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## Ninth International Symposium on Experimental Mineralogy, Petrology and Geochemistry

### STATE OF A MATURING METHODOLOGY: GIGAHERTZ ULTRASONIC INTERFEROMETRY

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Experimental determination of the anisotropic elastic properties of minerals plays an important role in interpreting Earth's seismic structure in terms of composition and mineralogy. Gigahertz ultrasonic interferometry (GUI) is a relatively new, rapidly evolving acoustical method that, when fully developed, will be used to measure P and S travel times in single crystals at non-ambient pressures and temperatures in a modified ultrasonic/X-ray diamond anvil cell. GUI has successfully reduced the sample-size requirements for single-crystal ultrasonics to about 50 microns, but has been essentially without the use of shear waves due to limitations in high-frequency piezoelectric shear transducers. A new method of generating shear waves with near-optical wavelength has been developed that features a P-to-S conversion inside a single-crystal MgO prism. Last year we demonstrated the exciting potential of this new method by measuring the ambient *P-T* elastic constants of single-crystal magnesiowüstite-(Mg,Fe)O with a variety of compositions. In the diamond cell, P-wave measurements have been made on (Mg,Fe)O of mid-range composition to 3.6 GPa at room temperature. A major challenge facing future single-crystal ultrasonic measurements at high temperature will be keeping the sample in contact with the acoustic transmitting anvil. In a pilot study, we measured P-wave travel times in San Carlos olivine to 250 °C at ~2.5 GPa without a bonding agent. We are also exploring the use of aerogel to act as a gentle transparent "spring". Recently, we produced a silica aerogel in 4:1 methanol:ethanol solution (alcohol), and loaded it along with a quartz (010) plate and several rubies in our ultrasonic cell. The samples in the new supportive pressure medium exhibited hydrostatic compression to the maximum pressure of ~5 GPa based on the axial ratio *c/a* of the quartz and peak widths of the ruby spectra, although we have yet to measure travel times in this configuration.

### PRESSURE DEPENDENCE OF OH RAMAN FREQUENCIES OF HYDROUS SILICATE PHASES

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The range of minerals in the Earth capable of hosting water in the form of H<sub>2</sub>O and/or OH ranges from ice to nominally anhydrous silicate phases. Proton site locations and hydrogen bonding in minerals can be investigated with different experimental methods: Vibrational and NMR spectroscopy, as well as x-ray and neutron diffraction techniques. Raman spectroscopic observations of OH stretching frequencies in conjunction with distance-frequency correlations are particularly important in

cases where bulk crystallographic methods cannot be used, such as disorder of protons or the small sample volume of high-pressure phases. We report pressure dependencies of OH stretching frequencies in highly hydrous phases: Phase E (synthesized by N. Ross) and clinocllore, measured in a diamond-cell to 19 GPa and 25 GPa respectively with Micro-Raman spectroscopy. OH stretching frequencies showing the common negative, but also unusual positive pressure dependencies, have been observed between 3400 cm<sup>-1</sup> and 3700 cm<sup>-1</sup>. A decrease in OH stretching frequency with increasing pressure can be attributed to a weakening of the intramolecular OH force constant, as for example caused by increased hydrogen bond interaction. H-H repulsion under compression might also be an important factor in the positive pressure dependencies of OH stretching frequencies. In the high-pressure Raman experiment on clinocllore we used moissanite anvils, which, unlike diamond-anvils, do not obscure the Raman signal in the OH stretching region.

### A MODIFIED DOUBLE CAPSULE TECHNIQUE FOR H<sub>2</sub>O UNDERSATURATED PISTON CYLINDER EXPERIMENTS IN CALC-ALKALINE SYSTEMS

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The experimental study of H<sub>2</sub>O-undersaturated Fe-bearing systems in piston cylinder apparatus is faced with iron loss to and H<sub>2</sub> diffusion through noble metal capsules. During the alloying of the iron with the noble metal container, the FeO from the experimental charge is reduced to form an FeX alloy (X = Au, Pt, AuPd, AgPd), liberating excess O<sub>2</sub> [1]. Hence, the loss of iron to noble metal capsules is ultimately related to the oxygen fugacity (*f*O<sub>2</sub>). Here we present a modification of the double capsule technique that permits us to perform experiments on H<sub>2</sub>O-undersaturated, iron-bearing systems under conditions naturally prevailing in calc-alkaline magmas. Au<sub>50</sub>Pd<sub>50</sub> or Au<sub>90</sub>Pd<sub>10</sub> capsules were preconditioned for 48 to 72h in a gas mixing furnace at QFM to QFM-1 using a synthetic basalt that was mixed from oxides and hydroxides. The recovered capsules were cut in half and mounted in epoxy, the other half was put into HF for ~48 hours to remove the silicate glass and then mounted in epoxy. The iron content of the capsules before and after treating with HF was determined along profiles of the bottom and the side wall of the capsules. Experiments at 1 GPa used a modified double capsule technique, where a preconditioned AuPd capsule is put into an outer Pt capsule which contains the same starting material. Two different NaCl-pyrex assemblies were used in order to impose different oxygen fugacities (*f*O<sub>2</sub>) on the experimental charges: (i) MgO inner sleeves and (ii) Pyrex-BN inner sleeves. Au<sub>90</sub>Pd<sub>10</sub> capsules contain ~0.3 wt% Fe after presaturation but before leaching. Microprobe traverses revealed no significant zoning indicating that equilibrium was achieved at the given temperature and *f*O<sub>2</sub>. However, after leaching with HF, the Fe content of the capsules was significantly higher (~0.45 wt% Fe), indicating that leaching the silicate glass with HF signifi-

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cantly changes the iron content of Au<sub>90</sub>Pd<sub>10</sub> alloys. High pressure runs at 1.0 GPa contained olivine+liquid. The fO<sub>2</sub> of these experiments was calculated using Fe-Mg Kd's (olivine-liquid) and the thermodynamic formulation of Kress and Carmichael [2]. Results indicate that preconditioned Au<sub>90</sub>Pd<sub>10</sub> capsules with a BN assembly are best suited for H<sub>2</sub>O undersaturated experiments at ~QFM+1.

Grove TL, *Contrib. Mineral. Petrol*, **78**, 298-304, (1982).  
Kress VC, Carmichael ISE, *Contrib. Mineral. Petrol*, **108**, 82-92, (1991).

#### VERIFICATION OF THE FAXEN CORRECTION FACTOR USED FOR VISCOSITY DETERMINATIONS

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In viscosity measurements of silicate melts with the falling sphere technique a correction factor (the "Faxen correction factor") is often employed to account for the finite diameter of the sample (Faxen, 1925). Due to very large samples, dynamic neutron radiography allows to determine the effect of the sample size and hence to verify the magnitude of the necessary correction. Dynamic neutron radiography is the time- and position-dependent measurement of the attenuation of a neutron beam due to the absorption of the neutrons. In such experiments, silicate melts are generally transparent for neutrons, while the spheres can be made of highly absorbing material. In our experimental set-up, the ratio of the diameters of the sphere and the sample can be varied between 0.06 to 0.25. Furthermore, the long distance (90 mm) over which the fall of a sphere can be observed, allows to discontinuously change this ratio by manufacturing crucibles whose upper half has an inner diameter of 39 mm, while the lower half has an inner diameter of 20 mm. In nearly all of our experiments the velocity change of the falling sphere due to the change in the diameter of the crucible was significantly smaller than suggested by the commonly used expression for the Faxen correction factor. We can exclude thermal gradients or inhomogeneities within the sample as the cause of this behaviour, and hence conclude, that most probably the often used correction overcompensates the influence of the finite sample diameter by 10-15%. A more detailed evaluation will be presented at the conference.

