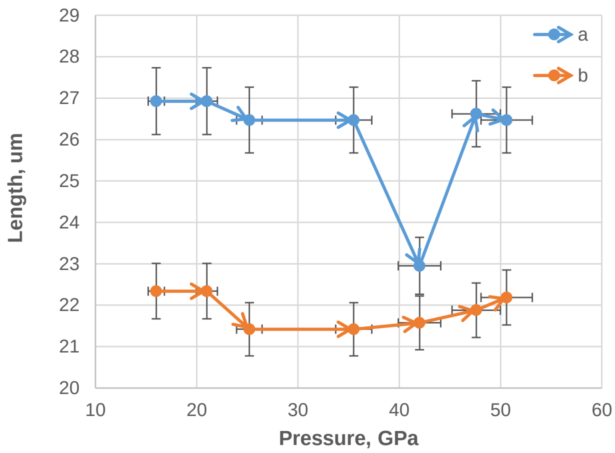
Images of the GFBO (GdFe3(BO3)4) crystal were measured in pressure range from 16 GPa to 51 GPa

The analysis of the images of the object at different pressures was as follows:

1. For each pressure value, the sample-crystal contour was drawn.
2. For each contour, its area in µm^2 was measured.
3. Two orthogonal directions ([100], [010]) were entered on the image of the object.
4. For each contour, the lengths of the rectangle describing the contour along the [100] and [010] directions were estimated.
5. The values of the areas and lengths of the object along the directions [100] and [010] were recorded in the table and the corresponding graphs were plotted.

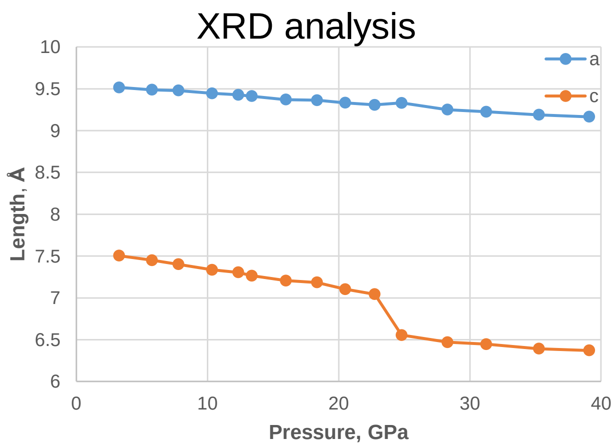
Experimental results





Microscopy analyses indicate the anisotropy of contraction

This anisotropy of contraction is not correlate with XRD data due to the an arbitrary orientation of the crystal in DAC



The second test sample is RbFeSe

The investigation of the behavior of the second sample occurred according to the same algorithm as the first sample,

