	Experiment title: Compressibility and structural response to pressure study of several rare-earth pyrochlore iridates $A_2Ir_2O_7$ (A = Nd, Sm, Er)	**Experiment number**: HC-4856
Beamline: ID15B	**Date of experiment**: from: 03/05/2022 to: 07/05/2022	**Date of report**: 08/08/2022
Shifts: 12	**Local contact(s)**: GARBARINO Gaston	*Received at ESRF:*

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Report:

The single-crystal and powder diffraction experiments were performed on selected rare-earth $A_2Ir_2O_7$ iridates using ID15b diffractometer and diamond anvil pressure cells (DAC) to determine their compressibilities and evolutions of structural parameters with applied pressure. During the span of 4 days (12shifts), 4 pressure cells (2 DACs at constant room temperature and 2 DACs at constant 20 K) enabled measurements of a total of 11 samples (6 single crystals, 5 powders). We acknowledge the local contact for his kind help and ability to fit 2 or 3 samples to the sample space. As we initially expected to measure only one sample per cell, this provided us with more flexibility, and we decided to measure more pressure points per run (prolonging each run to 1day, up to 60-100 pressure points per run), as well as to measure selected samples as both polycrystals and single crystals. Of the 4 cells, 3 reached the 20 GPa safe upper limit successfully and 1 reached a maximum of 17.5 GPa (gasket collapsed, we were unable to continue to higher pressures), still enabling to draw complete conclusions on investigated samples compresibilities. The total amount of data gathered exceeded our expectations. The data are of sufficient quality for presentation and publication.

The measured data were analyzed: We employed the Dioptas program for initial masking and integration of both powder and single crystal data. The integrated intensities were refined, providing us with lattice evolution for all studied analogues. In all the analogues, the pyrochlore structure remained stable up to the highest pressures, that is, no structural transitions were observed, as expected. The data were subsequently fitted with different equations of state (Murnaghan, Birch-murnaghan, and Vinet). For example, Figure 1 shows the integrated intensities and the lattice evolution of the $Pr_2Ir_2O_7$ powder and how all three EOS fits behave very similarly in the measured pressure range. Measurements details, investigated samples and temperatures of the

measurements are listed in Table 1, which also contains the preliminary bulk moduli gathered from the EOS fitting of the lattice evolution.

The single crystal diffraction images were also analyzed with suitable software – CrysAlis for peak hunting and indexation, and Jana for single crystal refinement. However, our attempts to refine the single crystal data on-site proved difficult. Despite having high-quality images that also clearly showed the high quality of measured single crystals, the data analysis software had trouble refining the data with the pyrochlore structure (or any different structure recommended by the software). One of the proposed explanations blamed the issue of parasitic diamond reflections, with a unit cell parameter and symmetry that resulted in significant overlap with our reflections ($a_{\text{iridate-pyrochlore}} \approx 2\sqrt{2} \cdot a_{\text{diamond}}$). A clear solution has not been found yet, making our efforts to analyze the single crystal data difficult (and leading us to intially process the data as a simple powder-like integration for lattice parameter extraction). In order to solve this issue, we've reached out to obtain room temperature single-crystal diffraction data from a 4-circle goniometer, without a pressure cell, to help troubleshoot.

Data processing is still ongoing, with the hope of extracting more details about the structural evolution with pressure, such as subtle bond distance and angle changes, in both our powder and single crystal refinements. The data are planned for publication in the near future and will be invaluable in the interpretation of pressure-dependent magnetic and electronic property measurements.
This work will also be included in the PhD. thesis of Daniel Staško.

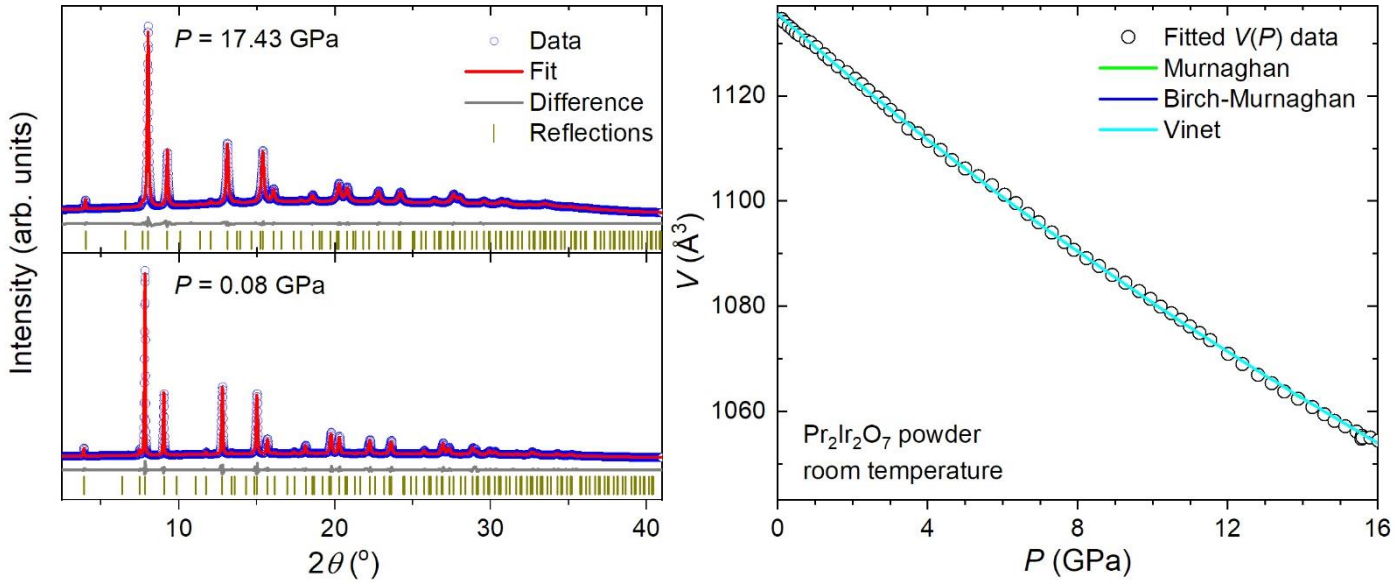

Figure 1: Integrated diffraction data with corresponding refinement fit of the $Pr_2Ir_2O_7$ powder at ambient temperature (left). Resulting volume evolution with pressure and preliminary EOS fits (right).

Table 1: Representative results from all measured samples. Each of the 5 analogues was measured at room temperature and 20 K, either in single crystal or powder form. The preliminary bulk moduli were determined from the EOS fits.

K_0 (GPa)	Ambient Temperature		Low Temperature (20 K)	
	powder	SC	powder	SC
$La_2Ir_2O_7$		150.4		173
$Pr_2Ir_2O_7$	178		254	
$Nd_2Ir_2O_7$	163.3		177.5	174.5
$Sm_2Ir_2O_7$		150.5		149.5
$Er_2Ir_2O_7$	187			141.5