Faxen H, *Ark. f. mat., astr. o. fys.*, **19A (No. 22)**, 1-10, (1925).

#### PARTIAL MELTING OF MUDSTONES AT 1 BAR; A CONSTRAINT ON THE PHASE DIAGRAMS FOR HAPLOGRANITE SYSTEM

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Partial melting of mudstones at P~1 atm during prolonged pyrometamorphic events was examined on the example of fused sedimentary rocks - clinkers from the Uralian burned dumps (Russia). During spontaneous coal combustion temperature obtained are high enough (700 - 1300°C) to cause partial or total melting of mudstones. Parent rocks contain SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O and H<sub>2</sub>O more than 90 wt.% taken together and less than 0.5 wt% of CaO, that allows to use haplogranite system as suitable for consideration of their melting. The melt compositions were determined by microprobe analysis of 100 glasses from the clinkers. The glasses being the quenching partial melts, vary in silica and alkalis contents. In the Qz-Ab-Or-diagram the figurative points of normative glass compositions trace the Qz-Fsp cotectic (P<sub>H<sub>2</sub>O</sub>=0.5 kb) and form two clusters according to tie-lines in the field of quartz stability. These individual sequences reflect the increasing of partial melting degree. The first cluster originates at the cotectic for P<sub>H<sub>2</sub>O</sub>=0.5 kbar (Tuttle, Bowen, 1958), and the second one at the dry cotectic of Schairer (1950). It is surprising, inasmuch as in both cases melting was realised under the same dry conditions at P~1 bar. There are several reasons of ambiguous reconstruction of melting conditions. The first reason is the inconsistency of the experimental data for low-pressure conditions. As P<sub>H<sub>2</sub>O</sub> decrease cotectics shift toward Qz corner. It allows to expect that dry cotectic should be widely spaced from the Fsp joint. In this case the first glass compositions cluster conforms to assumed dry cotectic that is contradictory to experimental data of (Schairer, 1950). The existence of two distinguished glass compositions clusters is attributable to either the singularity of melted substrate compositions, or Na- and Si-evaporation from melt during the prolonged heat treatment. Financial support of RFBR (grant 01-05-65060).

Tuttle OF & Bowen NL, *Geol.Soc.Am.Mem.*, **74**, 145, (1958).

Schairer JF, *J. Geol.*, **58**, 512-517, (1950).

#### MODELING OF THERMODYNAMIC CONDITIONS OF METAMORPHIC-METASOMATIC FORMATIONS NEAR ORE-BEARING COMPLEXES

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In order to study the evolution of metamorphic processes and evaluate their thermodynamic parameters, late magmatic and superimposed metasomatic mineral assemblages were investigated near the contacts of ore-bearing metamorphic garnet-biotite-amphibole rocks of the Fedorovo-Panskiy massif. The massif is located in the central part of the Kola peninsula and contains two stratified platinum-bearing layers horizon. On the basis of a detailed research of structures of coexisting minerals parageneses, three consequent stages of crystallization and

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mineral formation have been distinguished. The observed natural process of transformation of rocks in time from the initial crystallization of magmatic substance up to the formation of mineral assemblages is justified by the carried out theoretical research, and the thermodynamic conditions are described by appropriate chemical reactions. On the basis of comparisons of phase interrelations and the obtained calculated material, some features of the evolution of parameters are established for the regressive metamorphism.

The conditions for the evaluation of the thermodynamic parameters obtained by using mineralogical geothermobarometers and by the date from the calculations serve as a basis for revealing of mineral facies of metamorphic and metasomatic processes of rocks of the investigated massif. The established orientation of metasomatism in near ore-bearing garnet-biotite rocks, reflected in P-T-t trends, a mineralogical of data modeling, testifies to local warming under corresponding rise of pressure in the natural system and a consequent drop of these parameters.

### WATER CONTENT IN DEEP SUBDUCTED CRUST: EVIDENCE FROM HYDRATED CLINOPYROXENE AND GARNET IN UHP METAMORPHIC ROCKS

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Input of water into the earth's interior is restricted to subduction zones. However, the subducted oceanic crusts are dehydrated in shallow depth, and then transformed to dry eclogite containing no hydrous minerals at temperatures over 700°C and pressures over 30 kbar. It is therefore believed that subducted crusts cannot carry significant water into deeper mantle. Recent studies reported that trace amounts of hydroxyl can be contained in nominally anhydrous minerals (e.g., Bell and Rossman, 1992). Trace water in these minerals is capable of recycling into the earth's interior at subduction zones. In order to evaluate how much water is transported into the upper mantle at deep subduction zones, we investigated the hydroxyl contents of clinopyroxenes and garnets in ultrahigh-pressure (UHP) metamorphic rocks in Kokchetav massif, which has been subducted to more than 150 km depth. As a result of infrared spectroscopic study, clinopyroxene and garnet in the eclogites contain hydroxyl up to 3020 ppm and 290 ppm, respectively. The hydroxyl absorbances increase with recrystallized pressure in both clinopyroxene and garnet. In clinopyroxene, the hydroxyl content correlates with increase of the CaEscola component, which contain vacancy on the M2 site. Same relation was reported from mantle xenoliths in Roverts Victor kimberlite pipe (Smyth et al., 1991). Because clinopyroxene and garnet represent about 40-50 volume% and 35-50 volume%, respectively, of eclogites, the bulk eclogites can contain approximately 1400 ppm hydroxyl, whereas these rocks do not contain any nominally hydrous minerals. Thus, subducted oceanic crust can carry H<sub>2</sub>O into the upper mantle, even in the absence of nominally hydrous minerals, and has an important bearing on physico-chemical properties of mantle dynamics.

Bell DR and Rossman, GR, *Science*, **255**, 1391-1397, (1992).

Smyth JR, Bell DR and Rossman GR, *Nature*, **351**, 732-735, (1991).

### CARBON SOLUBILITY IN OLIVINE

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The mass of carbon stored in the Earth's mantle probably far exceeds the combined total amount of carbon in the atmosphere, hydrosphere and sediments. However, quantifying the carbon storage capacity of the mantle is nearly impossible because data on carbon solubility in major mantle minerals are lacking. Previous attempts to measure carbon solubility in olivine proved unsuccessful because of serious experimental and analytical problems (e.g. Tingle et al., 1988).

We have carried out a series of experiments in which olivine was crystallized from a stoichiometric starting mixture of oxides in the presence of a sodium carbonate melt. Experiments were carried out at 1200°C and 10 to 35 kbar. The experiments yielded inclusion-free, clear and euhedral olivine crystals up to several 100 micrometers in size. To facilitate the unambiguous detection of dissolved carbon in these crystals, the sodium carbonate melt was enriched to contain 99 percent of the isotope 13-C.

Measurements of blank standards using a Cameca ims 6f ion probe always yielded significant amounts of 13-C due to the presence of vacuum contaminants. However, measurements of the carbon-saturated olivine samples yielded a clearly detectable signal of excess 13-C corresponding to carbon dissolved in the olivine. Some preliminary calibrations using carbon-containing glasses suggest a carbon solubility in olivine in the order of 5 to 10 ppm by weight under the conditions studied. Ongoing work will rely on reference single crystals which have been ion implanted by known doses of 13-C.

Tingle TN, Green, HW & Finnerty AA, *J Geophys Res*, **93**, 15289 - 15304, (1988).

### GRAIN SIZE DISTRIBUTION AND MICROSTRUCTURES OF GRANITOID CATACLASITES

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Deformation processes at the frictional-viscous transition are important for the understanding of earthquakes and fault mechanisms. Despite numerous field and experimental studies these processes are still poorly understood. Fracturing usually is the dominant deformation processes, but for small grain sizes diffusion creep might be of importance. The aim of this study is to compare the grain size distribution and microstructures of experimentally and naturally deformed granitoid in order to establish the deformation mechanisms.

