Physical Properties of Earth Materials
Newsletter

December, 2009

http://www.ppem.org

A Note from the Chair

Steve Hickman, USGS

With the Fall AGU meeting fast upon us, it is time once again for the annual PPEM Newsletter. Many thanks to those of you who contributed articles, and thanks also to the Newsletter editor, Shenghua Mei, for soliciting contributions and pulling them together into the finished product.

Speaking of the AGU meeting, once again Steve Blair and Brian Bonner have put their heads (and appetites) together and come up with a fine restaurant for the PPEM dinner. We will be eating at the New Delhi Restaurant, 160 Ellis Street in San Francisco on Monday December 14, with cash bar at 6 pm and dinner at 7:30 pm. The New Delhi was named one of the finest Indian restaurants in the U.S. by the New York Times and was featured on the Galloping Gourmet TV Show. For details see http://www.ppem.org/ppemdinner.

Numerous people from the PPEM and MRP Communities of AGU have received awards this year in recognition of their outstanding scientific achievements. David Kohlstedt was elected to the U.S. National Academy of Sciences in 2009 (this country's top scientific honor) and was awarded the 2009 Murchison Medal of the Geological Society of London. Yves Guéguen received the European Geosciences Union's Louis Néel Medal for 2009. Alexandra Navrotsky received the 2009 Roebling Medal and Ronald Cohen received the 2009 Dana Medal, both from the Mineralogical Society of America.

In this issue

Note from the Chair

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Please also congratulate new 2009 AGU Fellows Jay Bass, Roger Buck, Katherine Cashman, Stuart Crampin, Don Dingwell, Kei Hirose and Frederick Ryerson.

There are several reports on recent PPEM-related meetings in this Newsletter, as well as announcements for upcoming meetings. Among the latter, please note in particular the Gordon Research Conference on Rock Deformation: *Transient And Transitional Behavior In Rock Deformation - Moving Away From Steady-State*, to be held August 8-13, 2010, at the Tilton School, New Hampshire. David Prior (Chair) and Peter Kelemen (Vice-Chair) are organizing this GRC, which is one of the key events for the PPEM community. The agenda for this Conference looks outstanding and I strongly encourage you to attend. Other meetings during the coming year (described in more detail below) include: *Short Course on Microstructures and Physico-Chemical Properties of Earth and Planetary Materials*, February 8-12, 2010, in Verbania Italy; the *13th International Conference on Experimental Mineralogy, Petrology and Geochemistry,* April 11-14, 2010, in Toulouse, France; and the *Consortium for Materials Properties Research in Earth Sciences (COMPRES)* Annual Meeting, June 22-25, 2010, in Stevenson, Washington.

I would also like to thank the new PPEM web master, Joe Morris, who this year redesigned and now maintains the PPEM web site (http://www.ppem.org/). Joe's efforts (and those of his predecessor, Andreas Kronenberg, who still sends out announcements to the PPEM mailing list) help assure that we keep abreast of job announcements, meetings, publications and other activities of special interest to the PPEM community.

Finally, two members of the PPEM Steering Committee, Ben Holtzman and Shenghua Mei, will be rotating off in the coming year (as will I). I would like to thank them and other members of the Steering Committee (Joanne Fredrich, Francois Renard, Joe Morris and Dave Goldsby) for their service to the community. If you have any nominations for people to replace Ben, Shenghua or me (as Chair), please send them to hickman@usgs.gov.

Annual PPEM Dinner

Steve Blair & Brian Bonner

Time: December 14, 2009 (Monday)
6:00 pm bar opens
7:30 pm dinner

Join in the merriment with colleagues at AGU interested in Physical Properties of Earth Materials! This year's venue is a critically acclaimed and conveniently located Indian restaurant: New Delhi.

Address: 160 Ellis St., San Francisco, CA 94102-2122
Web: www.NewDelhieRestaurant.com
Phone: (415) 397-8470

Conference Reports

Euroconference 2009 on Rock Physics and Rock Mechanics Ascona CSF center

Following the example of the first Euroconference on Rock Physics and Geomechanics, organized by Yves Guéguen and Maurice Boutéca in Aussois France 1998, a conference was organized in the Alps, a little more than ten years later, gathering scientists from different academic and industrial environments. In the meantime, seven conferences were held all across Europe: in Edimburgh UK, 1999; in Badhonnef Germany, 2000; in Kijkduin, The Netherlands, 2003; in Potsdam Germany, 2004; in Oleron France, 2005 and finally in Erice Italy, 2007.

This 2009 Euro-Conference was held Sept. 13-18 in Ascona, Switzerland and focused on *Thermo-Hydro-Mechanical Couplings and the Applications of Rock Physics and Geomechanics Knowledge and Methods in the Society* with particular attention to problems like Scientific Drillings, CO2 sequestration, waste disposal and oil exploration/exploitation. Its program included a mixture of keynote lectures, oral and poster contributions on theoretical models, laboratory experiments and field applications, covering a broad range of topics spread over five sessions.

The largest part of the conference was devoted to thermo-hydro-mechanical couplings during rock deformation. New data on high-velocity friction, time-dependent deformation and compaction localization were presented. A wide variety of experimental techniques, such as acoustic emissions and X ray computed tomography were used to study damage development and its effect on fluid flow in volcanic and reservoir rocks. A session on modelling was the opportunity to celebrate Yves Guéguen's 60^{th} birthday and to acknowledge his contribution to the rock physics community and to the Euroconferences. Following his keynote lecture on *elastic wave anisotropy and dispersion in cracked rocks*, new theoretical studies related to fractures at various scales were presented.

Another session was dedicated to reservoirs and resources. Several presentations gave an overview of recent results on the mechanical compaction in porous carbonates. For example, a new approach to the micromechanics of cataclastic pore collapse in limestone was presented. Most of the other contributions related to reservoirs focused on 2D and 3D imaging of deformation and fluid flow in porous sandstone as well as on physical and geomechanical properties of shales.

Intense cross-disciplinary collaboration has developed around fault drilling projects and the questions of underground radioactive waste disposal. New petrophysical and mechanical data related to SAFOD and ICDP projects as well as several studies on the Mont Terri Rock laboratory and Meuse Haute-Marne URL were presented. Several presentations were also related to the fundamental question of CO2 storage. The poromechanics of in-pore crystallization and the role of geomechanics in leakage risk management were for example presented. About 100 scientists gathered from all over the world (including USA, Canada, Australia, Japan, Indonesia and India) to attend this conference at the Centro Stefano Franscini in Ascona, Switzerland. 15 PhD and Master students were fully supported thanks to our various sponsors (BP, Schlumberger, Andra, Nagra, IUGS, IFP, CNRS-INSU, EOST, INGV and ETH). More than 20% of the conference attendants were students, and 30% were under 33. The 9th Euroconference will be organized in Trondheim, Norway, in 2011.

(*Submitted by Patrick Baud, Luigi Burlini, and Alexandre Schubnel*)

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DRT International Conference

In September 2009 the 17th meeting of the bi-annual Deformation Mechanisms, Rheology & Tectonics international conference was held in Liverpool, in northwest England. This year the DRT meeting was a joint venture between the universities of Liverpool and Manchester.

