

EUG XI



Symposium OS08

Geochronology and Stable Isotopes

Convenor

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OS08 Geochronology and Stable Isotopes

Sunday PM Session

OS08 : SUPm25 : G7 New Advances in Lu-Hf Geochronology

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The advent of multi-collector inductively-coupled plasma mass spectrometry (MC-ICP-MS) has enabled the routine high-precision analysis of Hf isotopes on significantly smaller samples (as low as 10 ng) than can be analyzed by thermal ionization mass spectrometry (TIMS). Until recently, however, the precision of Lu concentrations determined by isotope dilution on the MC-ICP-MS was not much improved over TIMS and consequently the precision of Lu-Hf ages was limited to ca. 1%. Furthermore, the accuracy of Lu-Hf ages is limited by the uncertainty of the ¹⁷⁶Lu decay constant. Here we describe our new achievements in improving both the precision and accuracy of Lu-Hf age determinations.

With their pioneering method, Blichert-Toft et al. (1997) utilized Yb that occurs naturally in samples to apply a mass bias correction to the measured ¹⁷⁶Lu/¹⁷⁷Hf, yielding an external reproducibility of ca. 1% for Lu concentrations and ¹⁷⁶Lu/¹⁷⁷Hf ratios. To improve on this level of precision, we first separate Yb from the Lu cut using an α HIBA ion exchange column, and then add Re to the purified Lu to correct for mass bias during the MC-ICP-MS run. External replicates of a solution prepared from Gore Mountain (NY, USA) garnet yield 2s.d. reproducibilities of $\pm 0.1\%$ for the ¹⁷⁶Lu/¹⁷⁷Hf ratio and ± 0.4 epsilon units for ¹⁷⁶Lu/¹⁷⁷Hf.

There is a ca. 4% discrepancy between the average λ^{176} Lu of three recent physical counting experiments (i.e., 1.86 x 10⁻¹¹yr⁻¹; Nir-el and Lavi, 1998) and the decay constant determined from a Lu-Hf isochron for ecrites that are known to be 4,550 m.y. old (1.94 x 10⁻¹¹yr⁻¹; TIMS data of Patchett and Tatsumoto 1980, refined by Tatsumoto et al., 1981). Using high precision MC-ICP-MS (Lu-Hf) and TIMS (U-Pb) techniques, we are attempting to resolve the discrepancy between these results by cross-calibration of λ^{176} Lu against the relatively well known decay constants of uranium in natural rock samples. Rapidly-cooled igneous samples were chosen to eliminate any effects caused by different closure temperatures of the U-Pb and Lu-Hf systems in the various minerals. The samples include: 1) a large (6x4x4 cm) gadolinite crystal from Evje, Norway (U-Pb upper int. age = 909.4 Ma, ¹⁷⁶Hf/¹⁷⁷Hf = 213.00 to 262.84), 2) apatite from the Phalaborwa carbonatite (South Africa; U-Pb upper int. = 2059 Ma, ¹⁷⁶Hf/¹⁷⁷Hf = 0.36022-0.36411), and 3) xenotime from Tvedestrand, Norway (U-Pb upper int. = 1091 Ma, ¹⁷⁶Hf/¹⁷⁷Hf = 0.38869-0.41714). The resulting decay constant derived from these three samples is 1.86 \pm 0.01 x 10⁻¹¹yr⁻¹, in excellent agreement with the mean value from counting experiments that was recommended by Nir-el and Lavi (1998). We are currently investigating the U-Pb-Lu-Hf systematics of additional samples (gadolinite, aeschynite, and xenotime) from other localities to verify these results and to improve the precision of the decay constant.

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OS08 : SUPm26 : G7 New Garnet Lu-Hf and Zircon SHRIMP U-Pb Data Confirm a Late Cretaceous Age and Fast Exhumation Rate for the Type-Locality Eclogites in SE Austria (Saualpe, Eastern Alps)

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Metabasic high-P assemblages from the Koralpe-Saualpe area in SE Austria are partly derived from Permian MORB-type gabbros which were eclogitized during early Alpine subduction/collision events (Thöni & Jagoutz, 1992; Miller & Thöni, 1997). We present new Lu-Hf, U-Pb SHRIMP and petrological data for two samples from the exact eclogite type-locality, Kupplerbrunn-Prickler Halt in the Saualpe (Haiÿ, 1822), in order to better constrain the crystallization history of these rocks. The mineral assemblages show dynamic recrystallization and are composed of grt + omp (Jd 40) + zo \pm ky \pm amp \pm phe + qtz + rt \pm zr. Overall retrogression (symplectite) is minor. PT estimates give 2 GPa/620°C (Miller, 1990). One ky-phe-bearing eclogite yielded a garnet-whole rock Lu-Hf age of 83.8 \pm 5.2 Ma and an eHf (t) value of +15.5, fully compatible with a MORB source. The mm-sized garnet grains show a uniform major element distribution. No diffusional retrograde resetting has been observed. From a second sample, the U-Th-Pb isotope systematics of two zircon grains have been analyzed by the SHRIMP technique. While core domains show strongly scattered ages (183-84 Ma), the weighted mean ²⁰⁶Pb/²³⁸U age for the zircon rims is 82.2 \pm 3.7 Ma (MSWD = 0.89). Together with published Sm-Nd, Rb-Sr, ⁴⁰Ar-³⁹Ar and K-Ar mineral ages for eclogites and their metasedimentary country rocks (Thöni & Miller, 1996), the new data indicate that high-P conditions in the eclogite type-locality may have persisted down to c. 85 \pm 5 Ma, followed by a fast exhumation for the time 84-76 Ma (cooling rate: c. 30-40°C/Ma, corresponding to an exhumation rate of some 3-6 km/Ma). Late zircon recrystallization may have been driven by fluids released during incipient isothermal decompression and rapid exhumation of the eclogites, following continental collision of Adria and the Austroalpine microplate some 85 Ma ago. If our garnet Lu-Hf ages are compared with those from different tectonic units of the Western Alps (Duchêne et al., 1997), it becomes evident that subduction of the SE parts of the Austroalpine basement occurred at least some 20-30 Ma earlier than in the more external zones of the future Alpine orogen.

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OS08 : SUPm27 : G7 Rb-Sr Geochronology by MC-ICPMS

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Techniques for Rb-Sr isotopic analysis by multi-collector inductively coupled plasma mass spectrometry (MC-ICPMS) have been developed that provide an important step towards improving the accuracy and precision of Rb-Sr geochronology. Simple and consistent mass fractionation of similar mass elements by ICPMS enables measurement of isotopic ratios of similar elements simultaneously. By doping Rb isotope dilution samples with natural Zr it is possible to correct the measured Rb isotope ratio for mass fractionation occurring during plasma ionization by comparison to variation between the measured value for the stable isotope ratio ⁹⁰Zr/⁹¹Zr and its natural

value (taken as 4.58824). Natural Rb analysed using this technique has yielded a fractionation corrected value of ⁸⁷Rb/⁸⁵Rb = 0.3854 \pm 2 (0.05%, 2sd). All solutions were introduced using Micromist glassware as the relative fractionation of Zr and Rb was observed to change with introduction system (e.g. desolvating nebuliser vs. wet nebuliser), probably reflecting variations in plasma conditions with differing amounts of introduced liquids. All standard measurements were interspersed with spiked samples with highly variable Rb isotopic compositions. An on-peak zeroes procedure prior to analysis is necessary to account for incomplete washouts of Rb. A typical Rb analysis lasts ~5 minutes, including washouts, and consumes ~20ng of Rb, allowing for rapid sample throughput. Careful chemical treatment of samples is required prior to analysis to remove isobaric interferences from ⁸⁷Sr.

Rigorous chemistry is also necessary for Sr isotope samples due to isobaric interferences from ⁸⁷Rb and doubly charged heavy rare earth elements. A simple clean up of the samples using one or more passes through Sr-spec resin is usually sufficient to provide clean analytes. An on-peak zeroes procedure is also necessary for Sr isotopic analyses to counteract isobaric interferences from Kr in the Ar plasma. A typical Sr analysis lasts ~15-20 minutes (including washouts) and uses ~300 ng of Sr. Replicate analyses of Sr standard SRM987, interspersed with natural samples (spiked and unspiked) yields an average of 0.71026 \pm 2 (0.003% 2sd). Preliminary investigations suggest a reproducibility of better than $\pm 0.3\%$ for the ⁸⁷Rb/⁸⁶Sr ratio. To test the technique we have dated a suite of samples (Proterozoic to Tertiary) and our results are in close agreement, and in general of better quality, than previously published age determinations. The observations made in developing this method using solution based techniques have important implications for in situ Sr isotope studies by laser ablation MC-ICPMS, in particular the potential interferences caused by doubly charged HREE, and has helped us to develop methods to monitor and account for these interferences. In addition, the increased precision of Rb isotope dilution determinations by MC-ICPMS may permit refinement of the Rb decay constant by comparison to the other geochronometers.

OS08 : SUPm28 : G7 NEPTUNE: A New MC-ICPMS For High Resolution Isotope Ratio Measurements

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The ThermoFinnigan NEPTUNE is a double focusing sector MC-ICPMS with high mass resolution capabilities. It basically combines the source of the high resolution sector ICP-MS ELEMENT2 and the multicollector of the latest ThermoFinnigan thermal ionization mass spectrometer TRITON.

The ICP source has become established during the last decade for isotope ratio measurements. It provides a high ionization efficiency for all elements, including elements with a high first ionization potential, such as Hf. It provides the possibility of external mass bias correction, successfully used for Pb and stable isotope measurements. However, isotope ratio measurements of elements of the lower mass range, such as Cr, Fe, Ca and Si are more difficult, if using an ICP source, since most isotopes are affected by molecular interferences. High mass resolution is the key to extend the power of ICPMS to these isotopic systems.

The ThermoFinnigan NEPTUNE is the first multicollector instrument capable to do high mass resolution measurements in true multicollector mode. Flat top peaks are achieved in the high resolution mode, which are essential for high precision isotope ratio measurements. In high resolution mode NEPTUNE's mass resolution is R = 4000 on several detectors at a peak plateau width of about 50-100 ppm. This is sufficient to resolve molecular interferences on Fe, Ca and Si, such as ArO, ArH or N₂H. In high mass resolution mode the transmission is reduced to about 10% relative to the transmission in low resolution setting (R=450).

Beside high mass resolution overall low background and high abundance sensitivity is substantial to high precision isotope measurements. The abundance sensitivity of

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NEPTUNE is 3 ppm under operating conditions (measured on mass 237 relative to ^{238}U). Using an additional energy filter (RPQ) the abundance sensitivity can be further improved by a factor of 10 on one channel. At 2 mass units distance in the high mass region (i.e. $^{232}\text{Th}/^{230}\text{Th}$) the abundance sensitivity drops by another factor of 10 resulting in an abundance sensitivity level of about 30 ppb on the RPQ-channel.

In this study we will demonstrate the performance of the NEPTUNE for high precision isotope ratio work for low and high mass resolution applications. The overall robustness and the stability of the system will be shown.

OS08 : SUPm29 : G7 Diffusion of Rb, Sr and Ar in Synthetic Micas

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A systematic investigation of tracer diffusion in micas has been undertaken. Various approaches have been used and results on Rb, Sr and Ar diffusivities in synthetic fluorophlogopite have been gathered. In the case of Rb and Sr, millimeter-sized single crystal were immersed in a source of fluorophlogopite stoichiometry containing the tracer. For Sr, additional experiments were performed wherein the tracer was introduced by evaporating a SrCl₂ solution on the mica surface (cleavage plane) before annealing. Both approaches yielded similar results (Hammouda and Cherniak, 2000). Samples were annealed at ambient pressure in a vertical furnace, investigated temperatures ranging between 550 and 1200°C (Sr) and 650 and 1050°C (Rb). Tracer distribution after annealing were measured in the micas by depth profiling in a direction normal to the cleavage planes using Rutherford Backscattering Spectrometry. For Ar study, fluorophlogopite single crystals were irradiated in a neutron flux in order to produce ^{39}Ar by the $^{39}\text{K}(\text{n,p})^{39}\text{Ar}$ nuclear reaction. Irradiated samples were subsequently submitted to various thermal cycles under vacuum (T between 600 and 1200°C) and ^{39}Ar fractions released at successive steps of the cycles were routinely analyzed by mass spectrometry. The following Arrhenius relationships have been obtained :

$D(\text{Sr}) = 2.7 \cdot 10^{-14} \exp(-136000/\text{RT})$ normal to cleavage,
 $D(\text{Rb}) = 3.0 \cdot 10^{-10} \exp(-240000/\text{RT})$ normal to cleavage,
 $D(\text{Ar}) = 5.6 \cdot 10^{-6} \exp(-209000/\text{RT})$

bulk exchange interpreted as being parallel to cleavage, where Ds are given in $\text{m}^2 \text{s}^{-1}$ and activation energies are in J mol^{-1} . The results show a positive correlation between tracer size and activation energy for diffusion. This correlation is still valid if O diffusivity data in natural OH-phlogopite taken from the literature are considered (Fortier and Giletti, 1991). This suggests that activation energy for diffusion in micas is dominated by elastic contributions which overcome electrostatic effects. As a consequence, Sr^{2+} diffuses faster than less charged, but larger tracers (Rb⁺, Ar). This also suggests involvement of the interlayer space of the mica where bonding is ionic, therefore weak.

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OS08 : SUPm30 : G7 The Influence of Topography on low-T Geochronology

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In thermal models that are used to explain geochronological cooling age data it is generally assumed that the isotherms are planar surfaces at depth. However, at shallow crustal levels isotherms will follow the surface topography in a damped manner and the lower the closure temperature of a chosen geochronological system is, the more important it is to consider the curvature of isotherms underneath topography for a meaningful interpretation of the data. Stüwe et al (1994) have shown that the interpretation of apatite fission track ages and ages derived from the closure of the (U-Th)/He system may be significantly influenced

by topography above 2 km, if the denudation rate is of the order of several millimeters per year or more. Their study was confined to the interpretation of a topography where the denudation rate is a constant. We have now expanded this study, in order to explore the influence of topography on low-T isotherms if the denudation rate is spatially variable, for example due to differential rain fall on the different sides of a range or due to asymmetry of the topography, for example at passive margins. In our approach, a semi-analytical solution was found that can be used to calculate the effect. We show that such asymmetric denudation should be recognizable in the record of low-T geochronological methods that date temperatures below about 150°C, if the denudation rate exceeds several millimeters per year. We expect to be able to document an increase in cooling rate with decreasing temperature on the high denudation side of a range and a decrease in cooling rate with decreasing temperature on the low denuding side of the range. Cooling ages may be twice as old on the slow denuding side than they are on the rapidly denuding side, even if the topography remains self similar through time. (This study is supported by FWF P12846-GEO.)

