

Experimental report

03/10/2024

Proposal: EASY-1205

Council: 4/2023

Title: Characterisation of new 1 GPa cell, FC770 vs Glycerol-D8/D2O and performances of XtremeD with PE cell

Research area: Methods and instrumentation

This proposal is a new proposal

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Experimental team:

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Samples: Fe₂O₃

Instrument	Requested days	Allocated days	From	To
XTREMED	96	96	18/09/2023	22/09/2023

Abstract:

Together with PSI, we have developed a double-wall pressure cell which should ease very much experiments up to 1 GPa. The objective is to characterise the cell (transmission, absorption, scattering) and compare it with the CuBe₂ clamp cell generally used on this pressure range.

The pressure transmitting media FC-770 is not anymore commercially available and we would like to compare the performances of this liquid with a mixture of glycerol-D₈ / D₂O. According to the literature, this mixture should be a very good replacement.

Finally, we would like to compare the performances of the new diffractometer XtremeD when using a Paris-Edinburgh press. For his purpose, we will work in the same conditions as during the DIR-146 experiment (Fe₂O₃ sample, TiZr gaskets, methanol/ethanol pressure transmitting media).

Here is the foreseen task list:

- PE measurements at 0 and 6 GPa at 300 K (1.3 and 2.4 Å)
- double-wall cell measured at 1.3 Å empty at 300 K, filled with FC-770 at 1 GPa / 2 K, glycerol mixture without sample at 1 GPa / 2 K, filled with sample and FC-770 and then glycerol mixture at 0 and 1 GPa/RT
- CuBe₂ cell measured at 1.3 Å with Fe₂O₃ sample at 1 GPa/RT for comparison

Experiment report EASY-1205

Characterisation of new 1 GPa cell, FC-770 vs Glycerol-D8/D₂O and performances of XtremeD with PE cell

Together with PSI, we have developed and built double-wall 1 GPa TAV6/CuBe₂ pressure cell which should ease very much experiments up to 1 GPa. The objective of this experiment was:

- to characterise this cell and compare it with the CuBe₂ clamp cell generally used on this pressure range.
- to compare the performances of the FC-770 pressure transmitting media which not commercially available anymore with a mixture of glycerol-D8/D₂O.

We present in figure 1 the signal obtained with an empty pressure cell and with the same cell filled with FC-770 or glycerol-D8/D₂O, and in figure 2 the diffraction pattern obtained with Fe₂O₃ powder.

The cell scatters neutrons a lot in the 60 - 100° range and is not appropriate to powder diffraction. We had to remove this range to refine the data properly (figure 2). However, the new TAV6/CuBe₂ cell will be preferable to a CuBe₂ clamp on INS spectrometers. With much less CuBe₂ in the beam, the signal/background ratio will necessarily be improved.

Glycerol-D8/D₂O creates a 0 to 40% additional background compared to FC-770, except at low angle where FC-770 presents a broad peak associated to local ordering. The absence of additional Bragg peak confirms that a 50/50 mixture is sufficient up to 1 GPa and down to 10 K.

Figure 3 shows the data collected with the PE press in its cryostat. Clearly, the diffraction pattern is much cleaner and easier to refine ($\chi^2 \approx 0.15$ instead of 2.05).

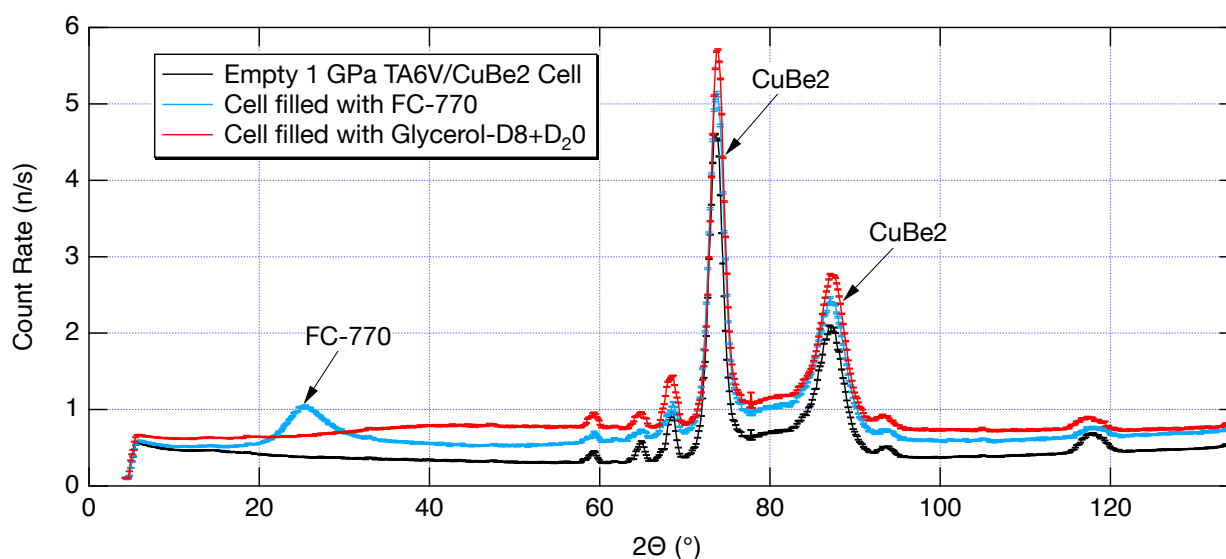


Figure 1: Comparison of FC-770 and Glycerol-D8/D₂O and scattering due to the TAV6/CuBe₂ pressure cell.