The DRT meetings are devoted to the study of rock and mineral deformation and rheology. This year the meeting was particularly special to our group in that it was dedicated to Dr. Martin Casey who's untimely death impacted us all last year. Martin had many friends and colleagues within our community and he will be sadly missed.

Topics and sessions for the meeting were selected to reflect and commemorate Martin's interests, but also to maintain the breadth of topics characteristic of DRT. Sessions included Crustal Kinematics & Mechanics, Localisation of deformation, Frictional-Brittle Processes, Folding, Boudinage & Deformation, Crystallographic Preferred Orientation, Mantle Anisotropy & Processes, Microstructures, Mechanisms & Mechanics and Melts & Deformation. Sessions were packed with talks and posters of new and original science, as well as some revisits of classic topics close to Martin – all attracting great feedback and discussion.

Field trips to two classic areas of British structural Geology: the Mam Tor landslide, Derbyshire, and Anglesey, north Wales were a great success with superb British autumn weather and active discussion.

Around 150 scientists travelled to Liverpool from across the globe, including Australia, Europe, north America, South Africa and Asia. Thanks to the Tectonics Studies Group support funds, we were able to award 6 research students with awards for best posters and best talks. Best talk prizes went to Erin Gray (Curtin), Verity Borthwick (Stockholm) and Daniel King (Minnesota), with mentions to Eddie Dempsey (Liverpool) and Robert Farla (ANU). Best posters were received by Sabrina Diebold (Utrecht), Sara Wassmann (Bochum) and Paulina Jaconelli (Stockholm) with mentions to Salah Elgarmadi (Liverpool) and Liz Cramer (Liverpool).

A selection of photographs from the conference and the DRT 2009 Conference Abstract Volume can be downloaded from www.liverpool.ac.uk/earth/drt2009. A thematic volume of papers entitled "Deformation Mechanism, Rheology & Tectonics: Microstructures, Mechanics & Anisotropy" will be published as a Special Publication of the Geological Society of London and will include many papers presented first at the Liverpool DRT. The volume will be dedicated to the memory of Martin Casey. The manuscript deadlines for this will be March 1st 2010 and further details are available from the web-address listed above.

(*Submitted by Dan Tatham, Dave Prior and Ernie Rutter*)

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Research Notes

A summary of "Viscous constitutive relations of solid-liquid composites in terms of grain boundary contiguity, 1, 2 & 3"

Yasuko Takei[1] & Benjamin Holtzman[2]

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Our collaboration has resulted in a series of three papers on a new model for grain boundary diffusion creep in partially molten rocks. The broad aim is to better understand the physical processes controlling the interactions of deformation, melt distribution and melt migration. Towards this aim, we saw the need for a 3-D, grain-scale model for viscosity in the presence of melt. The constitutive relations resulting from this model enable us to study dynamic processes at longer length scales, based on micro- and meso-scale textures observed in experimental samples and natural rocks. In this model, grain scale melt geometry is described by grain boundary contiguity. Contiguity is the fraction of the total grain area that is in contact with neighboring grains, and is a useful structural state variable for describing granular media. It can be quantitatively related to melt fraction for specified values of surface energies, for texturally equilibrated materials (e.g. Takei, 2002). This geometric basis is the same as the contiguity model for homogenization of elastic properties of partially molten rocks (Takei, 1998). Thus, the viscous model is an extension of the elastic contiguity model and builds on the original work of Cooper & Kohlstedt (1984) and Cooper et al. (1989) for grain boundary diffusion creep, referred to below as the CK model. In an *ab initio* approach, we derived viscous constitutive relations by solving the mass and mechanical balances and reaction and diffusion kinetics for each grain, with boundary conditions determined by the 3-D distribution of grain boundary contiguity. The many questions and findings that emerged in the process of developing the model have taken the form of these three companion papers (cited below as TH1, TH2, and TH3 and listed in references). The first focuses on the general model and its potential applications, the second on the creep behavior at very small melt fractions, and the third on the causes and consequences of viscous anisotropy.

TH1, "Grain boundary diffusion control model": The contiguity-based approach demonstrates that the 3-D pathways for matter diffusion through grain boundaries and melt, as illustrated in Fig 1a, are critical for understanding the nature of diffusion creep in partially molten rocks. The solution predicts that as long as melt forms connected pathways, matter will take the shortest path through the grain boundary, maximizing the path length through melt pockets. This model leads to several interesting results:

1) When plotted as a function of contiguity and compared to the contiguity-based model for elasticity, viscosity is more sensitive to contiguity than elasticity (Figure 1b), because of the strong dependence of viscosity on diffusion length. We also show that viscosity is more sensitive to microstructural anisotropy than elasticity. This ability of our model to calculate elastic and viscous properties from the same geometric basis will be useful for mapping between measurements or predictions of seismic properties and effective viscosity of partially molten regions of the Earth, discussed further below.

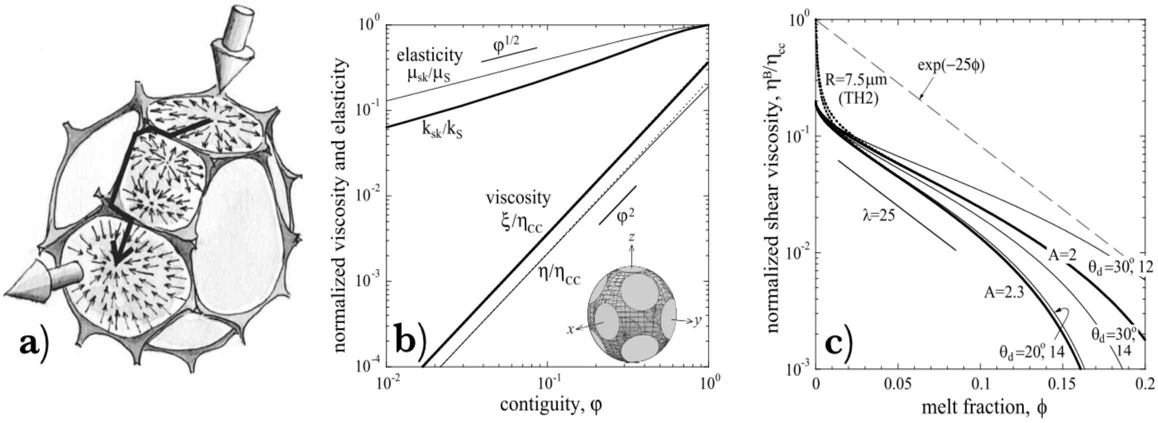

Figure 1. From TH1: a) A schematic illustration of a result of the contiguity model, that in 3D matter diffusing from grain boundaries under compression to grain boundaries under relative tension will take the shortest path to the melt and the longest path within the melt tubules. b) Results for isotropic shear and bulk elastic and viscous properties as functions of contiguity. Inset shows the 3-D geometric basis for contiguity. c)Viscosity as a function of melt fraction for various relationships between melt fraction and contiguity. The dotted line at low melt fraction shows the solution from TH2, that effectively smoothes the singularity at zero melt fraction in the grain boundary diffusion control model. Also, the λ = 25 line shows the empirical fit to experimental data, with which our model is consistent.

