EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Key amorphous intermediates in thermal decomposition of multimetallic coordination compounds	**Experiment number**: CH-5683
Beamline: ID15A	**Date of experiment**: from:03.02.2021 to:06.02.2021	**Date of report**: 16.02.2022
Shifts: 6	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Experiment has been performed in the mail-in mode.

Dr. Kirill Yusenko (BAM, Berlin, Germany) and Dr. Sergey Gromilov (Institute of Inorganic Chemistry, Novosibirsk, Russia) were allocated as remote users.

Report:

Due to CORONA regulations, our experiment has been performed using mail-in mode. Samples were sent to ESRF and measured by beammline scientist. Experimentalists participated remotey.

In the frame of our experimennt we measured thermal decomposition of $[Pd(NH_3)_4]_2(Mo_8O_{26})$ in a hydrogen flow. Several experimets were performed using 5 and 10 K/min heating ramp up to 1000 °K. PDF curves includinng air and empty cappilaries were collected to substract background. Mixtures of $[Pd(NH_3)_4]Cl_2$ and $(NH_4)_4(Mo_8O_{26})$ were also measured as references to characterise mechannistic aspects of thermal decomposition. Several amorphous and semi-crystalline inntermediates specific for double complex compounds were obtained. We show that inn hydrogen flow Mo and fcc-structured Pd_xMo_{1-x} alloys can be obtained. In inert flow, minnor admixture of Pd_2Mo_3N can be detected.