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.

Work supported under contract AC02-76CH00016 with US DOE Div. of Chem. Sci., Office of Basic Energy Sciences, and the by the Danish Natural Science Research Council.

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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. Oversluzien[1], G. Sankar[2], J. M. Thomas[2]. CCLRC, Daresbury Laboratory, Warrington, WA4 4AD, UK, [1]Netherlands Organisation for Scientific Research, The Hague, The Netherlands, [2]Royal 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. Kvick, European Synchrotron Radiation Facility, BP 220 38043 Grenoble, France

The advantages of synhrotron 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 Amonium Dihydrogen Phosphate (ADP) using the SIEMENS SMART system. Both measurements gave an R-int of the order of 3%, with data extending to sin θ/λ=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] Synhrotron Radiation Crystallography; P. Coppens, Academic Press Limited, 1992.
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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 μm^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 μm^3 volume.

MS01.05.05 APPLICATION OF MICRO-BEAM TO MINERALS IN A THIN SECTION OF METEORITE AND STRUCTURE REFINEMENT. K. Ohsumi[1], M. Uchida[2], K. Hagiya[3], M. Miyamoto[4] and M. Ohmasa[3]. [1]Photon Factory, National Laboratory for High Energy Physics, Japan; [2]Synchrotron Radi. Sci., Graduate School for Advanced Studies, Japan; [3]Department of Life Science, Himeji Institute of Technology, Japan; [4]Mineralogical 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 prove 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, KEK[1]). 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

refinement, or to obtain diffraction data from submicron crystal grain with high signal to noise ratio.

The microbeam has been applied to olivine ($(Mg,Fe)_2SiO_4$) and two unknown crystals included in a thin section of ureilite (meteorite) with thickness of 30 μm, in order to (1) obtain site occupancies of Mg and Fe in cation sites (M1 and M2) which indicate thermal history of the sample, (2) identify two unknown crystals in the rim of the olivine covered with carbonaceous material. The results are summarized below.

(1) Structure refinement was successfully carried out in an usual manner based on more than 300 Laue intensities, and site occupancy of Mg in M1 site was determined as 0.908(4) under the constraint of Mg and Fe abundant which was analyzed by EPMA.

(2) One of the unknown crystal is determined as diamond, and the thermal parameter(B) was refined to be 0.19(3)A^2 by the least-squares method based on 12 Laue diffraction intensities. The other was revealed to be a face-centered cubic phase of iron (Fe) which was crystallized, topotaxial to the olivine, of Fe atoms from M1 and M2 sites under reduction by carbon. The thermal parameter (B) was also refined to be 0.06(1)A^2 based on 8 Laue diffraction intensities.

1) K. Ohsumi et al.: *Rev. Sci. Instrum.*, 66(2), 1448-1450, 1995.

MS01.05.06 STRUCTURAL INVESTIGATIONS ON MICROCRYSTALS WITH A HIGHLY COLLIMATED SYNCHROTRON BEAM. A.Bram, S. Fiedler, C. Riekel, European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France

A endstation for single crystal microcrystallography has been installed at the ESRF microfocus beamline [1]. A monochromatic beam from an undulator is demagnified by an ellipsoidal mirror. Background reduction is effected by a post collimator. A liquid N_2 cooled CCD detector with converter screen and optical demagnification is used for data collection on a K-goniometer. In order to minimize the influence of air scattering the whole goniometer can be placed in a vacuum vessel. First experiments have been performed on inorganic crystals, proteins and small organic fibres. The present setup will be shown together with the results of selected experiments.

[1] P. Engstroem, S. Fiedler, C. Riekel, Rev. Sci. Instrum., 66(2), 1348 - 1350 (1995)

MS01.05.07 MICRODIFFRACTION ON Be SINGLE CRYSTALS WITH SYNCHROTRON RADIATION. S. Fiedler, A. Bram, C. Riekel[1] and M. Burghammer[2]. [1]European Synchrotron Radiation Facility, BP 220, 38043 Grenoble Cedex, France, [2]Institut f. Kristallographie, Theresienstr. 41, 80333 Muenchen, Germany.