2) When plotted as a function of melt fraction (for a range of relationships between contiguity and melt fraction), the slope is similar to that of the experimentally determined dependence of viscosity on melt fraction (i.e. $\eta \propto \exp(-\lambda\phi)$), between melt fractions phi ~ 0.005 to 0.1, as shown in Figure 1c. Also, bulk and shear viscosity are predicted to be quite similar, with parallel slopes, a subject of importance for magma dynamics theory. The relative behavior of bulk and shear viscosity at small melt fractions is addressed in TH2.

3) The model predicts a singularity at zero melt fraction that is about a factor of 5 lower than the melt-free reference viscosity. In the grain boundary diffusion control model, this singularity is due to the simplifying assumption that melt diffusivity is infinitely high. Thus the flux in the melt is not limited, even at infinitesimally small melt fraction. Because the singularity, indicating a significant effect of very small amount of melt on viscosity, has important geological implications, we further refined the model to predict the detailed behavior of viscosity at nearly zero melt fraction, which is the subject of TH2.

TH2, "Compositional model for small melt fractions": The physical interpretation of the singularity at zero melt fraction in the diffusion control model is developed by describing the processes that occur at the microscopic scale as triple-junction tubules become infinitesimally small. While the grain boundary diffusion control model explored in TH1 assumes that melt diffusivity and reaction rates are infinitely high, the "compositional" model developed here treats finite melt diffusivity and finite reaction rates. This refinement of the model requires to specify the chemical and thermodynamic heterogeneity of melt in the system subjected to a differential stress by solving the mass balance and reaction and diffusion kinetics in the melt phase (as illustrated in Fig. 2a,b). Importantly, this model removes singularity at zero melt fraction in the grain boundary diffusion control model, as shown in Figure 1c (and 2c). The singularity is smoothed because the rate-limiting process changes from the diffusion though grain boundary to the diffusion through melt as the cross-sectional area of the pores decreases. Viscosity increases rapidly as melt fraction drops below a critical value (φ_c). In the mantle, this φ_c is predicted to be ~0.0001; in experimental conditions, $\varphi_c \sim 0.02$. This sharp change in viscosity could help explain the large fraction of the differences in creep strength between "nominally" melt-free olivine aggregates (i.e. Hirth & Kohlstedt, 1995a,b) and truly melt-free aggregates of synthetic olivine (i.e. Faul & Jackson, 2007). The model also predicts that φ_c for bulk viscosity is smaller than that for shear viscosity and bulk viscosity rapidly increases to infinite below φ_c. The compositional model further predicts the heterogeneous pore geometry under stress, which could establish the initial conditions for the growth of anisotropic melt distribution at the grain scale during deformation, the theme of TH3.

TH3, "Causes and consequences of viscous anisotropy": In TH1, the effects of a simple form of anisotropic contiguity on elastic and viscous properties were demonstrated. In TH3, a range of anisotropic melt distributions (or tensorial values of contiguity) were explored, designed to correspond to experimental observations of melt distribution under applied deviatoric stress (i.e. Takei, 2001, 2009; Zimmerman et al., 1999), in which melt pockets align at 0-25 degrees to the principal compressive stress direction. Under these forms of anisotropy (with an obliquity relative to the shear plane), the anisotropic viscosity tensor causes a softening in the least compressive stress direction (σ_3), which leads to a coupling between the shear and isotropic components of the stress tensor, as illustrated in Figure 3a. This coupling has very interesting consequences for meso-and macroscopic dynamics of melt migration, explored in forward models of the macroscopic two-phase flow equations. First, it predicts that melt can migrate up stress gradients in the solid matrix, as observed in experiments on solidifying metals (Gourlay & Dahle, 2005). Second, it predicts that this coupling can enhance the driving force for the segregation of melt into bands, as observed in experiments (e.g. Holtzman & Kohlstedt, 2007), and stabilize those bands at low angles to the shear plane (without a stress exponent greater than unity, e.g. Katz et al., 2006). Finally, it predicts that in geodynamic settings, melt will be attracted to the regions of highest shear stress. In a mid-ocean ridge, this region forms a lobe pointing towards the ridge axis, dipping 45 degrees (Fig. 3b); in a subduction zone corner flow, this suction effect will keep melt or fluid focused along the top of the

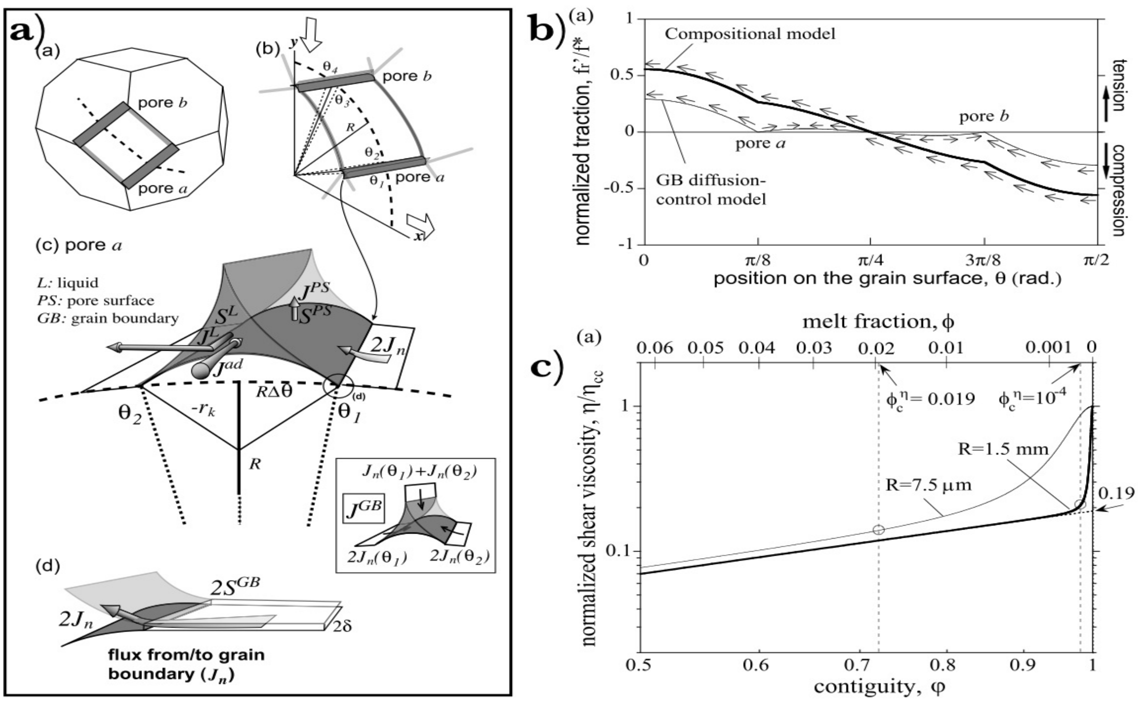

Figure 2. From TH2: a) Illustration of the model geometry and fluxes in the melt phase considered in the composition model for small melt fractions. b) Tractions on the 1-D (1/4 circular) grain surface for near-zero melt fraction, for the GB diffusion control model and the compositional model. The difference between the two models shown here corresponds to the presence and absence of the singularity at zero melt fraction. Arrows show the flux directions of matter. c) Solutions for shear viscosity as a function of contiguity for the compositional model at two different grain sizes, approximating earth- and experimental conditions.

slab (Fig. 3c). Melt migration to these interfaces will enhance the reduction of effective viscosity, causing a lubrication effect at a geodynamic scale.

