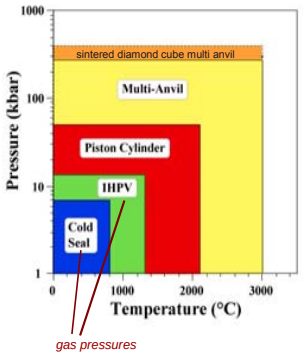
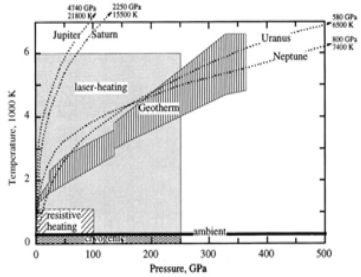
Experimental techniques

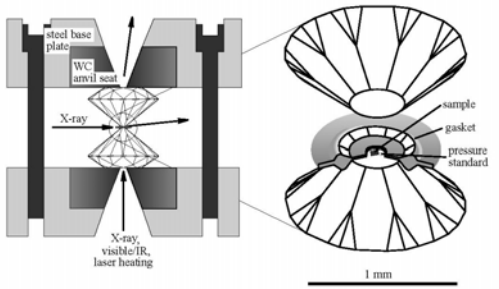
- Experimental Petrology (0 – 40 GPa, 25-3000°C)
- Experimental Mineral Physics (0-350 GPa, 25-7000°C)

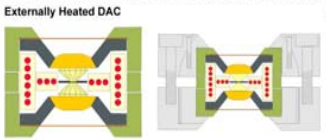




Diamond Anvil Cells (DAC)

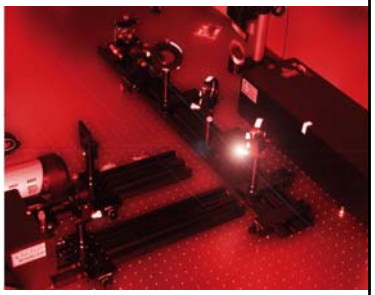
Tools I: Diamond Anvil Cells

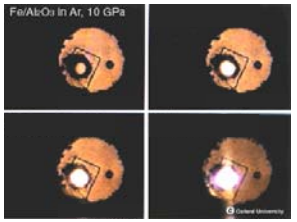




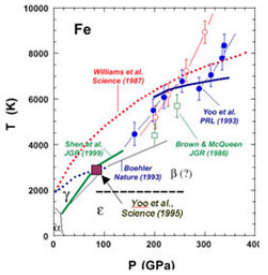
0 – 350 GPa, 7000°C

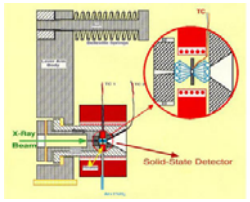
Tools I: Diamond Anvil Cells

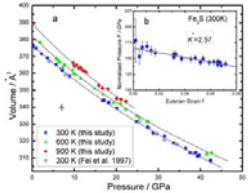


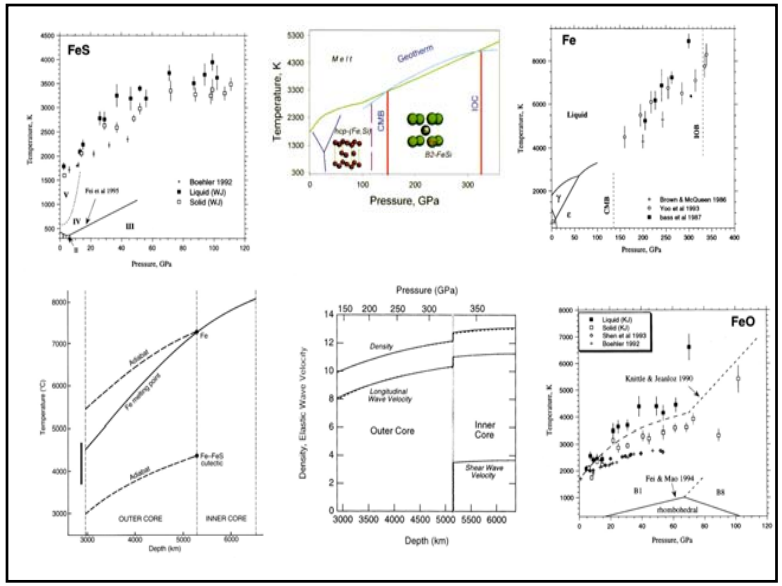


Tools I: Diamond Anvil Cells









Tools I: Diamond Anvil Cell

extreme conditions
in-situ observations (spectroscopy, X-ray diffraction etc etc)
extreme temperature gradients
open chemical system
micron-size samples

good for simple systems and mineral physics questions
not so good for petrology and phase diagrams

