

MS13-P15**Structural characteristics of vermiculites treated with alcohol**Zulema del Rio Rivadulla¹, Celia Marcos Pascual¹¹. Department of Geology, University of Oviedo (Asturias, Spain)email: zulema1699@hotmail.es

As a result of its lamellar structure, vermiculite exhibits various properties related to structural features, such as the sheet loading associated with the numerous isomorphous substitutions or the ability to dehydrate and rehydrate. It is an interesting mineral in physics, chemistry and biology and very attractive due to its numerous thermal applications and as an insulator. In addition to water, vermiculite can adsorb inorganic or organic substances in the expandable interlayer. Among the various studies conducted on the intercalation of polar organic molecules by clay minerals, the most studied are those related to the adsorption properties of alcohols (Bergaya et al., 2006).

Two vermiculites from China and Libby were treated with different alcohols (methanol, ethanol, propanol and butanol), at room temperature, for 1 month, and subsequent microwave irradiation, for 20 seconds. This treatment caused structural changes and expansion of the vermiculites. The structural changes were characterized by x-ray diffraction, infrared spectroscopy and scanning electron microscopy. The results indicated: 1) The appearance of extra interstratified phases during the transformation from 2- to 1-WLHS (Water Layer Hydration States); 2) the improvement of the crystallinity and order of the most phases, which are the same phases of the untreated vermiculites or even some more. The expansion, k , was measured by the change of the apparent density ($k = \text{density of the raw sample} / \text{density of the treated sample}$) (Justo et al., 1989). Expansion is related to the water loss, composition and distribution of vermiculite cations. Thus, the Fe^{2+} ions would facilitate the fixation of the K^+ ions but not the water, so that the amount would be lower (Marcos and Rodríguez, 2010). The expansion with microwaves irradiation of the samples treated with alcohol varied related to starting sample and time of alcohol treatment. It was also observed that some particles expanded and others did not, as reported previously by Marcos and Rodríguez (2011). The objective of this work was to relate the expansion to structural changes induced in the investigated samples and to the potassium migration.

References:

- Bergaya, F., Theng, B.K.G., Lagaly, G. (Eds.), 2006. Handbook of Clay Science, Elsevier.
 Justo, A., Maqueda, C., Pérez-Rodríguez, J.L. and Morillo, E., 1989. Expansibility of some vermiculites. *Appl. Clay Sci.* 4, 509-519.
 Marcos, C. and Rodríguez, I., 2010. Expansion behaviour of commercial vermiculites at 1000°C. *Appl. Clay Sci.*, 48, 492-498.
 Marcos, C. and Rodríguez, I., 2011. Expansibility of vermiculites irradiated with microwaves. *Appl. Clay Sci.* 51, 33-37.

Keywords: vermiculite, alcohol, exfoliation

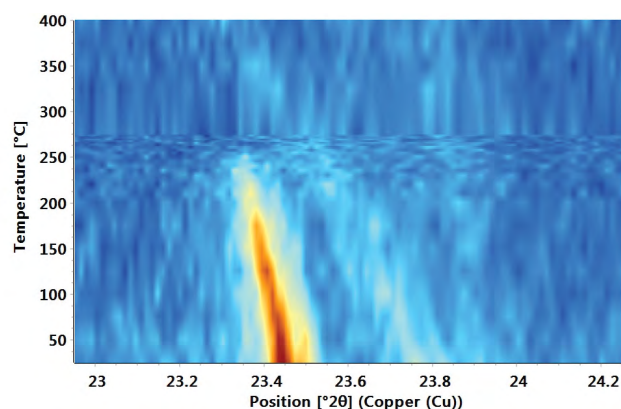
MS13-P16**Piezoelectric phase transition in KSrVO_4 investigated using the Aeris diffractometer**Jan Gertenbach¹, Gwilherm Néner¹¹. Malvern Panalytical, Almelo, Netherlands (Holland, Europe)email: jan.gertenbach@panalytical.com

The rich crystal chemistry of $\text{A}^1\text{B}^{\text{II}}\text{XO}_4$ ($\text{A}^1 = \text{alkali ion}$, $\text{B}^{\text{II}} = \text{alkali-earth ion}$, $\text{X} = \text{P, V, As}$) leads to numerous polymorphic phases that have related structural families, such as olivine (e.g. LiMnPO_4), arcanite ($\beta\text{-K}_2\text{SO}_4$), glaserite, tridymite, $\alpha\text{-K}_2\text{SO}_4$, $\beta\text{-Na}_2\text{SO}_4$ and $\gamma\text{-Na}_2\text{SO}_4$ [1]. Among the various families ($\text{X} = \text{P, V, As}$), the phosphates have been most widely studied. In addition to their interesting crystal chemistry behaviour, research on this family of materials is mainly driven by their ferroelectric-, and ferroelastic properties as well as possible applications as phosphors for LEDs [1].