Applications to Earth: In TH1, we demonstrated a simple approach to the application of this contiguity model to upper mantle situations, namely the base of an oceanic plate, by forward modeling the effects of various melt-distribution profiles on viscous and elastic properties (Fig 4a,b). The first direct application of this model to seismic observations was recently published in Kawakatsu et al., 2009. They detected large amplitudes of P-to-S and S-to-P converted waves, indicating a 7-8% contrast in shear wave velocity at the base of oceanic plates in the western Pacific. The contiguity model was used to calculate the velocity contrast and the corresponding viscosity contrast at the lithosphere-asthenosphere boundary. A range of solutions for different melt fractions and distributions were calculated, from a homogeneous partially molten asthenosphere to a

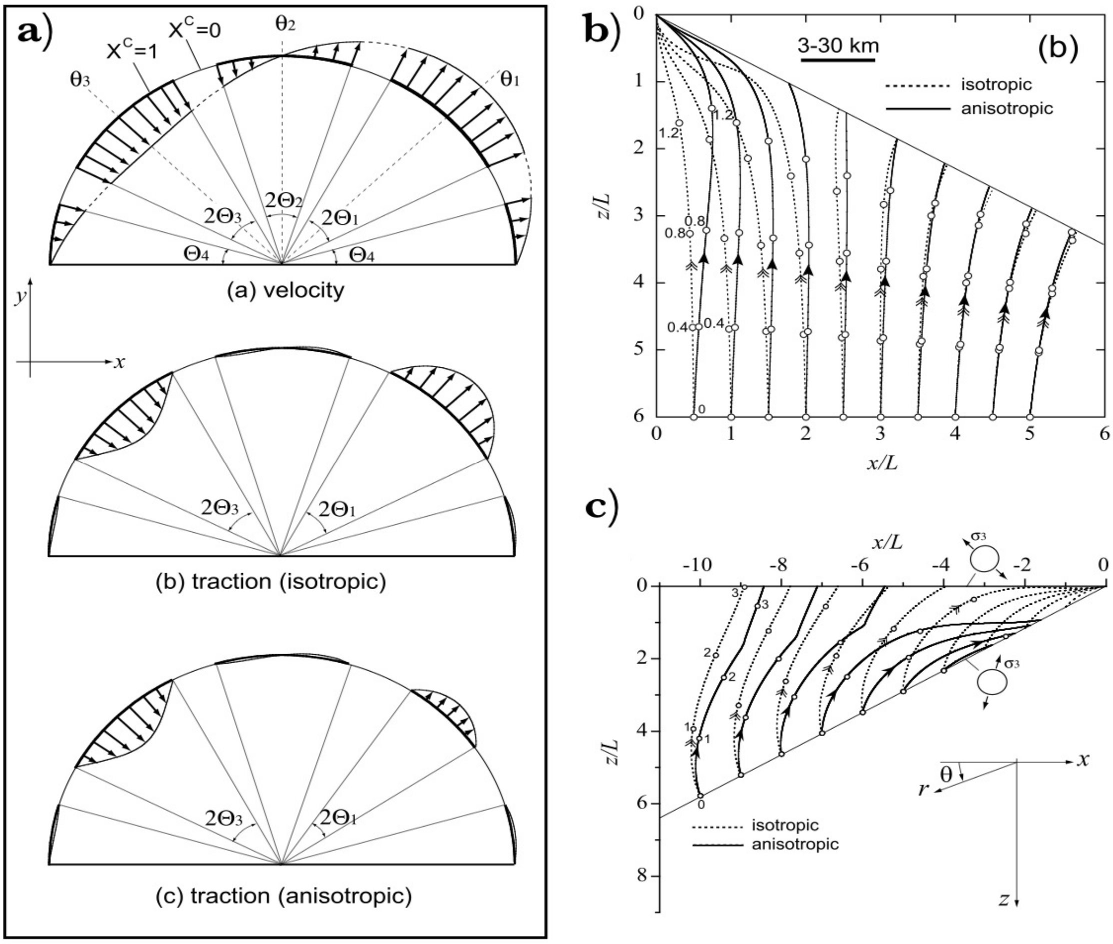

Figure 3. From TH3: a) Instantaneous velocity of grain surfaces due to deformation imposed upon a 2-D half-grain. Xc is the contact function, which equals 0 where the surface touches melt and 1 where it is a grain boundary. The middle figure shows the resulting tractions for isotropic melt/contiguity distribution, followed by that for a grain with an anisotropic melt/contiguity distribution. The asymmetry in the traction leads to a coupling between shear and isotropic components of the stress tensor. b) The melt flow trajectories calculated for a passive mid-ocean ridge flow for isotropic and anisotropic viscosity. The difference is due to the suction force towards regions of elevated shear stress, due to coupling between shear and isotropic components of the stress tensor (i.e. melt migration up stress gradients in the solid). c) Melt flow trajectories calculated for the mantle wedge of a subduction zone, for isotropic and anisotropic.

multi-scale layered model consisting of horizontal melt-rich and melt-free layers. The multi-scale model, but not the homogeneous model, is consistent with several diverse constraints: 1) the observed large velocity contrast from receiver functions, 2) dynamical models of plate-mantle interactions that indicate a need for a large viscosity contrast (10^3) across the LAB (e.g. Hoeink & Lenardic, 2008; Takaku & Fukao, 2008), and 3) geochemical studies that indicate only a small melt fraction (<1%) resides within the asthenosphere (references within Kawakatsu et al., 2009). While there is much yet to learn, mapping from seismic velocity measurements to viscosity estimates is becoming possible. As we began to explore in TH3, the contiguity model also enables us to simulate the formation of such multi-scale structures based on microstructural processes.

