V22G MCC: 3004 Tuesday 1600h

Centennial Celebration of Radioisotopic Geochronology: Dates, Rates, and New Debates II

Presiding: **S P Kelley,** Open University**; S B Shirey,** Carnegie Institution of Washington

V22G-01 1600h INVITED

New Advances in Re-Os Geochronology of Organic-rich Sedimentary Rocks.

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Geochronology using ^{187}Re-^{187}Os is applicable to limited rock and mineral matrices, but one valuable application is the determination of depositional ages for organic-rich clastic sedimentary rocks like black shales. Clastic sedimentary rocks, in most cases, do not yield depositional ages using other radioactive isotope methods, but host much of Earth's fossil record upon which the relative geological timescale is based. As such, Re-Os dating of black shales has potentially wide application in timescale calibration studies and basin analysis, if sufficiently high precision and accuracy could be achieved. This goal requires detailed, systematic studies and evaluation of factors like standard compound stoichiometry, geologic effects, and the ^{187}Re decay constant. Ongoing studies have resulted in an improved understanding of the abilities, limitations and systematics of the Re-Os geochronometer in black shales. First-order knowledge of the effects of processes like hydrocarbon maturation and low-grade metamorphism is now established. Hydrocarbon maturation does not impact the ability of the Re-Os geochronometer to determine depositional ages from black shales. The Re-Os age determined for the Exshaw Fm of western Canada is accurate within 2σ analytical uncertainty of the known age of the unit (U-Pb monazite from ash, conodont biostratigraphy). This suggests that the large improvement in precision attained for Re-Os dating of black shales by Cohen et al (ESPL 1999) over the pioneering work of Ravizza & Turekian (GCA 1989), relates to advances in analytical methodologies and sampling strategies, rather than a lack of disturbance by hydrocarbon maturation. We have found that a significant reduction in isochron scatter can be achieved by using an alternate dissolution medium, which preferentially attacks organic matter in which Re and Os are largely concentrated. This likely results from a more limited release of detrital Os and Re held in silicate materials during dissolution, compared with the inverse aqua regia medium used for Carius tube analysis. Using these "organic-selective" dissolution techniques, precise depositional ages have now been obtained from samples with very low TOC contents (~0.5%), meaning that a greater range of clastic sedimentary rocks is amenable for Re-Os age dating. Well-fitted Re-Os isochrons of plausible geological age have also been determined from low-TOC shales subjected to chlorite-grade regional metamorphism. These results further illustrate the wide, but currently underutilized, potential of the Re-Os geochronometer in shales. The precision of age data attainable by the Re-Os system directly from black shales can be better than ± 1% uncertainty (2σ, derived from isochron regression analysis), and the derived ages are demonstrably accurate.

V22G-02 1615h

Application of the Rhenium-Osmium Isotopes to the Geochronology of Diamonds

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The advent of the modern era of high sensitivity and accuracy measurements of Re and Os isotopic compositions by negative thermal ionization mass spectrometry (N-TIMS; Creaser et al, 1991; Volkening et al, 1991) has led to numerous applications of Re-Os isotopes in tracer studies and geochronology. Recent developments in processing blanks (e.g. Richardson et al, 2001) by miniaturization of chemistry (Re <40x10^{-15}g; Os <2x10^{-15}g) permit single sulfide inclusions in minerals such as diamond to be analyzed for their Re-Os isotopic systematics (Pearson et al, 1998; Pearson and Shirey, 1999). Such data on syngenetic inclusions can provide ages on individual macro-diamonds. The microchemistry technique analyses the entire grain, thereby minimizing problems from exsolution. In addition, the low blanks, combined with high sensitivity of N-TIMS allows the analysis of single eclogitic sulfides that are intractable by laser-ICPMS methods This method of diamond geochronology is being applied to diamonds from ancient terranes such as the Kaapvaal-Zimbabwe, Siberian, Slave, and Australian cratons. The work depends on the distribution of mined, diamond-bearing kimberlites, the frequency and size of sulfide inclusions in respective diamond suites and the beneficence of diamond mining companies. A goal of obtaining ages on diamonds is to place diamond formation episodes into the broader framework of the geological processes that create and modify the continental lithosphere. Additionally, diamonds and their inclusions have long held general interest as the most robust containers of ancient minerals from the mantle at depths of 150 km or more. The most detailed application of Re-Os sulfide inclusion ages has been to the evolution of the Kaapvaal-Zimbabwe craton where there exists the widest distribution of mined kimberlites in diverse geologic terrains, the most extensive dataset on silicate inclusion ages and diamond compositions, and recent seismic tomography of the diamond source region in the lithospheric mantle. Diamond ages track the geological evolution of the craton throughout most of its history. Geographically restricted, 3.2-3.3 Ga Sm-Nd ages on harzburgitic garnet inclusions in diamond document early cratonic nuclei development likely by subduction but involving severe mantle depletion and concomitant light REE enrichment. Widely distributed, circa 2.9 Re-Os Ga ages (e.g. Richardson et al, 2001) on eclogitic sulfide inclusions document the subduction accretion that put older cratonic blocks together. Proterozoic ages often unique to each locality and seen in both silicate and sulfide inclusions suites testify to re-fertilization of the cratonic lithospheric mantle by magmatic, metasomatic and subduction-margin processes. This age-framework appears applicable to the more restricted datasets from other cratons and allows us to clearly relate diamond genesis to the dynamics of craton creation and assembly.