Future:
since a few years chemical analyses of run products with
FIP - analytical TEM

Tools II: Gas pressures

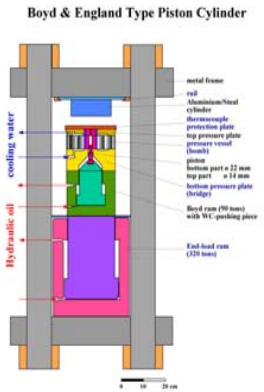
Typically 5 kbar, 600 °C

Very slow quench

Two types: externally heated (pressure vessel at high T)
internally heated (furnace inside the water cooled pressure vessel)

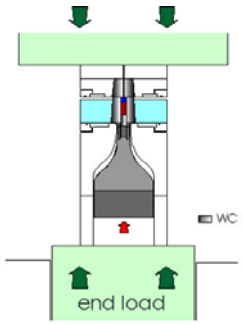
typically 4-5 kbar, 1300 °C, a few (one ?) to 12 kbar, 1200 °C

Tools III: Piston Cylinder Solid Media Presses

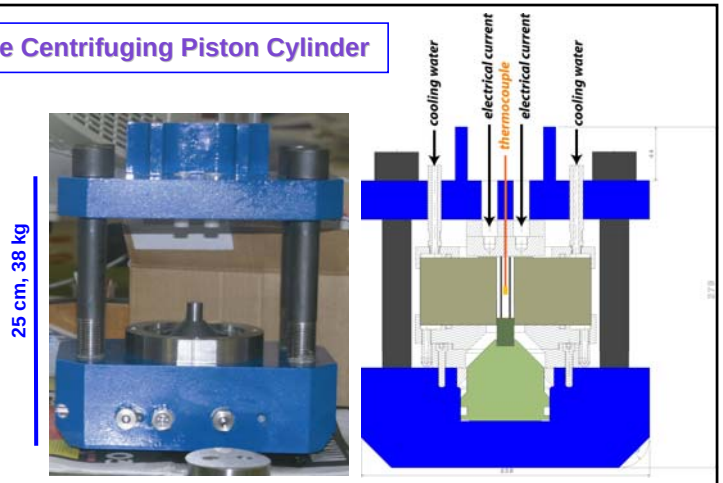


0.5 - 4 GPa, 2000°C

Tools III: piston cylinders

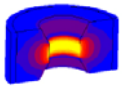


The Centrifuging Piston Cylinder



- typical magmatic pressures: 2-18 kbar (5 - 55 km depth)
- standard apparatus: piston cylinder

(with standard size of 80-200 cm height and 200-500 kg weight)



conical steel ring pressed into matrix provides lateral stress/pre loading

Tools 3: piston cylinders

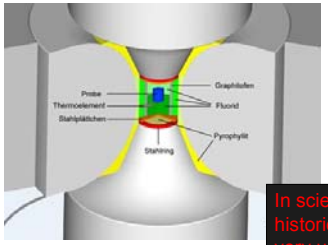
Endloaded = two hydraulic cylinders, one for piston, one to pre-constrain ("loading") the pressure vessel axially

Single stage = one hydraulic cylinder to drive piston

piston cylinders are
- relatively low-tech,
- reliable (>95% technically successful experiments)
- not so expensive (200 SFr/experiment)

- the work horses of experimental petrology

Belt apparatus - concave shaped bore/cell - max. 70 kbar

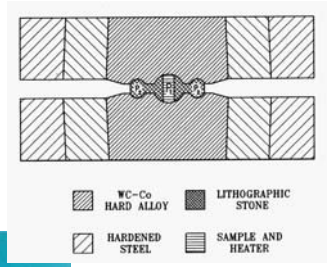


photos: Frankfurt - G.Brey

In science in a few labs still present as a historical (pre-multi anvil) developement, very usefull, but a big and expensive apparatus to cover the 5-7 GPa range

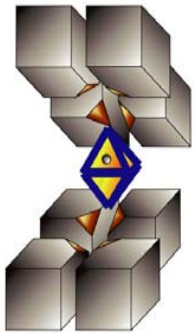
industrial diamond making - all with Belt-type apparatus

