



ISC₀C

International School of Crystallography

Director: Sir Tom Blundell, FRS FMedSci



49th Course

**High-pressure crystallography:
status artis and emerging opportunities**

Erice, Italy • 27 May - 5 June 2016

Course Directors: Francesca P.A. Fabbiani • John B. Parise • Malcolm Guthrie

**Programme,
Lecture Notes & Poster Abstracts**

Ettore Majorana Foundation and Centre for Scientific Culture
President and Director: Professor Antonino Zichichi



High pressure behaviour of $\text{AlPO}_4\text{-5}$ in penetrating/ non penetrating pressure medium

Davide Comboni¹, P. Lotti², G.D. Gatta¹, L. Pastero³

¹Dip. di Scienze della Terra, Univ. degli Studi di Milano, Milan, Italy

²Elettra, Sincrotrone Trieste S.c.P.A., Trieste, Italy

³Dip. di Scienze della Terra, Univ. degli studi di Torino, Turin, Italy

Aluminophosphate are objects of a growing research interest due to their potential technological and industrial applications [e.g 1,2]. Their large channels serve as ideal host for organic compounds and small polymers. Among those, $\text{AlPO}_4\text{-5}$ is a synthetic zeolite characterized by an open-framework of (P,Al) O_4 tetrahedra. The tetrahedra are connected to form six- and twelve-membered rings, in such a way that a large channel ($\text{O} \sim 7.3\text{\AA}$), parallel to the [001] direction, occurs. Klap et al. [3] underlines that every crystal of $\text{AlPO}_4\text{-5}$ is built up by three different microdomains, in which the positions of the framework oxygen atoms are slightly different; the main effect of the structural disorder is the very large anisotropic displacement parameters of the framework oxygens. We performed two *in situ* single-crystal synchrotron X-ray diffraction experiments using both penetrating (*methanol:ethanol:H₂O mix, m:e:w*) and non-penetrating (*silicon oil*) pressure media [4]. The structure refinements showed that: 1) for compression in m:e:w mix, H_2O molecules are absorbed at low- P regime, forming a H_2O -network by H-bonding interaction; 2) the elastic parameters of the super-hydrated $\text{AlPO}_4\text{-5}$ are different if compared to the one compressed in silicon oil; 3) the structural deformation mechanisms of super-hydrated and regular $\text{AlPO}_4\text{-5}$ are different; 4) evidence of a incommensurately modulated structure occur (according to [3]), and there is an evolution of the non-Bragg reflections with pressure.

The author acknowledges the Italian Ministry of Education, MIUR-Project: "Futuro in Ricerca 2012 - ImpACT- RBF12CLQD".

[1] Tang Z.K. et al. Applied Physics Letters 1998; 73, 2287-2289.

[2] Yang W.S. et al. Microporous and mesoporous materials 2016; 219,87-92.

[3] Klap G.J. et al. Microporous and mesoporous materials 2000; 38, 403-412.

[4] Gatta, G.D. Microporous and Mesoporous Material 2010; 128, 78-84.

Keywords: zeolites, $\text{AlPO}_4\text{-5}$, high pressure.



POSTER 24

High-pressure behavior of natural borate colemanite. An *in situ* synchrotron single-crystal X-ray diffraction study

Paolo Lotti^{1,2}, G.D. Gatta¹, D. Comboni¹, M. Merlini¹,
H-P Liermann³

¹Earth Sciences Dept., Univ. degli Studi di Milano, Milan, Italy

²Elettra Sincrotrone Trieste S.c.P.A., Trieste, Italy

³Photon Sciences, Desy, Petra-III, Hamburg, Germany

Colemanite is an inoborate compound and a common constituent in natural borate deposits. In addition, it is an economically relevant mineral commodity, not only as a primary source for B, but also for its applications in the production of lightweight concretes and ceramics. Despite its relevance in industrial applications, its elastic behavior, phase stability and structure evolution with pressure have never been investigated. Here we report the high-*P* behavior of a natural colemanite based on an *in-situ* synchrotron single-crystal X-ray diffraction study performed at the P02.2 beamline at PETRA-III, Hamburg, Germany.

Colemanite, which crystallizes in the monoclinic $P2_1/a$ space group ($a = 8.712 \text{ \AA}$, $b = 11.247 \text{ \AA}$, $c = 6.091 \text{ \AA}$, $\beta = 110.12^\circ$, $V = 560.4 \text{ \AA}^3$), undergoes a reconstructive phase transition between 13.95 and 14.91 GPa, toward a monoclinic polymorph (S.G.: $P2_1/n$, $a = 11.726(11) \text{ \AA}$, $b = 10.206(1) \text{ \AA}$, $c = 23.45(3) \text{ \AA}$, $\beta = 95.07(9)^\circ$, $V = 2796(4) \text{ \AA}^3$, at 14.91 GPa). A III-order Birch-Murnaghan EoS fit leads to a refined bulk modulus at ambient conditions of 76(8) GPa [$K_V' = 4.4(10)$], for colemanite in the phase stability field: 0.0001-13.95 GPa. The structure of colemanite is made by infinite chains of corner-sharing B-polyhedra alternated by chains of corner-sharing Ca-polyhedra (coordination number 8). Of the three crystallographically independent B sites, one shows a triangular coordination and the others a tetrahedral coordination. In the high-*P* polymorph, only three over eighteen independent B-sites (1/6) show a triangular coordination, the other fifteen being $B(O,OH)_4$ tetrahedra. Two independent corner-sharing borate chains are interconnected through corner and edge-sharing chains of Ca-polyhedra (C.N. 8 or 9). X-ray diffraction patterns collected during *P*-release show that the phase transition is completely reversible and colemanite fully recovers its starting structural features.

Status of
"Xpress"

B. Joseph¹

G. Bais², S.

¹IISc-ICTP

²Elettra Sincrotrone Trieste

³Bhabha Atomic Research Centre

⁴Indian Institute of Technology

Recently we

diffraction

experiment

partnership

IISc Bangal

x-ray diffrac

supercondu

nitrogen co

splitter chan

(SCW) and

keV. At this

to the perma

beam is focu

with a Pt co

mirror is fur

intense and

x-ray diffrac

ruby fluoresc

stage. The pr

high pressure

Anvil Cells (I

a controllable

positions is a

single crystal

[1] <http://www.icts.ac.in>

[2] <http://www.icts.ac.in>