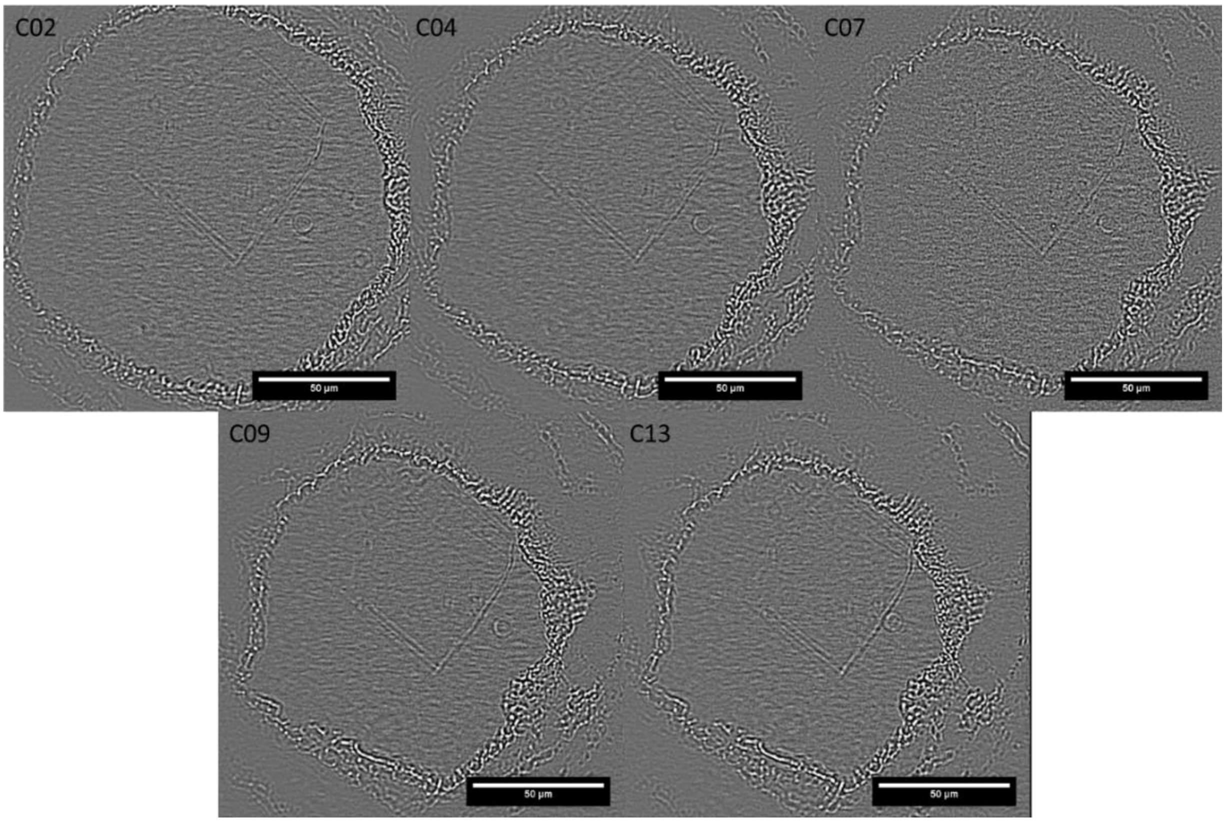


Figure 2: X-ray images of the RbFeSe crystal after Fourier filtration in order of increasing pressure

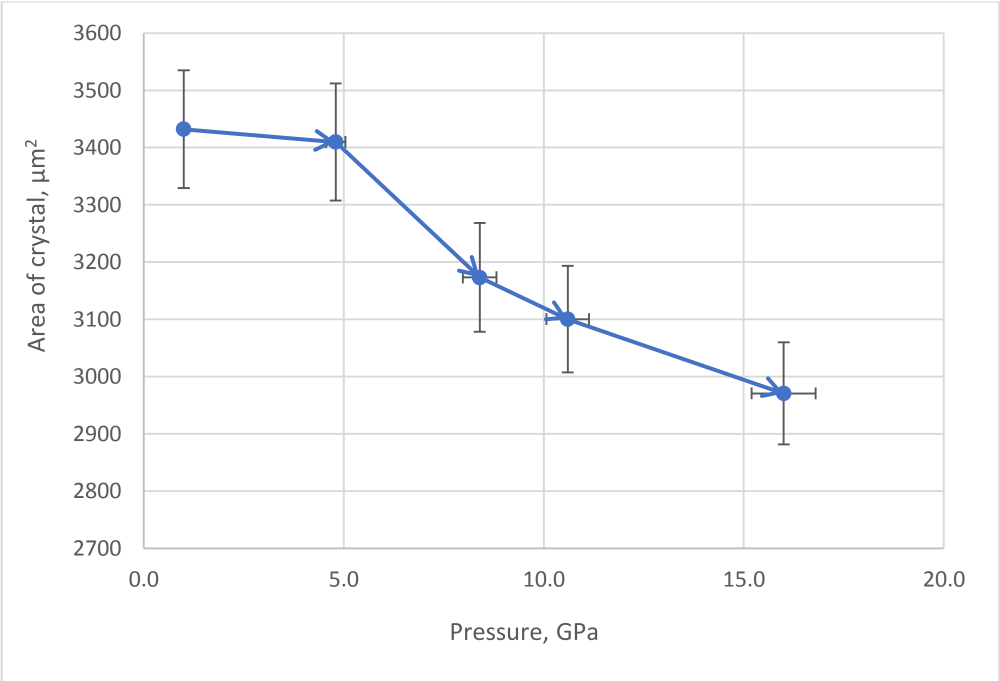


Figure 3: Graph of the change in the area of the RbFeSe crystal from the applied pressure.