Creaser, R., Papanastassiou, D., and Wasserburg, G. (1991) GCA, 55, 397-401. Pearson, D.G., and Shirey, S.B. (1999) D.D. Lambert, and J. Ruiz, Eds. Rev. in Econ. Geol. 12, 143-172. Pearson, D.G., Shirey, S.B., Harris, J.W., and Carlson, R.W. (1998) EPSL, 160, 311-326. Richardson, S.H., Shirey, S.B., Harris, J.W., and Carlson, R.W. (2001) EPSL, 191, 257-266. Volkening, J., Walczyk, T., and Heumann, K. (1991) Int. Jour. of Mass Spectr. & Ion Proc., 105, 147-159.

V22G-03 1630h INVITED

U-series Isotopes and the Time Scales of Magmatic Processes

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The first published record of a radioactive decay chain was 100 years ago this year, and the sequence of isotopes in the U and Th decay chains were largely determined in the following ten years. Isotopes of U, Th, Pa, Ra and Pb with half-lives in the range 75,000 to 22 years have had a major impact in our understanding of magmatic processes, because the time scales of magmatic processes are similar to the half lives of these isotopes, and physically realistic models of natural processes require information on the rates at which those processes occur. U-Th-Pa-Ra isotopes can now be measured by mass spectrometry, routinely with errors of less than 1%. A key characteristic is that U-series isotopes can change significantly by radioactive decay while the crystals and rocks were forming. At subduction zones fluids may be transferred from the downgoing slab in a few 1000 years. In most tectonic settings the magmas and the peridotite matrix spend different lengths of time in the melt zone, and typically the observed isotope fractionation implies some form of dynamic melting process. New U-Pa isotope data for 40 young lavas from 7 different arcs worldwide have, with one exception, (^{231}Pa/^{235}U) > 1, and extend to values as high as 2.48. Their U/Nb ratios are < 9.0 and so > 80% of the U has been added from the subducting slab, and large enrichments of Pa over U occurred during melting and melt transport. The ages of phenocrysts and the time scale of differentiation of the host magma can be different, and in a number of cases it has been shown that the phenocrysts formed after the fractional crystallisation responsible for the whole rock composition. Different approaches are therefore used to investigate the crystallisation history and the differentiation of magmatic suites: crystallisation rates are 10^{-10} to 10^{-11} cm/s, whereas differentiation to high silica magmas may take up to 2 x 10^5 years. The ages of crystals at the time of eruption can range back to 2-3 x 10^5 years, the older ages tend to be in the more evolved rock types, and it can take 10^5 years for high silica magmas to be generated at individual volcanic centres. Thus, the generation of evolved magmas is often thermally controlled, and the rates of fractional crystallisation have, for example, been linked to volcanic power outputs. In contrast, crystallisation in response to magma degassing or decompression, may be too fast for much fractional crystallisation to take place.

V22G-04 1645h

Zonation-Dependent α-ejection Correction by Laser Ablation ICP-MS Depth Profiling: Toward Improved Precision and Accuracy of (U-Th)/He Ages