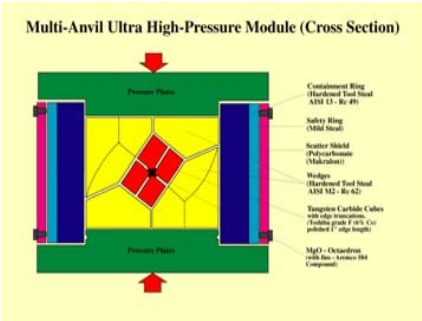
Torroidal apparatus (invented in Russia) - can be used for deformation in torsion geometry



Tools IV: multi-anvils

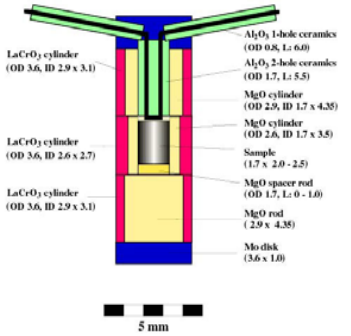
Prinzip einer 6/8 Multi-Anvil Apparatur

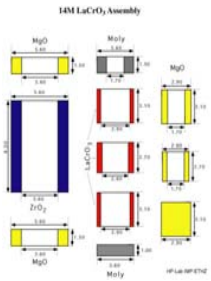




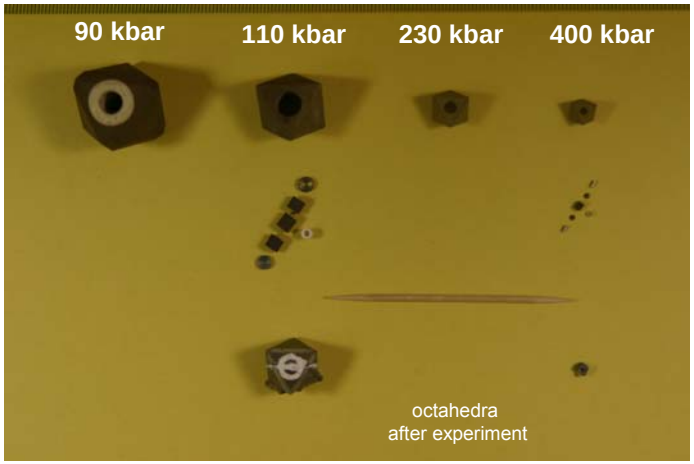
Assembly-Parts

$LaCrO_3$-Furnace Multi-Anvil Assembly for 8 mm TEL (usable to 15 GPa, 2500°C)

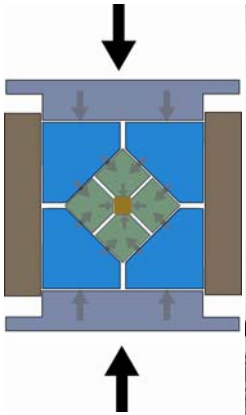


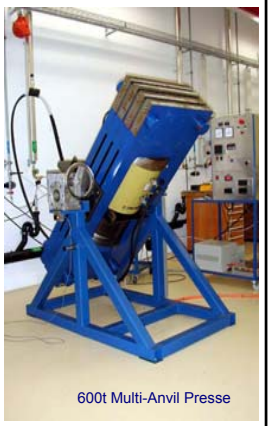


UNIAXIAL Multi-Anvil Press: Assemblies



The Tools II: Uniaxial Multi-Anvil Presses



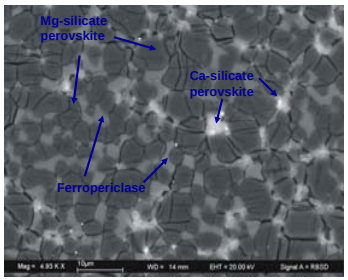


3 – 25 GPa, 2500°C

Tools IV: multi-anvils - limit

The inner anvils (8 cubes) are made of WC which best qualities start to deform plastically at 25 GPa

→ use of different anvil materials → sintered diamond (to 90 GPa possible)
cubic Bornon Nitride (cBN)



$(Mg,Fe)(Si,Al)O_3$
$(Mg,Fe)O$
$CaSiO_3$

~1 wt% Fe-rich metal
$3\ FeO_{(system)} = Fe_2O_{3(MgPv)} + Fe^0$

(Frost et al. 2004)

