PS18.03.24 COMPARATIVE CRYSTAL CHEMISTRY OF SILICATES BEYOND 20 GPA. Li. Zhang, H. Ahsbahs, A. Kutoglu, S. S. Hafner, Center of Materials Sciences, University of Marburg, Germany

The crystal chemistry of Mg^{2+}, Co^{2+}, Fe^{2+}, Mn^{2+}, Ca^{2+}, Fe^{3+} and Al^{3+} at high pressure in relatively incompressible silicates is not well studied. Present knowledge is largely based on limited experimental results at relatively low pressure. As a result, a systematic description of the high pressure crystal chemistry for the above cations is not available. Mg^{2+}, Fe^{2+}, Ca^{2+}, and Al^{3+} cations are major constituents in minerals of the Earth's deep mantle. For understanding especially the chemical processes and the physical properties in that region it is essential to obtain information about the high pressure behavior of the above cations in the mantle minerals.

The high pressure crystal chemistry of those cations was studied by considering their electronic structures, polyhedral coordination as well as the crystal structure. Experiments were performed on synthetic endmember garnets, $Mg_3Al_2(SiO_4)_3$ $Fe_3Al_2(SiO_4)_3$ $Mn_3Al_2(SiO_4)_3$ $Ca_3Fe_2(SiO_4)_3$ $Ca_3Al_2(SiO_4)_3$, olivines, Mg_2SiO_4, Fe_2SiO_4, Mn_2SiO_4, Co_2SiO_4 and pyroxenes, $CaMgSi_2O_6$, $CaFeSi_2O_6$. The crystals were loaded in diamond anvil cell with rare gas solids, argon, neon and helium as well as mixtures of ethanol-methanol serving as pressure transmitting media. The unit-cell parameters determined up to 212 GPa show that polyhedral coordination of individual cation controls primarily their crystal chemical behavior. The 6-fold coordinated polyhedra with cations of similar electronic structures like Co^{2+}, Fe^{2+}, Mn^{2+} do possess compression behavior which obeys prediction of bulk modulus - volume systematics, whereas those with cations of different electronic structure like e.g. Fe^{2+}, Mg^{2+} do not follow such predictions. This is evidenced in olivine and pyroxene. The high pressure crystal chemistry of these cations in garnet, olivine and pyroxene as well as its implication to the bulk modulus - volume systematics will be discussed.

PS18.03.25 PRESSURE-INDUCED PHASE TRANSITION OF $AgGaS_2$. H. Kitahara, N. Ishizawa, F. Marumo* and Y. Noda** Research Laboratory of Engineering Materials, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-ku, Yokohama 226, Japan, *Department of Earth Sciences, Nihon University, 3-25-40 Sakurajosui, Setagaya-ku, Tokyo 156, Japan **Department of Materials Science, Faculty of Engineering, Tohoku University, Aramaki, Aoba-ku, Sendai 980-77, Japan

Single-crystal X-ray diffraction study under high-pressures has been carried out on $AgGaS_2$ to obtain crystallographic information of the high pressure phase in the range 4.2-11.6 GPa employing a diamond-anvil cell. Single-crystals of $AgGaS_2$ with chalcopyrite structure were grown with the Bridgman method. The crystal is tetragonal, I42d, a=5.7626(5) and c=10.3128(9) Å at ambient pressure. Above the transition point 4.2 GPa, the systematic condition of 2h+1=4n for hhl reflections was broken, showing that the diamond glide planes parallel to {110} have been disappeared in the high-pressure phase. In addition, most diffraction spots except 001 split into two or more in the high-pressure phase. They turned into single ones again when the pressure was released. These results indicate that (1) above 4.2 GPa the diamond glide planes are lost, and (2) a twin or a quadruplet seems to be formed in a way which keeps the c direction constant, and (3) the transition is essentially reversible, though the glide twins were left in the pressure-released crystals. The phase transition looks like a displacive type and the high-pressure phase apparently possesses a symmetry of C2, one of the non-isomorphic subgroups for I42d. The compressibility along the c axis is larger than; that along the a axis under pressures up to transition point. Since the intensities of 001 reflections were scarcely affected by the transition, the structural change may involve the atomic displacements on (001).