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Digital images of cut hand specimens and thin sections obtained by optical and electron microscopy were analysed for their grain size distribution. The obtained 2D-radius of grains is recalculated to a 3D-radius. It was tested whether the grain size distribution is log-normal or power law distributed. The microstructures were also studied with light and electron microscopes. Bulk rock and phase composition of the undeformed and cataclastic granitoids were determined with XRF, XRD and EDX.

The grain size distribution for the naturally deformed cataclastic granitoids is similar on all scales of investigation. The range of grain sizes is very large (from centimetre to 0.1 micrometer). The grain size distribution qualitatively shows the same trend as found for experimentally deformed rocks (Marone & Scholz, 1989) and for naturally deformed rocks whose grain size distribution were determined by sieving (Anderson et al., 1983). Microstructural studies of the rocks show intercrystalline and intracrystalline fractures, angular to rounded particles consisting of one or more phases, mostly straight grain boundaries, but also some pressure solution rims and undulatory extinction. Equidimensional grains make up the matrix of the cataclasites. Healed fragments indicate multiple cataclasis within one cataclasite, which is visible by clasts consisting of cataclastically deformed rocks.

Anderson JL, Osborne RH & Palmer DF, *Tectonophysics*, **98**, 209-215, (1983).

Marone C & Scholz CH, *J. Struc. Geol.*, **11**, 799-814, (1989).

### PHASES FORMATION IN THE SYSTEM WITH CALCIUM, GADOLINIUM, MANGANESE, TITANIUM, AND URANIUM OXIDES: APPLICATION TO RARE EARTH - ACTINIDE WASTE IMMOBILIZATION

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Ceramics within the compositional series  $\text{Ca}_{4-x}\text{Gd}_x\text{Mn}_2\text{Ti}_2\text{O}_{20+x/2}$  ( $X = 0, 1, 2, 3, 4$ ) and a sample with  $\text{Ca}_2\text{U}_2\text{Ti}_7\text{O}_{20}$  formulation were studied as promising matrices for immobilization of rare earth and actinide constituents of high level waste (HLW). The samples were prepared by cold pressing of oxide mixtures in pellets at 200 MPa, followed by their sintering at 1400°C or melting at 1500°C, and examined with X-ray diffraction, scanning and transmission electron microscopy. At  $x=0$  and  $x=1$  a perovskite – pyrophanite assemblage occurred. The sample with  $x=2$  consisted of murataite and perovskite. Murataite was a major phase in the sample with  $x=3$  (pyrochlore and perovskite were minor phases) and the only phase in the sample with  $x=4$  prepared under oxidizing conditions (in air). The latter was composed of two murataite varieties with seven- and five-fold fluorite unit cells. The sample with the same formulation, but synthesized under reducing conditions, contained pyrochlore as an extra phase.  $\text{Ca}^{2+} + \text{U}^{4+}$  ions heterovalent substitution for  $\text{Gd}^{3+}$  ions resulted in formation of pyrochlore as major phase. The ceramic samples corresponding to the compositional series studied are composed of murataite – perovskite and murataite – pyrochlore assemblages. Gadolinium is considered as trivalent rare earths and actinides surrogate. In the first case both phases are hosts for trivalent rare earth and actinides and these HLW

constituents are partitioned among them. In the second case murataite is the host phase for rare earths and actinides (mainly trivalent, including Pu(III), Am(III), and Cm(III)) and corrosion products (manganese, iron, aluminum) whereas pyrochlore is the host phase for rare earths and tetravalent actinides (U(IV), Np(IV), Pu(IV)). This makes the system of calcium, gadolinium, manganese, and titanium oxides prospective for immobilization of rare earth – actinide fraction of HLW.

### TEMPERATURE DEPENDENCY OF Sm, Eu, Gd, AND Ga PARTITIONING BETWEEN CLINOPYROXENE AND GARNET AND ITS POSSIBLE USE AS GEOTHERMOMETER

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During the last years, the techniques of in-situ microanalytical trace element analyses (e.g. ion microprobe or laser ablation ICP-MS) have been improved so that it becomes possible to use trace element partitioning between solid phases as a geothermometer. For this reason, the temperature dependency of Sm, Eu, Gd, and Ga partition coefficients between garnets, clinopyroxenes, and silicate melts ( $D^{\text{cpx/gar}}$ ,  $D^{\text{cpx/l}}$ ,  $D^{\text{gar/l}}$ ) were experimentally investigated using a piston cylinder apparatus. Experiments were conducted at pressures and temperatures ranging from 2 to 3 GPa and 950 to 1100°C, respectively.  $D^{\text{gar/l}}$  values vary in a wider range than  $D^{\text{cpx/l}}$  ones. In terms of the lattice strain model of Blundy & Wood (1994) this is due to the fact that the size of the unstrained lattice site  $r_0$  incorporating the rare earth elements (REE) is more variable in garnet than in clinopyroxene (Klein et al. 2000). That is, the more  $r_0$  is shifted to lower values the more the partition coefficients of a fixed cation decrease. As a result, variations of  $D^{\text{cpx/gar}}$  are mainly controlled by the garnets.  $D^{\text{gar/l}}$  values of the REE are much more sensitive to changing run conditions than those of Ga. This is also supported by the observation that strongly zoned garnets are homogeneous with respect to Ga and it can be explained by the fact that the ionic radius of Ga is very close to  $r_0$  of the lattice site in octahedral coordination in garnets and clinopyroxenes. The results reveal a negative, linear correlation between the reciprocal temperature and  $\ln D^{\text{cpx/gar}}$  at fixed pressure and an increase of  $D^{\text{cpx/gar}}$  with decreasing pressure at constant temperature. Sm partitioning shows the strongest change with temperature and pressure.

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### THE GARNET-SPINEL TRANSITION IN THE SYSTEM $\text{MgO-Cr}_2\text{O}_3\text{-SiO}_2$

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The transition from spinel lherzolite to garnet lherzolite is one of the most important phase boundaries in the Earth's upper mantle. For example, our understanding of the generation of mid-ocean ridge basalts requires a sound understanding of the position of the garnet-spinel transition as a function of pressure, temperature and composition.

The garnet-spinel transition is well understood in simple (e.g.  $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ , c.f. Klemme and O'Neill, 2000a) and complex systems in fertile compositions (see e.g. Robinson and Wood, 1998), but there is scant information about the transition in refractory compositions. Moreover, rigorous thermodynamic modeling of spinel-garnet reactions in the upper mantle over a range of temperatures, pressures and compositions requires reliable thermodynamic data for Cr-bearing minerals such as Cr-spinels, Cr-bearing pyroxenes and Cr-bearing garnets, the latter of which are, unfortunately, rather unconstrained. The present study aims to fill this gap.

The simplest reaction describing the transition from spinel lherzolite to garnet lherzolite may be written as follows



However, the present study experimentally investigates the analogue reaction to (1) in an Al-free, but Cr-rich system, i.e.



Reversal experiments on reaction (2) were performed in a multi-anvil apparatus at pressures between 4.5 GPa and 11 GPa and at temperatures between 1200°C and 1600°C. The starting material contained all four phases that were synthesized prior to the commencement of the study. Analysis of run products with X-ray diffraction and electron microprobe showed which phase assemblage grew and which was consumed.

Whilst the garnet spinel transition in Cr-free (reaction 1) and in fertile compositions is known to have a positive Clapeyron slope (e.g. Klemme and O'Neill, 2000a, Robinson and Wood 1998), experimental results on reaction (2) seem to indicate a negative slope in pressure-temperature space at considerably higher pressures. Thermodynamic evaluation of our experimental results enable calculation of thermodynamic properties of khorringite garnets and, in conjunction with data for Cr-spinels and Cr-bearing pyroxenes (Klemme et al. 2000, Klemme and O'Neill 2000b), calculations of phase equilibria in Cr-rich compositions.