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OS08 : SUPm31 : G7 Alpha-Recoil Track Dating (ART-D) – Method and Application

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Alpha-recoil-tracks (ART) in mica were first described by Huang et al. (1967). These lattice defects are caused by the alpha decay of ^{238}U (10^6 times more frequent than spontaneous fission), ^{235}U , ^{232}Th and daughter products. The surface density of ARTs would allow dating Quaternary geological as well as archaeological materials (Gögen & Wagner, 2000). ARTs in dark micas are temperature sensitive and the geological closure temperature is in the range of 60°C. Dark mica contains U and Th in trace concentrations (1-5 ng/g). Their alpha disintegration releases energies of several MeV, part of which is transferred to the daughter-nucleus as recoil-energy ($\sim 10^2$ keV). The alpha-recoil nucleus slows down when interacting with the lattice atoms producing ~ 1000 lattice defects which equals one single ART. The size of latent ART in dark mica is in the range of 30 to 100 nm (Glasmacher et al., 2000). To visualise ARTs, dark mica (grain size > 5 mm) was etched with 40% HF at 25°C between 1 and 25 s. This etching procedure creates small shallow triangular shaped etch pits at the surface of the cleavage plane. The etch pits are visible and countable in the light of phase-contrast microscopy. A linear relationship of surface density of ART etch pits with etching time (t_e) was observed for phlogopite from volcanic rocks of the Eifel volcanic field, Germany, and the East-African-Rift system. Based on the experiments on phlogopite from volcanic rocks of the Eifel region, a model for the accumulation of ARTs in mica was proposed (Gögen & Wagner, 2000). The age equation combines the volumetric density (ρ) of ART with the U and Th contents. Dark micas having been below 60°C for a time range of more than 10^5 years reveal an etched ART surface density where individual tracks cannot be resolved by phase contrast microscopy any longer. Visualization of latent ART by AFM-technique enables to commit track densities beyond 10^8 cm^{-2} and thus extend the new ART-dating technique (Glasmacher et al., 2000) to an age range > 10^6 a.

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OS08 : SUPm34 : G7 ^{13}C and ^{18}O Determinations from Carbonates and DIC using Continuous Flow Techniques

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Stable Isotope analyses have been initiated and developed initially by the geochemical community. ^{13}C and ^{18}O analyses on carbonates has played a significant role in the development of the isotopic techniques. The initial analyses were done by offline preparation systems and analyses by classical dual inlet IRMS, where the sample and the reference are introduced alternatively through a change over valve in the source of the mass spectrometer. Then automated preparation systems online with the dual inlet were developed to improve performances and throughput. More recently continuous flow techniques appeared where the sample is measured as a gas pulse in a helium stream. This was first applied to EA-IRMS and then to GC-IRMS. The advantage of the continuous flow technique is generally simpler systems, high throughput and smaller sample amounts. What is presented here is a continuous flow prep system that allows different types of measurements that used to be performed through dual inlet prep systems. Besides the analyses of D/H and ^{18}O using water equilibration with respectively H₂ and CO₂ on 200 microlitre aliquots of water the MultiFlow II system is capable of performing the analyses of ^{13}C and ^{18}O on CO₂ gas released from the reaction of carbonates with phosphoric acid. The sample is loaded into the reaction vessel then put on the sample tray and then the analyses can be performed automatically including automatic delivery of the phosphoric acid. This technique can be applied to solid carbonates like calcite or dolomite but also to dissolved inorganic carbon DIC in waters. Accuracy and reproducibility runs are presented here to evaluate the technique on different types of sample sizes. International references analyses (NBS19, NBS18) are shown to illustrate the performances of this configuration. Comparison is made with Dual Inlet performances on the same type of samples. For the analyses of ^{13}C on Dissolved Inorganic Carbonates with acidification techniques, data are presented here to illustrate the reproducibility of the MultiFlow II at this configuration. Experiments are shown using artificial DIC made from known bi-carbonates to demonstrate the quality of the analyses performed with that configuration. Carbonate analyses using the MultiFlow II offer a valuable alternative to classical dual inlet techniques. Differences still remain between both approach as described in this presentation but the advantage of such a prep system is that not only it addresses some of the major applications of stable isotopes in geology, but it also allows higher sample throughput and much shorter analyses times using a much simpler system with a good level of precision on the measurements.

OS08 : SUPm35 : G7 Evidence for Archean Seawater Interaction in Komatiites from Kambalda, Western Australia Based on the Use of Hydrogen and Oxygen Isotopes

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The hydrogen isotopic composition of Archean seawater has been inferred from δD values of the ultramafic komatiite lavas (2.7 Ga) from Kambalda, Western Australia. These rocks are part of the basal section of the Kambalda Komatiite Formation. The nickel-sulphide komatiite lavas are products of large degrees of mantle melting and are deposited on the ocean floor. Seawater interaction with the cooling lava flows occurs immediately on emplacement resulting in the formation of serpentinites. The original δD characteristics of the infiltrating fluid are largely retained because hydrogen is consumed in the mineral-forming hydration reactions, therefore the hydrous mineral phases such as serpentine, chlorite and amphiboles formed during this early interaction will record the isotopic composition of the seawater at that time unless subject to further fluid-rock interaction. δD values of whole rocks and mineral separates range from -97.5‰ to -38.1‰. These

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results are in accord with the results of a similar study by Lécuyer et al. (1996) on the mafic-ultramafic Chukotat Group. They found that the δD values for the Proterozoic rocks were similar to the results from more recent seafloor alteration, implying that the δD for seawater has changed little over geological time with the assumed $\delta D = 0\text{‰} \pm 20\text{‰}$. Using the serpentine formation fields developed by Wenner and Taylor (1973), and the corresponding $\delta^{18}\text{O}$ values of the same samples, the data fall into four groups: a clearly seawater group with average values of $\delta^{18}\text{O} = +4.4\text{‰}$, and $\delta D = -62.3\text{‰}$, these indicate the serpentinisation occurred on the ocean floor. A clearly continental group with average values of $\delta^{18}\text{O} = +8.4\text{‰}$, and $\delta D = -77.6\text{‰}$, which show the influence of a continental, possibly metamorphic fluid in the formation of the serpentine. In the third group, average values of $\delta^{18}\text{O} = +7.7\text{‰}$, and $\delta D = -53.9\text{‰}$, the $\delta^{18}\text{O}$ value reflects extensive carbonate alteration that has occurred in these samples, while the δD value represents the value of serpentinisation on the ocean floor. The final group, with average values of $\delta^{18}\text{O} = +4.1\text{‰}$, and $\delta D = -86.4\text{‰}$, represents samples where the serpentine minerals have totally recrystallised while preserving the original serpentine $\delta^{18}\text{O}$ values. This study illustrates that the preservation of the earliest phase of alteration in ultramafic komatiite lavas, seafloor hydrothermal interaction, is recognisable from δD values when the $\delta^{18}\text{O}$ values have been modified by later hydrothermal events.

Lécuyer C, Gruau G, Frueh-Green GL, and Picard C, *Geology*, **24**(4), 291-294, (1996).
Wenner, DBand Taylor, HPI, *Geochimica et Cosmochimica Acta*, **38**, 1255-1286, (1974).

OS08 : SUPm36 : G7 The Oxygen Isotopic Composition of Eclogitic Rocks from the Umba-Kolvitsa Suture Zone (Kola Peninsula): A Key for Tracing the Evolutionary History from Mantle Source to Mid-Crustal Levels

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The oxygen isotopic composition of eclogitic rocks within the Por'ya Guba nappe unit of the Umba-Kolvitsa suture zone has been studied to reconstruct the fluid regime during the evolution of the rocks and to understand the significance of oxygen isotopes as tracers of processes in eclogitic rocks. Field and petrological data indicate the following evolutionary history of the rocks: (1) formation of gabbro-norite-ultrabasic complexes and related rocks through intrusion and fractional crystallisation of mantle-derived magmas (ca. 2.45 Ga); (2) granulite facies metamorphism (ca. 1.94 Ga) represented by the assemblage Opx+Grt+Pl+Cpx,Hbl (8-9 kbar, 850-900°C); (3) downloading of the metagabbro-noritic complexes to depths of about 50 km, leading to eclogitisation of basic granulites with pervasive development of the assemblage Cpx+Grt+Rt (14-16 kbar, ca. 950°C); (4) Cooling of the rocks during up-thrusting of the nappe units (ca. 1.90 Ga) accompanied by extensive hydration at amphibolite facies conditions (Hbl-Pl/Scap assemblages, 700-650°C) proceeding from pegmatite dykes and along ductile shear zones.

According to their bulk chemistry and oxygen isotope composition, the studied rocks can be subdivided into two groups: (i) basic members of the metagabbro-noritic complex (BC) ($\text{SiO}_2 < 50 \text{ wt.}\%$, $\text{MgO} > 12 \text{ wt.}\%$, and $\text{Na}_2\text{O} < 1 \text{ wt.}\%$) are characterised by "mantle-like" $\delta^{18}\text{O}$ values in the narrow range between 5.5-5.8 ‰. (ii) (normative) plagioclase-rich members of the metagabbroic complex (PC) (SiO_2 , 50-52 wt.%, MgO 7-9 wt.%, and Na_2O 2.2-2.5 wt.%) exhibit higher $\delta^{18}\text{O}$ values between 6.5-7.3 ‰.

The oxygen isotope composition is considered separately for (i) the refractory phases of the rocks, represented by garnet which preserved its original isotopic signature throughout the superimposed metamorphic processes and, (ii) the other minerals (Cpx, Opx, Pl, Amp, Rt) which appear to have exchanged oxygen during the polyphase metamorphic evolution. $\delta^{18}\text{O}$ of garnet varies from 4.6 ‰ in ultramafic rocks (40 wt.% SiO_2) to 6.1 ‰ in the basic rocks (52 wt.% SiO_2). Accounting for isotopic fractionation between garnet and the igneous assemblage during garnet growth, the estimated $\delta^{18}\text{O}$ of the igneous precursors were 5.1-6.1 ‰ (BC) and 6.7-6.9 ‰ (PC). The isotopic variations are within the range predicted for fractional crystallisation of a mantle-derived common magma.

The oxygen isotope ratios of less refractory minerals in the rocks bear evidence of a two-stage history. During hydration under amphibolite facies conditions, these phases were involved in oxygen isotope exchange with infiltrating fluids that were isotopically uniform at least in the scale of 10 to 100 metres. The calculated isotopic composition of the fluids (7.5-7.8 ‰) suggests sialic crust as the source of the fluids, presumably the underlying gneisses of the Archaean Belomorian complex. The last stage traced by the oxygen isotope systematics is characterised by a closed system behaviour of oxygen, and reflects micro-scale isotopic exchange between the less refractory minerals during cooling down to temperatures of 500-400°C.

The study was supported by grants INTAS 97-0172 and DFG-Ra205/23-1,2.

OS08 : SUPm37 : G7 High Temperature Geospeedometry Based on $^{18}\text{O}/^{16}\text{O}$ Exchange between Minerals. Application to Adirondack Uplift Rate

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We have developed a purely analytical model allowing to calculate the initial cooling rate ($s = dT/dt$) of deep rocks uplifted from an initial peak temperature T_0 to the surface. This model is based on Lasaga's analysis (1983) and was used to re-interpret Edwards and Valley (1998) (E&V) data of $^{18}\text{O}/^{16}\text{O}$ partitioning between diopside grains and a surrounding abundant calcite matrix. The model uses a mathematical analysis close to the one developed by Jaoul and Sautter (1999) and also takes advantage of the well established compensation rule for oxygen diffusion in diopside, the mineral that controls the oxygen exchange with calcite in the present case.

We use the mean isotopic concentrations of each grain measured by E&V as a function of its radius d , to calculate the associated mean temperature $= T$ of each particular crystal.

One of the new items of our model is the expression of $1/T$ that we demonstrate to be a linear function of $1/d$: that allows a solid estimate of T_0 by direct extrapolation of $1/T$ toward $1/d=0$. To is a very critical parameter to calculate s and E&V had difficulty to estimate its precise value. Our linear relation holds only when d is superior to a critical size d_c , under which grains are too small to keep the memory of T_0 in their core. From the slope of the plot $1/T$ vs $1/d$, we can extract the activation energy E of oxygen diffusion in the specific diopside of the studied rock.

The second new item is the use of the compensation rule for all oxygen diffusion data in various diopsides that linearly correlates E to the logarithm of the pre-exponential factor D_0 . From this correlation and using the value of E previously found, we can extract the corresponding D_0 , necessary for the calculation of s . Hence all diffusion parameters are directly known from the rock itself and it eliminates the problem of making a difficult choice among scattered published data.

From the plot $1/T$ vs $1/d$ one can also deduce d_c , the critical grain size, under which the linear trend of the plot is lost. The value of d_c corresponds to the case $\gamma=10$ in Lasaga's model (1983).

Knowing the values for E , D_0 , d_c and the sensitivity of the partitioning coefficient to temperature, it is now easy to calculate s at the beginning of the cooling event using for instance Lasaga's equation (40): we find $s = 80 \text{ K/Ma}$ (and $T_0 = 1100\text{K}$) while E&V estimated an average value of 1.5 K/Ma over the total duration of the uplift event (with $T_0 = 973\text{K}$).

Such an analytical approach has the huge advantage to check the sensitivity of s to each parameter included in the model.

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Jaoul O & Sautter V, *P. E. P. I.*, **110**, 95-114, (1999).

OS08 : SUPm38 : G7 A Stable Isotope Study of Smithsonite with Application to Pb-Zn Deposits of SW Sardinia, Italy

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We determined the $^{18}\text{O}/^{16}\text{O}$ fractionation factor α between smithsonite and CO_2 gas produced by reaction with phosphoric acid at 50 and 72°C, as the reaction is very sluggish at temperatures of 25°C and incomplete yields (less than 25% after 5 days) result in a low precision of oxygen and carbon isotope values (>0.3‰). The reaction of smithsonite with phosphoric acid in sealed vessels is, however, complete within 60 hours at 50°C and within 1.5 hours at 72°C. The reproducibilities of isotope values at these temperatures are generally better than 0.15‰. The α value for oxygen isotope fractionation between smithsonite and CO_2 produced by phosphoric acid reaction is 1.01044 at 50°C and 1.00979 at 72°C. Between 25 and 72°C, the temperature dependence of α is given by $1000 \ln \alpha = 5.38 (\pm 0.07) + 5.21 (\pm 0.07) * 10^5/T^2$ with T in Kelvin. In the regression we included the α value at 25°C from Sharma & Clayton (1965). We applied the techniques described above to measure the stable isotope composition of smithsonites from various oxidized Pb-Zn deposits in SW Sardinia (Italy). The $\delta^{18}\text{O}_{\text{VSMOW}}$ values range from 25.6 to 28.1‰. These values indicate formation temperatures of 25 to 32°C assuming a water isotope composition of modern local meteoric waters ($\delta^{18}\text{O}_{\text{VSMOW}} = -6.5\text{‰}$), and applying the smithsonite-water fractionation equation of Zheng (1999). The carbon isotope compositions of the smithsonites are extremely variable with $\delta^{13}\text{C}_{\text{VPDB}}$ values from -10.5 to -1.8‰. This indicates variable contributions of carbon in smithsonites from reduced organic and marine carbonate carbon. The lack of correlation between oxygen and carbon isotope values suggests that the marine carbon component in smithsonite probably derived from dissolution of marine limestones hosting the Pb-Zn ores by infiltrating meteoric waters.

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Sunday PO Session

OS08 : SUpo01 : PO
Atmospheric Lead 210 Flux Variations for
North-Eastern China during the Last 200 Years

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High resolution ²¹⁰Pb measurements on an annually laminated lacustrine sediment record recovered from Lake Sihailongwan (Long Gang Shan mountains, Jilin province) provide the first attained conception of the variability of the atmospheric ²¹⁰Pb flux in NE China within the dating frame of the lead-210 method.