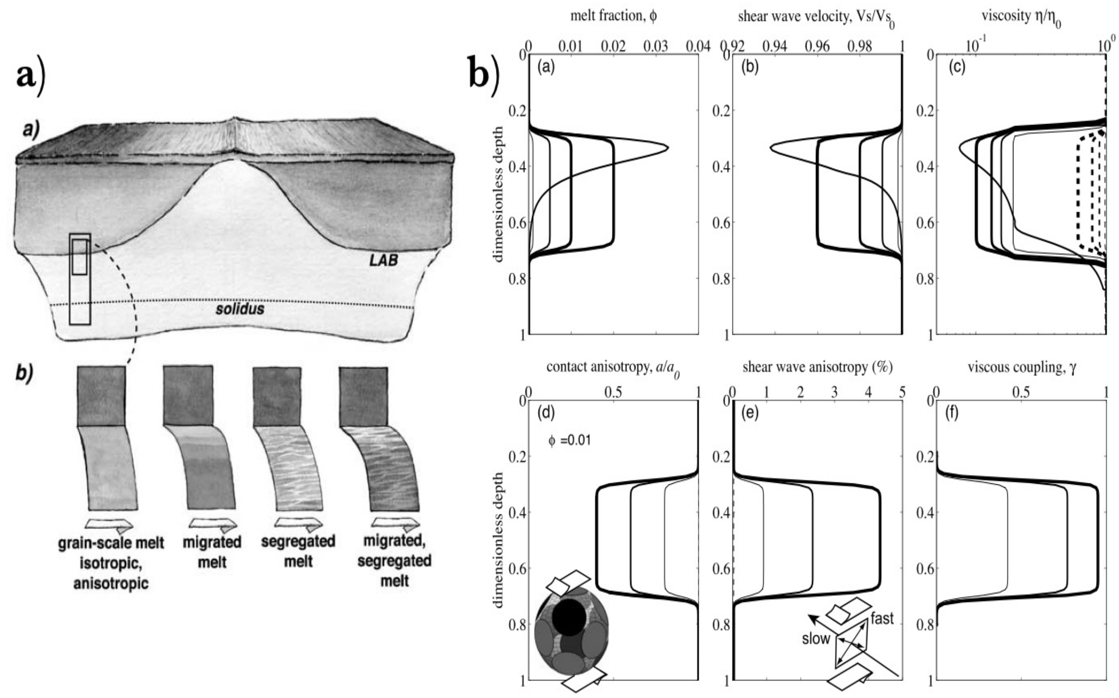

Figure 4. Applications to Earth, from TH1: a) Schematic illustration of a mid-ocean ridge and a suite of hypothetical melt distributions beneath the plate. b) Top row: hypothetical isotropic melt fraction distributions beneath the plate and resulting shear wave velocity reduction and corresponding viscosity reduction. The dashed lines represent the empirical relation from experiments on melt-bearing samples. The difference between the empirical curves and model predictions indicates the potential importance of the difference between nominally melt-free and truly melt-free rocks, discussed in TH2. Bottom row: hypothetical distributions of contact anisotropy in the contiguity model, resulting shear wave anisotropy and degree of viscous coupling between the shear and isotropic parts of the stress tensor.

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The benefit of BIB/FIB-cryo-SEM techniques for characterization of elusive microstructures in clay-rich geomaterials

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Abstract

Detailed investigation of the morphology of the pore space in clay is a key factor in understanding the sealing capacity, coupled flows, capillary processes and associated deformation present in clay-rich geomaterials. Actually, the combination of ion milling tools, cryogenic techniques and SEM imaging offers a new alternative to study in-situ elusive microstructures in wet geomaterials and has the high potential to make a step change in our understanding of how fluids occur in pore space. By using this range of techniques, it is possible to stabilize in-situ fluids in pore space, preserve the natural structures at nm-scale, produce high quality polished cross-sections for high resolution SEM imaging and reconstruct accurately microstructure networks in 3D by serial cross sectioning.

1. Introduction

Clay-rich geomaterials form seals for hydrocarbon accumulations, aquitards and chemical barriers. The sealing capacity is controlled either by the rock microstructure or by chemical interactions between minerals and the permeating fluid. A detailed knowledge about the sealing characteristics is of particular interest in the storage of anthropogenic carbon dioxide and radioactive waste in geologic formations. A key factor to understanding the sealing by clay-rich geomaterials is a detailed understanding of the morphology of the pore space. The morphology of porosity has a strong effect on many mechanical and transport properties of claystones. Though the bulk expression of these properties are relatively well known for a number of clay-rich geomaterials, the relation between nanostructures and macroproperties are poorly studied for a complete understanding of the fluid-rock interactions. There is a considerable body of literature on the characterization of porosity in clay-rich geomaterials using many different techniques (metal injection porosimetry; magnetic susceptibility measurement; SEM; TEM; neutron scattering; NMR spectroscopy, CT scanner; ESEM…). However, the pore space characterization has been mostly indirect until now.

To investigate directly the meso-porosity (from mm to about 10 nm in pore size) in clay-rich geomaterials, SEM imaging is certainly the most direct approach but, on one hand, it is limited by the poor quality of the investigated surfaces (mainly broken or mechanically polished surfaces including the decoration of porosity by colored resin embedment), which make observation difficult, and the interpretation of the microstructures complicated; and on the other hand, most of conventional methods require dried samples in which the natural structure of pores could be damaged due to the desiccation and dehydration of the clay minerals. The recent development of ion milling tools (Broad Ion Beam, BIB and Focussed Ion Beam, FIB) and cryo-SEM allows respectively producing exceptional high quality polished cross-sections suitable for high resolution porosity SEM imaging at nm-scale and investigating samples under wet conditions.

In order to summarize the benefit of using BIB/FIB-cryo-SEM techniques, this contribution reports on investigation of pore space in Boom (Mol site, Belgium) and Opalinus (Mont Terri, Switzerland) clays, Rotliegend sandstone and a SAFOD sample, which are important clay-rich materials since they are respectively investigated for nuclear waste storage, tight gas reservoir production and detailed understanding of earthquake processes.

2. Ion beam cross sectioning

SEM imaging of pore space at high resolution in geosciences is undergoing rapid development since ion beam milling tools has been demonstrated as a powerful technique to prepare smooth, damage free cross-sectioned surfaces at the state-of-the-art SEM resolution ([1], [2], [5], [6] and [9]). We distinguish two main type of ion source: (1) a broad ion beam (BIB, up to few mA, Argon source) is suitable to produce large polished cross-sections area of few mm^2 (Figure 1), while the focused ion beam (FIB, 1pA - >50nA; Gallium source) is better used for fine and precise polished crosssections area of about few μm^2 (Figure 3). Because BIB is not focused, a shielding plate is placed on the top surface of the sample in order to create flat cross-section (Figure 1). Up to now, on the market, BIB cross sectioners are usually available as stand-alone machines able to produce single polished cross-section; while FIB cutters are now mainly integrated in SEM offering the possibility to perform serial cross sectioning suitable for 3D microstructures reconstruction (Figure 4, [1], [2], [5], [6] and [9]). However, the big advantage of BIB is that it is able to produce larger polished surfaces. This feature is of relevant interest for geosciences since it fits better to the typical size-range of microstructures and representative elementary area of geomaterials (Figure 2, [2]).

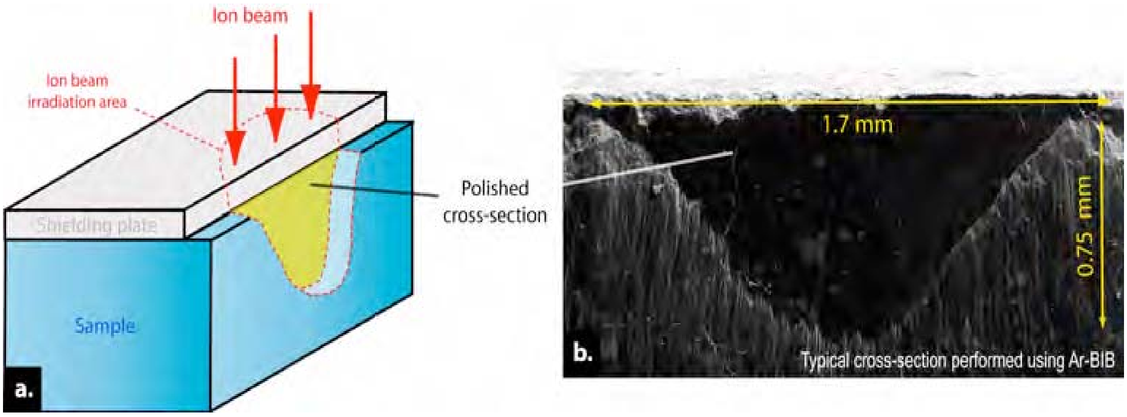

Figure 1: The principle of BIB cross-sectioning. (a) the ion beam irradiates the edge of sample un-masked by the shielding plate to create mirror-polished cross-sections suitable for SEM imaging. (b) Overview of a typical cross section performed in SAFOD sample by BIB (6 KV, 6 hours, 150-200 µA).