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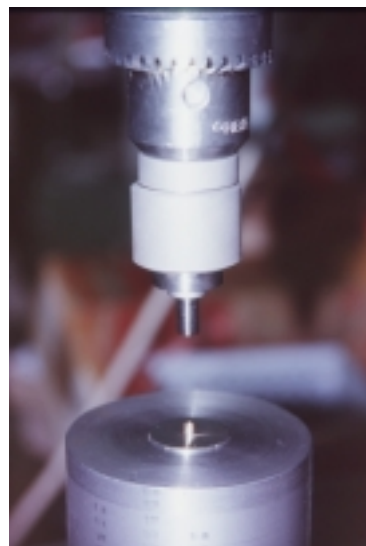
### ORBITAL HEADFORMING: A COLD FORMING PROCESS TO SEAL SAMPLE CAPSULES

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Orbital headforming is a non-impact and vibration-free cold forming process, which is widely used in industrial production. This technology can be advantageously used for sealing precious metal sample capsules for high pressure experiments. Due to the high heat input, conventional arc welding technique suffers from a high fault rate, especially if large fluid amounts should be sealed. Headforming is a cold forming process, which seals malleable metal cylinders of any size certainly and fast. The sample capsules closed in this way can be used in any kind of high pressure experiment, i.e. piston cylinder, cold seal vessel, internally heated vessel or multi anvils. Orbital headforming can be used as an alternative to conventional hammering, crimping, welding and other closing operation. Industrial orbital assembly systems are highly developed units, which are too expensive for laboratory purpose in most cases.

I present a simpler design: the capsule-closing-system (CCS) has all features of the orbital process, but runs on a simple workshop drilling machine. CCS consists of a capsule fixture and three form tools for each desired capsule diameter. The form tools are mounted off-center in a revolving chuck. The chuck and the drilling machine can be used for all desired diameters. The first tool forms the cylindrical capsule container conically without closing the capsule yet. The second tool has a flatter inner shape and seals the capsule. Finally, the third tool produces a planar surface. The capsule fixture allows a vertical adjustment of the capsule during the entire process. We run by routine gold capsules of 4 and 6 mm outer diameter, respectively. Each sealing takes about 5 minutes at a fault rate of practically zero, even if large fluid amounts are encountered. Depending on the pre-existing equipment, CCS is available from 1415 EURO up. For further information contact [www.geolab.de](http://www.geolab.de).



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### STABILITY OF SPINELLOID PHASES IN THE SYSTEM $Mg_2SiO_4$ - $Fe_2SiO_4$ - $Fe_3O_4$ UNDER UPPER MANTLE AND TRANSITION ZONE CONDITIONS

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Experiments were conducted at 4-14 GPa and 1100-1200°C in the  $Mg_2SiO_4$ - $Fe_2SiO_4$ - $Fe_3O_4$  ternary system. Mixtures of olivine<sub>ss</sub> + magnetite were used as starting materials, with olivine having  $X_{fay} = 0.1, 0.3, 0.5, 0.8$  or  $0.9$ . Particular attention was paid to the presence or absence of spinelloid III, since this polytype is isostructural with  $Mg_2SiO_4$ -wadsleyite.

Spinelloids II, III, and V are stable within the ternary system. In general the addition of Mg shifts the spinelloid stability fields towards higher pressures compared to the Mg-free system (Woodland & Angel, 2000). Spinelloid II is the first intermediate phase to appear at low pressures (~3 GPa) in Mg-poor compositions, with olivine<sub>ss</sub>+spinel<sub>ss</sub> stable in more Mg-rich compositions (e.g.  $X_{fay} \approx 0.5$  in olivine). With increasing pressure, spinelloid V effectively replaces spinelloid II between 6.5 and 7 GPa. Spinelloid III can coexist with either spinelloid II or V depending on pressure and contains less  $Fe_3O_4$  than either II or V (up to 0.94 cpfu). The most Mg-rich spinelloid synthesised to date contains 0.56 cpfu Mg and has the spinelloid V structure, which is stable up to 9 GPa. This indicates that the stabilities of the spinelloid phases are limited to the  $Fe^{2+}$ -rich half of the ternary system. In experiments with high Mg, olivine<sub>ss</sub> always coexists with a  $Fe^{3+}$ -rich spinel<sub>ss</sub>.

Magnetite-rich spinel can incorporate significant amounts of Si and Mg when coexisting with the spinelloids. However, coexistence with olivine<sub>ss</sub> in Mg-rich compositions causes a shift in the spinel composition to include a Mg-ferrite component (Si < Mg). Thus this system is pseudo-ternary since Mg substitution can be decoupled from Si in spinel. Phase relations in Mg-rich bulk compositions suggest that, in the absence of garnet and cpx, introducing even a small amount of  $Fe^{3+}$  into a depleted mantle composition could stabilize a  $Fe^{3+}$ -rich spinel coexisting with olivine.

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### HIGH-PRESSURE IR-SPECTRA AND THE THERMODYNAMIC PROPERTIES OF CHLORITOID

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Using IR radiation from a synchrotron source, high-quality absorbance spectra were obtained from polycrystalline powder of chloritoid (cld) from ambient conditions up to pressures of 10 GPa over 50 to 4000  $cm^{-1}$ . The idealized chemical composition of the chloritoid group is  $M_2Al_4O_2(SiO_4)_2(OH)_4$  where M = Fe or Mg in our experiments. All of the 42 expected fundamental IR modes were observed. These data, combined with the response of the IR bands to substitutions of Fe for Mg, and of D for H, constrained the band assignments. Heat capacity ( $C_p$ ) and entropy ( $S_o$ ) for the triclinic and monoclinic polymorphs of Fe- and Mg-cld were calculated from Kieffer-type model, using our detailed band assignments. The calculated heat capacity and entropy for the monoclinic and triclinic polymorphs differ negligibly. The results at temperatures of above 298 K are described by the following polynomial expressions in J/mol-K:  $C_p = 7.835 \cdot 10^2 - 5.170 \cdot 10^3 T^{-0.5} - 1.648 \cdot 10^7 T^{-2} + 1.934 \cdot 10^9 T^{-3}$  for Mg-cld and  $C_p = 7.848 \cdot 10^2 - 5.185 \cdot 10^3 T^{-0.5} - 1.548 \cdot 10^7 T^{-2} + 1.783 \cdot 10^9 T^{-3}$  for Fe-cld. At room temperature,  $S_o = 293$  J/mol-K for Mg-cld and 335 J/mol-K for Fe-cld. These values differ somewhat from entropy estimated from various internally consistent data bases (-3 - -9% for Mg-cld and -9 - +5% for Fe-cld). However, using our new  $S_o$  and  $C_p$  values in conjunction with the enthalpy of formation,  $H_f = -7101$  kJ/mol for Mg-cld or  $H_f = -6422$  kJ/mol for Fe-cld (estimated in this study), we can closely reproduce the experimental data for the reactions Mg-chloritoid + talc = clinocllore + kyanite (Chopin, 1985) and Fe-chloritoid = almandine + diaspore + water (Vidal et al., 1994).