A view into the lower mantle

Heterogeneity in the lower mantle

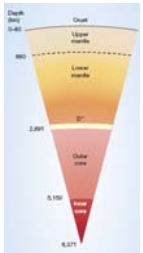
From mineral physics and HP-experiments: no futher phase changes until D'':

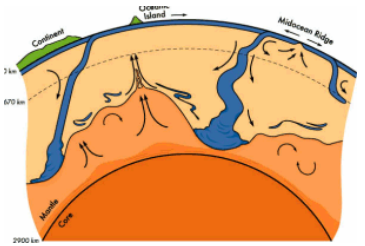
MgPv → ‚post-perovskite' at ~125 GPa (Murakami et al. 2004, Oganov & Ono 2004)

..."geophysical evidence indicates that compositionally distinct, hence convectively isolated, mantle domains may exist in the bottom 1000 km of the mantle."

van der Hilst & Karason 1999

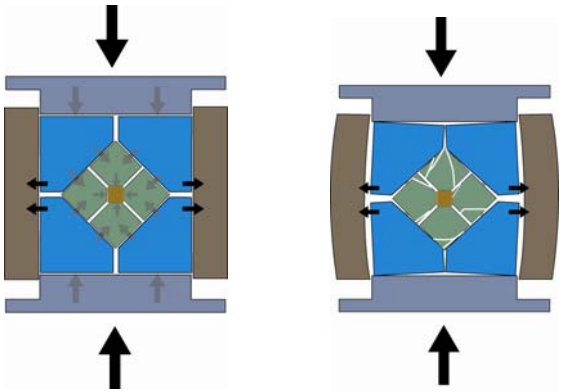
- Seismic tomography, variations from PREM
- Noble gasses; mantle is not completely outgassed
- ‚missing' heat producing elements (U, Th)

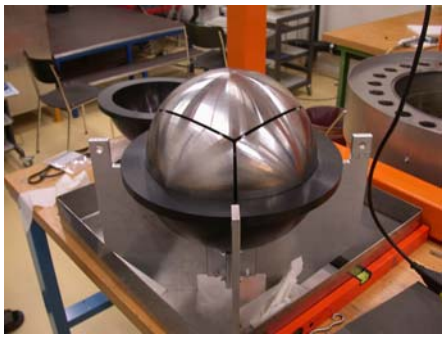


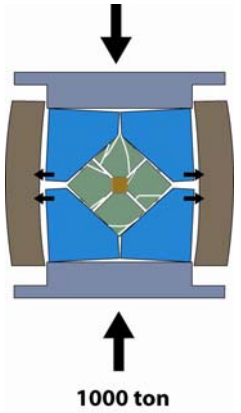


The uniaxial multi-anvil design is limited to WC (hardmetal) cubes

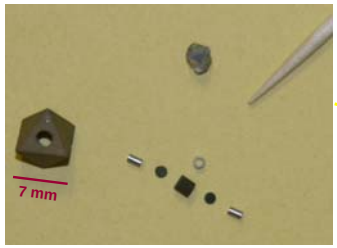
- WC-cubes deforms plastically at 23-25 GPa
- Thus, lower mantle pressures need better materials (sintered diamond) but a different design !!!

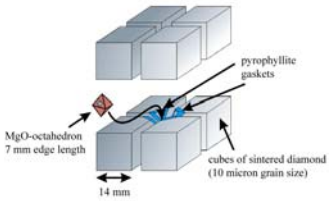


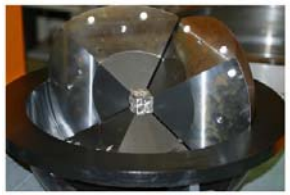


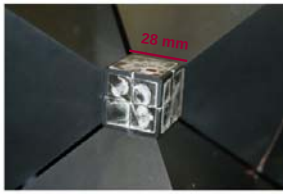


The Tools III: Spherical multi-anvil with diamond-cubes – to 400-500 kbar / 2500°C