In recent years, we have investigated several structural combinations of the $\text{A}^1\text{B}^{\text{II}}\text{VO}_4$ type [2] and a new crystal type within this family was identified, namely a form of NaSrVO_4 that is isostructural to larnite [2]. In this work, we continue our exploration of the $\text{A}^1\text{B}^{\text{II}}\text{VO}_4$ family by investigating the composition of KSrVO_4 . While NaSrVO_4 crystallizes in the $\text{P2}_1/\text{n}$ space group, divergent results are reported for KSrVO_4 . Single crystal work suggests Pnma symmetry [3], but powder diffraction studies show evidence for $\text{P2}_12_12_1$ symmetry with distinct reflections present that are not consistent with Pnma symmetry [3]. These contradictory results prompted us to study the structural composition of the compound as a function of temperature.

Using the Aeris benchtop diffractometer equipped with the BTS 500 non-ambient chamber, the crystal structure of KSrVO_4 was studied in the 25-500°C range. At room temperature the structure is best described as having $\text{P2}_12_12_1$ symmetry, allowing for piezoelectricity in this material. By increasing the temperature, it is possible to observe a phase transition to Pnma symmetry. This phase transition appears to be related to the piezoelectric nature of the material and its associated spontaneous strain.

This work reports on the first purely gyrotropic phase transitions in the $\text{A}^1\text{B}^{\text{II}}\text{VO}_4$ family.



References:

- [1] Isupov, V.A. (2002) *Ferroelectrics*, 274(1), 203 and Choi, S., Yun, Y. J., Kim, S.J. & Jung, H.-K. (2013) *Optical Lett.*, 38, 1346
 [2] Nénert, G., O'Meara, P. & Degen T. (2017) *Phys. Chem. Minerals*, 44, 455 and Nénert, G. (2017) *Z. Kristallogr.* 232(10), 669
 [3] Klement, R. & Kresse, P. (1961) *Z Anorg Allg Chem*, 310, 53 and Azrour, M., El Ammari, L., Le Fur, Y. & Elouadi B. (2000) *Materials Research Bulletin*, 35, 263

Keywords: phase transitions, spontaneous strain, benchtop XRD

MS14- Combined approaches for structure characterization of modulated and complex structures

Chairs: Prof. Joke Hadermann, Dr. Phillipe Boullay

MS14-P01

Structure elucidation of a zeolite with multi-dimensional disorder by combining HRTEM and XRPD analyses

Magdalena O. Cichocka¹, Yannick Lorgouilloux², Stef Smeets¹, Philippe Caullet³, Nicolas Bats⁴, Lynne B. McCusker⁵, Jean-Louis Paillaud³, Xiaodong Zou¹

1. Department of Materials and Environmental Chemistry, Stockholm University, Stockholm, Sweden
2. Univ. Valenciennes, EA 2443 - LMCPA – Laboratoire des Matériaux Céramiques et Procédés Associés, Valenciennes, France
3. Université de Haute-Alsace, CNRS, Axe Matériaux à Porosité Contrôlée (MPC), IS2M UMR 7361, Mulhouse, France
4. IFP Energies nouvelles, Rond Point de l'échangeur de Solaize - BP 3, Solaize, France
5. Department of Materials, ETH, Zurich, Switzerland

email: magdalena.cichocka@mmk.su.se

We present a detailed structure analysis of zeolite IM-18 with a complex disordered framework structure that was unknown for more than 10 years.^[1,2] IM-18 has a novel framework topology and a three-dimensional 8x8x10-ring channel system, making it ideal for the shape selective catalysis of small molecules. The unique disorder in the structure makes the structure analysis complicated, so we combined three complementary techniques, including single-crystal electron diffraction (SCED), high resolution transmission electron microscopy (HRTEM) and synchrotron X-ray powder diffraction (XRPD) to reveal the details of the disorder.

Solving the structure of a polycrystalline material that is also disordered can be very challenging as there are no standard procedures to follow. Rotation electron diffraction (RED) allowed us to solve the average structure of the material, the local ordering was observed from the HRTEM images (Fig. 1B-E), but the fine details of the structure and location of the OSDA and Ge could only be derived from XRPD data (Fig. 1A). We found that IM-18 contains two different types of stacking faults along two directions, which were difficult to observe using HRTEM because of the beam sensitivity of the material. We could describe the structure as 1D long-range ordered and 2D disordered, which culminates in a mixture of four types of domains.

By using such an approach, we not only demonstrated the general procedures for *ab initio* structure elucidation of disordered nanocrystals, but also the potential that the combination of SCED, HRTEM and XRPD offers.