Lake Sihailongwan is a unique site for this object of investigations: (1) The regional atmospheric ²¹⁰Pb flux is very high. (2) The lake is situated in a wooded region with only slight human influence. The palaeo-vegetation cover in the surrounding of the lake, as indicated by palynological investigations, shows only minor variations for the last 250 years. (3) The catchment of the lake is very small. The lake receives no inflow via streams. Only slight contributions from the inflow of mineralised groundwater are indicated by the low salinity of the lake water. There are no indications for soil erosion by surface run-off in the surrounding of the lake. (5) Allochthonous and autochthonous deposition show distinct seasonal variability, which made the microscopic identification and counting of annual sediment layers easier. The annual sediment laminations are neither destroyed by bioturbation nor by the release of methane bubbles. (6) The ratio of unsupported ²¹⁰Pb vers. supported ²¹⁰Pb is about 70 for the recent sediment. (7) The allochthonous minerogenic sediment component is around 75%. It is highly contributed by quartz and seems to originate mainly from the direct deposition on the lake surface.

The derived mean locally unsupported ²¹⁰Pb flux between 1790 and 1970 is 517 Bq m⁻² yr⁻¹ with maximum deviations of -22% and +26% of this average. Disregarding the variability of the atmospheric flux of unsupported ²¹⁰Pb relates to a maximum radiometric dating error of 8 years which exceeds the analytical uncertainty in dating about three times. A positive correlation between the unsupported ²¹⁰Pb flux and the input of remote dust is shown. The detrital flux and ²¹⁰Pb flux variations probably reflect seasonal and spatial variations of the east Asian monsoon cycle.

OS08 : SUpo02 : PO
Environmental Conditions Reflected on Shells
Mytilus galloprovincialis from Eastern Coastal
Area of Adriatic Sea

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In order to estimate the influence of environmental conditions on the excretion of resistant part of the organism samples of recent *M. galloprovincialis* were taken from entire eastern coastal area of Adriatic Sea from polluted and reference unpolluted locations in October 1998. Isotopic composition and metal concentrations in shells were measured.

M. galloprovincialis deposits daily accretionary growth bands in the outer prismatic calcite layer of its shell, which contains a continuous record of seawater temperature and chemical conditions.

Since aragonite shell layer is found to be enriched in ¹⁸O and ¹³C relative to coexisting calcite shell layer at relevant temperatures, the shell layers were sampled separately for isotopic analysis. On average, aragonite was enriched by 1.07 ‰ in ¹³C and by 0.15 ‰ in ¹⁸O. The ¹⁸O / ¹⁶O ratio of carbonate reflects the δ¹⁸O of water from which it was deposited and the temperature at which the deposition occurred. The calculated temperatures of the seawater were between 17.7 - 24.7°C for calcite and from 20.5 - 28.3°C for aragonite, which coincide with measured temperatures

on investigated locations. According to our results of δ¹⁸O and δ¹³C in shell layers of *M. galloprovincialis*, the locations of the investigated area could be separated into three groups: those with more influence of fresh water, those with less influence of fresh water and those of marine environments. Results of isotopic composition of carbon indicate that shells from locations influenced by fresh water inflow are depleted with ¹³C.

The highest contents of Mn, Ba, B, As, Ni in the shells of *M. galloprovincialis* were found in locations, where chemical and heavy industry is located and close to the sewage outlets. The highest concentrations of manganese were determined in the samples from mouth of the Neretva River, which is contaminated by industrial sewage. The highest concentrations of zinc, lead, copper were measured in samples collected close to bigger ports. The shell of *M. galloprovincialis* has proven to be a good indicator of pollution and environmental conditions of Adriatic coastal area. Palaeoenvironmental conditions of the habitat were detected. Therefore, carbonate shells can be considered as very important palaeoenvironmental indicators.

OS08 : SUpo03 : PO
Isotopic Carbon and Oxygen Composition of
Siderites as Indicator of Conditions of their
Phosphatization

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Widespread in the Sysola Depression (northeast of the Russian Plate) filled with a powerful sequence of terrigenous deposits are Jurassic siderites. Data on isotopic oxygen and carbon composition enables one to reconstruct formational environments of phosphate mineralisation paragenetically associated with siderite formation

The siderites are confined to clays, more rarely to sands and silstones. They occur as concretions to 0.8 m in diameter and layers to 0.3 m thick. Their chemical composition varies widely due to abundant isomorphism. They are commonly found as polymineral objects consisting of a large number of different carbonates. The most abundant among the minerals are Ca-Mg-siderite, Ca-siderite, Mg-Ca-siderite, and siderite. X-ray diffraction studies show a high degree of crystallinity, structural ordering of the phases and indicate the presence of more than one phase in their composition (main reflections 2.79; 2.80; 2.81; 2.82 angstrom). Where CaCO₃ account for more than 17%, free calcite is formed (main reflection 3.02 angstrom).

Isotopic composition of oxygen and carbon in siderites was studied by a conventional procedure making use of orthophosphoric acid. δ¹³C_{carb} (PDB) and δ¹⁸O_{carb} (SMOW) values for the majority of samples vary from -16.8 to +0.9 ‰ and from +21.0 to +32.7 ‰, respectively, which is characteristic of diagenetic siderites.

Lightening of the isotopic composition of oxygen is observed in samples with increased phosphorus concentration reaching 5% P₂O₅. The δ¹⁸O_{carb} value in this case varies from +13.9 to +16.2 ‰ (SMOW).

Accumulation of the phosphate-bearing sediments occurred in desalinized conditions. Siderite phosphatization must have taken place mostly at the early stages of diagenesis, in an aqueous environment enriched in phosphorus supplied by continental washdown.

OS08 : SUpo04 : PO
Recognising Heterogeneous Distribution of
Platinum Group Elements (PGE) in
Geological Materials by Means of the Re-Os
Isotope System

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The identification of uncertainties caused by sample inhomogeneity on one side and by sample preparation and measurement on the other side is a challenging task. Using chemometric methods to separate and estimate these contributions to a combined standard uncertainty of measurement (uc) of an analytical result requires complex experiments. The very difficulty of platinum group element measurement makes this task even more complex. But unless it cannot be clearly demonstrated that the nugget effect domi-

nates the reproducibility of PGE concentration determination one should be cautious interpreting the results. In this work we are able demonstrate without variation of the sample mass that it is possible to evidence a real nugget effect in one pyroxenite sample causing a large variation of replicate measurement of Os (86% RSD) relative to Re (7% RSD). Replicate measurements of several pyroxenite from Kraubath (Austria) gave an Re-Os age of 554 ±46 Ma, which is a geologically meaningful age. Estimation of the uncertainties for sample preparation and measurement by quantification of individual components, even with independent spike solution and two different instruments in the case of the pyroxenites, lead in the case of ICP-MS to a combined standard uncertainty of < 3% RSD which is considered satisfactory. Thus it is possible to identify contributions from components other than sample preparation and measurement. In another example, komatiites from the Pilbara Craton (Western Australia), we are able to demonstrate that high Re (91% RSD) and Os (50% RSD) variations are also due to the nugget effect but that the high variations in Os are actually caused by radiogenic ingrowth (and not to an a priori nugget effect of Os). A true isochron age of 2876 ±38 Ma is determined by the replicate measurement of one komatiites sample. In either case it was not possible to assign an average concentration, since the variabilities of replicate measurements are too large (e.g. through heterogeneity and/or segregation). But only by a holistic approach the concentrations and isotopic ratios of several subsamples make geochemical interpretations possible. Identifying the reasons for the nugget effect helps to understand the geochemical history and formation of the described rock samples.

OS08 : SUpo05 : PO
Combined Lu-Hf, Sm-Nd and REE Analytics to
Demagnify the Effect of Heterogeneously
Distributed Accessories

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A serious problem for Lu-Hf studies in crustal rocks is the different geochemical behavior of Lu and Hf. Most of the Hf is hosted in zircon, which is difficult to dissolve. Conventionally this problem has been solved by using HF in the attack as well as in the chemical separation of Hf. However, Lu (as well as the other REE) has a strong complexing behavior in HF, which can inhibit complete sample spike homogenization in HF bearing solutions. In order to demagnify these analytical problems, we have set up a HF-free digestion method and for combined Lu-Hf, Sm-Nd and REE analyses on whole-rock samples based on Na₂O₂ peroxide sintering. The respective elements are subsequently extracted from one homogenized sample solution. A further problem in multi isotope studies results from the fact that powder aliquots of geological rock samples tend to be isotopically distinct due to heterogeneous distribution of accessory phases. Discrete constituents with different amounts of the analytes such as zircon or garnet can be hardly homogenized down to the micrometer-level in geological rock samples. The effects with respect to the Sm-Nd system and Lu-Hf system can be seen in the overall uncertainty budget of the analyses. Reproducibilities of elemental ratios of dry aliquots from the zircon-poor basaltic rock standard JB-1 (Sm/Nd: 0.6%, Lu/Hf: 0.8%) approach those defined for pure standard solutions (Sm/Nd: 0.3%, Lu/Hf: 0.8%). (The higher uncertainty of the Lu/Hf ratio is due to cumulative weighting errors resulting from the usage of single standard solutions instead of a mixed standard solution as used for Sm-Nd. Thus, the reliability of the digestion and separation technique is demonstrated. The same reproducibility (0.5%) applies for the Sm/Nd ratio on the zircon-rich dioritic rock standard DR-N. The Lu/Hf ratio varies by 4.1% due to variable Hf contents. Relative to the aliquot size (100 mg) this variation can be modeled by the addition of 0.0015 mg zircon with a Hf content of 11000 µg/g. Therefore it can be concluded, that using this

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technique, the largest source of uncertainties for Lu-Hf determinations is found in sample heterogeneities, which made it very difficult to determine the Lu/Hf ratio of a sample. This indicates the importance of replicate analyses in particular for Archean rocks, where the accuracy of the Lu/Hf ratio significantly affects the geological interpretation of the resulting initial Hf isotopic composition.

OS08 : SUPO06 : PO U-Pb Dating of Detrital Zircon from Sediment Provenance Studies- A Comparison of Laser Ablation ICPMS and SIMS Techniques

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New developments in the U-Pb dating of zircons by laser ablation ICPMS are described and, for the first time, a direct comparison of detrital zircons dated by LA ICPMS and SIMS methods is presented. Aspiring a solution containing enriched ²³⁵U and natural Th isotopes at the same time as laser ablation makes true real-time mass bias correction. Correction for laser-induced Pb/U elemental fractionation from the laser ablation is based on mathematical treatment of time resolved data that is independent of laser ablation characteristics and does not require external standardisation. Internal corrections for mass bias and elemental fractionation eliminate the effects of variable sample matrix on isotopic ratios and improve the accuracy of U-Pb dating by laser ablation ICPMS. With the proper error propagation, the precision of U-Pb age determinations is comparable to SIMS-based ion probe dating but LA ICPMS is capable of more rapid analysis of the large number of zircons required for sediment provenance studies. Comparison of concordant laser ablation ICPMS and SIMS analyses of detrital zircons extracted from lower Silurian meta-sandstone from the Ulven Group in the west Norwegian Caledonian nappes suggests a good agreement between the two techniques.

Meta-sedimentary rocks that were accreted to the Caledonian ophiolitic terrain of western Norway during an Ordovician (~ 470 Ma) arc-continent collision have been analysed by LA ICPMS. These meta-sedimentary rocks show a distinctly different age spectra than Baltic continental margin sedimentary rocks that we also have analysed with this technique. The U-Pb detrital age spectra suggest that the Caledonian ophiolites of western Norway were not obducted onto the Baltic shield in Ordovician times. A high proportion of Achaean detrital zircons in the sedimentary rocks associated with the ophiolitic terrain rather suggests that the arc collided with the Laurentian margin at this time.

OS08 : SUPO07 : PO Dating of Serpentinization: Combined Fluid Inclusion Data and U-Pb Age of Zircon from Rodingite Blackwall (Sudetic Ophiolite, SW Poland)

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Serpentinization is a common low-grade metamorphic process affecting most of the ultramafic rocks at different geological settings. However, the age of serpentinization is typically unknown, unless it is a present-day process. The serpentinization is accompanied by Ca-metasomatism producing calc-silicate rock, rodingite, at the expense of

lithic inclusions in parent ultramafic rocks and simultaneously produced outer metamorphic aureole, i.e. blackwall. The serpentinite host rock generally represents lizardite-rich pseudomorphic variety formed from the mantle tectonite. The rodingite is composed of clinzoisite and grossularite garnet with accessory relict chess-board albite. Large transparent football-like crystals of zircon, up to 2 mm in size, were found in a chlorite-corrensite blackwall (Dubinska et al. 1995) surrounding the rodingite boudin in the serpentinite from the Sudetic ophiolite. Abraded single zircon grains were used to U-Pb isotopic analyses that yielded the crystallization age of 400±4/±3 Ma. Primary fluid inclusions in zircon crystals contain syngenetic aqueous solutions and liquid carbon dioxide; both were used to estimate by means of crossed-isochore method the temperature 280-300°C, and pressure ca. 1 kbar. U-Pb dating combined with pT data that the Sudetic ophiolite age is Early Devonian or older. Consequently, the 353±21 Ma Sm-Nd age of the Sudetic ophiolite (Pin et al. 1988) should be discarded as the age of the ophiolite formation. Dating of minerals combined with the precise studies of conditions in which the minerals form may give significant information on the ophiolite nature and evolution.

Dubinska E, Bylina P & Sakharov BA, *Clays Clay Miner*, **43**, 630-636, (1995).

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OS08 : SUPO08 : PO Early Palaeozoic Rifting, Devonian Crustal Thickening and Carboniferous Exhumation of the Variscan Orogenic Root System: Evidence from U-Pb Zircon Dating

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Key lithological units for the pre- and syn-collisional evolution of the late Variscan orogenic root were dated with U-Pb zircon ages in order to establish a geodynamic framework. The samples include highly metamorphosed and strongly deformed felsic volcanic rocks, orthogneisses, granulites, and granites. The structure of the root is characterised by an eastern Neo-Proterozoic buttress that was overthrust by an external lower crustal unit metamorphosed at 750°C and 14-15 kbar. The middle crustal unit (Svratka Complex and Gföhl unit at the base of the Varied Group, both metamorphosed at 650°C and 9 kbar) slid westwards from the external lower crust. It was overthrust from the west by the lowermost internal granulites (900°C, 16 kbar) and eclogites of the Gföhl unit (800°C, 18-20 kbar).

Subhedral and prismatic zircons from granitoid rocks of the Svratka Complex yielded U-Pb ages of 471±29 Ma and 515±9 Ma; euhedral grains from a felsite of the Varied Group gave 526±22 Ma. The deep root system comprises felsic orthogneisses and migmatites of the Gföhl unit. Single euhedral and long-prismatic grains with bipyramidal terminations from both an orthogneiss and a migmatite sample yielded a common Concordia intercept age of 394±6 Ma that is interpreted as the time of emplacement. An upper intercept age of 1374±36 Ma indicates Mesoproterozoic inheritance supported by the Nd model age of 1.64 Ga for the whole-rock. Short prismatic and stubby zircons from a Gföhl migmatite, however, yielded a significantly younger age of 353±16 Ma suggesting Pb-loss during Variscan high-grade metamorphism.

Three near spherical, multifaceted grains with no internal zoning from a felsic granulite yielded a nearly concordant age of 383±3 Ma, which combines with slightly discordant data points of additional five metamorphic grain fractions to an age of 387±11 Ma. This age is interpreted as first evidence for a granulite event some 40 Ma earlier than that determined for other granulite terrains elsewhere in the Bohemian Massif. However, zircons from a melt patch occurring within granulitic amphibolites from the external lower crustal section of the root system, yielded a U-Pb age 343±16 Ma similar to the age of high-grade metamorphism known in Bohemia.