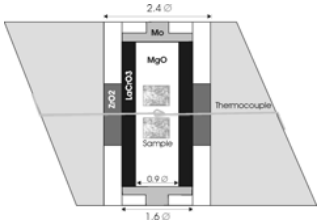


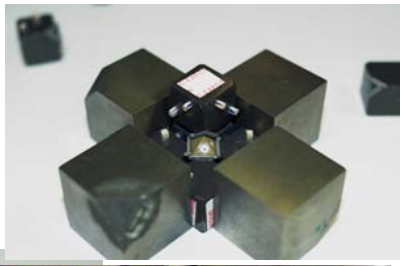


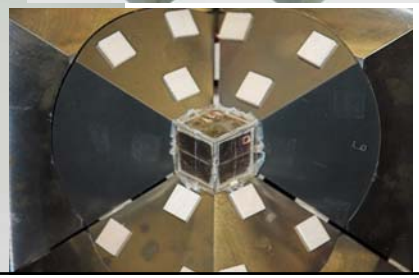


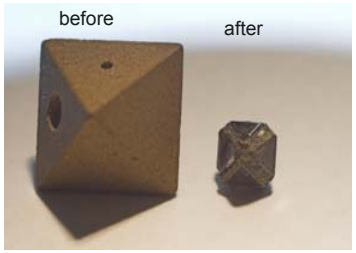


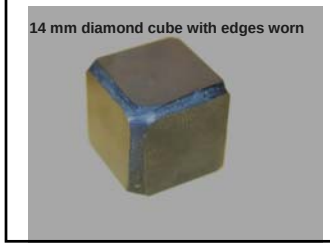
Doing experiments in ‚The Bucket'

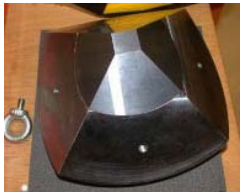




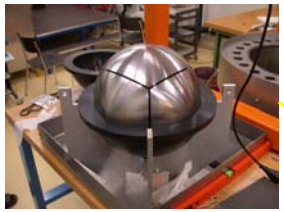


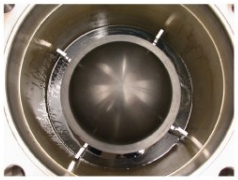






Spherical multi-anvil for use
with sintered diamond-cubes

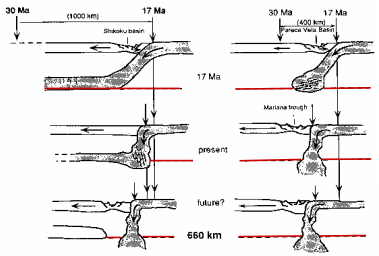




40 GPa = 600 km into the lower mantle of Earth, core pressure of Mercury and Mars

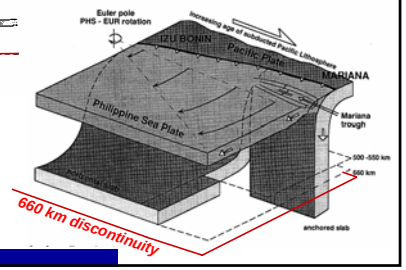
Lower Mantle and Core Formation (1)

subduction into the lower mantle



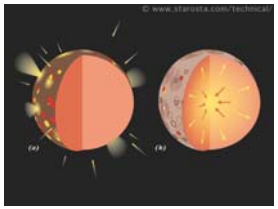
660 km discontinuity

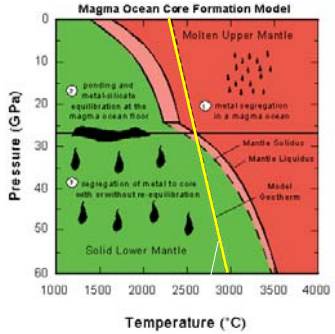
van der Hilst + Seno (1993)



Lower Mantle and Core Formation (3)

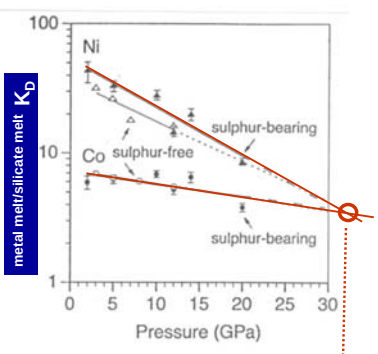
"magma ocean" during accretion of the Earth

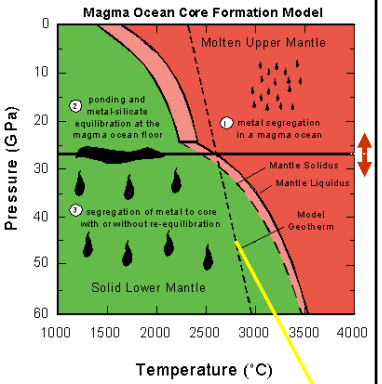




Magma ocean depth from Co-Ni fractionation

Co and Ni did not fractionate from each other in the silicate Earth (with respect to Chondrites), i.e. are equally partitioned between metal and silicate Earth.





