| ESRF | **Experiment title:**<br>**STRUCTURAL TRANSFORMATIONS INDUCED BY HIGH PRESSURE IN ZEOLITE A, WAIRAKITE AND YUGAWARALITE** | **Experiment number**:<br>CH-1019 |
|---|---|---|
| **Beamline**:<br>ID09 | **Date of experiment**:<br>from: 28-03-2001 to: 31-03-2001 | **Date of report**:<br>1/08/2002 |
| **Shifts:**<br>12 | **Local contact(s)**: Alessandra Sani | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

**O. Ferro*, G.Vezzalini*, R. Arletti, Dipartimento di Scienze della Terra, via S. Eufemia 19, 41100 Modena, Italy**

**S. Quartieri*, Dipartimento di Scienze della Terra, Salita Sperone 31, 98 166 Messina S.Agata, Italy**

**A. Sani*, ESRF, Grenoble, France.**

## Report:

Introduction

Zeolites are framework aluminosilicates, whose structure is characterised by cavities and channels, where exchangeable cations and water molecules are hosted. These structural features are responsible of the wide industrial applications of zeolites as catalysts and selective absorbers. The literature on the effects of pressure on zeolites is rather limited [see for instance 1-4] and only few quantitative information on the structure and cell parameters of the compressed phases are reported [see the recent results obtained by our group on scolecite and bikitaite reported in 5, 6]. Here we report the preliminary results of an investigation on the pressure-induced structural modifications in yugawaralite, wairakite and zeolite Na-A.

Experimental details

The diffraction experiments were performed at ID09 beamline, at the fixed wavelength of 0.4103 Å. High pressure structures were obtained placing the powdered zeolite samples in a Diamond Anvil Cell, using silicon oil as non penetrating pressure medium [2,4-6]. Data collection was performed from 0 to about 10 GPa and some pressure points were collected decompressing the samples. The pressure calibration was performed by the ruby-fluorescence method [7]. The patterns, obtained by the integration of the image plate images, using FIT2D software [8] have been employed for the determination of the cell parameters using GSAS package [9]. The peak intensities of the collected patterns are not reliable since they are significantly influenced by a number of uncontrollable effects, such as the poor statistics of the diffraction data - due to the low mosaicity of the crystallites and the small volume hit by the beam - and the development of strong preferred orientations. As a consequence, crystal structure refinements are prevented and only the unit cell parameters were extracted from the powder patterns by means of the Rietveld method.

Results

In all studied samples no appearance of new peaks is observed, whereas weakening and broadening of the diffraction peaks with increasing pressure is a common characteristic. However, complete amorphization was not observed up to 10, 9 and 7 GPa for yugawaralite, wairakite and zeolite Na-A, respectively.

For yugawaralite and zeolite Na-A some powder patterns were collected in decompression. From the analysis of these data it can be concluded that the P-induced effects on these two zeolites are reversible (see Fig. 1 for yugawaralite patterns).

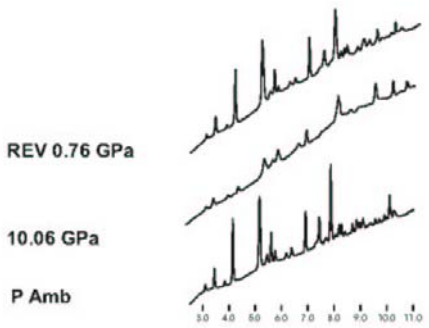



Figure 1

Figures 2-4 report the pressure-dependence of the lattice parameters for the three zeolites. The isothermal bulk modulus of yugawaralite [K0 = 36(1)] and zeolite Na-A [K0 = 22(1)] were determined up to 5.3 and 6.8 GPa, respectively, using a second order Birch-Murnaghan equation of state. All the zeolites here investigated show a high compressibility in the in the studied P range. On the contrary, zeolite Na-A and wairakite are rather rigid during dehydration under high temperature conditions.

Figure 2

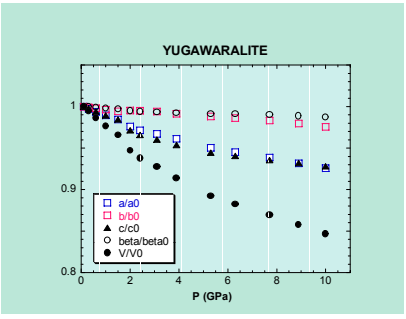



Figure 3

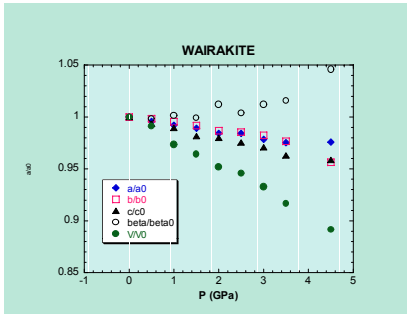
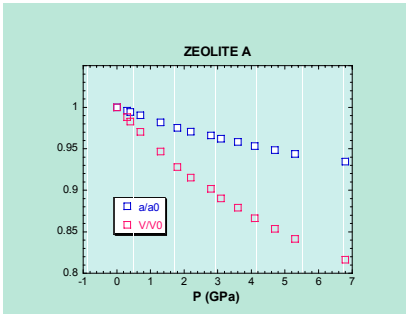



Figure 4




**References**

[1] Rutter M.D., Uchida T., Secco R.A., Huang Y., Wang Y. (2001) *J. Phys Chem. Sol*., 62, 599-606

[2] Gillet, P., Malézieux, J.M. and Itiè, J.P., (1996) *Amer.Mineral.* , 81, 651-657.

[3] Comodi P., Gatta G.D., Zanazzi P.F. (2001) *Eur. J. Mineral.,* 13, 497-505 and (2002) *Eur. J. Mineral.,* 14, 467-574

[4] Vezzalini G., Quartieri S.**,** Sani A., Levy D. (2001) "Structural modifications induced by high pressure in scolecite and heulandite: in-situ synchrotron X-ray Powder diffraction study." in "Studies in surface science and catalysis." Proceedings of the 13 International Zeolite Conference. July 8-13 2001, Montpellier, France. Elsevier B.V.

[5] Ballone P., Quartieri S., Sani A., Vezzalini G. (2002) High-pressure deformation mechanism in the zeolite scolecite: a combined computational-experimental study. *Amer. Mineral*., 87, 1194.

[6] Ferro O., Quartieri S., Vezzalini G., Fois E., Gamba A., Tabacchi G. (2002) High pressure behaviour of bikitaite: an integrated theoretical and experimental approach. *Amer. Mineral*. In press

[7] Mao H.K., Xu J., Bell P.M. (1986) *J.Geophys.Res.,* 91, 4673-4676.

[8] Hammersley A.P., Svensson S.O., Hanfland M., Fitch A.N., Hausermann D. (1996) *High Press.Res.*, 14 235-248.

[9] Larsson A.C.and von Dreele R.B. (2000) GSAS. General Structure Analysis System. Report LAUR 86-748, Los Alamos National Laboratory, Los Alamos, New Mexico