The Cambrian/early Ordovician zircon ages from mid-crustal complexes are interpreted to reflect magmatic activity related to continental break-up. Thermally rejuvenated and weakened lithosphere was thickened and heated before extensive crustal melting and high-grade metamorphism took place from 390 to 380 Ma. Accompanied by thrusting lithologies of the lowermost part of the root over those of the external root, the exhumation of thickened lower crust occurred 350 to 340 Ma ago as suggested by the age of mid-pressure re-equilibration of a Gföhl migmatite and the age of a melt patch from the external lower crust section.

OS08 : SUPO09 : PO Episodes of Palaeozoic Sedimentation Constrained by SHRIMP Dating of Single Zircons from Paragneisses of the Schwarzwald (Germany)

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Metasedimentary amphibolite-facies gneisses and migmatites of pelitic to psammitic compositions are the most abundant rocks in the internal zone of the Variscan belt in central Europe. These rocks are hence the key to understanding the sedimentary, metamorphic and deformational evolution of a major part of the Variscan orogenic belt. However, high-grade metamorphism and deformation during Devonian and Carboniferous collision have largely obliterated documents of earlier geological records. Time constraints on pre-Variscan sedimentation periods are one of the prerequisites to unravel the premetamorphic evolution. For this reason we analysed single zircons from high-grade metasediments collected from the three different tectonometamorphic units (I-III) of the Moldanubian part of the Central Schwarzwald Gneiss Complex (Hanel et al. 1999). Dating of the individual grains was performed by ion microprobe analysis (SHRIMP). The results indicate that most of the investigated detrital grains in all three units were derived from Neoproterozoic sources (650-540 Ma), with only minor contributions of Archean (2.9-2.5 Ga) or Palaeoproterozoic components. In the studied metasediments of unit I, Phanerozoic detrital zircons are almost completely absent. Metasediments of units II and III bear zircon domains formed during magmatic events in Ordovician times (460-490 Ma). In metasediments of unit III, a detrital sub-population indicates sedimentation during Upper Silurian to Lower Devonian times. The Variscan high-grade metamorphism is recorded in zircons newly grown in granulite-facies metasediments of unit III and in rocks of unit I that experienced melt- or fluid-present metamorphism (Kalt et al. 2000). The ages of the detrital populations of the investigated rocks as a whole support Palaeozoic sedimentation of the protoliths. Most likely the Precambrian zircons were derived from sources of northern Gondwana. The Ordovician zircons found in rocks of units II and III evidence the formation of new crust at this time related to the break-off of small terranes from the northern margin of Gondwana. Based on the zircon age data of this work each of the three Schwarzwald gneiss units experienced an individual Palaeozoic evolution.

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OS08 : SU010 : PO
Ion Probe Dating of Complex Zircon from the 0.85 Ga Old Allochthonous Vistas Granite, Swedish Caledonides: A Study Aimed at Reconstructing Ancient Continental Margins

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This contribution is intended to illustrate the benefit of high spatial resolution dating technique in palaeogeographic work. The Swedish Caledonides are a stack of crustal sheets thrust onto a margin of Baltica during Early Palaeozoic closure of the Ögir Sea (Torsvik & Rehnström, in press) and Iapetus Ocean. Lower sheets derived from the volcanic rifted margin of Baltica. Dating of rocks within such sheets could accordingly inform about the extension and configuration, in the Neoproterozoic North Atlantic tract, of orogenic belts transected by the Caledonide belt i.e. the Sveconorwegian (1.2-0.9 Ga); the Transscandinavian Igneous Belt (1.81-1.65 Ga); the Svecofennian (1.91-1.85 Ga) and Archaean belts. However, because of polyphase Caledonian collision, isotope systems of rocks involved are often reset, and information obtained from complex zircons is vital.

The Vistas Granite (Seve-Kalak Superterrane) occurs as rare lenses of fairly massive syenogranite passing into augen gneiss. The granite and a coeval gabbro were emplaced into metasedimentary rocks now occurring as psammitic gneisses of high metamorphic grade. The granite-gabbro complex was intruded by a swarm of dolerite dykes with affinity to transitional basalts of continental rifts. Ion-probe (NORDSIM) U-Pb dating of two populations of zircon from the granite was performed. The first group of crystals, with oscillatory growth zoning and length:width ratios of 4-8, yielded an age of 845 ± 14 Ma (MSWD = 1.15), interpreted to date magmatic crystallization. Crystals of the second group are complex with zoned cores and rims and length:width ratio of 2. An age of 1778 ± 11 Ma (MSWD = 0.86) was obtained from cores, while rims yielded ages of c. 800 Ma. However, one core yielded an age of 2836 ± 110 Ma (MSWD = 2.5). Crystals of both populations have thin overgrowth rims yielding a good concordia age of 600 Ma, interpreted as reflecting the emplacement of the dyke swarm. Th/U ratios of 0.4-0.8 for zircons from the two groups and 0.03 for overgrowth rims support interpretations of magmatic and metamorphic events respectively.

A magmatic complex of such age and country rock is absent in the Baltic Shield basement of the Caledonide belt; however, the ion-probe data allow correlation within the belt. On equivalent tectonostratigraphic level (Kalak) but 300 km to the north (Seiland), a gabbro-granite complex of 830-700 Ma (Daly et al., 1991) occurs hosted in high-grade psammitic gneisses and Archaean orthogneiss. At 600-525 Ma, a continental riftmagmatic suite of gabbros, ultramafites, alkaline basaltic dykes, nepheline syenites and carbonatites intruded the gabbro-granite complex (Reginussen et al., 1995).

The ion-probe data also invite to correlation with the opposite (Siberian) margin of the Ögir Sea. In Central Taimyr, 850 Ma old granites of continental crustal derivation and 1800-1900 Ma model age occur in high-grade metasedimentary rocks and associated with an ophiolite complex hosting c. 740 Ma old plagiogranites and (Vernikovskiy et al., 1998).

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OS08 : SU011 : PO
U-Pb SHRIMP Geochronology of Eclogites and Orthogneisses from the Furgg Zone (Western Alps)

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The Furgg zone is an intensely deformed tectonic element of the Western Alps, separating the Monte-Rosa nappe from surrounding units. It consists of eclogite-facies metabasic boudins and a matrix of leucocratic gneisses and schists. The nature, origin and age of both the boudins and matrix rocks are a matter of controversy. An eclogite boudin within carbonatic schists of interpreted Mesozoic age (Keller, 2000) in the area of Andolla, and a leucocratic orthogneiss at Mattmark (Saas valley) were collected for SHRIMP-dating. The zircons of the eclogite are relatively euhedral, ca. 120 µm large equidimensional crystals. Cathodoluminescence (CL) pictures reveal the same type of pattern for all zircons: they consist of large magmatic cores surrounded by narrow (a few µm wide), U-poor rims formed by recrystallization during metamorphism. Ionprobe dating of the magmatic domains yielded a weighted mean age of 510 ± 5 Ma, interpreted as the crystallization time of the gabbroic protolith. The metamorphic age was not resolvable at this stage, due to the limited thickness combined with very low U-contents (1-15 ppm) of the metamorphic rims. However, various degrees of lead-loss, due to a younger metamorphic event, were identified. The youngest of these "ages" (142 ± 14 Ma; 2σ) defines a maximum age for metamorphism. Hence, HP metamorphism of these eclogite boudins should be Alpine. The zircons of a leucocratic orthogneiss at Mattmark show, in CL, a well developed magmatic core and a thin, U-poor recrystallization rim formed during metamorphism. SHRIMP-dating of the magmatic cores revealed that the protolith crystallized at 272 ± 4 Ma. Like for the eclogite zircons of Andolla, the metamorphic, narrow and U-poor rims of the Mattmark orthogneiss zircons did not yield a metamorphic age, but show lead-loss due to a younger metamorphic event which is clearly Alpine.

Our SHRIMP-results exclude a Piemont-Ligurian or a Valais origin for the eclogite precursor. The Cambrian protolith age of the eclogite boudin of Andolla and the Permian protolith age of the orthogneiss of Mattmark indicate heterogeneity of rock constituents within the same geotectonic zone. Thus, based on the SHRIMP results, the Furgg zone may be interpreted as ordinary Hercynian basement but would also be in agreement with the view that it constitutes a tectonic mélange, as has been supported mainly by lithostratigraphic arguments (e.g. Milnes et al., 1981; Froitzheim 1997).

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OS08 : SU012 : PO
⁴⁰Ar/³⁹Ar Dating of White Mica from Eclogites of the Tauern Window (Eastern Alps, Austria) and the Problem of Excess Argon in Phengites

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We carried out a combined ⁴⁰Ar/³⁹Ar laser-probe dating and electron microprobe study on white mica from the Eclogite zone and the Rote-Wand - Modereck nappe of the Tauern Window, Eastern Alps (Austria) in order to constrain the age of eclogite-facies metamorphism and deformation within this area. Mica compositions are discussed with respect to the respective muscovite (Ms), celadonite (Cel), paragonite (Pg), and margarite (Mrg) components. Chemical investigations on four white mica concentrates separated from three samples of eclogite-mylonite show similar chemical composition, with an average of

c. $Ms_{40}Cel_1Pg_{49}$. ⁴⁰Ar/³⁹Ar dating of these four concentrates yielded similar flat Ar-release patterns, with ages ranging between 31.9 ± 0.4 Ma and 33.3 ± 0.4 Ma. ³⁶Ar/⁴⁰Ar vs. ³⁹Ar/⁴⁰Ar isotopic correlation plots yielded ³⁶Ar/⁴⁰Ar intercepts ranging between 0.003446 and 0.003665, which is close to atmospheric composition (³⁶Ar/⁴⁰Ar atmos. = 0.003384), and might be interpreted in terms of minor isotopic loss instead of incorporation of extraneous ⁴⁰Ar-components. Another sample, also separated from an eclogite of the same tectonic level, can be regarded to as paragonite (Ms₄Pg₉₃Mrg₂). This sample yielded an age of 37.9 ± 2.1 Ma. The relatively large error of the age results from the low K- and therefore Ar-content of the sample. For comparison, one sample has been separated from Triassic calcite-marble of the Rote-Wand - Modereck nappe, which experienced blueschist-facies metamorphism, followed by a greenschist-facies metamorphic overprint. Analyses of the concentrate indicate that these micas can be regarded to as phengitic muscovites (Ms₆₆Cel₁₂Pg₁₂), and yielded an age of 38.9 ± 0.4 Ma. ⁴⁰Ar/³⁹Ar dating of both samples yielded flat Ar-release patterns with similar ages, which are significantly older than the ages obtained from the eclogite-mylonites. Isotopic correlation plots yielded ³⁶Ar/⁴⁰Ar intercepts of 0.003302 and 0.003108 respectively. Again, this value is close to atmospheric composition and indicates that incorporation of extraneous ⁴⁰Ar-components is negligible within these samples. Our results indicate that the Ar-isotopic system in these white micas have only slightly been influenced by incorporation of extraneous ⁴⁰Ar components or ⁴⁰Ar loss after initial closure. Therefore, the integrated ages are interpreted to be geologically meaningful, and the age difference of c. 6 Ma between white micas separated from eclogite-mylonites and those from fine-grained eclogite and calcite-marble is interpreted to be significant. We conclude that the in the area of investigation regional metamorphism at c. 38 Ma was followed by deformation under eclogite-facies metamorphic conditions at c. 32 Ma. Furthermore, our study shows, that isotope correlation plots significantly help to understand and interpret results obtained by ⁴⁰Ar/³⁹Ar age dating, and that phengites separated from eclogites do not always contain extraneous ⁴⁰Ar components.

OS08 : SU013 : PO
Rb-Sr and K-Ar Dating of Fault Gouges from the Ulsan Fault Zone, Southeastern Korea

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Dating fault activities is of great importance in evaluating structural stability of the site of public facilities such as nuclear power plants and waste repositories. Here we report Rb-Sr and K-Ar dating results of fault gouges from the Ulsan fault zone, southeastern Korea where nuclear power plants have been constructed. The gouge samples were collected from two representative fault outcrops with geological evidences of Quaternary deformation; the Ipsil and the Wonwonsa fault. The clay fractions mainly consisting of smectite with minor illite and kaolinite were separated by centrifugation and size fractions of 2-5 µm, 1-2 µm and <1 µm were analyzed for Rb-Sr and K-Ar dating. We also applied 1N and 6N HCl-leaching to <1 µm fractions, and yielded a large spread of Rb/Sr ratios. The three size fractions of the Ipsil fault samples show limited variation in ⁸⁷Sr/⁸⁶Sr and ⁸⁷Rb/⁸⁶Sr ratios. The residues after acid leaching, however, have higher ⁸⁷Sr/⁸⁶Sr and ⁸⁷Rb/⁸⁶Sr ratios than the leachates and untreated bulk samples. Six samples from the Ipsil fault show consistent Rb-Sr age of around 30 Ma and 24 Ma with reasonable initial ⁸⁷Sr/⁸⁶Sr ratios (ca.0.705). One sample shows the same Rb-Sr age (30 Ma) for all the three size fractions. The reproducibility of Rb-Sr ages suggests that the two ages are not fortuitous but geologically meaningful. Three samples from the Wonwonsa fault show Rb-Sr age of 30 Ma and 8 Ma in their acid leaching data, but the bulk samples yield an errorchron age of 49.5 ± 4.8 Ma (initial ⁸⁷Sr/⁸⁶Sr=0.7048±0.0003) which is in good agreement with the age of host granites (49.7 ± 0.1 Ma, initial ⁸⁷Sr/⁸⁶Sr=0.7048±0.00001; Kim and Kim, 1997). This means that the closed Rb-Sr system has been maintained in bulk gouge samples in spite of recurrent fault activities and associated hydrothermal alteration. The reproducibility of 30 Ma Rb-Sr age is confirmed once again in the Wonwonsa fault. The K-Ar ages both in the Ipsil and the Wonwonsa fault, ranging from 33 Ma to 45 Ma, are consistently older

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than the Rb-Sr ages, implying the presence of detrital radiogenic Ar. Ar-Ar technique and cation-exchange treatment will be undertaken to test the presence of inherited Ar and to investigate the behavior of cations during acid leaching. This study indicates that the Rb-Sr and K-Ar systems of the analyzed fault gouges were not reset during surface faulting in the Quaternary period that is suggested by ESR method (Lee and Schwarzc, 2000).

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OS08 : SuPo14 : PO Post-Hercynian Thermal History of the Easternmost Pyrenees: K-Ar Constraints and Some Methodological Consequences

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Twenty K/Ar age determinations have been made on several rock types of the Cap de Creus peninsula (Easternmost Pyrenees). The area consists of Palaeozoic sediments and igneous rocks deformed, metamorphosed and intruded by calc-alkaline granitoids and pegmatites during the Hercynian orogeny. They are deformed by mylonitic shear zones, that have been considered late-Hercynian in age by the previous authors (Carreras et al., 1980). K-Ar analyses were made on 18 minerals (muscovites, biotites and potassic feldspars) and on 2 whole rocks from pegmatites and other intrusives.

Muscovites from Pegmatites at Cap de Creus area recorded one of the most comprehensive thermal histories never found in a single mineral species. Cores of large muscovites and biotites (up to 8 cm of diameter) and bulk small muscovites and biotites (< 0.5 cm of diameter) have been sampled. The inner core of the coarse muscovite from a pegmatite has an age of 285±3 Ma that may be close to the Hercynian intrusive age. Other muscovites yield ages that range from 74 to 281 Ma. Coarse-grained biotites from pegmatites have ages ranging from 208 to 217 Ma and fine-grained biotites yield ages as young as 91±1 Ma. A perfect relationship between age and grain size was found, and it is interpreted as due to protracted cooling (1.5 to 0.3 K/Ma) of the high-grade K-feldspar-sillimanite-migmatite zone.