The microdiffractometer installed on the Microfocus beamline at the European Synchrotron Radiation Facility has been used to study single crystals of Be as a function of crystal volume in order to derive accurate structure factors. Crystals of conical shape have been produced by chemical etching techniques. The smallest tips obtained had diameters between 1-2 micrometer. Crystal volumes down to a few cubic micrometer have been examined by monochromatic radiation (λ=0.0687nm) which corresponds to a minimum scattering power of $S=8*10^{11}$ (defined as in [1]). Datasets with a resolution down to 0.04nm have been collected with an online area detector. Background scattering had to be reduced as far as possible by using microcollimators in combination with a focused beam. Results of the refinement of the extinction and the temperature factor will be reported.

W. Rieck, H. Euler, H. Schulz; Acta Cryst., A44, 1099, (1988)

MS01.05.08 DIFFRACTION ANOMALOUS FINE STRUCTURE AT THE ESRF BY USING DISPERSIVE & MONOCHROMATIC DIFFRACTION. J. L. Hodeau[1,2], J. Vacinova[1], H. Renevier[1], P. Wolfers[1], J. F. Berar[1], M. Hagelstein[2], A. San Miguel[2], [1]Lab. Cristallographie CNRS, BP 166, F-38042 Grenoble, France, [2]ESRF, BP 220, F-38043 Grenoble, France

We present several Diffraction Anomalous Fine Structure (DAFS) studies performed at ESRF and at LURE on platinum or iron oxydes and on multilayers, using two different experimental modes: the **Dispersive Diffraction** mode (DD) which uses the combination of X-ray energy dispersive optics, sample goniometer and a two dimensional position sensitive detector and the **Multi Monochromatic Diffraction** mode (MMD) performed with classical monochromatic optics. We discuss our last results obtained at ESRF by using these two experimental modes for DAFS data collection. The DD technique would be precious for time dependent investigations and in-situ experiments and it could be used not only for DAFS experiments but also for other anomalous scattering experiments.

The DAFS spectroscopy provides in the same experiment information regarding the local atomic environment through X-ray absorption processes and long-range order information through diffraction processes. It can provide **site selective** and **chemical selective** structural information. Up to now, most results obtained with this method concern materials where structural information can be extracted from pure, site-selective Bragg reflections. Our studies apply the technique to highly absorbing "real" materials and to multilayers containing several anomalous atoms which do not respect the latter restriction. An accurate empirical absorption correction procedure for small highly absorbing single crystals necessary for the DAFS analysis of this kind of samples is developed. We present a **simultaneous multi-wavelength refinement** procedure of several Bragg reflections with the anomalous terms f' of different crystallographic sites as unknowns.

MS01.05.09 HELICITY SWITCHABLE HIGH ENERGY X-RAY PHASE RETARDER. J. C. Lang, G. Srajer, S. Shastri, D. Haeffner, D. Mills, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

We have developed a high-energy (E > 50 keV) phase retarder capable of producing both left- and right-handed circularly polarized photons simultaneously. This phase retarder was constructed from a single Ge crystal in a monolithic Bragg-Laue design, similar to that studied previously (D. M. Mills, Nucl. Instr. and Meth. A266, 531 (1988)). This previous phase retarder while able to produce photons with a relatively high degree of circular polarization (P_c>90%), had a major drawback in it's inability to conveniently reverse the photon helicity. The phase retarder in this study overcomes this limitation by simultaneously exciting the (220) and (2-20) crystalographically equivalent and perpendicular reflections. Each of these reflections induces an equal and opposite phase retardation between the σ and π wave fields in the Laue portion of the crystal, thus producing two x-ray beams with opposite senses of helicity. These beams are spatially separated by approximately 2 mm therefore helicity reversal is easily achieved by chopping between the two beams. A major advantage of this technique is that it retains the non dispersive geometry between the preliminary monochromator (Bragg portion) and the Laue phase retarding crystal therefore preserving the maximum possible throughput of the optic. Details of the construction and characterization of this optic will be presented.