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### PETROGENESIS OF OCEANIC PLAGIOGRANITES BASED ON NEW EXPERIMENTAL DATA OF HYDROUS THOLEIITIC SYSTEMS

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Evolved rocks composed of quartz diorites, tonalites, trondhjemites, so-called plagiogranites exist in the recent oceanic crust and are also present in the plutonic sections of most ophiolites. For their genesis at mid-ocean spreading systems, two models are under discussion: (1) Late-stage differentiation of a MORB-type melt (e.g., Dixon-Spulber and Rutherford, 1983) and (2) partial melting of pre-existing gabbros within high-

temperature shear zones (e.g., Flagler and Spray, 1991). In this study, we have applied recent experimental data of the experimental lab in Hannover to the plagiogranite petrogenesis in order to test both models. The role of water during the genesis of these rocks (presence of amphibole as mafic phase in many natural plagiogranites) was assessed by including water to the systems. Crystallization experiments were performed in a MORB system doped with different water contents at different redox conditions at 200 MPa (Berndt et al., submitted), and hydrous partial melting experiments were performed at 200 MPa on typical oceanic gabbros. For the experiments we have used an internally heated pressure vessel for high temperatures (up to 1250°C) equipped with a rapid-quench system and a hydrogen membrane for controlling the oxygen fugacity. Liquid lines of descent obtained via crystallization experiments are mainly controlled by oxygen fugacity and only to a little extent by water activity. SiO<sub>2</sub>-rich residual melts can be obtained under both oxidizing and reducing redox condition, but at least one fractionation step is required to reach plagiogranitic (in the narrower sense) residual melt compositions (SiO<sub>2</sub> > 70 wt%; Na<sub>2</sub>O = 4 wt%; K<sub>2</sub>O = 1 wt%). The experimental obtained liquid lines of descent are compared with differentiation trends of natural plagiogranitic suites. The results of partial melting experiments show that the process of re-melting of oceanic gabbros is ineffective in generation of SiO<sub>2</sub>-rich liquids.

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Dixon-Spulber S & Rutherford MJ, *Journal of Petrology*, **24**, 1-25, (1983).

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### AN EXPERIMENTAL STUDY ON THE INFLUENCE OF WATER AND OXYGEN FUGACITY ON THE DIFFERENTIATION OF A FERROBASALTIC SYSTEM (SKAERGAARD PARENTAL LIQUID): EXPERIMENTAL OUTLINE AND FIRST RESULTS

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The ferrogabbroic layered intrusion of Skaergaard/Greenland is one of the most impressive and best investigated rock suites on Earth. One of the proposed compositions of the parental liquid of the intrusion ("SC1") was used as starting material for extensive experimental studies under "dry" condition at 1 atm both in a system open (Toplis and Carroll, 1995) and close to oxygen (Lattard and Partzsch, 2001). The present study investigates the role of water under controlled oxygen fugacities on the phase relations and phase compositions in the same system. Thus, experiments with "SC1" under dry and hydrous conditions will result in a unique, system-consistent data set, well suited for testing quantitatively our ideas and models how the extensive parameters T, P, water activity, and oxygen fugacity will influence the differentiation of ferrobaltic systems. The crystallization experiments are performed at a pressure of 200 Mpa in an internally heated pressure vessel equipped with a rapid quench system. To control the oxygen fugacity, this apparatus is equipped with a Shaw membrane made of Pt for high temperatures enabling the accurate determination of the hydrogen fugacity prevailing in the vessels. Within the sample

capsule the oxygen fugacity is fixed due to the in-diffusion of hydrogen and the water equilibrium ( $H_2 + 0.5 O_2 = H_2O$ ). Therefore, oxygen fugacity is dependent of water activity in runs with water activity < 1, and the exact oxygen fugacity can be determined only after the experiment (after calculation of the prevailing water activity). To minimize the Fe-loss by diffusion into the capsule material, Au<sub>80</sub>Pd<sub>20</sub> pre-saturated in iron is used for the experiments. First results are presented.

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### IN-SITU X-RAY STUDY OF (Mg,Fe)O UNDER HIGH PRESSURE AND TEMPERATURE

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Magnesiowustite, (Mg,Fe)O, is one of the major minerals in the earth's lower mantle. The decomposition of ringwoodite and the possible further transition to oxide mixtures (Saxena et al., 1996) would produce oxides in the deep mantle. The stable phases and physical properties of magnesiowustite at the lowermost mantle condition are still debated and important to investigate the dynamics and structure of the core-mantle boundary. The high pressure behaviors of FeO and MgO are quite different in both crystal structure (Fei & Mao, 1994) and electronic structure (Knittle & Jeanloz, 1986). Recently, the decomposition of (Mg,Fe)O was reported (Dubrovinsky et al., 2000). However, the phase boundary and its dependence on composition are unknown. In order to clarify these problems, we performed high pressure experiments using a diamond anvil cell with both external heating and laser heating using multi-mode Nd:YAG laser. Starting materials were synthetic (Mg<sub>x</sub>Fe<sub>1-x</sub>)O (X=0, 0.1, 0.2, 0.4, 0.6). In situ X-ray diffraction experiments under high pressure and high temperature were made at Photon Factory (BL-13B2 and BL-13A) in KEK, Tsukuba. The monochromatized X-ray with energy of about 30 KeV was collimated to 30 micron square beam. The diffracted X-ray was detected using an imaging plate. Rhombohedral distortions probably due to a magnetic transition were observed for all compositions, but the initiation pressure of the distortion has a positive dependency on the Mg composition. No further phase change or decomposition was observed up to about 100 GPa and about 800 K by external heating for (Mg<sub>0.1</sub>Fe<sub>0.9</sub>)O and (Mg<sub>0.2</sub>Fe<sub>0.8</sub>)O. However, B1 phase was observed at higher temperature condition about above 1500 K using laser heating for (Mg<sub>0.1</sub>Fe<sub>0.9</sub>)O. We have also observed B1 phase above the stability field of B8(NiAs) phase in FeO. These results indicate that the stable phase of (Mg,Fe)O have B1 structure in the lowermost mantle condition.

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Dubrovinsky, L.S., Dubrovinskaia, N.A., Saxena, S.K., Annersten, H.,  
*Science*, **289**, 430-432, (2000).

### EXPERIMENTAL INVESTIGATIONS ON THE ROLE OF PARTIAL MELTING DURING THE FORMATION OF HIGH-P/HIGH-T GRANULITES

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High-P/high-T granulites are a prominent rock type in the Moldanubian Zone of the Variscan Orogen in Lower Austria. These rocks are thought to have formed in a Carboniferous subduction setting at 950 - 1050°C and 15 - 19 kbar from mainly granitoid protoliths [1 and refs. therein]. During subsequent rapid exhumation, the original peak P-T assemblage garnet + ternary feldspar + quartz ± kyanite ± clinopyroxene was variably retrogressed. One important question with considerable petrological implications concerns the role of partial melting during the formation of the South Bohemian high-P/high-T granulites [2]. In order to place further constraints on this particular problem, piston cylinder experiments were conducted in the T-range of 800 - 1000°C and in the P-range of 12 to 16 kbar. The experimental conditions should simulate the metamorphic P-T path of these granulites, with peak conditions of 1000°C, 16 kbar and the subsequent stage of nearly isothermal compression (950°C, 14 kbar and 900°C, 12 kbar). In addition, experiments at 800°C, 12 kbar and 850°C, 16 kbar were performed to investigate the upper T-limit of biotite stability. We used a natural felsic granite gneiss as starting material with the assemblage plagioclase + K-feldspar + biotite + sillimanite + quartz which is geochemically similar to the main granulite type in South Bohemia. The experiments in the temperature range of 900 - 1000°C all yielded the assemblage garnet + ternary feldspar + quartz + melt ± kyanite. Biotite is only stable in the experiment at 800°C and 12 kbar. The amount of melt in the 1000°C, 16 kbar experiment is approximately 20 volume% and decreases with temperature. The obtained melt is strongly peraluminous granitic in composition. Garnets are almandine-pyrope solid solutions but show unusually high Ti contents of approx. 1 - 2 wt% TiO<sub>2</sub>. Ternary feldspars are close to ab<sub>40</sub>or<sub>50</sub>an<sub>10</sub>. The high Ti-contents found in the experimental garnets is consistent with the presence of rutile exsolutions in some granulite garnets.