A whole-rock analysis on a basaltic dyke cross-cutting a mylonite in the Roses area yield a late-Tertiary age. Therefore it does not constrain a Hercynian age for the mylonites, as previously reported. One whole rock analysis from one sample of quartz-feldspathic mylonite has an upper Cretaceous age (79±1 Ma). This age is interpreted as related to the last time of hydrothermal circulation associated to the mylonitic event. The ages of 56±1 and 61±1 Ma obtained from coarse pegmatitic K-feldspars are interpreted as reset of these minerals during the early stages of Eocene compression in the Pyrenees.

From the data obtained, it is concluded that the Cap de Creus peninsula remained at depth at least for 200 Ma, and their uplift must be associated to collision between Iberian and Eurasian plates at late-Cretaceous and early Tertiary times.

Finally, some conclusions about diffusion parameters for muscovite and biotite are discussed based on our observations. If recently published muscovite diffusion parameters are correct (Kirschner et al. 1996), the diffusion in biotite in the absence of large fluid circulation must be less than previously assumed, and in fact comparable to phlogopite (Giletti, 1974).

The quality of the data obtained led us to suggest that classical K/Ar technique must be retrieved from a widespread unfortunate criticism. With appropriate sampling and convenient geological understanding it can provide precise information.

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OS08 : SuPo15 : PO Petrological Evolution of the Cryptomelane Rich Deposit at Serra do Navio, Brazil

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The possibility of using ⁴⁰Ar/³⁹Ar method on K-rich cryptomelane to date supergene weathering events (Vasconcelos et al., 1992, 1999; Henocque et al., 1998, 1999) opens the mind field of considering laterites as paleoclimatic indicators. Situated on both sides of the Atlantic Ocean, the Azul (Brazil) and Tambao (Burkina Faso) manganese deposits result from lateritic weathering of Precambrian volcano-metasedimentary manganeseiferous rocks (Mn-carbonates and Mn-garnet quartzites). The Azul ⁴⁰Ar/³⁹Ar datings suggest that during late Cretaceous (69-65 Ma) and Eocene (56-51, 43-40 Ma), wet events have led to manganese oxide occurrences (Vasconcelos et al., 1994). The absolute ages of Tambao cryptomelanes are coherent with those obtained at Azul (Henocque and al., 1998, 1999). Then, the ages suggest the occurrence of intense tropical humid periods during Eocene (58-46 Ma), at the Oligocene-Miocene boundary (25-22 Ma) and more recently (17-12, 7.5 Ma).

Our studied site, the Serra do Navio deposit (North Brazil) presents the same geological context as Azul and Tambao. The purpose of our study is to evaluate if Serra do Navio manganese deposit was formed during the same (or not) lateritization periods prevailing in the past at Azul and Tambao. In a first investigation, we define the petrological patterns of fresh and weathered rocks and the main manganeseiferous ores leading to the development of cryptomelane.

Serra do Navio primary rocks consist of three types of protoses: rhodocrosite-quartz rocks, Mn-Si-rich carbonate rocks, and gametiferous-quartzites (Bello et al., 1981). The manganese ores are mineralogically and structurally heterogeneous. However, in the deeper part of the deposit, three main mineralogical assemblages ending to cryptomelane formation are observed: 1-Rhodocrosite - manganite - cryptomelane, 2-Rhodocrosite - manganite - pyrolusite - cryptomelane 3-Spessartite - lithiophorite - cryptomelane. Towards the topsoil, the weathering of pyrolusite to cryptomelane is the main supergene process.

In the whole lateritic mantle, many cryptomelane generations of different textures are detected but it is not easy to evidence the relations between them. In the lower part and at the surface of the profile, the main cryptomelane textures are respectively, compact (absolute manganese enrichment) and porous (manganese leaching). Dissolution and recrystallization features, such as colloform and karstic structures, are observed at all depths. The younger cryptomelane generation fills up cracks crosscutting the other Mn-ores.

Datations will now be undertaken on the cryptomelane generations on the basis of our petrological data.

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OS08 : SuPo16 : PO Time Constraints for Cameroon Line Magmatism from Single Crystal Laser ⁴⁰Ar/³⁹Ar Dating of the Hossere Nigo Anorogenic Plutonic Complex

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Hossere Nigo is one of the anorogenic plutonic complexes in the northeastern part of the Cameroon line (CL). It is a dominantly gabbroic ring complex with a core formed by mangeritic and syenitic intrusive bodies.

Previous studies for the timing of magmatism in the CL indicate a time span from 67 to 31 Ma for the plutonic activity of the anorogenic complexes and from 42 Ma to Recent for the volcanic fields of the CL. No age data for Hossere Nigo were available. Therefore, six whole rock samples of Hossere Nigo were dated by the conventional K/Ar method. The whole rock ages which range from 82 to 69 Ma are, however, in conflict with the relative succession of emplacement. Therefore we performed ⁴⁰Ar/³⁹Ar laser fusion analyses on single crystals of phlogopite from one of the conventionally dated rocks. The mean ⁴⁰Ar/³⁹Ar and related isochron ages date back to the K/T boundary and are about 7 Ma younger than the K/Ar whole rock date of the sample. The overestimation in conventional K/Ar dating of whole rocks is explained by hydrothermal overprinting. The reported Ar/Ar age can serve as precise fix point for the earliest CL intrusive magmatism.

OS08 : SuPo17 : PO Permian Granite Magmatism in the Western Carpathians: New U-Pb Single Zircon Grain Results on S-Type Granites and Orthogneisses from the Velka Fatra Mountains

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In this study, we present new evidence on Permian S-type granite magmatism in the Velka Fatra Mountains. This area, located in the central part of Slovakia belongs to the Tatric unit of the Central Western Carpathians (CWC) where Permian granites were not recognized until now. The studied granite plutons ranging from tonalitic and granodioritic rocks to two mica granites and orthogneisses represent a major part of the pre-Alpine basement of the Fatra Mountains. These rocks were investigated in detail by geochemical, isotopic and geochronological methods. The granites are classified as S-type peraluminous granites with alumina saturation index (ASI) 1.05 - 1.45 and can be described as MPG granites after Barbarin (1999). The tonalites are little evolved calc-alkaline subaluminous igneous rocks and their ASI range from 0.90 to 1.15. Isotope analyses of Sr, Nd and common Pb show generally continental crust signatures for both granite types. New U-Pb single zircon grain dating results show that the two mica S-type granite crystallised 260 Ma ago. The orthogneisses show 280 Ma age, interpreted as a crystallization age for the granitic protolith of the orthogneisses. These geochronological data report the Permian age of granitoid magmatism in the Tatric unit of the Western Carpathians for the first time. Variscan syn- to post-collisional processes in the CWC generated voluminous felsic granitic magmas mostly during Carboniferous time. Permian A-type granites and the special ore-bearing S-type granites are relatively scarce. However, in the more south-eastern regions of the CWC (Veporic and Gemeric units), Permian granite magmatism and acid volcanism have been documented (Finger & Broska, 1999). Although the Permian period is generally connected with extension and/or rifting accompanied by A-type granites, our study suggests also the generation of S-type granites in the CWC. Geodynamically, these magmatic events could be connected to extension or rifting of the Variscan crust. The occurrence of S-type granites can be explained by melting of continental crust due to heat flow from the mantle.

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OS08 : SUpo18 : PO Rb-Sr Age of Kimberlites of the Kola Peninsula

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The alkaline, ultrabasic-alkaline and kimberlitic magmatism terminate the Phanerozoic evolution of the Baltic shield. The giant deposits of apatite and rare elements and diamond occurrences were formed in that period. The diamond-bearing kimberlite are revealed now in Finland and Russia. The time span of Phanerozoic magmatism was believed firstly very narrow - 380-360 Ma (Kramm et al., 1993). Then it was broadened by geochronological studies of ultrabasic-alkaline intrusions (Kurga, Sebl'yavr) - 410-360 Ma (Arzamastsev et al., 1997; Gogol et al., 1998). The age of kimberlites from Ermakovka kimberlitic field - 406±20 Ma - fallen into that time span (Gogol, Delenitsyn, 1999). The Rb-Sr age of kimberlites of Por'ya Bay is 420±5 Ma (Antonov et al., 1999). It is known that kimberlites from different provinces have the variable ages of emplacement (Krivoson, 1997). The time span of kimberlitic magmatism in North-Western Territories, Canada, is 47-539 Ma (Zinichuk, Mityukhin, 2000). The variations in isotope composition of Sr and Nd, and in model ages (TDM=0.79-2.00 Ga) indicate on different magmatic sources for kimberlites and melilitites of Arkhangelsk province (Parsadanyan, Zhuravlev, 1997). From geological data the age of Arkhangelsk kimberlites is estimated as 400 Ma (Kharkiv et al., 1998). For Rb-Sr dating two samples of medium-grained phlogopite-olivine kimberlite of crater facies were taken from Ermakovskaya-7 pipe. The samples are very similar in mineralogical and petrochemical composition. The olivine and phlogopite was picked out the one sample, and phlogopite out the second sample. The purity of phlogopite fractions is 99.5% with impurity of magnetite, and the purity of olivine is 95%. The isotope analyses were performed in Geological Institute KSC using the mass-spectrometer MI-1201T. The Rb-Sr isochrone age of kimberlites is 465±12 Ma. The similar age of 461 Ma was obtained at recalculation on three points from single sample (phlogopite, olivine, whole rock). The age obtained is different from the age of the same kimberlite, published earlier (Gogol, Delenitsyn, 1999). Most likely the low purity of the mineral fractions in early case was the reason of isotope data distortion. Initial Sr ratio is 0.7044 which is intermediate between the I and II kimberlitic groups of Arkhangelsk province. The age obtained corresponds to the onset of magmatic activity at 180-200 km depth before the main alkaline magmatic phase. The time sequence of magmatic events in NE Baltic shield is follow: kimberlitic magmatism - 465-410 Ma; alkaline-ultrabasic magmatism - 410-370 Ma; alkaline magmatism - 375-360 Ma.

OS08 : SUpo19 : PO Albian Magmatism in the Karakorum Arc (Hunza Valley, Pakistan): New Bracketing Ages for the Karakorum-Kohistan Accretion

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Dominantly mid-Cretaceous magmatism in the Karakorum Batholith marks accretion of the Kohistan Complex to Asia after northward subduction of the northern Tethys Ocean. Accretion-associated deformation developed between 102 and 75 Ma along the North Kohistan Suture. Better understanding of the arc-arc collision between the Kohistan Island Arc Complex and the Karakorum Paleo-active Margin requires precise dating of rocks involved in the collision-related deformation. U-Pb dates from single-grain zircon geochronology provide additional evidence for Cretaceous magmatism in the Hunza region of the Karakorum Arc. The magmatic ages of two deformed plutons located a few hundred meters above the locally called Karakorum Thrust help to better bracket the age of the arc-arc collision along this North Kohistan Suture. Zircon fractions were separated from weakly deformed, garnet-bearing orthogneisses (one metadiorite and one granite) that crop out along the Kohistan-Karakorum

Highway between the city of Hunza and the North Kohistan Suture. The metadiorite is a prominent mylonitic sill associated with garnet-staurolite schists in the immediate hanging wall of the Karakorum Thrust (74.573 E and 36.269 N). The metagranite is a folded, thick dyke at the very southern edge of the Hunza city. We selected euhedral, long-prismatic zircon grains whose morphology supports the igneous origin of both bodies. Other crystal types found in subordinate amounts are not essential to this study. The magmatic zircons were further selected by transmitted-light microscopy to exclude core-bearing grains. The best crystals were mechanically fragmented to remove domains influenced by the rock foliation and/or containing alien mineral inclusions such as biotite or apatite. The metadiorite is characterised by unusually big zircons (up to 50 micrometre each) with widespread internal tunnels and inclusion-bearing tips. The zircons have relatively low U concentrations (238-447 ppm) and Th/U ratios 0.52-0.81. Four data points yielded perfectly concordant ages that we consider as the magmatic one at 106.1±0.1 Ma, 95% c.l. In contrast, the granite contains relatively small and internally cleaner zircon grains. In two cases we had to combine a few very small grains with the same morphology for a single analysis. Zircons have U concentrations of 866-1579 ppm with Th/U ratios ranging from 0.14 to 0.30. Three data points are concordant and two grains (75 and 83% of concordance) show the same ²⁰⁶Pb/²⁰⁷Pb age as the concordant ones. This is interpreted as the intrusion age at 100.1±0.1 Ma, 95% c.l. In conclusion, our high-resolution/precision results document two distinct and analytically non-overlapping Albian intrusions in the then-active Karakorum Margin, with and early diorite and a slightly later granite. As a corollary, collision-related deformation that has foliated both bodies is younger than 100 Ma, which better brackets this event than previous dating.

OS08 : SUpo20 : PO U-Pb Zircon SHRIMP Geochronology of Orogenic Cycles and Supercontinents in the Southern Brazilian Shield

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A large data-base of 687 concordant (95-105%) SHRIMP U-Pb analyses of 576 zircon crystals from 43 rocks and ores, all supported by field geology and geochemistry, establishes four Precambrian orogenic cycles and two, possibly three, Proterozoic supercontinents. The investigation of one backscattered electron and one cathodoluminescence image of each zircon crystal, integrated with the isotopic results, makes evident the open-system geochemistry of zircon by domains. Zircon may recrystallize during metamorphism inwards from the rims, outwards from the cores or along thin euhedral zones, because of radioactive defect-enhanced porosity. The Uruguayan Cycle (3.4-3.0 Ga) and the Jequié Cycle (2.9-2.5 Ga) occurred in the Archean and is associated with magmatism and deformation of granite-greenstone belt terrains and extensive granulites. The Trans-Amazonian Cycle (2.25-2.00 Ga) is most significant in northern South America, where it forms granite-greenstone belts with large gold deposits, but is also widespread in the entire Brazilian Shield. This Paleoproterozoic cycle has the Encantadas Orogeny (2.25-2.10 Ga) which contains voluminous tonalites and greenstone belts, and the Camboriú Orogeny (2.10-2.00 Ga) with a predominance of K-rich granitic rocks and mostly flat-lying structure. The Brasiliano Cycle extends from 900 Ma to 500 Ma, and includes the Passinho Orogeny (900-800 Ma) and the São Gabriel Orogeny (750-700 Ma), both with abundant juvenile tonalites and andesites. Late in the Neoproterozoic, the Dom Feliciano Orogeny (650-590 Ma) produced the largest volume of granitic and volcanic rocks and created foreland basins, which were followed by the Rio Doce Orogeny (580-540 Ma). One last compressive event occurred about 530-500 Ma, the Búzios Orogeny, not observed in the southern Brazilian Shield. The absence of zircons formed in two intervals from 2.6 to 2.25 Ga and from 2.0 to 0.9 Ga is most significant, because it indicates that the southern Brazilian Shield was in the interior of three supercontinents, the first unnamed, the second recognized as Atlantica and the third Gondwana. Only a large data base of high quality SHRIMP U-Pb ages make possible such a far reaching inference. The integrated investigation of field relationships, rock petrography, zircon petrography - optical microscopy, backscattered electron and cathodoluminescence imaging, chemical analyses of zircon crystals by electron microprobe, and voluminous, high-quality, concordant U-Pb isotopic SHRIMP analyses of zircon crystals led to the

identification of orogenic cycles and supercontinent cycles in the southern Brazilian Shield, which has wide application to South America. Only the investigation of the internal structure of zircon crystals and diffusion processes involved made possible this advance in SHRIMP geochronology and impact on the understanding of the tectonic evolution of the southern Brazilian Shield.

