

transition with change of space group symmetry, and the systematic rearrangement of T-O-T bonds in the high-temperature phase. To our knowledge, this is the first direct experimental evidence of a zeolite framework disruption during dehydration caused by increased cation coordination to framework oxygens.

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MS01.05.02 THE EFFECT OF VARIOUS NUCLEATING AGENTS UPON THE CRYSTALLISATION OF CORDIERITE GLASS CERAMIC. S. M. Clark, G. N. Greaves, M. Oversluzien¹, G. Sankar², J. M. Thomas². CCLRC, Daresbury Laboratory, Warrington, WA4 4AD, UK, ¹Netherlands Organisation for Scientific Research, The Hague, The Netherlands, ²Royal Institution of Great Britain, 21 Albermarle Street, London, W1X 4BS, UK

Cordierite glass ceramics are of considerable relevance to the electronics packaging industry due to their high dielectric constant and their low coefficient of thermal expansion. Interest has also been shown in their optical properties for use in tunable lasers and solar concentrators. They are presently being produced in bulk quantities for use as catalyst supports in catalytic converters for car exhaust systems.

Cordierite glass ceramics can be made by heating a glass of the appropriate stoichiometry ($Mg_2Al_4Si_5O_{18}$) above the glass transition temperature. An intermediate crystalline phase known as μ -cordierite is first formed as the final product alpha cordierite crystallises.

We have studied this reaction using time resolved powder diffraction, EXAFS and small angle scattering as well as conventional static measurements. The results of a detailed kinetic analysis will be presented together with a study of the effect of adding a number of nucleating agents to the reaction mixture.

An extension of the classical kinetic equations governing series reactions will also be presented.

MS01.05.03 HIGH RESOLUTION SINGLE CRYSTAL DIFFRACTION USING SYNCHROTRON RADIATION H. Graafsma, O. Svensson, A. Kvik, European Synchrotron Radiation Facility, BP 220 38043 Grenoble, France

The advantages of synchrotron radiation for high resolution single crystal diffraction, e.g. for electron density distributions, are well known [1]. The high energies reduce systematic errors such as absorption and primary extinction. The same is achieved by using small samples, possible due to the high flux at the sample. This high flux also permits the measurement of higher order data, as well as weak reflections [2]. An other advantage beside accuracy is the high speed. The combination of 2D-detectors and the high flux at the sample allows to record a full data set within hours. The results of two measurements at 56 keV (0.22 Å), performed at the materials science beamline of the ESRF will be presented. The first is the determination of the electron density of Magnesium Formate Dihydrate, using a Princeton Instruments slow scan CCD coupled to an Image Intensifier. The second is an electron density

study of Ammonium Dihydrogen Phosphate (ADP) using the SIEMENS SMART system. Both measurements gave an R-int of the order of 3%, with data extending to $\sin \theta/\lambda=1.4$. The first data set was measured in 2 hr, using an oscillation per frame of 4 degrees. The data was integrated both with DENZO and the Seedskewness package. The second data set was obtained in 9 hours using an oscillation of 0.05 degrees per frame, and integrated with the SIEMENS SAINT program using 3-dimensional profile fitting.

[1] Synchrotron Radiation Crystallography; P. Coppens, Academic Press Limited, 1992.

[2] A. Kirfel and K. Eichhorn, *Acta Cryst.* A46,271 (1990).

MS01.05.04 STRUCTURE DETERMINATION OF MICROMETER AND SUBMICROMETER SINGLE CRYSTALS WITH SYNCHROTRON RADIATION. Neder, R. B., Burghammer, M., Grasl, T., Schulz, H., Institut für Kristallographie und Mineralogie, Universität München, Theresienstr.41, 80333 München, Germany

We have mounted individual submicrometer sized single crystals of kaolinite to thin glass fibers. We developed a novel micromanipulator for usage within a scanning electron microscope. This micromanipulator uses a combination of stepper motor controlled microtranslation units with piezo drives and is capable of nanometer resolution. The glass fiber supports are pulled from massive glass rods to a diameter of 0.5 μm .

The sample volume is estimated at $< 0.1 \mu\text{m}^3$. Diffraction experiments are carried out at the microfocus beamline, ESRF, under vacuum conditions. The combination of a vacuum chamber and submicrometer sized sample-supports effectively reduces the experimental background.

As another example we present the results of the determination of hydrogen positions from a hydrous barium oxalate. Despite the presence of a heavy metal, the positions, as well as, thermal parameters of the hydrogen could be refined.

A third example is optically anomalous topaz. An individual microcrystal was prepared from within an optical zone. Single crystal diffraction experiments at the ESRF were carried out on a crystal of $2 \mu\text{m}^3$ volume.

MS01.05.05 APPLICATION OF MICRO-BEAM TO MINERALS IN A THIN SECTION OF METEORITE AND STRUCTURE REFINEMENT. K. Ohsumi¹, M. Uchida², K. Hagiya³, M. Miyamoto⁴ and M. Ohmasa³. ¹Photon Factory, National Laboratory for High Energy Physics, Japan; ²Synchrotron Radi. Sci., Graduate School for Advanced Studies, Japan; ³Department of Life Science, Himeji Institute of Technology, Japan; ⁴Mineralogical Insti., Graduate School of Science, Univ. of Tokyo, Japan.

Polychromatic SR microbeam with diameter of 1.6 μm and with divergence of 40 μrad . was produced by a micro-pinhole technique for structure refinement using Laue method. This size of micro-area on the sample is comparable to those examined by optical microscope, scanning electron microscope, electron probe micro analyzer (EPMA), Raman spectroscopy and so on. The X-ray diffraction method with this micro-pinhole provides crystallographic information from the exact same micro-area as analyzed by various methods mentioned above. The micro-pinhole was installed in the Laue camera which was developed at the beam line 4B of the Photon Factory, KEK¹). One of the application of this method is given below.

In crystallographic studies of meteorite which must be affected by shock as presumed from its origin, the microbeam is indispensable to search micro-area that is good enough for structure