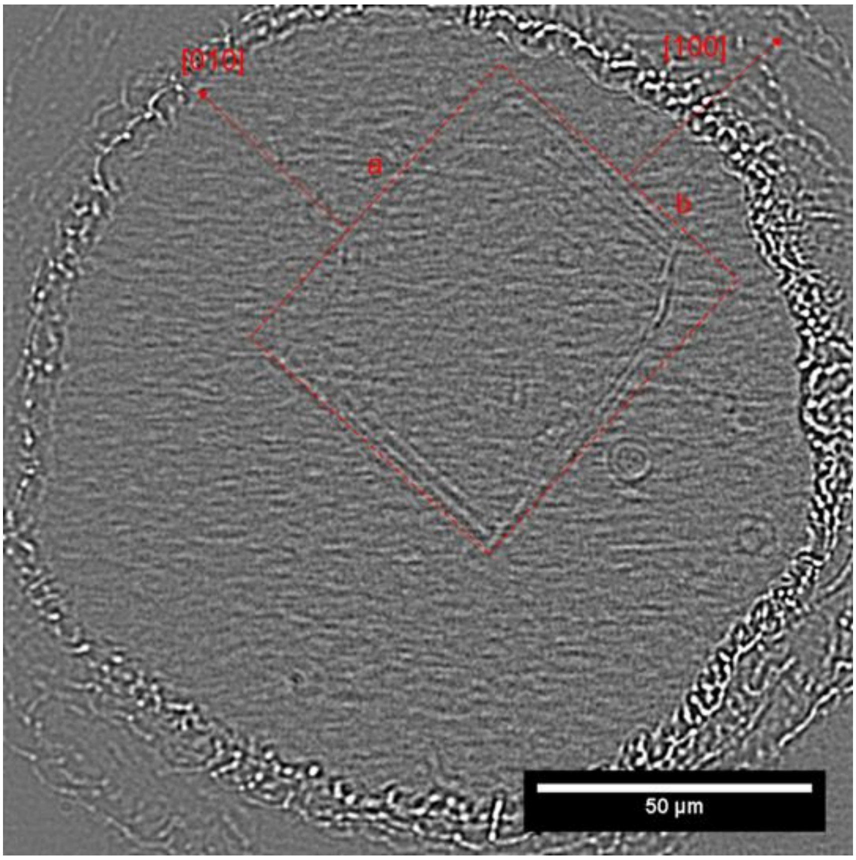
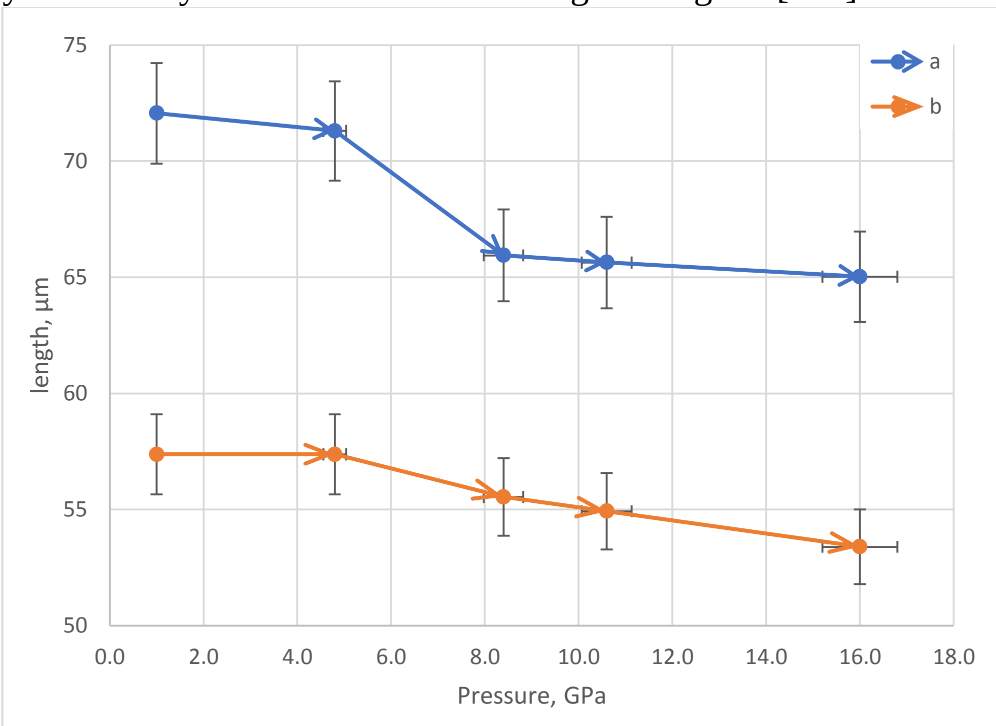


Fig. 4. Selection of [100] and [010] directions in the X-ray image of the RbFeSe crystal sample and estimation of the linear dimensions of *a* and *b*.

Figure 4 shows that with an increase in pressure from 5 GPa, there is a jump in area.When analyzing the changes in linear dimensions, it is seen that the jump at 5 GPa occurs anisotropically and mainly due to dimensional changes along the [100] direction.



Conclusions

We assembled at ID15B station a scheme of high-resolution x-ray microscopy based on a refractive x-ray lens with the following parameters:

- X-ray energy - 30KeV, (0.41328 Å);
- The spatial resolution is 0.306 µm.

The high-resolution x-ray microscope allowed to measure the behavior of crystals in diamond anvils during their loading and release of pressure. Two crystals were studied: GFBO and RbFeSe.

An analysis of the area change with increasing pressure showed that GFBO underwent two changes in area from 22 GPa to 25 GPa and from 35 GPa to 42 GPa. The first jump occurred relatively isotropically, and on the second jump a pronounced anisotropy of the size change was observed - the crystal narrowed only in one direction. When the pressure was released after 40 GPa, an isotropic exponential increase in the area of the crystal was observed, which was similar to a microexplosion with the separation of a part of the crystal.

An analysis of the area change with increasing pressure for the RbFeSe crystal showed that it underwent one jump in the area change: from 5 GPa to 10 GPa. The jump at 5 GPa occurred anisotropically and mainly due to changes in the dimensions along the [100] direction.