OS08 : SUpo21 : PO First Isotopic Age Determination of Mid- Proterozoic Granulite Facies Metamorphism in the Epupa Complex, NW Namibia

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The Epupa Complex of NW Namibia, situated at the southwestern margin of the Congo Craton, consists of pre-Pan-African high-grade metamorphic gneisses which are intruded by the large anorthositic Kunene Intrusive Complex. Granulite facies rocks of various compositions occur in major parts of the study area (Brandt et al., 2000). P-T conditions for the granulite facies metamorphism were calculated at about 900-1000°C and 7-9 kbar (see accompanying abstract by Brandt et al.).

Garnet-bearing granulite samples were selected for isotopic age determination by the Pb-Pb stepwise leaching method (Frei & Kamber, 1995; Frei et al., 1997) applied to garnet. Metapelitic granulites comprise the peak metamorphic paragenesis: garnet-sillimanite-perthitic K-feldspar-plagioclase with rutile, zircon and Hercynite as accessory phases. Charnockitic gneisses display coexisting garnet and orthopyroxene (I) porphyroblasts in a quartz-feldspar matrix. In some samples garnet is replaced by cordierite-orthopyroxene (II) coronas. Accessories are zircon, ilmenite and Hercynite.

Garnet fractions of metapelitic and charnockitic granulite samples yielded similar ages of 1430±68, 1446±31 and 1461±59 Ma. All samples show increasing radiogenic Pb release in each leach step with the last, residual step (step 5) being by far the most radiogenic. The garnet leach pattern from step 1 to 4 indicates rather low U/Pb but increasing contents of thorium-bearing phases. In contrast, the residual step has high U/Pb and low Th/Pb contents which is most probable due to submicroscopic inclusions of zircon. However, judging from the lead isotope ratios, the zircon inclusions in the garnet grains do not influence the slope in the ²⁰⁶Pb/²⁰⁴Pb vs. ²⁰⁷Pb/²⁰⁴Pb plots of all samples. This implies that these zircon inclusions have grown during the same high temperature event as the garnets.

In summary, a major granulite facies event has been dated in the Epupa Complex at 1450±50 Ma. To place further constraints on the tectono-metamorphic evolution of the Epupa Complex additional age determinations on the granulite and associated amphibolite facies rocks in the study area are in progress.

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OS08 Geochronology and Stable Isotopes

OS08 : SUPO22 : PO Geochronological Evidence for Late Proterozoic Magmatic and Metamorphic Events in the Eastern Ghats Belt, India: Implications for the India-East Antarctica Correlation

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Extending along the eastern coast of India, the Eastern Ghats Belt (EGB) forms an integral part of the once continuous high-grade terrain of Precambrian Gondwanaland. In paleotectonic reconstructions of the supercontinents Rodinia and Gondwana, the EGB is considered as a segment of the global SWEAT orogen (Moore, 1991) and juxtaposed with the Rayner Complex and Northern Prince Charles Mountains of East Antarctica, implying similarities of their crustal evolution. To test this reconstruction, U-Pb zircon dating of enderbite and charnockite intrusive complexes as well as Th-U-total lead dating of monazite in the Western Charnockite Zone (WCZ) of the EGB south of the Godavari graben has been carried out. SEM and CL studies of morphology and internal structure of the zircons indicate their magmatic (type I) and metamorphic (type II) origin. The discordia constructed for two abraded fractions of type I zircon from enderbite has an upper intercept with the concordia at 1720±6 Ma that corresponds to the crystallisation age of the enderbite protolith. Type I zircon from charnockite yields a discordia with an upper intercept with the concordia at 1716±8 Ma and a lower intercept at 882±117 Ma (MSWD=0.1). Type II zircons are concordant with an age of 1595±2 Ma. The U-Pb zircon data thus show that the protoliths of enderbites and charnockites intruded the supracrustal granulites at ca. 1720 Ma and underwent granulite facies metamorphism at ca. 1600 Ma. The Th-U-total lead ages of monazites from diatexitic granulites, leptynites and charnockites range from 1638±29 to 1543±16 Ma. These data clearly show that the WCZ, in contrast to the extended eastward domains of the EGB (Mezger and Cosca, 1998, was not affected by high-grade metamorphism during the Grenvillian and Pan-African events. The separating Sileru lineament (Ramakrishnan et al., 1998) thus represents a major terrane boundary. The WCZ has no counterpart in East Antarctica. Its magmatic and metamorphic events, however, correlate with 1.7-1.6 Ga events in the south-western United States and Australia. The eastward domains of the EGB and the Rayner Complex and Northern Prince Charles Mountains of East Antarctica, on the other hand, show a comparable crustal evolution with mainly Paleoproterozoic (2.4-2.2 Ga) and late Archaean (2.8-2.6 Ga) Nd model ages, a major tectono-thermal-magmatic event at ca. 1.03-0.94 Ga and a marked Pan-African (ca. 0.63-0.5 Ga) overprint (cf. Mezger and Cosca, 1989; Kovach et al., 1998; Rickers et al., 2001), which confirms their juxtaposition in the Rodinia and Gondwana supercontinents.

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OS08 : SUPO23 : PO Age of the Marunkeu Metamorphic Complex in the Polar Urals: Neoproterozoic or Palaeozoic?

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Statistic processing of isotopic dates (K-Ar, Pb-Pb) on metamorphic rocks in the Marunkeu block highlighted a distinct age maximum in the 800-600 Ma range. The maximum is based on eclogite and their likely protoliths (peridotites, gabbro) dates, so it seems reasonable to link it to eclogite-grade metamorphism. In recent years, however,

Sm-Nd and Rb-Sr isochronous mineral dates have been published (~360 Ma) that are interpreted as indicating the time of eclogite formation while completely ignoring available data, though even K-Ar amphibole dates are commonly equal to 600 Ma and Pb-Pb zircon dates can be as old as 750 Ma. Such interpretation seems premature, because it does not take into account geochronological data on other rocks in the block as well as data on endogenic evolution of the nearest geologic environment. If it is remembered that the estimated temperature of eclogite and amphibolite formation is 550-650°C, i.e. it virtually coincides with the K-Ar isotopic system's closure in amphiboles, then the eclogite and amphibolite dates are real ones, maximum close to true eclogite and amphibolite ages. In order to check this inference, we did Rb-Sr whole rock dating of metamorphic rocks from Slud'anaya Gorka and obtained the following data. Amphibolites were dated at 772±16 Ma, peridotites - 808±140 Ma. Eclogites yielded three ages: 626±20, 478±15, and 399±37 Ma. This seems to complicate the situation, because the new dates fall into the Palaeozoic; however, being obtained on muscovite-bearing rocks, they, in all probability, reflect more recent processes in the metamorphic evolution of the eclogites. This suggestion is confirmed by K-Ar dates on muscovites from the eclogites and the enclosing rocks, ranging 480-340 Ma. Furthermore, the rocks that yielded the 360 Ma date, also contain mica. The younger age may be due to substance remobilisation resulting from eclogite exhumation or tectonic events in the evolution of uranides on the Urals' eastern slope. It is only a suggestion, but one that even at this stage is supported by factual data. Thus, peaks of post-ophiolite granitoid magmatism in Shuch'ya palaeo-island arc system are attributed to 400 and 360 Ma, and it is hardly possible that the observed age correlation in the west and east of the Polar Urals is mere coincidence.

OS08 : SUPO24 : PO Isotopic and Chemical Indications on the Origin of the Mixtequita and the Chiapas Batholiths in SE Mexico: Evidences for Inherited Grenville and Panafrican Basement?

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During the past few years a model has been widely accepted, which groups all Grenville-age rocks in Mexico within one Proterozoic microcontinent called Oaxaquia, that underlies Mexico from the NW to the SE (Ortega-Gutierrez et al., 1995). The easternmost exposure of Proterozoic granulites in Mexico is the Guichicovi complex at the western edge of the Tehuantepec isthmus (Weber & Köhler, 1999). These granulites are intruded by the Mesozoic Mixtequita batholith (MB). The Chiapas batholith (CB) extends from the east of the Tehuantepec isthmus to the border of Guatemala, and is composed mainly of deformed granulites. Almost nothing is known about basement in the CB, however, we observed orthogneisses, amphibolites, paragneisses, and marbles intercalated within the CB granulites. In order to test possible similarities between MB and CB, and to get some information about basement intruded by the CB, we present Rb-Sr, Sm-Nd isotope, and geochemical data from both intrusive complexes, and preliminary results from U-Pb zircon dating of an orthogneiss from the CB.

Rb-Sr biotite/whole rock ages of granulites from the MB are between 228±8 and 239±12 Ma. A gabbro has a biotite/whole rock age of 181±9 Ma. These age data, together with trace and RE element distributions, indicate two separate igneous events in the MB. CB samples yielded similar biotite/whole rock ages (237±12, 177±9 Ma). Nd- and Sr-isotope data and Nd model ages (1.15-1.25 Ga) of granulites from the MB show an influence of reworked Precambrian lower crust. The CB granulites and gneisses display a stronger variation in their Sr- and Nd- isotope compositions and Nd model ages (1.04-1.27 Ga). Some CB rocks indicate a similar lower crust influence than the MB, but some gneisses display more upper crust like isotope signatures. One amphibolite has a Nd model age of 1.4 Ga, indicating that its precursor is of Precambrian age. U-Pd isotope data of four zircon fractions from a CB orthogneiss are strongly discordant. They yield a lower intercept age at

227±(8-11) Ma which is within the errors of the biotite/whole rock age (237±12 Ma). The upper intercept age at 664±84-76 Ma is not well constrained, but it shows that a late Precambrian or Panafrican event has affected the CB basement, and inherited lead of Grenville age may be present in these zircons, also.

To date, it has not been possible to demonstrate granulite facies rocks in the CB, but our data indicate that at least part of the CB formed within the same magmatic arc than the MB, which doubtless intruded Grenville basement. The possible influence of a Panafrican event, strong deformation, and some upper crust isotope signatures demonstrate, however, that the CB is much more complex and not simply the continuation of the MB towards the east.

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OS08 : SUPO25 : PO Crustal Evolution of Indo-Chinese Region: A Perspective from Northern Vietnam

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The crustal history of Indochinese region as viewed from northern Vietnam was initiated in Archean. Nd model ages suggest that a continental crust as old as 3.4-3.1 Ga may have been separated from a depleted mantle source. Since then, the region has experienced a complicated tectonic evolution - three thermal episodes during Precambrian and four episodes during Phanerozoic. Late Archean magmatism (2.9-2.8 Ga) with TTG geochemical characteristics, a Middle Proterozoic (1.8-1.3 Ga) event corresponding to the major episode of crustal formation in the South China Block and a Neoproterozoic (850-740 Ma) mantle plume event that initiated the breakup of Rodinia are the major thermal events in Precambrian. The result was a series of collision and extrusion events. Silurian (428±5 Ma) magmatism occurred to relate to the Caledonian orogeny. The recognition of a ca. 280-240 Ma magmatic arc along the northern margin of the Indochina Block and a ca. 240 Ma metamorphic belt in the Song Ma area suggests the collision of Indochina with South China has occurred in the Early Triassic. After suturing, widespread intraplate magmatism affected Vietnam, during Late Jurassic to Cretaceous (145-75 Ma) time, which most likely corresponds to the Yanshanian "orogeny" in South China Block related to lithospheric relaxation and extension. Finally, a mid-Tertiary (ca. 40-25 Ma) event is recorded by gneisses exposed within the Red River shear zone owing to the continental extrusion resulting from the India-Asia collision.

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OS08 Geochronology and Stable Isotopes

OS08 : SUpo26 : PO The Kontum Massif (Central Vietnam): A Composite Indosinian and Older Basement

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The Indochinese peninsula is a composite of several continental blocks which have been amalgamated during the Indosinian Orogeny. The territory of Vietnam, to the East, is itself composed of several terranes which have experienced a complex succession of tectonometamorphic and magmatic events. The northern half of Vietnam is mainly occupied by the Truong Son belt which comprises metamorphic series, either exposed as mylonites along linear NW-SE dextral strike-slip fault zones or as dome structures of similar trend. The age of the major tectonometamorphic episode, which affects these terranes, is 240-250 Ma, as determined by numerous Ar-Ar datings. This means that the Truong Son belt has essentially acquired its present-day structure during a Permian-Early Triassic event of the Indosinian Orogeny. However, at least two areas in this belt have been heavily reworked during the Cenozoic, i.e. between 20 and 35 Ma: the Red River Fault Zone and the Bu Khang-Phu Hoat massif. To the south of the Truong Son belt, the Kontum Block exhibits some peculiar tectonic and petrological characters. Four main petrological units are identified: - An autochthonous basement of granulitic metamorphic facies rocks, showing decompression LP granulitic corona-forming textures. Garnet-biotite geothermobarometry confirms the HT-LP peak, which has been reached during the growth of the specific assemblages. - A mesozonal orthoderived formation, may be in allochthonous position, occupies the western side of the Massif (Dak To Formation). It is a calc-alkaline to sub-alkaline formation containing granitic to dioritic orthogneisses and some gabbros-derived amphibolites. Both, the granulitic and the mesozonal Dak To units yield Paleozoic ages up to 450 Ma, with different degrees of rejuvenation linked to subsequent magmato-metamorphic events. The granulitic series are widely intruded by charnockitic magmas. Many of them are more or less differentiated, giving rise to acidic to intermediate and basic rocks. Only one assemblage occurs in these charnockites. Biotites from these charnockites yield ages ranging from 226 to 264 Ma. - Two other major metamorphic formations are represented in the Kontum massif by schists and micaschists formed under greenschist amphibolite facies conditions. They more or less overprint the granulitic and charnockitic suites. All dated biotites, amphiboles and muscovites from these series yield ages between 230 and 250 Ma. These geochronological data imply that the tectonometamorphic event represented in SE and S Asia and related to the Indosinian Orogeny, also occurred in the Kontum Block, which therefore was not at that time a distinct and separate block with respect to the northern segment of the Truong Son belt. Moreover, the Kontum Block, in its whole, cannot longer be considered as an Archean fragment of the Gondwana, on the simple argument of the occurrence of charnockites.

OS08 : SUpo27 : PO Thermal Evolution of a Late Archean Terrane: Sm/Nd, Rb/Sr, ⁴⁰Ar/³⁹Ar and U-Th/total Pb Geochronology of the Isorssua Complex (West Greenland)

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The Isorssua complex is predominantly gneiss, grt-crd-sil micaschist and amphibolite metamorphosed to the upper amphibolite facies (700-750°C; 4-6Kb) during the late Archean (~2620-2680 Ma, total Pb zircon, Masson et al., 1971). These rocks have been geochronologically re-investigated in order to constrain their late metamorphic cooling evolution and to test the possibility of dating biotite inclusions in garnet, by *in-situ* ⁴⁰Ar/³⁹Ar UV-laser probe. Whole rock-garnet Sm/Nd analyses yield ages between 2474-2487 Ma. Chemical zonation in garnets indicates retrograde chemical re-equilibration suggesting the geochronological data record cooling ages. In addition, ⁴⁰Ar/³⁹Ar data from hornblende give ages of 2449-2504 Ma, consistent with the Sm/Nd ages. A sample containing large garnets (1.5-2 cm) preserving prograde chemical zonation produced a Sm/Nd age of 2639 Ma (in agreement with the previous total Pb zircon ages). Later cooling has been estimated by *in-situ* U-Th/total Pb dating (electron microprobe analyses) of small monazites, indicating ages of 2361-2379 Ma. The low temperature cooling history is difficult to estimate because of the thermal overprint during the Ketilidian orogenesis. Ar/Ar and Rb/Sr data on biotites indicate that the isotopic system was partially reset, providing ages between 2200 and 1800 Ma. However, an ⁴⁰Ar/³⁹Ar age on phlogopite (2221 Ma) could represent the latest cooling stage recorded in these rocks prior to the Ketilidian overprint. *In-situ* ⁴⁰Ar/³⁹Ar UV-laser analysis of biotite inclusions in garnet did not yield prograde metamorphic ages, probably because the garnets are highly fractured which is sufficient to permit argon loss. These geochronological data are consistent with a continuous cooling rate of ~1°C/Ma, from 2600 to 2200 Ma. This slow cooling rate is equally consistent with erosional denudation with a typical paleo thermal gradient of ~40°C/km.

