# Experimental report

02/04/2021

**Proposal:** 5-22-793 **Council:** 4/2020

**Title:** Neutron diffraction study of bismuth nanowires incorporated within powder ZSM-12 and ZSM-22 zeolites

**Research area:** Materials

**This proposal is a new proposal**


**Main proposer:** Julien HAINES

**Experimental team:**

**Local contacts:** Thomas HANSEN


**Samples:** ZSM-22
ZSM-12

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D20 | 1 | 1 | 26/09/2020 | 27/09/2020 |


## Abstract:

Bismuth is the one of the best thermoelectric materials for Peltier cooling,
we hope to improve its properties by reducing its dimensionality and by designing
bismuth nanowires for which large enhancement of the thermoelectric properties have been predicted.
Porous materials are ideal for designing a network of nanowires.
The objective of this experiments is to confirm successful synthesis of two zeolite/Bi composites with
1D pore structures (ZSM-22 and ZSM-12) and to determine the structure of Bi nanowires within the zeolites
and how the structures of the zeolites are modified by the incorporation of bismuth. Neutron diffraction is
suitable for such bulk sample due to its high penetration depth, which will permit to probe the whole sample
despite of the presence of residual bismuth outside the zeolite.
25mm3 samples will be prepared from zeolite powder and Bi at 6 GPa and 300Â°C in a belt press for this experiment.

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The zeolite-Bi mixture are prepared in Glovebox, put in the PTFE capsules (6 mm Ø) and sealed in vacuum ampoule. $SiO_2$:Bi ratio is circa 1:2 /w. The resulting mixture is a powder-like material very similar to the original zeolite powder, slightly grey for the presence of Bi.
The capsules are pretreated at 310°C for 1h before the HP experiment.
Two samples of ZSM-22 + Bi treated in the press "Conac28" with insulating material (in contact with the PTFE capsule) of calcite.

- **TP184**: heated to 400 °C at 1 GPa and then compressed up to 5 GPa, 1h waiting time and then quenched.
- **TP185**: heated to 320 °C at 1 GPa and then compressed up to 3.7 GPa then heated up to 400°C then compressed until 5 GPa and then quenched.

Two samples of ZSM-12 + Bi treated in the press "Conac40" with insulating material (in contact with the PTFE capsule) of BN.

- **TP186**: ZSM-12 + Bi: heated to 320°C at 1 GPa and then compressied up to 3.7 GPa and then heated up to 400°C then compressed until 5 GPa + quenching.
- **TP189**: ZSM-12 (2eme source) +Bi: heated to 320°C at 1 GPa and then compressied up to 3.7 GPa and then heated up to 400°C then compressed until 5 GPa + quenching.

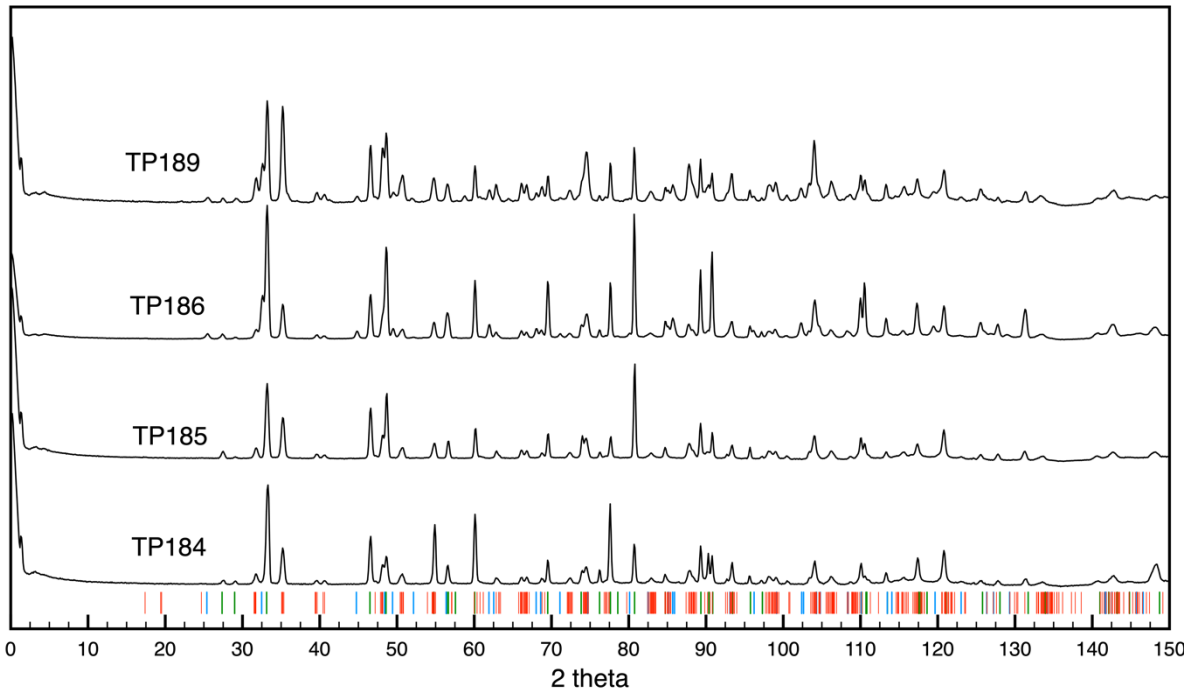


**Figure 1.** Neutron diffraction pattern of the zeolite samples after the P-T treatment. The pattern of Bi (green), quartz (blue) and coesite (red) are also reported.

The neutron diffraction shows the presence of silica condensed phases such as quartz and coesite, and the absence of the original zeolites. The conversion of the zeolites is possibly due to the incomplete filling of the cavities which are collapsed when the pressure is applied.