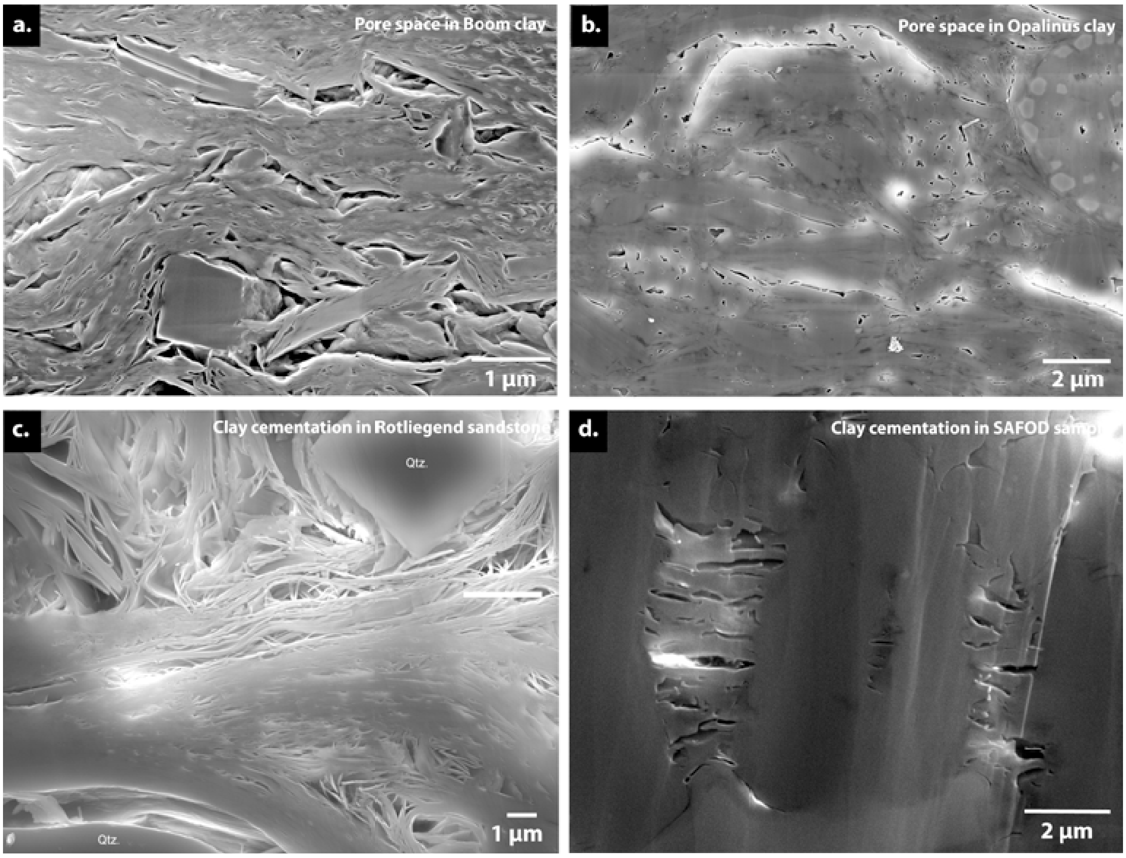

Figure 2: SEM micrographs (SE) from BIB polished cross-sections performed on different types of rich-clay geomaterials. (a) Boom clay (Mol site, Belgium) and (b) Opalinus clay (Mont Terri, Switzerland) show the 2D pore space. The morphology of pores depends on clay particle arrangement and mineralogy. (c) Rotliegend tight gas reservoir sandstone: pore at quartz-grain triple junction where the illite cementation is arranged tangential at the direct vicinity of the detrital grain edges and as "hairy/fibrous" toward the pore center containing an "floating" angular quartz fragment. (d) SAFOD sample: porosity in clay-filled channels connecting calcite veins.

3. Cryo-SEM

A Cryo-SEM is the combination of cryogenic techniques to stabilize wet media at cryo-temperature with state-of-the-art SEM to image the stabilized microstructure and in-situ fluids at high resolution (Figure 3). Though the cryo-SEM method is widely used in Life Sciences ([3] and [8]), applications in Geosciences are up to now limited to few studies of fluid inclusions and wettability ([4], [7] and [10]) since investigations are performed on poor quality mechanically prepared surfaces. However, the actual standardization of FIB source embedment in SEM chamber and its compatibility with cryogenic methods solves this problem of surface preparation and is opening novel cryo-SEM applications for fluid-rock interaction studies ([1], [2], [5], [6] and [9]).

For rich-clay materials, FIB-cryo-SEM method is of special interest since it allows preparing samples without dehydration and dessication processes which could disturb the natural structures of clay phases, stabilizing in-situ fluids to investigate their distribution in pore space (Figure 3 and .

[2]), producing high-quality polished cross-sections for high resolution SEM imaging and reconstructing accurately the pore space networks in 3D by serial cross sectioning (Figure 4, [2] and [5]).

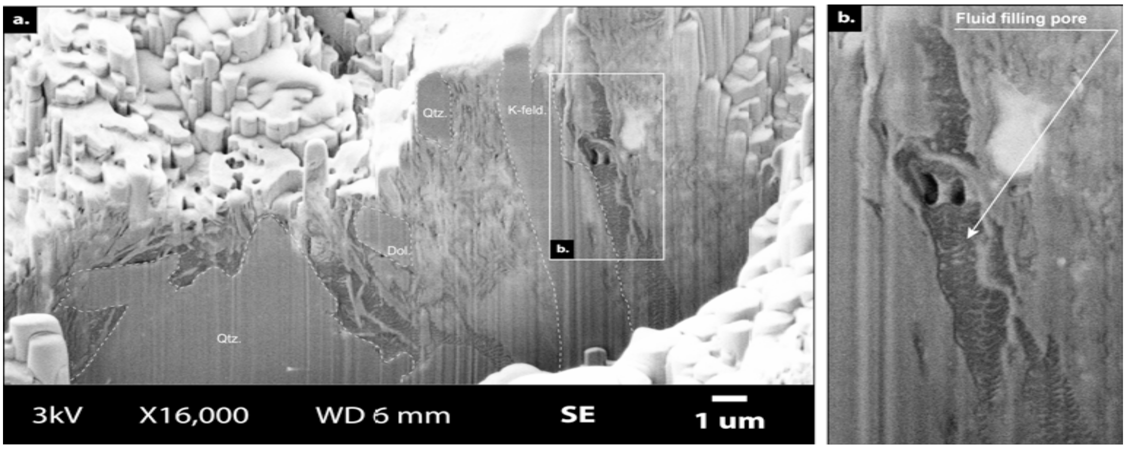

Figure 3: SEM micrograph (SE) of FIB cross-section in Boom clay performed under cryo-condition. In-situ fluids are clearly visible in the biggest pores. (a) The biggest pores are located around the non-clay minerals (Quartz = Qtz, Dolomite =Dol., K-Feldspar = K-feld) and the smallest pores in the clay aggregates (b) Close-up showing *in-situ* fluid.