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Monday AM Session

OS08 : MOam01 : G7 The Fate and Memory of Zircon Xenocrysts in Melted Metasediment

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Ion probe zircon dating in combination with cathodoluminescent and backscattered electron images and laser ablation microanalysis has improved our understanding of the type of petrogenetic events that can be dated using zircon (Cornell et al. 1999). An important issue is the question of survival or resetting of older zircons which are taken up in younger magmas. In this contribution three different examples are discussed, all concerning melted >1583 Ma metasediments from the Stora Le-Marstrand Formation of the Southwestern Baltic Shield (Åhäll et al. 1998). At Ålgön an 916 Ma intrusive norite melted metagraywackes to form a 10 m wide rheomorphic felsic contact migmatite (Schersten et al. 2000). Thin new magmatic rims developed on most xenocryst zircons, which generally retained their much older ages, from ~1600 up to 3.54 Ga (Cornell et al. 2000), but lead loss due to the 916 Ma intrusion is recognised in some grains. At Vrångö an intrusive 1550 Ma gabbro melted and mixed with metagraywacke to form hybrid magmatic rocks. Thick metamorphic rims developed on zircon xenocrysts, but with a few exceptions the xenocrysts show major lead loss for the time of hybridization. The Rönäng Tonalite is a large 1583 Ma body which intruded the foliated graywackes from depth, showing no field evidence that it originated from or assimilated them. Nevertheless its zircons show distinct cores and rims with very similar cathodoluminescent and backscattered electron characteristics to those seen at Vrångö. However the cores and rims give indistinguishable ages. In this case it seems that the xenocrystic cores have undergone complete lead loss during the magmatic event. These well-characterised cases are used to develop criteria for distinguishing unaffected from reset zircon xenocrysts, based on detailed SEM-CL and BSE images and microchemical analyses.

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OS08 : MOam02 : G7 Trace and Rare Earth Element Geochemistry of Magmatic and Metamorphic Zircon: Caveats for Geochronological Interpretation Based on Th/U and Morphology Only

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U-Pb dating of zircon is one of the most important techniques used on magmatic, metamorphic and also sedimentary rocks (provenance studies). Interpretation of zircon ages as magmatic or metamorphic growth events mostly uses arguments based of grain morphology and Th/U. Some 'rules of thumb' have thus been established, e.g. that metamorphic zircon has very low Th/U, that recrystallised zircon have significantly lower ages and may be discordant etc. It has also recently been proposed that REE patterns of zircon do not vary enough to be useful in distinguishing different zircon populations/samples.

Our studies on zircon from the regional metamorphic aureole in Rogaland, SW Norway, combines *in-situ* U-Pb zircon dating by SHRIMP with trace element characterisation of the dated zircon by electron- and ion-microprobe (e.g. Y, Hf, REE). Our results suggest that these 'rules' for zircon interpretation have to be used with caution: e.g. recrystallised areas may be perfectly concordant and yield

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ages within the uncertainty of a magmatic event. Th/U were found not to be correlated with magmatic/metamorphic age groups. Instead, analyses of each sample have a distinct range of Th/U regardless of magmatic or metamorphic growth and plot in distinct fields on a Y vs. Th/U diagram. This type of plot can be used to detect inherited xenocrystic cores and/or recrystallised areas (low Th/U, low Y) within zircon grains.

It can be concluded that not all metamorphic zircon has to have low Th/U. Where low Th/U occurs in metamorphic zircon, it may be the result of growth of competing phases (e.g. monazite, allanite) or of open-system behaviour (transport and fractionation of Th and U by fluids) in the rock, whereas unchanged Th/U indicates closed-system behaviour or solid-state recrystallisation. Independent arguments need to be found to distinguish these mechanisms.

REE measured with a 'standard', non-dedicated, ion-microprobe yielded fairly uniform strongly enriched (norm. to chondritic abundances) and steep LREE patterns, with pronounced positive Ce- and negative Eu-anomalies and an almost flat HREE pattern for most magmatic and metamorphic growth zones. Some types of zircon differ clearly: recrystallised areas characterised by lack of zoning, high CL-brightness and high Hf/Y generally have lower overall abundances coupled with much steeper HREE patterns; metamorphic zircon intergrown with xenotime on the other hand is characterised by higher overall abundances (no competition with xenotime).

We conclude that geochemical characterisation of zircon with trace elements has the potential to become a useful interpretative tool and should deliver powerful arguments for the interpretation of the growth environment of zircon when the mechanisms controlling trace element behaviour are better understood.

OS08 : MOam03 : G7 Low P-T Disturbance of Titanite and Uranium-Rich Paleoproterozoic Zircons, Central Sweden

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The U-Pb systems in zircon and titanite from a high-grade shear zone in Paleoproterozoic rocks east of the present Caledonian front in central Sweden display a strong Paleozoic disturbance. The shear zone is composed of mixture of lithologies that were deformed and juxtaposed during a Paleoproterozoic deformational and metamorphic event. Brown titanite that formed or recrystallized during ductile shearing yield a concordant U-Pb TIMS age of 1799±7 Ma. However, titanite with pale rims yield discordant ages with Paleozoic lower intercepts, and uranium-rich zircons from this zone show an almost complete resetting of the U-Pb system (>70% discordant) in Paleozoic time.

Uranium-rich zircons occur as large brown cloudy prisms and as small milky-white irregularly shaped grains. Also present are clear prismatic zircon of lower uranium content, which yield mildly discordant to concordant U-Pb ages. U-Pb SIMS analysis of 40 zircons range continuously from concordant at 1852±8 Ma to 98% discordant at 384±15 Ma. Regression through all zircons yields an upper intercept at 1871±11 Ma. The slight discrepancy between this intercept and the age of concordant zircons is attributed to the poly-lithological nature of the deformation zone. Raman laser spectroscopy reveals that dark cathodoluminescent areas in brown zircons have a highly metamict lattice. In contrast, the dark cathodoluminescent milky-white grains with equal or higher U+Th contents than the metamict zircons have significantly higher degree of crystallinity, comparable to that observed in the bright cathodoluminescent clear prisms. This suggests that the milky-white zircons are a younger population than both the other zircon types.

The strongly discordant zircons clearly have suffered severe disturbance at about the time of the Caledonian orogeny. However, Caledonian metamorphic temperatures and pressures in this region did not exceed 150-200°C and 1-3 kbar, too low to strongly disturb the U-Pb systematics in non-metamict zircon and in titanite by thermal means.

Independent evidence indicates that saline fluids were circulating in the Paleoproterozoic basement rocks at this time, possibly driven by hydrological gradients generated in front of the encroaching Caledonian orogenic wedge. These low-temperature saline fluids are inferred to be responsible for causing strong Pb loss in the mostly metamict brown zircons via a diffusive process, the formation of a new generation of small milky-white zircon via a low-temperature recrystallization or dissolution/re-precipitation process, and the alteration or re-equilibration of the rims of non-occluded titanite grains.

OS08 : MOam04 : G7 The Timing of Pre-Alpine High-Pressure Metamorphism in the Eastern Alps: Constraints from U-Pb SHRIMP Dating of Eclogite Zircons from the Austro-Alpine Silvretta Nappe

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Partly retrogressed eclogites of MORB-origin are present in the Austroalpine Silvretta Nappe as lenses and pods within amphibolite-facies orthogneisses. The eclogite assemblage comprises omphacite + garnet + phengite + kyanite + barrosite + rutile + quartz. PT conditions derived from garnet, omphacite and phengite geothermobarometry yielded at 2.8 GPa (500°) according to Schweinehage & Massonne (1999). Zircons were separated from a quartz-rich domain containing the typical eclogite assemblage. The zircons are euhedral in shape and may contain inclusions of quartz, rutile, omphacite and barrosite, indicating at least partial growth during the HP-event. All analyzed zircons show complex cathodoluminescence (CL) patterns including irregularly shaped cores with low CL-intensity, oscillatory sector zoning and overgrowths with high CL-intensity. Zircon ages were determined using ²⁰⁶Pb/²³⁸U-ratios with a common-Pb correction according to Tera-Wasserburg, 1972. Three different age groups could be distinguished that also correlate with distinct ranges in zircon Th/U-ratios: A low-CL irregular core with a Th/U ratio of 0.72 and an age of 507 ±11 (1σ) Ma is interpreted as a relic core. The age is consistent with intrusion ages of gabbros, tonalites and granites from the Silvretta Nappe (Schaltegger et al. 1997, Poller 1997) and the adjacent Ötztal Crystalline Basement (ÖCB) (Miller & Thöni 1995). Broad sector-zoned zircon areas with Th/U-ratios in the range 0.35-0.58 yielded a weighted mean age of 437 ±7 Ma (n=11). This age is thought to reflect magmatic growth of the zircons in the eclogite precursor, reflecting a Silurian/Ordovician magmatic event within the Eastern Alpine basement. The youngest event recorded led to the formation of narrow, irregular rims around zircons with very low Th/U-ratios in the range 0.01-0.29. The weighted mean age of these rims is 351 ±22 Ma which is interpreted as the age of the HP-metamorphic overprint. This would be consistent with Sm/Nd-mineral isochron ages of eclogites from the adjacent ÖCB that are in the range 370-340 Ma (Miller & Thöni 1995). The U-Pb zircon SHRIMP ages presented here clearly support the assumption of a widespread Variscan HP-event W of the Tauern Window.

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OS08 : MOam05 : G7 U-Pb SHRIMP-Dating of Zircon Domains from Eclogites of Antrona (Western Alps): Evidence for a Valais-Ocean Origin

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Mafic/ultramafic rocks in the Alps often occur as remnants of Mesozoic oceanic crust metamorphosed during Alpine subduction to HP/UHP conditions. The age of both formation and metamorphism of the main Alpine ocean basin, the Piemont-Ligurian ocean, is well constrained at ca. 164 Ma and ca. 44 Ma, respectively (e.g. Rubatto et al., 1998; Desmurs et al., 1999). However, it is likely that the Mesozoic mafic/ultramafic rocks in the Alps formed in more than one ocean and were subsequently subducted at different times. We dated amphibolitized eclogites from the Antrona ophiolites ('passo del Mottone') applying the U-Pb SHRIMP technique on zircon domains. The zircons dated are usually prismatic and ca. 60 μm long. In cathodoluminescence (CL) they show poorly preserved, dark magmatic cores surrounded by broad metamorphic rims. Ionprobe dating of the metamorphic rims yielded a weighted mean age of 38.5 ±0.9 Ma (error at 95% c.l.) for the time of HP metamorphism. SHRIMP II-dating (at ANU, Canberra) of the magmatic cores was complicated by the fact that they are small and show no distinct characteristic CL-patterns. In an attempt to get a protolith age, we obtained scattering 'ages' clustering around 133 Ma. Based on the model involving northwestwards-propagating subduction episodes of continental and oceanic crust (see Gebauer, 1999 for a review), the 38.5 Ma old HP metamorphic event recorded by the Antrona eclogites would fit in time to subduction of Valais oceanic crust. This Valais subduction zone would then be located between the Briançonnais microcontinent and the 35 Ma old subducted European margin (e.g. Adula-Cima Lunga and probably also Monte Rosa). According to the same model, the Briançonnais should, and also does, record metamorphic ages between 38.5 Ma (age of HP metamorphism of the Antrona ophiolites) and 44 Ma (HP/UHP metamorphic age of the more internal, SE-lying Piemont-Ligurian ocean). This scenario agrees well with recent SHRIMP-data for inherited zircons of the S-type granite of Novate (Liati et al., in press). These SHRIMP-data point to at least two episodes of continental rifting, at ca. 142 Ma and ca. 133 Ma, related to the break-off of the Briançonnais microcontinent from the European margin and subsequent opening of the Valais ocean. It seems to be also in agreement with our preliminary SHRIMP-data for the Antrona eclogites, which yield a protolith age cluster around 133 Ma.

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OS08 : MOam06 : G7 Timing of Metamorphism in the Central High Himalaya (Makalu Region) Constrained by In-Situ Th-U-Pb Dating of Monazite by Laser Ablation ICPMS

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Th-U-Pb dating of accessory monazite offers an excellent opportunity to constrain the timing of metamorphic events in amphibolite and higher-grade facies rocks. High Th and U concentrations in monazite, combined with a low diffusion rate of Pb and hence the relatively high closure temperature (> 750°C; Spear and Parrish 1996) of Th-Pb and U-Pb isotopic systems, make this mineral suitable for dating prograde metamorphism in young rocks.

In this study we utilized a VG PlasmaQuad 2 S+ instrument coupled to an in-house built NdYAG laser at Memorial University to measure Pb/Th and Pb/U isotopic ratios in monazites in standard petrographic thin sections.

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Monazites were exposed to a stationary laser beam while the sample stage was rastered, producing a 20x20 micron wide, ca. 10 micron deep ablation pit in the grain. Using He as a carrier gas, 10 Hz laser frequency, laser energy of 0.015 mJ/shot and a defocused laser beam, we were able to achieve stable Th, U and Pb signals for 1-2 minutes. Internal correction for instrument mass bias based on measurements of solution containing enriched ^{233}U (>99%) and Tl isotopes that was aspirated to the plasma during laser ablation, together with the mathematical approach to correct for laser-induced Pb/Th and Pb/U elemental fractionation, make the technique largely independent of matrix effects, which degrade analytical precision and accuracy of monazite ages.

Monazite inclusions in biotite, sillimanite, feldspar and quartz from the High Himalayan gneisses of the Makalu region in eastern Nepal record a polyphase metamorphic evolution between 35-21 Ma. The older monazites constrain a maximum age limit of ca 35 Ma for the sillimanite grade Barrovian metamorphism, while the younger monazite ages (ca. 21 Ma) reflect a thermal event associated with the intrusion of the Makalu granite at 21.7-24.4 Ma (Schärer 1984). Pressure and temperature conditions of the later event, recorded by garnet composition and Fe-Mg exchange between garnet and biotite, were 3.2-5.0 kbar and 600 \pm 25°C, respectively. We have found no correlation between the size of monazite inclusions in different metamorphic minerals, their ages, and location of samples relative to the granite intrusion. Maximum age of the Barrovian metamorphism recorded by Th-U-Pb isotopic systems in monazite from the Makalu region corresponds to the timing of prograde metamorphism reported from the Everest region (36-28 Ma; Simpson et al. 2000) and Garhwal and Zaskar Himalaya (37-29 and 35-25 Ma; Vance and Harris 1999). It is, however, somewhat younger compared to monazite ages from the Nanga Parbat - Haramosh Massif (44-36 Ma; Foster et al. 2000). The age difference may be a result of propagation of deformation and metamorphism from west to east related to diachronous collision of India and Asia.