O'Brien PJ, Carswell DA, *Geol.Rundschau*, **82**, 531-555, (1993).  
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### EXPERIMENTAL INVESTIGATIONS ON THE FORMATION OF PHOSPHORAN OLIVINE IN PARTIALLY MOLTEN GNEISSES FROM A PREHISTORIC SACRIFICIAL PLACE, TYROL, AUSTRIA

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Natural olivine with significant P<sub>2</sub> contents (>1 wt.%) is extremely rare and only known from very low fO<sub>2</sub> environments (Agrell 1998). P- and Fe-rich olivine, however, may be present in slags and is known from prehistoric to medieval smelting operations (Heimann et al., 1998). In this study we describe an occurrence of unusually P-rich olivine with up to 9 wt% P<sub>2</sub>O<sub>5</sub> from partially molten metapelitic gneisses recovered from a presumably La-Tène (450-15 B.C.) age sacrificial place in the Ötz Valley, Northern Tyrol, Austria, where ritual immolation of animal offerings took place. Thus, apatite from animal bones is the likely source of phosphorus. To place constraints on the conditions of olivine formation, 1 bar melting experiments were conducted in the temperature range 900-1300°C under oxidizing (Pt-crucible) and reducing (C-crucible) conditions. The investigated gneiss samples were collected from the immediate vicinity of the firing site and have a protolith assemblage biotite + plagioclase + K-feldspar + quartz. During partial melting, foamy patches of glass containing an assemblage olivine ± clinopyroxene + witlockite + plagioclase formed at the surface of the rocks. In an attempt to reproduce the observed assemblages and textures, bone material was sandwiched between small slabs of gneiss. Under reducing conditions, melting starts around 900°C with the formation of small patches of eutectic melt. Complete breakdown of biotite with concomitant formation of olivine + witlockite + Ti-magnetite + K-rich melt was observed in experiments at 1000°C. At 1100°C clinopyroxene may be present as additional phase with large fractions of melt. Above 1200°C the observed assemblage is whitlockite + Ti-magnetite + quartz + melt. Significant amounts of P in olivine in the range 0.3 - 1.2 wt% P<sub>2</sub>O<sub>5</sub> were observed at 1100°C but only in the immediate vicinity of the bone layer. This confirms the supposed olivine formation mechanism of incongruent melting of biotite under reducing conditions in the presence of organic apatite and also indicates strong disequilibrium P-partitioning between olivine and melt.

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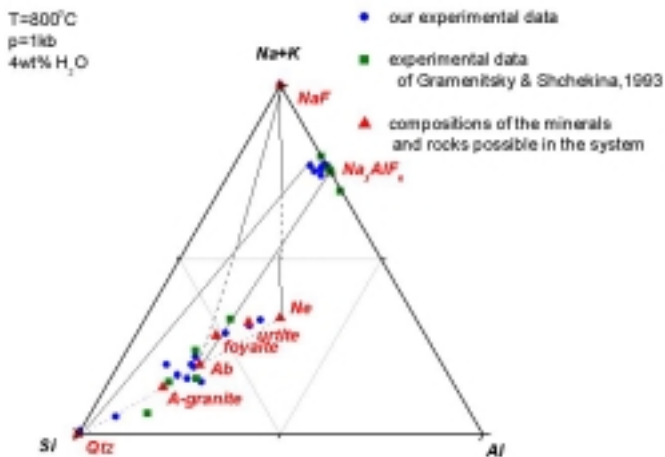
### EXPERIMENTAL INVESTIGATION OF THE MELT PHASE RELATIONS IN THE SYSTEM Na-Al-Si-O-F-H<sub>2</sub>O AT 800°C AND 1 kb

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Phase equilibrium relations have been experimentally obtained in the system Na-Al-Si-O-F-H<sub>2</sub>O at 1 kb, 800°C and water contents of 4 and 7 wt%. All experiments were performed in the Tuttle cold-seal pressure vessels inside of sealed platinum capsules. Start materials were supersaturated with the fluorine to attain the aluminosilicate and salt melts two phases equilibrium under experimental condition. The experiments were quenched and the runs products were analyzed by X-ray and Electron Microprobe Analysis. The amount of the fluorine in the aluminosilicate glass in the equilibrium with salt phase was accepted as the maximum solubility of the fluorine in the melt under experimental conditions. In the experimental products the salt melts is represented by globules, which have cryolite (Na<sub>3</sub>AlF<sub>6</sub>) or villiaumite (NaF) compositions. It is shown the nepheline-normative aluminosilicate melt coexists with villiaumite salt melt and the quartz-normative aluminosilicate melt coexists with cryolite one. In the space of (Na-Al-Si)<sub>x,y</sub>-(O-F)<sub>z</sub> triangular prism cryolite-aluminosilicate melt connods dispose over villiaumite-aluminosilicate melt ones (Figure 1).



Aluminosilicate melt in the equilibrium with two salt melts has near albite-normative composition. The solubility of the fluorine in the aluminosilicate melt is dependent on the contents of the water and silica in the system. In the nepheline-normative glass the fluorine content is about 0,5 at% in the system with 4 wt% H<sub>2</sub>O while it is about 3 at% in the system with 7 wt% H<sub>2</sub>O. The solubility of the fluorine in the aluminosilicate glasses increases gradually as the silica content is increased. In quartz-normative glasses of near-haplogranite composition fluorine contents is about 1,5 at% in the system with 4 wt% H<sub>2</sub>O and about 4,5 at% in the system with 7 wt% H<sub>2</sub>O. In extreme cases of near-silica composition of the glass the fluorine content peaks at about 6 and 14 at% in the systems with 4 and 7 wt% H<sub>2</sub>O respectively. Obtained phase relations are correlated well with the geological observational data. Cryolite is found only in the association with granitic rocks: Ivigtut (Greenland), Pitinga (Brasil), East Siberia (Russia) while villi-

aumite is only seen in the association with nepheline syenite: Lovoserskii and Khibinskii massives (Russia), Illimausak (Greenland). This work is supported by grants RFFI #00-15-98504, #01-05-64530, #01-05-64512.

### THE MECHANISMS OF FLUID MIGRATION IN LOW-PERMEABILITY ROCKS

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Rocks undergoing subduction and prograde metamorphism are subject to devolatilization reactions, fluid escape and associated compaction. High-grade metamorphic rocks are generally left with no free fluid phase or a disconnected pore space, and are impermeable to fluid flow. Fluid infiltration into such rocks during uplift and retrogressive metamorphism generally requires permeability generating mechanisms in order to reconnect the existing inclusion space or create new pore space by some deformation-related dilation process. On a large scale, the introduction of external fluids requires the presence of high-permeability channels like fractures resulted from the external stress. However, extensive interaction between the externally derived fluids and fractured rocks requires efficient mass transport away from the initial fracture into the adjacent rocks. This often occurs over distances much longer than expected from grain boundary diffusion. Here we present examples of 2 fundamentally different fluid migration mechanisms, constrained from field observations, microtextural studies and electron microprobe data. The importance of grain boundary dilation and hydrofracturing during the infiltration of non-wetting fluid is illustrated by an example of hydrated mantle-derived peridotites at the Almklovdalen, Western Gneiss Region, Norway.

Fluid migration facilitated by dilation connected to the PδV terms of overstepped metamorphic reactions is demonstrated by an example of serpentinization of a troctolite from the Duluth complex, Minnesota, USA. Results from numerical modelling of extensional microcracks in plagioclase formed due to volume change and "pressure generation" during the serpentinization of the adjacent olivine are presented.

