EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

ESRF	**Experiment title:** Kinetics and dynamics of fast transformations at extreme conditions. Combined XFEL and synchrotron study.	**Experiment number**: CH-6476
Beamline: ID27	**Date of experiment**: from: 07.09.2022 to: 09.09.2022	**Date of report**:
Shifts: 6	**Local contact(s)**: Mohamed Mezouar	*Received at ESRF:*

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Report:

The aim of the experiment was to study products of chemical reactions, which kinetics were previously studied at the HED station of EuXFEL. Typical experiment at ID27 included X-ray diffraction mapping of the heated / X-ray exposed areas of various samples (mainly metal nitrides) and collection of single-crystal X-ray diffraction patterns at the selected points of interest. One of the examples of such mapping is shown on the Figure 1. In this experiment the kinetics of temperature-induced decomposition of sodium azide NaN_3 was studied.

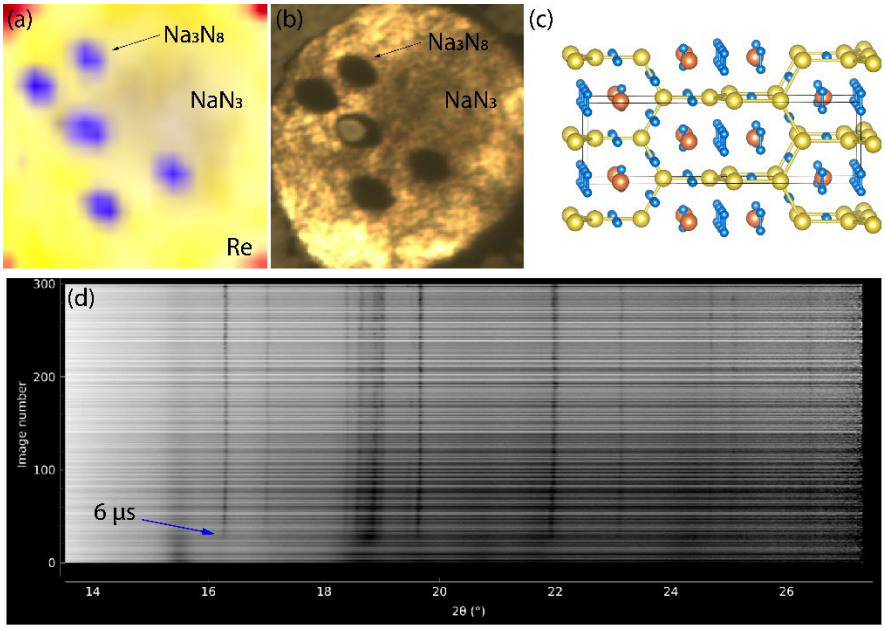

Figure 1. (a) X-ray diffraction map of the NaN_3 sample previously studied at EuXFEL. (b) Optical image of the sample chamber. (c) Crystal structure of the product (Na_3N_8) identified by single-crystal X-ray diffraction. Orange and yellow balls represent sodium atoms, while blue atoms show the positions of nitrogen atoms (d) Example of time-dependent diffraction measurement at EuXFEL, showing the onset of transformation.

EuXFEL setup does not allow rotation of diamond anvil cells and also does not allow the collection of high-quality diffraction data, which is necessary for the identification of new phases. In this regard, the combination with the synchrotron radiation is essential for the full characterization of the system. In case of the NaN_3 sample, we could find out that the product of its decomposition is a compound with the chemical formula Na_3N_8. It has an unprecedented structure type. Two symmetry-independent sodium atoms occupying Wyckoff sites 4a and 8e form a substructure isostructural to α-$ThSi_2$. Two nitrogen atoms N1 and N2 occupy Wyckoff sites 16h an 16f respectively and form N1-N1 and N2-N2 dinitrogen dumbbells with d(N1-N1) = 1.147(3) Å and d(N2-N2) = 1.149(3) Å at 28 GPa.

The aim of the experiment has been achieved however, due to the technical problems of the EuXFEL, the total number of screened DACs was smaller than initially expected. Mapping and SCXRD was performed on DACs containing NaN_3, Ta+N_2, W+N_2, Fe+NH_3, Fe + N_2. The results of the experiments are currently being prepared for publications.