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OS08 : MOam07 : G7 U-Pb Dating of Speleothems from the Spannagel Cave, Austria

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The Spannagel Cave is located in the western Tauern Window in the Austrian Alps, approximately 30 km SSE Innsbruck. It developed in metamorphosed basinal limestones of Jurassic age (Hochstegen Formation) sandwiched between granitic gneisses. The carbonate unit is a 20-30 m thick calcitic marble with cherty intercalations and a distinct content of disseminated graphitic material (metamorphosed organic matter). Despite its high altitude (main entrance 2531 m a.s.l.) the cave hosts both active and fossil speleothems, including flowstones, stalactites, stalagmites and soda straws. Compositionally, these speleothems are coarsely-crystalline columnar low-Mg calcite (some have coatings of gypsum). A conspicuous feature of Spannagel speleothems is their high U content (up to >300 ppm) which is also reflected in the elevated U concentration of modern dripwaters in the cave (up to 33 ppb). ^{230}Th - ^{234}U disequilibrium dating yielded a wide range of ages extending from essentially modern to > 350 ka, beyond the range of this method. We are investigating the older speleothems using the ^{238}U - ^{206}Pb method. As well as high uranium concentrations this method also requires sufficiently low initial lead concentrations and results to date show the Spannagel speleothems are, so far, uniquely favourable in both respects with μ -values ranging up to more than 5 million. This results in highly radiogenic lead even in samples as young as 300 ka, with measured $^{206}\text{Pb}/^{234}\text{U}$ ratios up to 200; two sub-samples yield an apparent age of 311 \pm 6 ka after correction for estimated initial $^{234}\text{U}/^{238}\text{U}$ disequilibrium. A second, older, sample yielded an age of 751 \pm 39 ka. The oldest speleothems in the cave include examples that are cut by neotectonic fractures

and analyses of these is in progress. The preliminary results obtained so far demonstrate great potential to extend the record of environmental change and landscape evolution preserved in speleothems back to a million years and beyond.

OS08 : MOam10 : G7 Submicroscopic Illitization and Excess Argon Incorporation in Phengite Related to Fluid Ingress during Late Miocene Extension in the Betic Cordilleras, South-Eastern Spain

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Well-crystallised phengite single grains from gneisses of the Mulhacén Complex yielded $^{40}\text{Ar}/^{39}\text{Ar}$ laser step-heating plateau ages ranging from 15.8 \pm 0.4 Ma to 90.1 \pm 1.0 Ma. Samples taken only a few metres apart may vary in age by as much as 50 Ma; ages of grain halves of crystals split over the basal plane may differ by 12%. As Rb-Sr mica dates in the literature fall in the 12.5-15.5 Ma range, the $^{40}\text{Ar}/^{39}\text{Ar}$ ages reflect ^{40}Ar uptake. The P-T path of the Mulhacén Complex shows an important late-stage reheating to over 500°C at 0.2-0.3 GPa, which has led to isotope resetting and an elevated transient partial argon pressure in the metamorphic fluid.

Despite the use of pristine grains, HRTEM images showed that at least 20% of the phengite lattice is affected by illitization, that is concentrated in several micrometre wide veins oblique to (001). The veins contain aggregates of 0.07-0.30 μm thick crystallites of three illitic micas types, that are chemically and structurally progressively closer to pure illite and occur in different textures. The oldest specimen is affected most severely as the veins contain newly formed (pseudo) illite that does not inherit its crystallographic orientation and chemistry from the host phengite, in contrast to the youngest sample. AEM analyses revealed that phengite and the illitic micas may be K-depleted. Despite the degassing of mixed phases the main release of Ar isotopes occurred between 800°C and 1000°C, without signs of $^{39}\text{Ar}_K$ recoil loss.

The oldest phengite is from a coarse-grained gneiss with extensively developed hydraulic cracks with a spacing of less than one millimetre, that are lacking in the youngest sample, a fine-grained mylonitic gneiss. Fluid-rock interaction and illitization were therefore more intense in the coarser-grained rocks. Recrystallisation of phengite and growth of the illitic micas in equilibrium with a high partial ^{40}Ar pressure enabled $^{40}\text{Ar}_{\text{XS}}$ incorporation in K-vacancies and other lattice imperfections. Variation in illitisation and associated textural dissimilarities in grains explain the age discordance. The 50-100°C/Ma cooling rate may have prevented equilibration of the different $^{40}\text{Ar}_{\text{XS}}$ levels.

The high atmospheric contamination in all grains may point to an interaction of meteoric water with the hot rocks during the final stages of their extensional exhumation in Late Miocene. Extension provides a rapidly changing interface between rocks and atmosphere, as well as fractures for the deep penetration of surface water.

OS08 : MOam11 : G7 Effects of Deformation on $^{40}\text{Ar}/^{39}\text{Ar}$ Geochronology – A Case Study of Defect-Enhanced Ar-Loss in Slowly Cooled Muscovite

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It is well established that isotopic dating methods are affected by diffusive element-loss during slow cooling and numerous geochronological studies have documented intragrain isotope concentration profiles readily interpretable in terms of volume diffusion. However, even these studies show remarkable deviations from Fickian diffusion gradi-

ents indicating that additional mechanisms are responsible for Ar-loss. In order to assess the role of deformation-induced defects on $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology we used the high-spatial resolution of the $^{40}\text{Ar}/^{39}\text{Ar}$ UV-laser microprobe for *in-situ* analysis of strongly deformed muscovite grains within the main mylonitic (S) foliation and mylonitic shear bands (C') of an upper greenschist facies mylonite. The sample studied comes from a crustal-scale shear zone (Pogallo Shear Zone, southern Alps) that accommodated oblique sinistral shear and exhumation of the Ivrea-Verbano Zone during Early Jurassic time. Quartz aggregates surrounding the muscovites show features of dynamic recrystallization by grain boundary migration and lack any evidence of post-mylonitic static annealing precluding a post-mylonitic thermal overprint. Muscovites within the studied sample, however, reveal (a) fragmentation of mm-sized porphyroclasts, (b) basal slip and disruption parallel to the (001) planes, and (c) segmentation, (sub-)grain formation and lattice misorientations across (001) planes often terminating in intragranular fractures. Both S and C' muscovites display a range of dates systematically younger than independent estimates for the timing of mylonitization at the Pogallo Shear Zone and conventional $^{40}\text{Ar}/^{39}\text{Ar}$ step-heating results of undeformed muscovite porphyroclasts from the same sample. The *in-situ* $^{40}\text{Ar}/^{39}\text{Ar}$ dates are highly variable when measured perpendicular to the (001)-direction of individual, deformed grains. This orientation allows a direct comparison of intragranular age variations and observed deformational microstructures. Chemical zonation as a possible cause for the observed range of *in-situ* $^{40}\text{Ar}/^{39}\text{Ar}$ dates is ruled out, as SEM imaging and detailed microprobe analyses reveal no detectable Ca and/or Cl zoning. We propose that deformational microstructures and associated defects that connect internal domains with the external surface of the grain may have played an important role in controlling the diffusion behavior of ^{40}Ar . Mylonitization induced heterogeneous segmentation of individual muscovite grains, with each segment bound by zones of high dislocation density or intragranular microfractures. Such lattice defects are inferred to reduce the physical grain size and hence the characteristic diffusional length scale for argon diffusion resulting in different closure temperatures for each segment and hence a range of cooling ages. Subsequently, the interaction of syn- and post-mylonitic thermal regimes induced diffusive ^{40}Ar -loss probably acting on the subgranular scale and Ar-loss occurred at temperatures well below those calculated for closed-system behavior at the observed grain size. The results of this study indicate that dislocations and microfractures might provide excellent examples for postulated fast diffusion pathways that facilitate Ar-escape during slow cooling of a geochronological system.

OS08 : MOam12 : G7 The Role of Deformation on Argon Diffusion in Muscovites

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Interpretation of $^{40}\text{Ar}/^{39}\text{Ar}$ data depends strongly on assumptions governing the distribution of argon in a mineral. Microanalyses performed using a UV-laser microprobe reveal that a relatively flat $^{40}\text{Ar}/^{39}\text{Ar}$ spectrum obtained by thermal heating in a furnace or infrared laser is not necessarily correlated with an homogeneous distribution of radiogenic argon within a mineral lattice. We present data that strongly supports the hypothesis that the presence of extended defects (dislocations, microfractures) greatly facilitate argon loss from within a mineral grain. Results are presented from two different areas where deformed muscovites occur. A combination of UV-laser $^{40}\text{Ar}/^{39}\text{Ar}$ microanalysis, furnace step heating $^{40}\text{Ar}/^{39}\text{Ar}$ analysis, electron microprobe and transmission electron microscope techniques were employed. The samples investigated come from gneiss and a pegmatitic intrusion of the metamorphic basement of the Siviez-Mischabel Nappe (Turtemann Valley, Penninic Alps, Wallis, Switzerland) and deformed samples of a late-Variscan granitic intrusion of the south Armorican shear zones (Brittany, France). In both geographic areas investigated, data obtained by UV-laser $^{40}\text{Ar}/^{39}\text{Ar}$ microanalysis (more than 300 individual analyses) plot on histograms with two separate age distributions (modes); the first corresponds to the original cooling age and the second is younger and plots with greater dispersion and has no geological meaning. In contrast, step-heated mineral concentrates from both study areas yield

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relatively flat $^{40}\text{Ar}/^{39}\text{Ar}$ spectra with integrated ages distinct from the age distributions observed on the histogram plots. The young ages with no geological meaning are interpreted as evidence for low temperature intragrain diffusion along fast diffusion pathways formed during deformation. Excellent evidence for such behaviour is shown in a detailed analysis (170 individual laser ablation ages) of a single Variscan (~350 Ma) deformed muscovite from the Siviez-Mischabel nappe. The deformed muscovite records clearly younger $^{40}\text{Ar}/^{39}\text{Ar}$ dates in zones subparallel to the shear plane (subparallel to C-surfaces). Microfractures and cleavage partings are often observed with an optical microscope in the deformed muscovites. Further microstructural analysis was performed using a transmission electron microscope and revealed the presence of networks of planar dislocations within the (001) plane. These results provide strong evidence that deformation induced microfracturing and cleavage partings reduce the effective length of diffusion (a) for argon diffusion. Furthermore, planar dislocations associated with microfracturing act as fast diffusion pathways for radiogenic argon (defect enhanced diffusion). Within such diffusivity paths the diffusion of argon is enhanced because the activation energy for diffusion is lowered. In such a cases where samples are variably deformed the closure temperature concept cannot be strictly applied as the system remains open to argon diffusion over a temperature range below that generally accepted as an argon closure temperature.

OS08 : MOam13 : G7 K-Feldspars: What Does Ar-Ar Date?

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K-feldspars (Kfs) showing staircase-shaped Ar-Ar age spectra can be interpreted following two mutually exclusive approaches: 1. Age spectra reflect the spatial distribution of Ar in a single, homogeneous mineral having discrete "domains" of variable size. It must be added that in geological situations in which the temperature history of a sample is independently known from non-isotopic constraints, the predictions of Discrete Domain Theory generally fall short by several orders of magnitude (Saltan Sea: [1], p. 421). 2. Discordant age spectra reflect the mixture of different minerals. It is well known that K-feldspars usually show patchy recrystallization due to interaction with fluids [2] and multiple Kfs generations are expected in virtually any magmatic or metamorphic rock. In a previous report [3] we analysed a Kfs from the Aar metagranite. The isotopic metagranite is ca. 1 m away from a mylonite, which was the site of intense fluid flow with mass transfer [4]. The Kfs age spectrum is a classical staircase and its apparent Ar diffusivity falls near the average of most Kfs reported in the literature. However, we observed linear correlations between Ar^*/K , Ca/K and Cl/K , and proposed that this requires a binary mixing between two heterochemical Kfs generations. Our new results on the same sample reveal that neither Sr nor Pb are in isotopic equilibrium among the two separate reservoirs that can be resolved by stepwise leaching. The $^{206}\text{Pb}/^{204}\text{Pb}$ ratios are 19.885 ± 15 and 19.726 ± 24 for leachate and residue, respectively. The residue has $^{87}\text{Sr}/^{86}\text{Sr} = 0.7333 \pm 1$, while the leachate has 0.7358 ± 1 , approaching that of mylonitic Kfs, 0.7434 ± 1 . Thus the fluid flow in the mylonite probably was the immediate cause for the formation of the high-Sr Kfs. The isotopic data all concordantly imply two diachronic, heterochemical Kfs generations rather than simple diffusion out of a mosaic of homogeneous "domains". We obtained additional petrographical data on this sample in an attempt to help explain the isotopic results. Back-scattered electron (BSE) imaging on a grain mount of the analysed fraction shows two separate Kfs with a resolvable brightness contrast, lobate boundaries and patchy replacement textures. Cathodoluminescence (CL) can provide further clues on deuteric alteration. On a thin section of sample Aar 10, fresh Kfs has a blue luminescence with different hues. Secondary Kfs (not identified as such under optical microscopy) shows pale blue CL. This secondary Kfs formed mainly along microcracks where fluids could access the pristine Kfs. The CL features are able to resolve

the two generations, whose main distinction is their history of interaction with an aqueous fluid. We conclude that it will be possible to more accurately reconstruct a K-feldspar's history if one can couple the geochronologic information on heterochemical mixtures offered by Ar-Ar with the additional constraints provided by BSE and CL imaging.

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OS08 : MOam14 : G7 The Thermal Evolution of a Deeply Exhumed Paleoproterozoic Orogen, Assessed by $^{40}\text{Ar}/^{39}\text{Ar}$ and U-Pb Geochronology

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A comprehensive study of orogenic processes requires reliable estimates of time and temperature at lower crustal levels during the development of an orogen. Rocks exposed in regional granulite-amphibolite facies terranes can be used to study deep crustal processes because they experienced conditions that appear to be typical for the middle-to-lower crust. Cooling and exhumation rates determined by thermochronometers allow us to distinguish between different modes of exhumational processes and can be interpreted in terms of large scale tectonic processes. Previous thermo-chronological studies of regional high grade terranes focussed on either regional implications on the basis of few thermo-chronometers or discussed details on the thermal evolution of a single or a few small area(s) on the basis of a series of thermo-chronometers. However, the reconstruction of the thermal evolution of an orogen requires a series of detailed time-temperature paths from different tectonic elements of an orogen. This study focuses on the hitherto poorly understood thermo-tectonic evolution of the Paleoproterozoic deeply exhumed Nagssugtoqidian orogen and its southern foreland, West Greenland. Based on the $^{40}\text{Ar}/^{39}\text{Ar}$ hornblende, muscovite and K-feldspar ages and U-Pb monazite, sphene and apatite age a series of cooling paths have been constructed. The proposed thermal evolution illustrates the thermal processes occurring at deep crustal levels during and subsequent to an orogenic event. We suggest that during peak metamorphism, between 1870 and 1850 Ma, temperatures higher than 540°C were restricted to the central part of the orogen, while heating to temperatures between 410°C and 540°C occurred in the southern foreland. Following peak metamorphism the core of the orogen cooled from ca. 800°C at ca. 5°C/m.y. to ca. 200°C at 0.9°C/m.y. by 1400 Ma. The northern part of the orogen cooled at 4 to 7°C/m.y. at 1740 Ma, diminishing to ca. 2°C/m.y. by 1660 Ma, when 200°C was reached. The slow and relatively homogeneous regional cooling of the Nagssugtoqidian orogen contrasts with the heterogeneous and faster cooling observed in many Phanerozoic orogens. These differences largely relate to the syn-orogenic cooling, caused by tectonic events, observed in Phanerozoic orogens, in contrast to the cooling in many high-grade terranes that post-dates orogenesis and where exhumation is driven by "static" erosion and isostatic adjustments.