### THE NEW LASER TIME-OF-FLIGHT MASS-SPECTROMETER FOR ANALYSIS OF GEOSAMPLE

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The new laser time-of-flight mass-spectrometer for panoramic isotopic analysis of elements in solids and powder samples (conductors, dielectrics, biological and mineralogy objects) is represented. The time of the analysis is about 10-15 minutes, an error of measurements less than 0.1%. The sensibility of is 3\*10<sup>-7</sup> %, range of mass is 1-400 a.m.u. The size of device (555\*550\*885 mm) and weight (100 kg) allow to analyze objects immediately without special preparing samples. The

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ion source uses Nd:YAG laser operating with 1064 mkm wavelength. The parameters of laser irradiation are following: 10-20 mJ pulse energy, 7 nsec pulse duration,  $(1-2) \cdot 10^9$  Wt/cm<sup>2</sup> irradiation power, 10-50 Hz pulse repetition frequency. Light optics allows laser beam to be focused into 50-70 mkm spot. Ion optics provides effective ion extraction from laser plasma, ion focusing and formation of nano-second ion packet duration. The electrostatic analyser is made in the form of cylindrical condenser. Inner and outer analyser electrodes radiuses equal 80 mm and 120 mm correspondingly. Height of analyser equals 180 mm. The special diaphragm (energy window) is installed within the analyser to decrease noise level. As a result it permits both to decrease noise level and to decrease duration of ion clouds at entrance of the detector. A new detector is used in the instrument. In fact one incorporated a couple of SEM. Each SEM is used for detecting ion packets having different intensities. Analog signals from every SEM after amplifying is digitised by a couple of 100 MHz 10 bit ADC also operation as single ADC with digital performans step of 5 ns. Both double channel mode and single channel mode can be used in the instrument. Data acquisition, accumulation and mass spectra processing is automated and carried out with the help of a Pentium-120 computer. Friendly user interface provides comfortable working condition in Windows environment. For improving accuracy of measurements authors propose to account multiply-charged ions of elements. It was introduced correction factor for avoiding discrimination of ions deferent masses. This factor includes potential of ionisation and cross-section. This innovation enables essentially to lower error.

### THE FORMATION OF K-RICH LAVAS AND ULTRABASIC NODULES OF VOLCANO VESUVIUS ACCORDING TO THE PARAGENETIC ANALYSIS AND SOME EXPERIMENTAL DATA

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The Somma-Vesuvius volcanic complex, located east of Naples in southern Italy, is a part of Thyrreinan volcanic province. It is characterized by association of silica undersaturated and potassium rich rocks. Through plinian eruptions different nodules with pyroclastic material are carried out. In this work we consider tephrites, basanites and ultrabasic nodules, which consist of olivine, clinopyroxene, phlogopite and titanian magnetite. During study of morphological features and chemical compositions of minerals, composing products of volcanic activity of Vesuvius (ultrabasic inclusions, tephrites, basanites) some parageneses of minerals were revealed. In nodules the next parageneses were studied: olivine + clinopyroxene + shpinel; clinopyroxene + phlogopite + titanian magnetite; clinopyroxene + phlogopite + titaniferous magnetite + apatite. In lavas - olivine + clinopyroxene + titaniferous magnetite; clinopyroxene + leucite + plagioclase + titaniferous magnetite; olivine + clinopyroxene + leucite + plagioclase + titaniferous magnetite. The evolution of mineral compositions and the cause of a variety of parageneses in volcanic products were determined. The occurrence of phlogopite and leucite in volcanic products proves the magmatic evolution extended with the potassium accumulation. The content of calcium increasing in pyroxenes from the ultrabasic nodules and the constant content of calcium in pyroxenes from lavas proves the calcium enrichment of magma. According to the Korzhinsky's

theory, the activity of one basic component increasing in magma promote the activity of another basic component. We explain such evolution by the magma assimilation of crustal components, which consists of dolomites and mica shales. Our data adjust with results of determination of <sup>87</sup>Sr/<sup>86</sup>Sr ratio and other isotopic characteristics. Presence of phlogopite in nodules and leucite in lavas suggests different fluid and pressure conditions of the rock formation. It is also confirms by the results of experimental study of fluid and melt inclusions in the minerals of nodules and lavas of Vesuvius.

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### SULPHATE-BEARING SODALITE STABILITY UNDER HYDROTHERMAL CONDITIONS AND T-X DIAGRAM OF Cl- SODALITE – NOSEAN SOLID SOLUTIONS

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Sulphur and chlorine play an important part in mineral forming fluid. Sodalites are the minerals which formation closely connected with the fluid conditions. The dependence of Cl-sodalite stability on concentration of NaCl in the fluid and temperature was studied earlier. The experiments on study of dependence of sulphate sodalite (nosean) formation on the fluid composition and temperature were desired at 600-800°C and P=2 kbar. The gel mixtures of nepheline and water solutions of Na<sub>2</sub>SO<sub>4</sub> were used as the start materials. The "large ampoule" method was used for the control of the salt concentration. On the experimental results the nosean is stable relatively nepheline at importance less concentrations of salt then Cl-sodalite. The phase diagram of Cl-sodalite – nosean system was constructed on data of the synthesis of these solid solution at the temperature 300-800°C and P=1-3 kbar. Starting material was a gel of nepheline. The composition of the sodalite solid solutions was set by a ratio (NaCl/Na<sub>2</sub>SO<sub>4</sub>) in an initial solution. The existing of wide immiscibility gap at the temperature 300-750°C for solid solutions of the (Cl,SO<sub>4</sub>-) bearing sodalites was shown. Solvus of this solid solution is nonsymmetric: at 700°C the sulphate mineral contains up to 24 mol.% of the Cl-sodalite, the chloride mineral of sodalite contains only 7 mol.% of sulphate one. The sulphur distribution between sodalite and fluid was studied at the same conditions. On the basis of the experimental data the analytical dependence for calculation of the sulphur mole fraction in mineral-forming solutions and for temperature determination using the compositions of the coexisting sodalite phases was derived. On this basis the estimation of temperatures and regime of fluids for the sodalite nepheline syenites of Lovozersky alkaline massif were conducted. *The work was supported by the RFBR grant 00-05-64680.*

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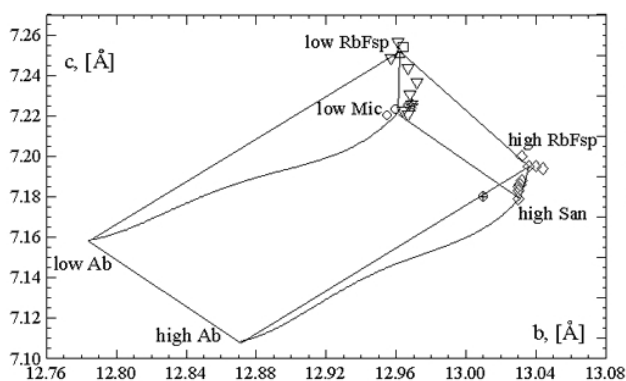
## Ninth International Symposium on Experimental Mineralogy, Petrology and Geochemistry