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(U-Th)/He dating of zircon and apatite constrains the timing and rates of shallow crustal exhumation by yielding ages corresponding to closure temperatures of about 180°C and 70°C, respectively. Relatively long α stopping distances in both minerals, however, typically require significant corrections for He ejected from dated crystals (α-ejection correction). Good reproducibility of α-ejection corrected ages from volcanic standards as well as predictable behavior of ages in intercalibration studies indicate that these corrections generally produce accurate and sufficiently precise corrected ages. However, the default α-ejection correction assumes homogeneous distribution of U and Th parent concentrations in dated crystals, an assumption that is often violated to varying degrees. Recent work has shown that heterogeneous U-Th distribution in single zircon crystals can produce both systematic age biases and poor precision in some crystal populations when a standard homogeneous α-ejection correction (HAC) is applied. We have developed a method for characterizing U-Th zonation in single crystals, prior to dating, by rim-to-core depth profiling by a 213 nm laser ablation ICP-MS. Compositionally homogeneous Sri Lankan zircon standards are used for standardization and to determine depth-dependent U/Zr and Th/Zr fractionation, which is insignificant relative to intragrain U-Th concentration variations. Time-resolved traces are converted to depth profiles using an empirically calibrated drilling rate of 0.6 μm/s. We calculate zonation-dependant α-ejection correction (ZAC) factors by inputting fractionation-corrected U-Th profiles into an α-ejection model of a spherical crystal with a surface-to-volume ratio equivalent to the analyzed grain, and assuming that compositional zonation has radial symmetry. Our data on zircons from a range of samples, including Fish Canyon tuff and Tardree rhyolite zircons, show that ZAC and HAC corrected ages can differ by up to 20%. Single-grain ZAC-corrected ages are always more accurate than HAC-corrected ages, typically by 5-10% relative to accepted ages. In some cases, even ZAC-corrected ages remain up to 5-15% different from accepted zircon ages. This suggests that crystal morphology and incomplete characterization of 3D zoning constitute significant sources of error. Ongoing developments include the use of multiple depth profiles to better characterize 3D zoning, and a new ZAC code with a more realistic bipyramidal prism morphology.

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V22G-05 1700h

Laser-Ablation (U-Th)/He Geochronology

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Over the past decade, ultraviolet laser microprobes have revolutionized the field of $^{40}Ar/^{39}Ar$ geochronology. They provide unprecedented information about Ar isotopic zoning in natural crystals, permit high-resolution characterization of Ar diffusion profiles produced during laboratory experiments, and enable targeted dating of multiple generations of minerals in thin section. We have modified the analytical protocols used for $^{40}Ar/^{39}Ar$ laser microanalysis for use in (U-Th)/He geochronologic studies. Part of the success of the $^{40}Ar/^{39}Ar$ laser microprobe stems from fact that measurements of Ar isotopic ratios alone are sufficient for the calculation of a date. In contrast, the (U-Th)/He method requires separate analysis of U+Th and ^4He. Our method employs two separate laser microprobes for this process. A target mineral grain is placed in an ultrahigh vacuum chamber fitted with a window of appropriate composition to transmit ultraviolet radiation. A focused ArF (193 nm) excimer laser is used to ablate tapered cylindrical pits on the surface of the target. The liberated material is scrubbed with a series of getters in a fashion similar to that used for $^{40}Ar/^{39}Ar$ geochronology, and the ^4He abundance is determined using a quadrupole mass spectrometer with well-calibrated sensitivity. A key requirement for calculation of the ^4He abundance in the target is a precise knowledge of the volume of the ablation pit. This is the principal reason why we employ the ArF excimer for ^4He analysis rather than a less-expensive frequency-multiplied Nd-YAG laser; the excimer creates tapered cylindrical pits with extremely reproducible and easily characterized geometry. After ^4He analysis, U and Th are measured on the same sample surface using the more familiar technique of laser-ablation inductively coupled plasma mass spectrometry (LA-ICPMS). Our early experiments have been done using a frequency-quintupled Nd-YAG microprobe (213nm), While the need to analyze U+Th and He in separate ablation experiments results in considerably worse spatial resolution than that typically possible for $^{40}Ar/^{39}Ar$ laser microprobe dating, it is possible to site the LA-ICPMS ablation pit within a few microns of the pit used for He extraction, or to simply re-occupy and enlarge the original ablation pit. The potential effective spatial resolution of the technique is thus on the order of a few tens to roughly 100 microns. As a proof-of-concept exercise, we have applied this technique to fluorapatite from Cerro de Mercado, Durango, Mexico, which has a generally accepted (U-Th)/He age of 32.1 $\pm$ 3.4 Ma (2 sigma) based on single-crystal fusion analyses reported by House et al. (2000, EPSL). Using the approach described above, we made 48 separate age measurements on a 12 mm polished section cut through a single crystal of Durango fluorapatite perpendicular to its c axis. The measured dates yield a mean of 34.9 $\pm$ 5.1 Ma (2 sigma), with a total dispersion of dates comparable to that reported by House et al. Much of the apparent age variation observed in both studies is due to documented U+Th heterogeneities in single crystals of the Durango fluorapatite. Nevertheless, the consistency of the laser ablation and conventional results for this material is striking. Compared to conventional laser and furnace methods of (U-Th)/He geochronology, the laser microprobe approach offers substantially improved spatial resolution, and the ability to avoid (or at least minimize) alpha-ejection corrections. In addition, the method affords improved sample throughput, such that age estimates for homogeneous materials can be made with considerably higher precision based on a larger number of analyses.

