In order to study the phase and structural behaviour in the La(Pr)AlO$_3$–TbAlO$_3$ pseudo-binary systems series of La(Pr)$_{1-x}$Tb$_x$AlO$_3$ samples with $x$ in the range of 0.1 – 0.9 were prepared from the oxides La$_2$O$_3$, Pr$_2$O$_3$, Tb$_2$O$_3$ and Al$_2$O$_3$ by a combination of solid state reaction and arc melting in Ar atmosphere. The crystal structures and of the solid solutions La$_{1-x}$Tb$_x$AlO$_3$ and their thermal behaviour in a wide temperature range of 12-1173 K have been investigated by using high-resolution powder diffraction applying synchrotron radiation (beamline B2, HASYLAB at DESY) and DTA/DSC methods. All crystallographic calculations (refinements of the lattice parameters as well as full profile structure refinements) were performed by means of the Windows version of the Crystal Structure Determination program package WinCSD.

From the results of the XRD phase and crystal structural analysis it was established that two kinds of solid solutions with rhombohedral and orthorhombic structures exist at ambient temperature. A wide immiscibility gap exists between these two perovskite-type phases. All lattice parameters decrease with temperature, mounting the MDAC to the cold finger of a specially designed helium flux cryostat (T$_{\text{min}}$ = 5K). In both cases, the pressure is measured in-situ (fluorescence of a ruby crystal). Data collection for structure determination (refinement) is possible thanks to the use of 2D detectors (Rayonix SX-165 or XPAD pixel detector[3]) and the implementation of the rotation method for data collection.

Several examples will be presented to illustrate the possibility of the beamline for HPC studies. They demonstrate that although CRISTAL is not specialized in high-pressure studies, HPC can be routinely and successfully performed in conditions analogous to those found at dedicated beamlines in other synchrotron facilities. In both cases, the pressure is measured in-situ (fluorescence of a ruby crystal). Data collection for structure determination (refinement) is possible thanks to the use of 2D detectors (Rayonix SX-165 or XPAD pixel detector[3]) and the implementation of the rotation method for data collection.

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Understanding the pressure effects on condensed matter is a growing field of interest. Pressure adds a new thermodynamic dimension to crystal-structure analyses: exploration of phase diagrams and phase transitions, structure-property relationships, investigation of higher-energy state of biomolecules... Therefore, High Pressure Crystallography (HPC) is a unique and powerful technique to reach a deeper understanding of matter at the atomic scale [1]. A large variety of materials (powders or single crystals) are concerned in fields as diverse as geophysics, biophysics, physics or chemistry. Furthermore, the availability of high brilliance synchrotron sources and the development of dedicated setups at diffraction beamlines have allowed one to study more complex samples (e.g. proteins), more smaller samples (few microns), and to reach higher pressure (>100 GPa). In that context, we have recently implemented a HPC setup on the CRISTAL beamline at SOLEIL synchrotron[2].

CRISTAL is a general-purpose crystallography undulator based. One of its three diffractometers can accommodate pneumatic or Membrane Diamond Anvil Cell (MDAC) for powder or single crystal samples. Pressure studies can be realized at ambient temperature and more recently at low temperature, mounting the MDAC to the cold finger of a specially designed helium flux cryostat (T$_{\text{min}}$ = 5K). In both case, the pressure is measured in-situ (fluorescence of a ruby crystal). Data collection for structure determination (refinement) is possible thanks to the use of 2D detectors (Rayonix SX-165 or XPAD pixel detector[3]) and the implementation of the rotation method for data collection.

Keywords: pressure; diffraction; diamond anvil high-pressure apparatus


Keywords: perovskites, crystal structures, phase transitions