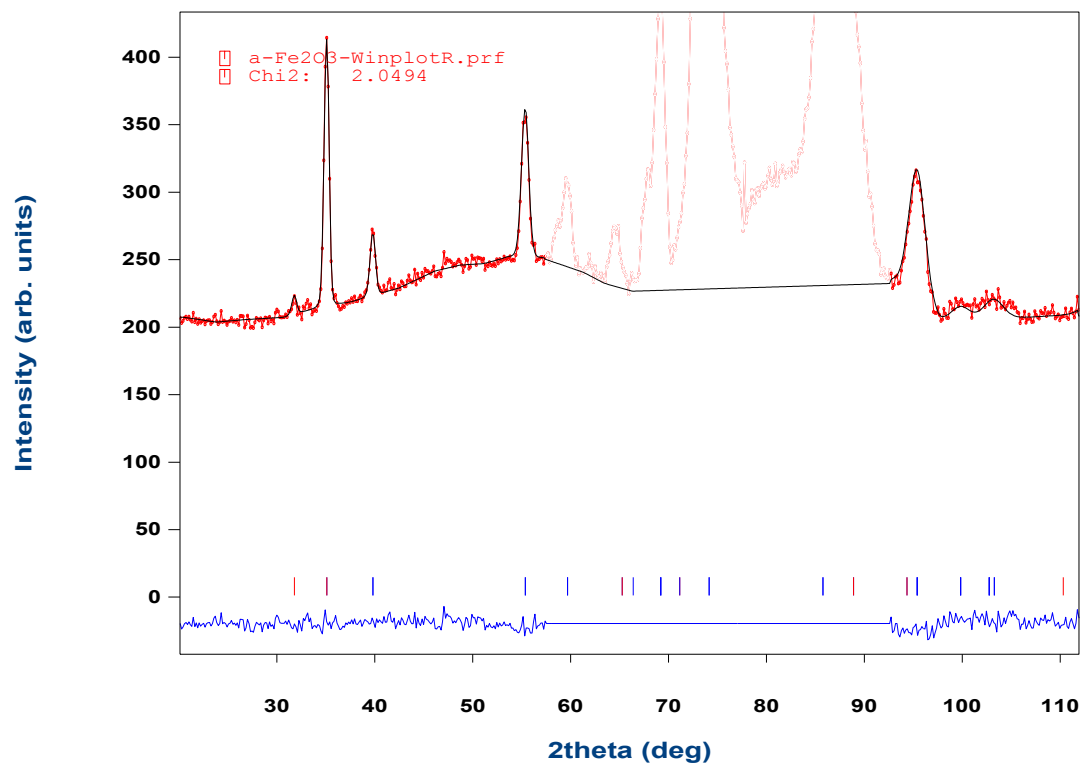


Figure 2: Fe₂O₃ in the CuBe₂ cell using glycerol-D₈/D₂O at 200 K and 10 kbar.

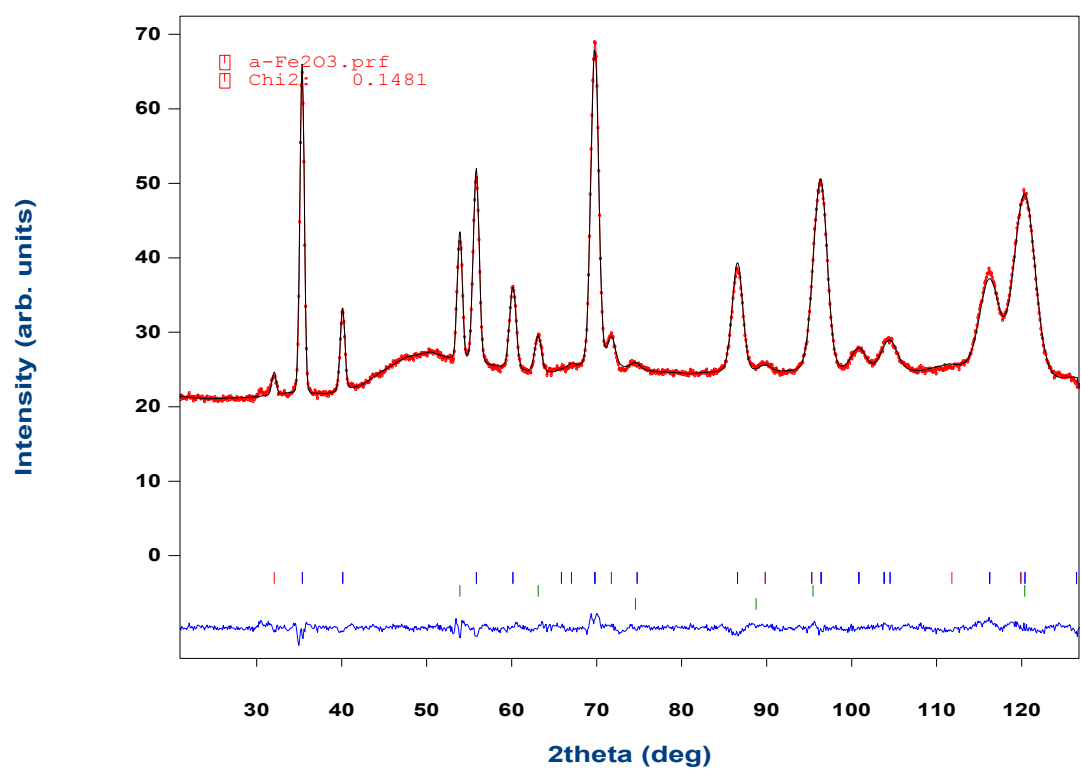


Figure 3: Fe₂O₃ in the PE press using d-EtOD-MeOD as pressure transmitting media and Pb as internal pressure calibrator.