V22G-06 1715h INVITED

Problematic Samples for Apatite (U-Th)/He Dating: Some Possible Causes and Solutions

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Seven years of experience with (U-Th)/He dating reveals a subset of granites and gneisses (perhaps 10%) which yield apatites problematic for the method. Here I document these challenging samples and provide possible explanations and solutions. Rocks from some areas (e.g., Himalayas and Taiwan) tend to yield apatites in which every grain is broken, precluding accurate α emission correction. We developed a protocol in which the grain fragments are abraded to eliminate the α-ejection-affected surfaces. The now opaque fragments are immersed in appropriate refractive index oil, rendering inclusions remarkably visible for handpicking. By eliminating the need to simultaneously have good morphology and freedom from inclusions, this technique greatly increases the population of datable grains. Results of a case study from Taiwan will be presented, as will modeling which shows how removal of outer edges must bias He ages, especially when cooling is slow.

More problematic are seemingly good apatites that yield irreproducible and anomalously old He ages. The following observations apply to these rocks: strong geographic control, with problem samples common in some areas (e.g., Transantarctic Mtns) but absent elsewhere (e.g., Coast Mtns); highly variable U,Th among apatite grains (up to 2-3x); REEs (now measured on every dated apatite) have LREE depletion, compared with LREE enrichment in most non-problematic apatites. These observations are consistent with problematic apatites occurring preferentially in S-type granites, which have precipitated monazite (Sha and Chappell, 1999). Flux melting indicates that inclusions cannot explain the aberrant ages, and modeling and ion probe measurements suggest that U,Th zonation is not a likely cause either. A possible role for implantation of He from neighboring monazites is consistent with abrasion experiments yielding younger cores than obtained from whole grains. If this explanation is correct, it may be impossible to obtain meaningful He ages on such samples. In any case it seems prudent to reproduce all He age determinations to insure data quality.

V22G-07 1730h INVITED

Cosmogenic ^{26}Al and ^{10}Be Depth Profiles in High-level Terrace Gravels Demonstrate Early Pleistocene Entrenchment of the San Juan River in the Canyonlands Region of Utah

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Cosmogenic ^{26}Al and ^{10}Be measured in a vertical profile can be used to date sedimentary deposits such as alluvial terraces, provided that the profile is deep enough to shield the lowest portion from secondary cosmic-ray nucleons and muons. Radioactive decay at depth lowers the $^{26}Al/^{10}Be$ ratio with time, allowing sediments to be dated up to 5 million years old. Accurate dating requires correction for post-depositional production of ^{26}Al and ^{10}Be, which can be important at depths up to 10-20 meters. Production at depth can be extrapolated from the upper portion of the profile. Here we present data from two terraces 150 meters above the San Juan River, Utah, near the towns of Bluff and Mexican Hat. The terraces are on the brink of the canyon upstream from the entrenched Goosenecks for which this river is known. These terraces and others grade evenly to the rim of Glen Canyon on the Colorado River, and thus constrain the age of canyon incision east of Grand Canyon. The terrace at Bluff is mantled with over 11 meters of gravel, while at Mexican Hat the gravel is 6 meters deep. These gravels have previously been correlated with glacial deposits in the San Juan mountains. The alluvium is capped by Stage IV-V pedogenic carbonate. We collected sediment from a profile 11.7 meters deep at Bluff, and 5.5 meters deep at Mexican Hat. We also collected a sample for cosmogenic surface exposure dating to compare with the profile dating method at Bluff. Our cosmogenic profiles yield an age of 1.36 (+0.20/-0.15) My for the deposit. The average incision rate of the San Juan River at this site is thus 113 +/- 16 m/My, a value that is similar to many others in the Colorado River system, but nearly 5 times slower than incision rates that have been inferred from nearby cosmogenic nuclide exposure dates on terraces and pediments. To test a possible source for this discrepancy, we measured ^{26}Al and ^{10}Be in a sample collected from the undisturbed terrace surface. We determined that the effective surface exposure age of this deposit is only 625 thousand years, only half the true age of the terrace. This discrepancy underscores the caution that must be taken when interpreting very old surface exposure ages, which are known to be sensitive to erosion at the surface. These data suggest that Glen Canyon on the Colorado River is at least 1.3 million years old, and that incision rates in the Canyonlands area are similar to those downstream in Grand Canyon, Marble Canyon, and on the Little Colorado River.