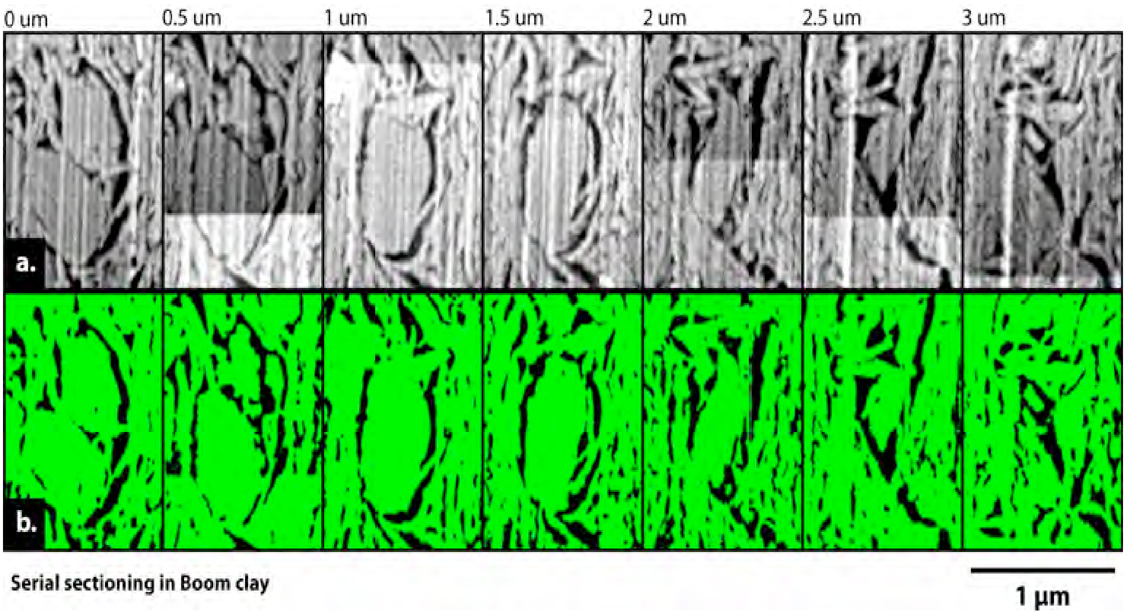

Figure 4: FIB serial cross-sections performed under cryo-condition around a quartz grain in Boom-clay, perpendicular to the bedding. The slice thickness is 500 nm. (a) Initial SE pictures and, (b) segmented pictures. The porosity is mainly connected along the direction parallel to the bedding.

4. Conclusions and further developments

Application of the BIB-FIB-cryo-SEM methods to study the porosity in clay-rich materials is clearly relevant for waste disposal, hydrocarbon production, basin modeling, fault zone studies and others. It has the potential to open a new field of investigation, producing an atlas of accurate pore models of mudstones. These methods offer a powerful combination for direct and *in-situ* investigations of elusive structures in rich-clay materials at the pore scale, opening a new field of investigations to study relations between nanostructures and macro-properties, which are poorly understood at present. As far as we know, this is the first time that fluids were directly imaged in in-situ conditions in mudrocks, offering new insights for the study of fluid-rock interaction (osmotic effect, ion exchange, pore alteration). The reduction of the slice thickness (down to 20 nm) for serial sectioning will produce high-resolution models of pore space with the possibility to model fluid flow and create microstructure-based models of transport in clays. This will provide a unique opportunity to model the flow through in-situ pore networks (Lattice Boltzmann method, [11]), study the effective interconnectivity, investigate the natural fluid distribution and quantify investigated microstructures ([2] and [5]). The next generation of ion beam-cryo-SEM instrument, especially designed for geomaterials, will probably include a BIB source instead of FIB. The first BIB-cryo-SEM machine is currently installing at RWTH Aachen University in Germany (Deutsche Forschungsgemeinschaft – Project UR 64/9-2) and will be dedicated to micro imaging of elusive microstructures in wet geomaterials.

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Report on the SAFOD Inter-laboratory Comparison

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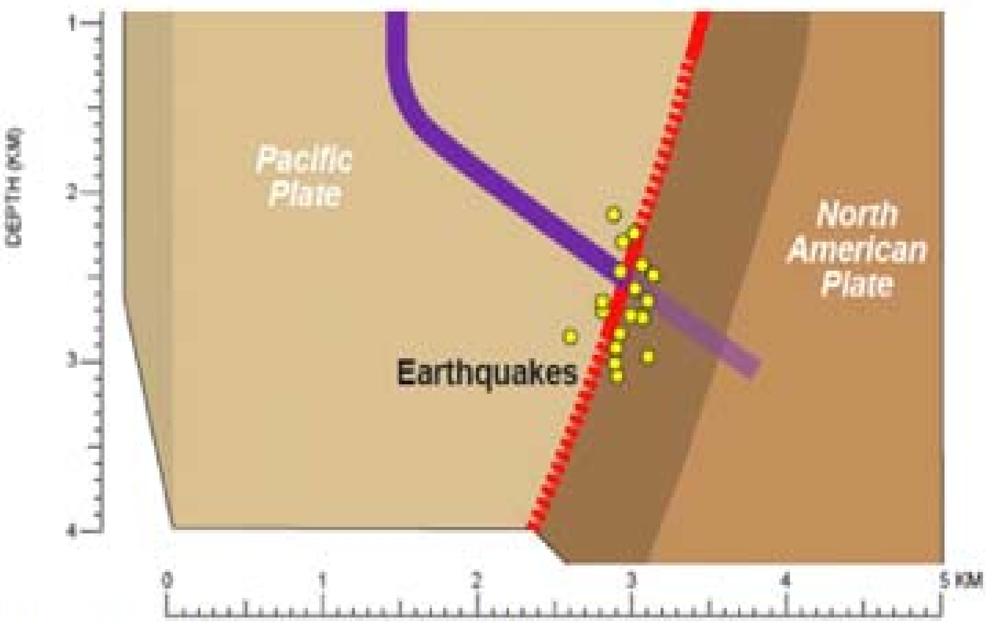
An international group of 20 laboratories have been carrying out tests on a suite of standards designed to allow detailed comparison between the results of physical property measurements on core recovered during drilling of the San Andreas Fault Observatory at Depth (SAFOD). The third phase of SAFOD drilling was completed in the summer of 2007 and included core recovery from the main trace of the San Andreas Fault at ~3 km depth. The SAFOD interlab comparison (SIC) exercise began in the fall of 2008 at the request of the US National Science Foundation. In all, SIC includes 37 investigators from nine countries. The work is divided into six areas: friction, permeability, elastic wave speed, rock strength, electrical resistivity, and thermal conductivity. Participants in the friction comparison are using a suite of five samples ranging from synthetic materials to a mixture of cuttings from SAFOD drilling in the vicinity of the main fault strand. Participants in the other areas are using five samples, including three sandstones, a marble and a granite. Preliminary data have been reported by six laboratories, with results on friction, permeability, strength, and ultrasonic velocity.

