ESRF	**Experiment title:** Synthesis and single-crystal X-ray diffraction characterization of novel yttrium nitrides under high pressures up to 175 GPa	**Experiment number**: CH-6473
Beamline: ID11	**Date of experiment**: from: 04.11.2022 to: 07.11.2022	**Date of report**: 14.12.2022
Shifts: 9	**Local contact(s)**: Eleanor Lawrence Bright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Andrii Aslandukov*—Department of Crystallography, University of Bayreuth

Report:

The aims of the proposed experiments were the synthesis of new Y-N compounds at multimegabar pressure, characterization of their crystal chemistry and compressional behavior, tests of theoretical predictions, and search for regularities in polynitrogen chemistry.

A diamond anvil cell, containing a sample composed of two pieces of yttrium embedded into molecular nitrogen, was compressed to 100(1) GPa and laser-heated to 3000(200) K. The precise 2D X-ray diffraction map, collected with a step of 0.5 µm at ID11 ESRF beamline from the bigger piece of yttrium after heating, revealed the crystallization of novel phases and allowed to pinpoint the location of crystallites most appropriate for single-crystal X-ray diffraction measurements (Fig. 1a). High-quality synchrotron single-crystal X-ray diffraction data were then collected from the sample. The subsequent crystal structure solution and refinement revealed the formation of two novel yttrium nitrides with chemical formulas of YN_6 and Y_2N_{11}. The distribution of the YN_6 and Y_2N_{11} phases shown in the 2D X-ray diffraction map (Fig. 1b) demonstrates that the heated area consists of many tiny crystallites and there is no obvious chemical gradient in the distribution.

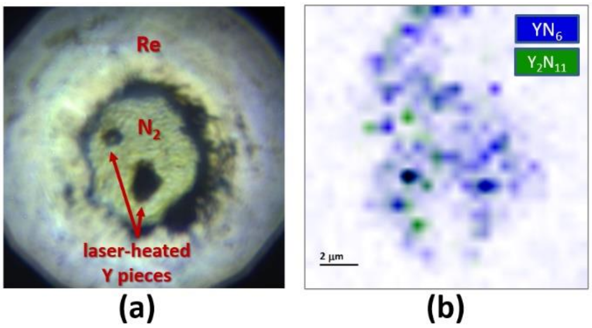

Figure 1. (a) Microphotograph of the sample chamber. (b) 2D X-ray diffraction map showing the distribution of the two yttrium nitrides phases within the heated sample.

High-pressure synchrotron single-crystal X-ray diffraction revealed that the crystal structures of YN_6 (space group *C*2/*m*) and Y_2N_{11} (space group $P6_222$) feature a unique organization of nitrogen atoms—a previously unknown anionic N_{18} macrocycle and a polynitrogen double helix, respectively (Fig. 2).

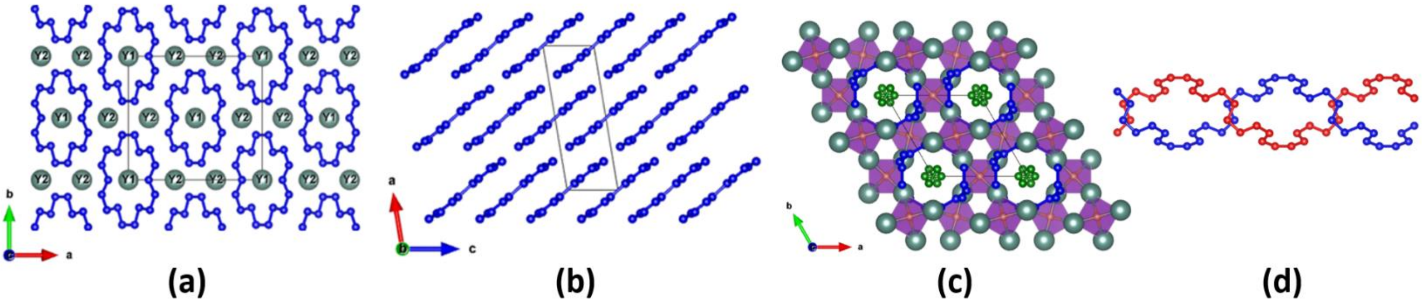

Figure. 2. Crystal structure of YN_6: a view of the crystal structure (a) along the *c*-axis and (b) along the *b*-axis, yttrium atoms are omitted. **Crystal structure of Y_2N_{11}:** (c) a view of the crystal structure along the *c*-axis, (d) a double helix built of two polynitrogen chains running along the *c*-direction around the 6_2 screw-axis. All Y atoms are greenish, all other colors correspond to N atoms.

The discovery of such compounds encourages further exploration of the remarkable inorganic chemistry of polynitrides. Moreover, the ability of nitrogen to form such structural units shows that perhaps humanity is on the verge of opening a new branch of chemistry—nitrogen organic chemistry under ultrahigh pressure.

The fruitful results of this experiment are published in a high-impact scientific journal:

A. Aslandukov, F. Trybel, A. Aslandukova, D. Laniel, T. Fedotenko, S. Khandarkhaeva, G. Aprilis, C. Giacobbe, **E.L. Bright**, I.A. Abrikosov, L. Dubrovinsky, N. Dubrovinskaia. (2022). Anionic N_{18} Macrocycles and a Polynitrogen Double Helix in Novel Yttrium Polynitrides YN_6 and Y_2N_{11} at 100 GPa. *Angewandte Chemie International Edition*, 61(34), e202207469