### THE B - C DIAGRAM OF TERNARY (Na,K,Rb)- FELDSPAR SOLID SOLUTIONS

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Ten samples of order and disorder (K,Rb)- feldspars were synthesized by the method of hydrothermal recrystallization of gel mixtures and cation-exchange of natural feldspars with molten salts. The cell parameters were refined for triclinic (Rb-microcline) and monoclinic (Rb-sanidine) series. Smith (1974) proposed the diagram in coordinates of the cell parameters b and c for determination of degree of ordering of binary (Na,K)-feldspars. In the present work the similar diagram sets up for ternary (Na,K,Rb)- feldspars based on the cell parameters of synthetic (Na,K)- feldspars (Kroll et al., 1986), Rb-microcline (Pentlinghaus & Henderson, 1979), Rb-sanidine, synthesized in the present research. The compositional dependences of the cell parameters b and c of the (Na,K)- feldspars describes by the third degree expressions (Kroll et al., 1986). The compositional dependences of these parameters for order (McMillan et al., 1980) and disorder (results of the present work) (K,Rb)-feldspars are linear. The expressions of b - c parameters for other solid solutions were accepted linear. The cell parameters of different Rb- bearing alkaline feldspars are plotted on the diagram. The data on Rb- bearing microcline by present research are close to results of McMillan et al. (1980). The parameters of natural Rb- containing microclines from Red Cross Lake pegmatite (Canada), Černý et al. (1985), are similar to the results of McMillan et al. (1980) for synthetic samples and data of the present work on natural Rb- bearing microclines from massif Orlovskiy (Zabaykalye, Russia). The natural Rb-microcline (rubicline) from San Piero in Campo (Elba, Italy) (Teertstra et al., 1998) by the cell parameters gets in area of disorder (Na,K,Rb)- feldspars. The computer program was created for degree of ordering estimation of Rb- containing alkaline feldspars using composition and cell parameters data. *The work was supported by the Junior grant of RAS # 329.*



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### AN IN-SITU X-RAY DIFFRACTION STUDY OF KINETICS OF THE POST-GARNET TRANSFORMATION

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The subducted oceanic lithosphere is mainly composed of basaltic crust and the underlying peridotite layer, in which garnet and silicate spinel are the major constituent minerals, respectively, at the depth of 500-600 km. As the slab descends into the lower mantle, garnet and spinel decompose to perovskite plus aluminous phase (post-garnet transformation) and perovskite plus ferro-periclase (post-spinel transformation), respectively. Metastability and changes of microstructures in these transformations under subduction zone conditions greatly affect on dynamics of the slab in the deep mantle. In order to clarify kinetics of these transformations, we performed high-pressure and high-temperature *in-situ* X-ray diffraction experiments combined with microstructural observations of the recovered sample. Here we report results on the post-garnet transformation kinetics of pyrope.

*in-situ* X-ray diffraction experiments were carried out using sintered-diamond multi-anvil apparatus "MAX-III" installed at KEK-PF, Japan. Two pyrope garnets with different chemical compositions were used as the starting material. One is synthetic polycrystalline pyrope  $Mg_3Al_2Si_3O_{12}$ , and another is natural polycrystalline pyrope with a composition of  $(Mg_{0.72}Fe_{0.17}Ca_{0.11})_3Al_2Si_3O_{12}$ . We observed kinetics of the post-garnet transformation at 27.0-31.0 GPa and 1273-1553 K. Time-resolved X-ray diffraction profiles of the sample were taken every 10-500 seconds during the transformation.

Dissociated post-garnet assemblages do not show the lamellar growth as observed in the post-spinel assemblages (e.g., Kubo et al., 2000), which suggests that the growth requires long-range diffusion. Obtained kinetic data indicates that the growth rate is time-dependent and significantly decreases with time contrary to the post-spinel transformation (Kubo et al., 2002). Consequently, the growth in the post-garnet transformation is much slower than that in the post-spinel transformation. Differences in kinetics of these transformations might have important implications for buoyancy of the subducted oceanic crust and formation of the garnetite layer at the top of the lower mantle.

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## EMPG IX

### Ninth International Symposium on Experimental Mineralogy, Petrology and Geochemistry

#### ALUMINUM POSITION IN SYNTHETIC Rb-FELDSPAR AS DETERMINED BY X-RAY PHOTOELECTRON SPECTROSCOPY

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Aluminums occupying two kinds of structural sites of synthetic Rb-feldspars have been detected by Al 2 p X-ray photoelectron spectra. Chemical compositions and properties of the synthetic Rb-feldspars were characterized using classical methods: electron microprobe analysis, X-ray powder diffraction analysis and Fourier transform infrared microspectroscopy. Resultantly small amounts of H<sub>2</sub>O molecule and of excess Si and Al have been detected for all single crystals of the Rb-feldspars. Moreover the quantities of excess-Al are on average 0.044 apfu and negatively correlated with those of Rb. Inasmuch as this negative correlation suggests the presence of excess-Al occupying non-tetrahedral sites of the feldspar structures, incorporation of the “Al(Al<sub>3</sub>Si)O<sub>8</sub> molecule” into the Rb-feldspars has led to presentation of appropriate endmembers for the unusual chemical compositions. Thus the representative EPMA allow the present Rb-feldspar to be identified as the non-stoichiometric feldspar with excess of Si and Al, resulting in a unit formula of (Rb<sub>0.831</sub> Al<sub>0.125</sub> Al<sub>0.044</sub>) Al<sub>0.963</sub> Si<sub>3.037</sub> O<sub>8</sub>, where the site is occupied by H<sub>2</sub>O molecule. In order to confirm excess-Al occupying the extra-framework sites of the feldspar structure, the effect of Al-Si ordering on Al 2 p X-ray photoelectron spectra of feldspars has been examined, using a monochromatized AlK $\alpha$  X-ray source, in comparison with the Rb-feldspars and several feldspar minerals. Al 2 p XPS signals of the Rb-feldspars reveal appreciably higher energy than any others of feldspars (sanidine, orthoclase, microcline and low albite) and higher one than that of bicchulite [Ca<sub>2</sub>Al<sub>2</sub>SiO<sub>6</sub>(OH)<sub>2</sub>], one of the exceptional minerals for the “aluminum avoidance rule”. Since the present Rb-feldspar is generally similar in Al-Si disordering to sanidine, shift of the Al 2 p X-ray photoelectron spectra toward the higher energy side provides a reasonable explanation for coexistence of the Al at extra-framework sites with the tetrahedral Al-O-Al linkage within the present Rb-feldspar.

#### CRYSTAL CHEMICAL CONTROLS ON RARE-EARTH-ELEMENT PARTITIONING BETWEEN EPIDOTE-GROUP MINERALS AND MELTS

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We have combined structural and physical data for epidote-group minerals (allanite, epidote s.s., clinozoisite and zoisite) with the elastic strain model of Blundy and Wood (1994) in order to qualitatively predict the changes of REE+Y mineral/liquid partitioning pattern with changing crystal structure. In addition, we present the first experimental data for partitioning of REE+Y between zoisite and hydrous silicate melt. Comparison of the predicted partitioning pattern with our new experimental zoisite/melt partitioning data and natural allanite/melt partitioning data suggests that REE+Y are preferentially incorporated into the A2 site of epidote-group minerals and that mineral/melt partitioning patterns are mainly controlled by the size and shape of the A2 site of these minerals. As a result all epidote-group minerals are expected to strongly fractionate the LREE from the HREE in mineral-melt systems. The moderately compatible behaviour of LREE and MREE in zoisite suggests that melts generated in the presence of zoisite are highly LREE and MREE depleted. In contrast, the available data for partitioning of REE between epidote-group minerals and fluids show no or little agreement with predicted REE partitioning pattern and suggest that fluid composition and oxidation state are likely to have a strong effect on the REE partitioning pattern. However, both mineral/melt and mineral/fluid partitioning data reveals that charge balancing mechanisms may exert important controls on absolute D<sub>REE+Y</sub> values for epidote-group minerals. The elastic properties of the allanite and zoisite A2 site, obtained by fitting our new and existing mineral/melt partitioning data for trivalent cations to the elastic strain model of Blundy and Wood (1994), indicate that the bulk elastic properties of epidote-group minerals are dominantly controlled by the large cation sites.

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#### DEFECT MICROSTRUCTURES AS A GENETIC FINGERPRINT OF DIAMONDS

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Diamonds occur in a great variety of natural environments such as ultra-high pressure metamorphic belts, kimberlite pipes, impact craters, and meteorites. These different occurrences of