The test protocols call for a range of conditions relevant to the brittle upper crust. Stresses range from hydrostatic to shear failure with mean stresses up to 150 MPa. Fluids range from distilled water to brines. Most of the standard conditions involve room temperature experiments but a few labs are looking at higher temperatures. Details of the protocols and initial results are available online at: http://www.geosc.psu.edu/~cjm/safod/ (but note that initial results are available only to labs participating in SIC).

This past summer The NSF EarthScope program, working with recommendations of the SAFOD Core Sampling Committee (SSC), authorized the second round of requests for SAFOD drill core. Initial distribution of samples began in September, 2009 and continues now. All investigators who are making physical properties measurements are required to complete the interlab comparison before SAFOD core samples are released to them.

Participants of SIC are planning to use the calibration exercise to aid the broader geophysical community. Comparisons will be especially useful to new laboratories and for those with newly developed equipment. As organizers of the inter-lab comparison, we hope to publish a complete set of comparisons. An informal SIC group meeting will take place at Fall AGU on Thursday during the lunch break: 12:30 pm, Moscone West room 2002. The meeting is open to everyone.



Phase III occured over for the summer of 2007, involving several multilateral core holes drilled off of the main hole to obtain continuous core within the San Andreas Fault Zone. A pilot hole was drilled at the SAFOD site in 2002 with financial support from the International Continental Scientific Drilling Program (ICDP) and NSF.

Meeting Announcements

Gordon Conference on Rock Deformation

August 8-13, 2010. Tilton School, New Hampshire.
(Chair: Dave Prior, Vice-chair: Peter Kelemen)

The Gordon Conference format provides for the most stimulating environment to discuss the cutting-edge of rock deformation research. The Conference will comprise 18 provocative and detailed invited talks, each followed by extensive discussion coordinated by expert chairs. There is also plenty of opportunity for more informal meetings and discussions, and added to this we hope that all those who attend will present their own research in poster form.

The theme of the eighth Gordon Conference on rock deformation is “Transient and transitional behaviour in rock deformation: moving away from steady-state.” Creep in the crust and mantle is commonly considered a steady-state process. This view prevails despite the fact that earthquakes do not represent steady-state and at the base of the seismogenic zone, for example, the stresses that drive creep must vary with the earthquake cycle. The contribution of transient versus steady-state behaviour is not easy to determine from naturally-deformed brittle or plastic rocks, and our view of steady-state depends on whether we consider geological or shorter time-scales. Perhaps we avoid a non steady-state picture because we lack appropriate descriptive or quantitative tools. The aim of the 2010 Gordon Research Conference (GRC) in rock deformation is to explore what we know about non steady-state deformation and how we might advance our understanding through geological and geophysical field investigations, laboratory experiments and modeling. This will require an appraisal of the applicability of steady-state concepts as well as an exploration of transient behaviour, in which processes and physical properties cycle between different states as might be the case during earthquake cycles, and transitions in behaviour, where finite strain or changing environmental conditions lead to changes in processes and properties. Conference sessions will cover:

- What is steady-state?
- Seismogenic faulting and brittle fault rocks.
- Episodic creep during the seismic cycle.
- Deformation in zones of temperature and stress cycling.
- Deformation, metamorphism and fluids.
- Mechanism and microstructure transitions during deformation.
- Mechanism and microstructure transitions related to mantle geophysics.

The role of fluids, including hydrous fluids, hydrocarbons and melts will be embedded within each topic area. The development of mechanical instabilities is also important to all of these areas and will have an impact that depends on the scale of the system.
The full program for the meeting is already available and can be viewed at

the GRC website: http://www.grc.org/. Use the same website for registration. The GRC format of focused, in-depth talks and extended discussion together with the opportunity for all participants to present research posters will provide a great opportunity for the geosciences community to discuss this difficult topic. Please mark the dates in your diaries and come to the GRC to contribute to discussion at the cutting-edge of rock deformation.

COMPRES Annual Meeting,

June 22 – 25, 2010, Washington, USA

The Executive Committee of COMPRES (COnsortium for Materials Properties Research in Earth Sciences) has voted unanimously to hold 2010 Annual Meeting from June 22 – 25 at the Skamania Lodge in Stevenson, Washinigton. The lodge is located on the north bank of the Columbia River Gorge; details at http://www.skamania.com/. For more information, please surf COMPRES webpage at: http://compres.us/.

EMPG XIII - International Conference

April 12-14, 2010 - Toulouse, France

The 13th International Conference on Experimental Mineralogy, Petrology and Geochemistry (EMPG XIII), which will be held in Toulouse, France, on April 11 to 14, 2010. Abstract submission and early registration will start on 1st December on the web-site: http://www.empg2010.com

Short Course on: Microstructures and Physico Chemical Properties of Earth and Planetary Materials

February 8 -12, 2010, Verbania, Italy
http://www.socminpet.it/Micro/index.html

Transport properties in the Earth and Planets are deeply controlled by microstructural arrangement. Viscosity, permeability, porosity, electrical conductivity, seismic attenuation, and chemical diffusion are influenced by grain size, shape preferred orientations, crystal preferred orientations and/or by a variety of microstructural heterogeneities on a dimension scale ranging from nanometers to meters or more. The microstructure of rocks and minerals records genetic processes testifying to incredible complexities in the formation of our planet. The unceasing game of strain and annealing processes in nature have led to an endless combination of the fabric observed in metals and ceramics. In the framework of the education activities of the Petrology Group of *the Italian Society of Mineralogy and Petrology* and of the *Marie Curie Research Training Network-c2c* (crust to core), this short course will review the fundamentals of microstructure development, moving from principles in material science to applications in mineralogy, petrology and geophysics. Original contributions from participants are also welcome for inciting discussion on "hot topics" in microstructural analysis.

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Book Announcements

Nonlinear Mesoscopic Elasticity: The Complex Behavior of Rocks, Soil, Concrete

Robert A. Guyer, Paul A. Johnson

This handbook brings together new data on the static and dynamic elastic properties of granular as well as composite materials. The authors present new and imported theoretical tools that have enabled our current understanding of the complex behavior of rocks. It is a comprehensive book of interest to researchers involved in civil engineering and geophysics.

Creep and Fracture of Ice

Erland M. Schulson, *Dartmouth College, New Hampshire*
Paul Duval, *Centre National de la Recherche Scientifique, Paris*

This book deals with the physics of the creep and fracture of ice, and their interconnectivity. The book discusses the relationship between structure and mechanical properties in ice. The book provides a road-map to future studies of ice mechanics. It is ideal for graduate students and scientists in Earth and planetary science.