PS18.03.26 KINETIC STUDIES ON THE CRYSTALLISATION OF COESITE USING THE HP/HT-DEVICE MAX80 AND SR. Zinn, P.[1], Hinze, E.[2] & Lauterjung, J.[1]; [1]GeoForschungsZentrum Potsdam, D-14473 Potsdam; [2]University of Giessen, D-35390 Giessen

The kinetic behaviour of the quartz - coesite transition has been studied in the P/T range of 3.2-5.2 GPa and 680-900°C by in-situ X-ray diffraction using synchrotron radiation and a MAX80 cubic anvil high-pressure apparatus at HASYLAB. The quartz - coesite equilibrium phase boundary has been determined and discussed using the experimental results. During the transition, X-ray patterns were collected, and the transformed volume fraction has been estimated from the diffracted intensities of the respective phases as a function of time. By fitting a fundamental rate equation for grain boundary nucleation and interface-controlled growth to the transformation-time data, rates of nucleation and growth have been estimated [1].

During the in situ observed phase transition over a small P-T stability region an intermediate non quenchable stage could be detected [2]. By constructing a Bragg-Bragg Mo-double-crystal-monochromator the resolution of the MAX80-device could be raised up to $\Delta d/d = 0.001\%$. With this configuration the intermediate phase shall be investigated in detail.

Furthermore, by quenching the samples, the reaction products could be investigated by TEM images. A strongly twinning with (100) reflection twins could be observed [3]. Its influence on the thermodynamic behaviour of the a-quartz - coesite phase transition is discussed. Coesite-nuclei in quartz crystals, and in a latter stage penetrating the quartz-crystals could be observed. The growth mechanism and the existing structural relationship between host-quartz and the coesite-nucleus were then analysed.

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High Pressure IV
Pressure Cells and Sample Conditions

MS18.04.01 LARGE VOLUME CELL AT HIGH P/T- NEW LIMITS AND FUTURE PROSPECTS. W.Utsumi, Department of Synchrotron Radiation Facility Project Japan Atomic Energy Research Institute Tokai-mura, Naka-gun, Ibaraki 319-11, Japan

In contrast to the diamond anvil cell, multi-anvil apparatus are capable of maintaining stable high temperatures under high pressure owing to the larger sample volume. More than 14 years have passed since the first multi-anvil apparatus for synchrotron radiation was installed at the Photon Factory, and similar apparatus have been installed at the HASYLAB and NSLS. These multi-anvil systems combined with synchrotron radiation have brought us a large number of excellent results. The recent biggest advance in the multi-anvil apparatus is the use of sintered diamond as an anvil material. The characteristics of sintered diamond such as high compressive strength and no cleavage plane has enabled us to extend the attainable pressure range greatly. Many attempts have been made to replace tungsten carbide anvils with sintered diamond for the various type of multi-anvil apparatus, and most of them have had successful results.

Among the several multi-anvil type apparatus, the DIA type apparatus, a variation of cubic anvil press, has been most commonly used for the synchrotron radiation experiment in Japan. This system has many advantages; 1) fairly large sample volume which has a simple symmetry, 2) stable high pressure and high tempera-

ture generation. 3)quasi-hydrostaticity, 4) simple x-ray diffraction geometry. By use of sintered diamond anvils with 3mm truncation, the DIA can generate pressures up to 20GPa.

Recently, the octahedral compression system with sintered diamond anvils has made great progress. This system has now reached pressures over 30GPa and temperatures up to 2000C in a sample volume of about 1mm3. In situ x-ray observation using this system have been successfully made, and various experimental results have been obtained. In this system, sintered diamond is used as an x-ray window. The fact that high energy x-ray can pass through the sintered diamond made this technique possible.

Although it might not be called multi-anvil, the simple opposed anvil type apparatus still has some advantages. Taking a Drickamer type sintered diamond anvil cell for example, it can generate more than 50GPa, and in situ observation has been successfully made at high P-T condition. There are some unreliability of the data because of the small sample volume and strong deviatric stress, but the opposed anvil devices are also very useful when they are used properly depending on the purpose and condition of the experiment.

In my talk, I will review the current status on the technical development in multianvil apparatus, and show a conception design of a new high pressure apparatus for the third generation synchrotron radiation (SPring-8).

MS18.04.02 T-CUP: A NEW HIGH-PRESSURE APPARATUS FOR X-RAY STUDIES D. J. Weidner, Y. B. Wang, M. T. Vaughan, C. C. Koleda (CHiPR and USB), I. C. Getting (CHiPR and U Colorado at Boulder)

We have designed and tested a new apparatus for *in-situ* X-ray diffraction studies under high pressures and temperatures. The system is a 6-8 two stage system, designed for a 200 ton load, with the possibility of sintered diamond second stage anvils. The first stage is a steel cylinder split into six parts and fastened onto the upper and lower guide blocks, enclosing a cubic cavity (19.5 mm edge length and the [111] axis of the cube stays vertical). The hydraulic press used for SAM-85 DIA guideblocks is used to apply up to 200 ton load. The second stage is assembled outside the press and consists of eight WC cubes each with 10 mm edge lengths.

