

**Experiment title:**Phonon-phonon interactions in the metallic transition metal oxide  $\text{ReO}_3$ **Experiment****number:**

HS-4736

**Beamline:**

ID09

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12

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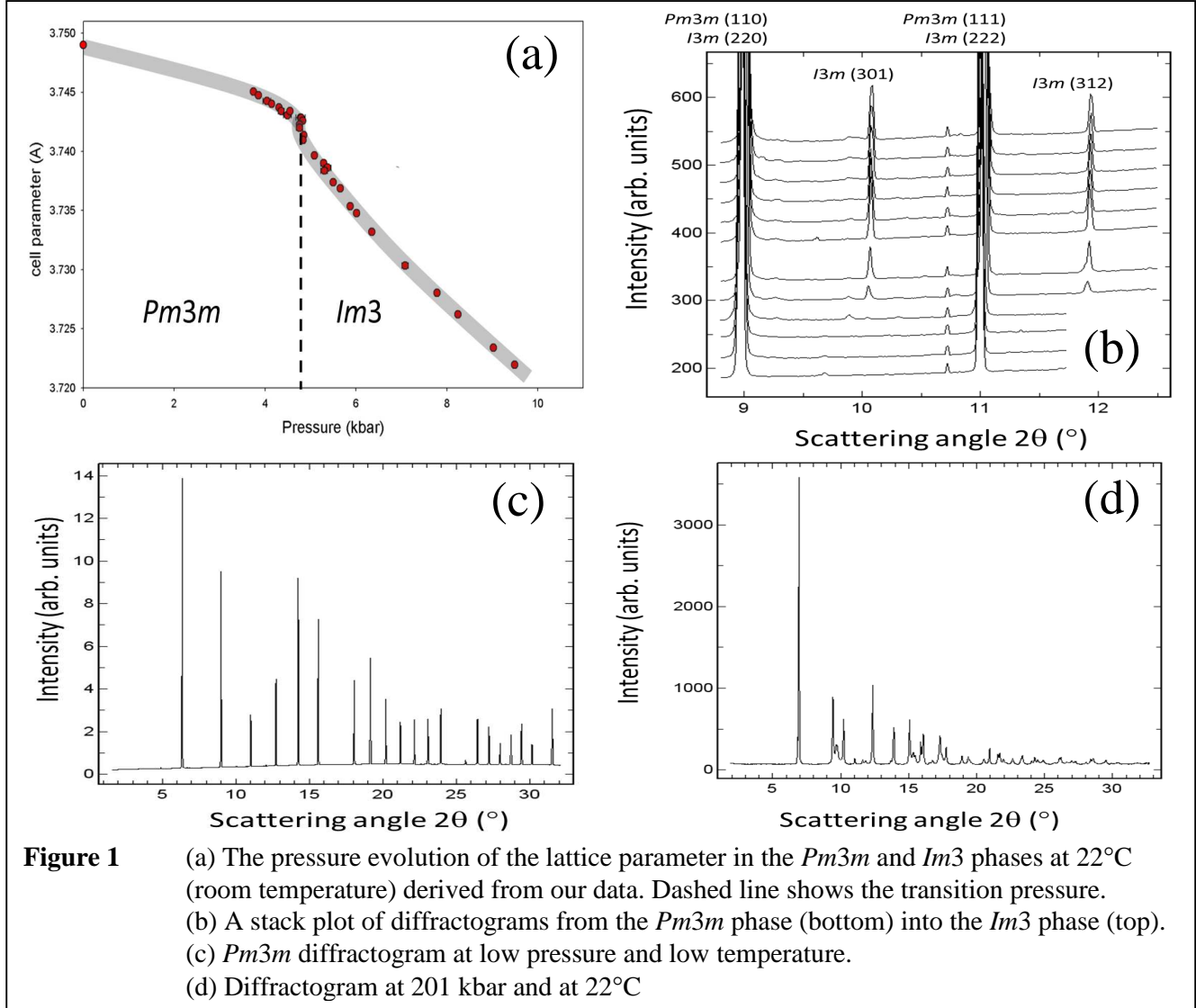
**Report:**

The aim of this experiment was to map out the high pressure and high temperature (HPHT) part of the phase diagram of  $\text{ReO}_3$  using powder X-ray diffraction (PXD). In particular we were interested in the behaviour of the lowest pressure phase transition, which at 22°C takes place at about 5 kbar. We measured up to a pressure of 227 kbar and a temperature of 283°C.

During the beamtime we used 4 different diamond anvil cell (DAC) loadings of  $\text{ReO}_3$  powders, and the pressures and temperatures that we measured at with each DAC are detailed below. We used helium, neon and silicon oil as different pressure transmitting media in different DACs, and fluorescence from both ruby and a Sm-doped crystal was used to measure the pressure at each point. The X-ray wavelength used was 0.414652 Å. All  $\text{ReO}_3$  powders were grown using chemical vapour transport, which produced many single-crystal samples of various dimensions which were then ground into a suitable powder.

DAC	Description of trajectory through P-T space
1	All data at 22°C, compression from 3 kbar to 227 kbar
2	Firstly heated at 1 kbar from 22°C to 283°C, then at 283°C the pressure was increased up to 227 kbar. Temperature was decreased to 98°C while maintaining the pressure, and then the pressure reduced to 27 kbar
3	Started at 22°C at a pressure below 1 kbar and increased pressure to 6 kbar. Decompression was then performed to below 1 kbar. Subsequent heating to 97°C and up to 22 kbar in pressure. Decompression performed once again, and finally heated to 188°C and up to 10 kbar in pressure.
4	Heated at constant pressure (1 kbar) from 22°C to 183°C, then cooled to 29°C. Temperature was then increased again to 189°C. At 189°C the pressure was increased to 160 kbar, and finally the temperature was lowered to 28°C at 160 kbar.

In Figure 1(a) the ‘compressibility collapse’ transition, first observed by Schirber and Morosin in 1979, can be observed. The very different compressibilities of the 2 phases allows us to accurately determine the pressure at which the transition occurs. Figure 1(b) is a stack plot showing how the diffraction pattern materially changes upon passing from the  $Pm3m$  to the  $Im3$  phase from bottom to top. Additional Bragg peaks (in this  $2\theta$  range we show the (301) and (312)) exist in the  $Im3$  structure. These exist only due to a change in the positions of the oxygen atoms and are therefore weak - but clearly visible - in PXD data.



Figures 1(c) and 1(d) show diffractograms measured at 22°C, the low pressure  $Pm3m$  phase in Figure 1(c) and an as yet undermined phase at 210 kbar in Figure 1(d). The data in Figure 1(d) cannot be explained by any high pressure structures suggested by previous studies.

In summary, we have mapped out the HPHT part of the phase diagram in  $ReO_3$  above room temperature up to over 200 kbar. We observe that the P-T dependence of the transition line between the  $Pm3m$  and  $Im3$  phases departs dramatically from the linearity observed previously below room temperature. We also find that several of the diffractograms that we observe at higher pressures are inconsistent with the published work and through the course of our careful data analysis (currently on-going) we aim to provide definitive information about the true structure of these high pressure phases.