V22G-08 1745h

Preliminary Estimate of Production Rates for Terrestrial Cosmogenic ^{38}Ar from Calcium

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Cosmogenic ^{38}Ar, dominantly produced from targets of Ca and K (and to a lesser extent from Fe and Ti), has been used in extra-terrestrial studies for decades. Recent measurement of terrestrial cosmogenic ^{38}Ar (Renne *et al.*, 2001) primarily produced by high-energy spallation on calcium has shown potential as a useful addition to stable noble gas cosmogenic geochronology. Terrestrial cosmogenic production rates for both $^{38}Ar_c$ and $^{36}Ar_c$ have not yet been empirically constrained, however, in part because simple atmospheric corrections to measured $^{38}Ar/^{36}Ar$ ratios are impossible. We have employed a different methodology, after Turner *et al.*, 1971, to quantify calcium derived $^{38}Ar_c$ production. Our method requires irradiation of mineral separates, using neutron activation to create ^{37}Ar as a proxy for the cosmogenic target calcium via the reaction $^{40}Ca(n,\alpha)\ ^{37}Ar$. The extent of conversion due to irradiation is monitored using co-irradiation of a standard with a known composition including [Ca] and [Cl], known age and no cosmogenic exposure, analogous to standards used in $^{40}Ar/^{39}Ar$ dating. Approximately 15-30 mg of sample is loaded into a mass spectrometer and degassed incrementally with a CO_2 laser. Measured isotopes are corrected for backgrounds, mass discrimination, radioactive decay and additional argon isotopes produced in the irradiation. Step-wise degassing allows construction of a "cosmochron" plot of $^{38}Ar_c/^{36}Ar$ vs. $^{37}Ar_{Ca}/^{36}Ar$, with a slope representing the $^{38}Ar_c/\ ^{37}Ar_{Ca}$, and an intercept ideally being that of atmospheric $^{38}Ar/^{36}Ar$ ($\sim$0.188). Deviations from atmospheric $^{38}Ar/^{36}Ar$ ratios imply sample disturbance or further complexity. We have used this irradiation method to successfully measure $^{38}Ar_c$ from calcium bearing minerals including diopside, clinopyroxene, garnet, sphene and apatite. A first order approximation can be made of the production of $^{38}Ar_c$ from calcium by using the better-known cosmogenic ^{3}He production rate. In this study, we collected samples primarily from the Antarctic Dry Valleys, where long exposure histories relative to low erosion rates and high latitude, along with a diverse range of lithologies maximizes cosmogenic dosage and potential target minerals. We have measured both ^3He_c and $^{38}Ar_c$ in samples with identical exposure histories, using mineral pairs where necessary due to the limited application of ^3He_c to olivine, clinopyroxene and apatite. ^3He_c is normalized to total mineral mass, reflecting its production from a range of target atoms, while $^{38}Ar_c$ is normalized to grams of Ca. Complementary measurement of these two cosmogenic isotopes yields a strong linear correlation of $^{38}Ar_c$ atoms/g Ca and ^3He_c atoms/g sample with a slope of $\sim$0.9 $\pm$ 0.4 (MSWD of 1.5), demonstrating that spallation dominated pathways and energies for formation of cosmogenic ^{3}He and ^{38}Ar are consistent to a first order. These data also provide an estimate of $^{38}Ar_c$ production on calcium as $\sim$100 atoms/g Ca-year at high latitude, sea-level, significantly lower than the 200 atoms/g Ca-year production estimate calculated by Lal, 1991. Further geological calibration and modeling are underway to refine these estimates.

URL: http://eps.berkeley.edu/~kimmer/38Ar.html

V22H MCC: 3008 Tuesday 1600h

Crustal and Mantle Processes in Ophiolites and Ocean Crust Generation V *(joint with GP, OS, T)*

Presiding: **H J Dick,** Woods Hole Oceanographic Institution**; J E Snow,** Max-Planck-Institut für Chemie

V22H-01 1600h

The Thetford Mines Ophiolite Complex: Focus on the Petrology, Mineralogy and Geochemistry (REE, PGE) of a Supra-Subduction Mantle Section

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