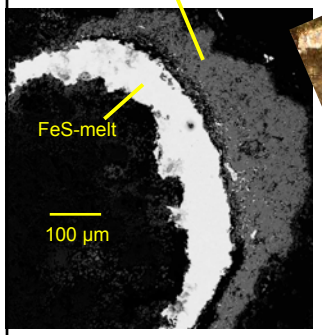
depths of magma ocean

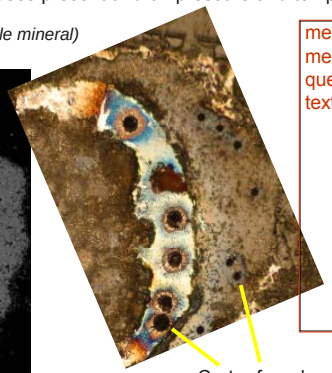
early geotherm

Trace element distribution between lower mantle minerals and metal melt

The partitioning (redistribution) of elements between the metal core and silicate Earth depends on the minerals/phases present and on pressure and temperature

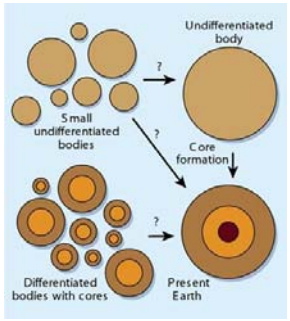
Ca-perovskite *(lower mantle mineral)*





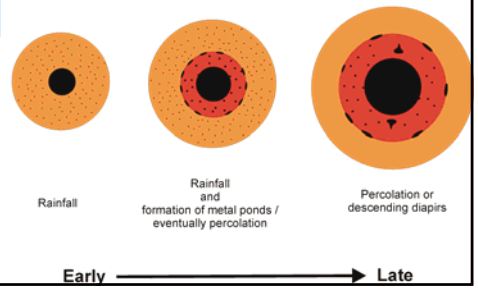
metal
melt
quench
textures

Crater from laser ablation ICP-MS

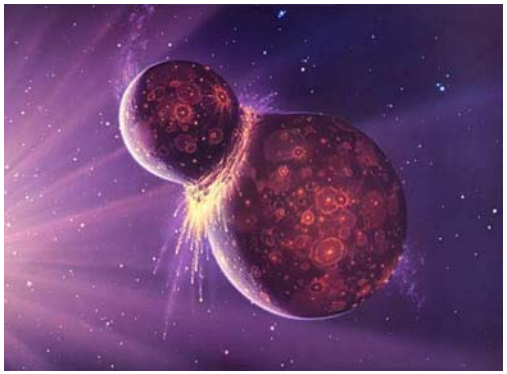


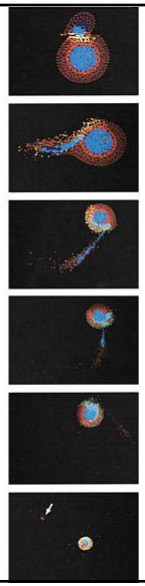
What are the conditions of core segregation ?
How did the Earth's magma ocean solidify ?

A homogeneously accreting Earth

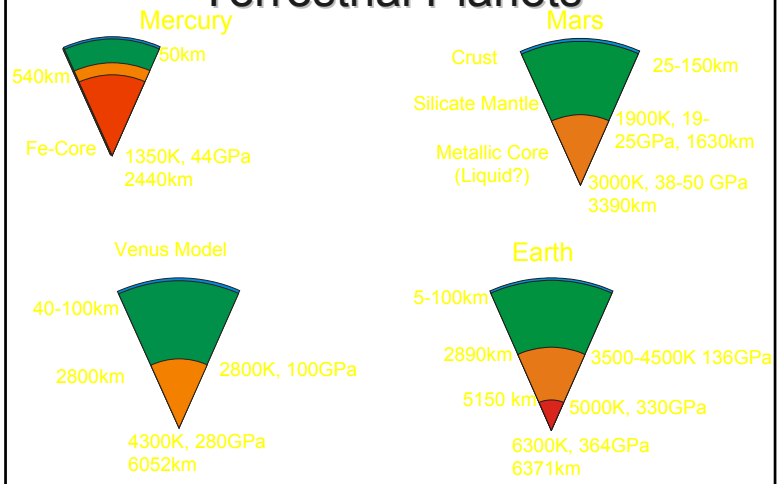


What are the conditions of core segregation ?
How did the Earth's magma ocean solidify ?

