

COMPRESSIBILITY BEHAVIOR OF AS-SYNTHESIZED HIGH-SILICA FERRIERITE

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Ferrierite (FER framework topology) is a well-known aluminosilicate zeolite mineral. An understanding of the structure and properties of FER remains important because of its role as a catalyst in commercial reactions. For example, it is important in the petrochemical industry, where it has been used as a shape selective catalyst for the production of isobutene. The thermal behavior of this phase (in its high silica form) was recently studied by Bull et al [1], while its compressibility has never been investigated before.

The high pressure (HP) behavior of synthetic high silica zeolite ferrierite (FER) was investigated by means of in-situ synchrotron X-ray powder diffraction, with the aim to understand the P-induced deformation mechanism. The microporous material was synthesized starting from pure silica and pyridine and propyl-amine as structure directing agents. Here we report the preliminary results on the compressibility of the as-synthesized phase. The study of the compressibility of the calcinated one will be carried out in the following steps of the project.

The crystal structure of ferrierite is built up of rings of fivecorner-shared SiO₄ tetrahedra (known as five-membered rings or 5MRs) building units, which form layers in the *ab* plane. The layers are connected to form a matrix of 10MR channels running parallel to the *c* axis, which are intersected by 8MR channels running parallel to the *b* axis. Six-membered rings connect the 10MRs along the *c* axis direction.

The HP diffraction experiments were performed at BM01a beamline (ESRF), at the fixed wavelength of 0.71 Å, using a modified Merrill-Basset DAC and a mixture of methanol:ethanol:water (16:3:1) as P-transmitting medium. The powder patterns were collected from P_{amb} to 6.2 GPa. Some patterns were also measured upon pressure release up to P_{amb}, to check the reversibility of the compression effects. The unit cell parameters were refined by means of Rietveld method.

The main results of this study are:

- 1) No complete X-ray amorphization is observed up to about 6.6 GPa;
- 2) No abrupt change of the elastic behavior is observed in the whole pressure range. Between P_{amb} and 6.2 GPa the reduction of the cell parameter are 4%, 5% and 6% for a, b and c respectively, accounting for a volume reduction of about 14 %.
- 3) The P-induced effects on the as-synthesized Si-ferrierite cell parameters are completely reversible.
- 4) The bulk modulus obtained using a second order Birch-Murnaghan equation of state and data weighted by the uncertainties in *P* and *V* was K₀ = 30.1(3) GPa. This compressibility is one of the highest when compared with the other natural and synthetic zeolites studied with “penetrating” aqueous media [2, 3] and is very similar to that of SAPO-34 [4], another microporous material studied at HP in its as-synthesized form containing the organic template.

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[4] L. Leardini, S. Quartieri, G. Vezzalini, MMM 127 (2010) 219-227..