The cell assembly is an octahedron made of semi-sintered MgO or boron-epoxy. The incident x-ray beam passes through the gaps between WC cubes in the [110] direction of the eight-cube assembly and diffracts in one of the (100) planes, through gaps between the WC anvils, with a diffraction vector 35.3° from the vertical plane. A special holder is built for a solid-state detector for energy dispersive mode at several 2q angles up to 7.5°We compressed NaCl at room temperature to 160 ton force and generated 18+ GPa pressure with 2mm truncations. Decker's equation of state for NaCl was used to determine pressures in the cell assembly.

MS18.04.03 NEW APPLICATIONS OF THE PARIS-EDINBURGH CELL: NEUTRONS AND X-RAYS S. Klotz, Physique des Milieux Condensés, Université P&M Curie, 4 Place Jussieu, B77, F-75252 Paris.

Until recently, the devices needed to compress large volume samples to pressures beyond 10 GPa were large and cumbersome. The development of the Paris-Edinburgh cell, which provides a thrust of 250 tonnes and has a mass of only 50 kg, thus represents a considerable reduction in size over comparable large-volume pressure cells. Equipped with two opposed anvils made of either tungten carbide or sintered diamond it will compress samples of up to ~ 300 mm^3 in volume to 6 GPa or ~ 30 mm^3 to 25 GPa. Originally designed for high pressure neutron powder diffraction, its compact size gives it advantages for a wide range of applications in both crystallography and solid state sciences. We give an overview of experiments which illustrates the potential of these techniques, covering: (1) Neutron powder diffraction to 25 GPa on solid, liquid and gaseous samples at ambient and low temperature on spallation sources, (2) inelastic neutron scattering on single crystals to 10 GPa on reactor sources, (3) in-situ x-ray powder diffraction at high temperatures to 1000 °C and 6 GPa, (4) in-situ EXAFS on solid and molten semiconductors at high temperatures and high pressures (5) density measurements by x-ray absorption experiments. We will also discuss current developments of cells with larger/smaller capacity and its future applications.

MS18.04.04 SAPPHIRE-ANVIL CELLS FOR NEUTRON SCATTERING STUDIES AT HIGH PRESSURES. A.S. Ivanov, Russian Research Center "Kurchatov Institute", Moscow 123182, RUSSIA

Recent progress on instrumentation for neutron scattering investigations of condensed matter at high pressures is reported. A set of high-pressure cells with maximal load up to 25 tons and ancillary equipment was constructed to conduct measurements of crystal structure and lattice dynamics at pressures of few dozens kbar and at different temperatures with sample volumes up to few cubic millimeters. Different size anvils (as a rule sapphire and sometimes diamond) in combination with various metallic gaskets serve to clamp a powder sample or a single crystal in liquid pressure-transmitting medium. A new type time-of-flight neutron scattering instrument with a full Debye-cone analysis was created and installed at the pulsed reactor in collaboration with Joint Institute for Nuclear Research, Dubna, Moscow Region. The instrument is dedicated to studies of microsamples and is adopted to measurements of both elastic and inelastic (using analyser ring) neutron scattering. The working abilities of the equipment are illustrated by examples of neutron scattering spectra registered at a number of fixed pressure values in the studied pressure range. The examples include measurements of pressure changes of crystal structure in ammonium halides and different mercury superconductors. Some data on phonon density of states, phonon dispersion and small-angle scattering are presented.

MS18.04.05 SINGLE CRYSTAL STUDIES IN THE Mbar RANGE. P.Loubeyre and R.LeToullec, PMC, Universite Paris 6, Paris, France

Single-crystal x-ray diffraction at Mbar pressure has been a major goal of high pressure physics. It is the essential technique to obtain diffracion from low -Z elements and can provide the most accurate structural data possible. But taking any single-crystals to very high pressures is extremely difficult as they usuallly break beyond use. Also, the diamond anvil cell should have an x-ray viewing angle of both sides of the sample as large as 80°, that is a mechanical challenge.

We will present a new x-ray cell with boron seats of half-sphere geometry that can hold the massive support on the diamond for the Mbar presures. The use of a soft hydrostatic cushion (helium at best and gold when it is impossible to load) will be shown to prevent the break up of the crystal. Already, single crystals of H_2, He, LiH, H_2O, $Ar(H_2)_2$, $Ne(He)_2$ have been studied in the Mbar range.

H_2 and H_2O will be selected as the most illustrative examples. Hydrogen because it is considered as the utmost difficult system for x-ray study at high pressure. H_2O because our single-cystal measurements can be compared to various powder x-ray diffraction works.