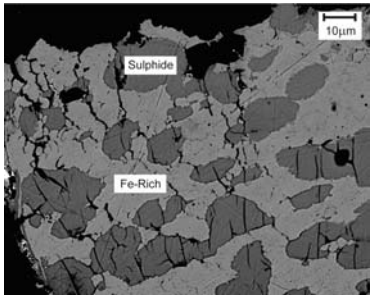




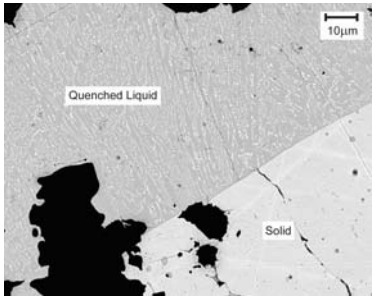
Terrestrial Planets



Samples at the centre of Mars

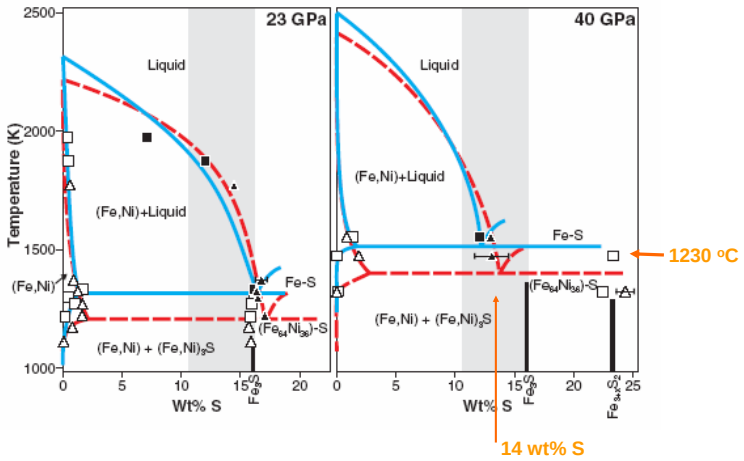


40 GPa, 1323 K (Fe-S)

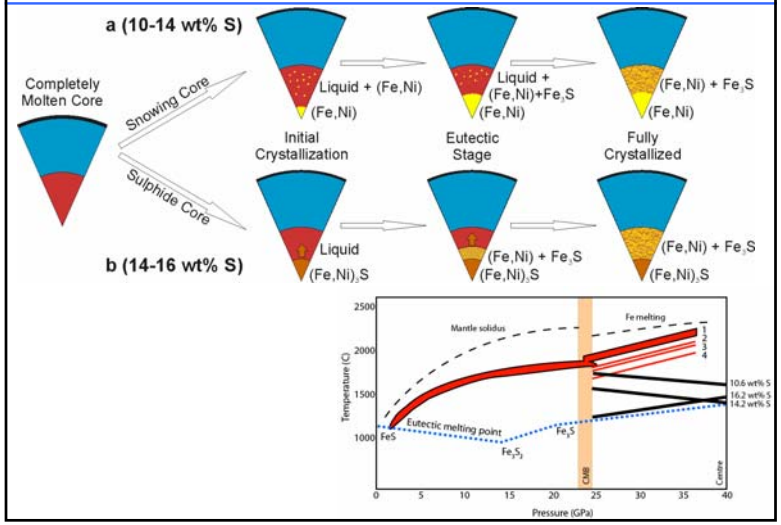


40 GPa, 1553 K ($Fe_{64}Ni_{36}$-S)

Fe-Ni-S phase diagrams



Current state and crystallisation regimes - Mars



Tools V: others

- Deformation apparati: Patterson rigg, Drickhammer, Grigg's
- Belt (similar to piston cylinder but with conical/curved piston and matrix) - makes all synthetic diamond !
- Toroidal geometries
- Different multi-anvil geometries, in particular for use on synchrotron
- Shock waves
- 1 atm furnaces with/without controlled atmosphere
- low pressure (<< 1 bar) evaporation devices

caveats and inconveniences of experimental petrology
or
"Why aren't all problems solved ?"

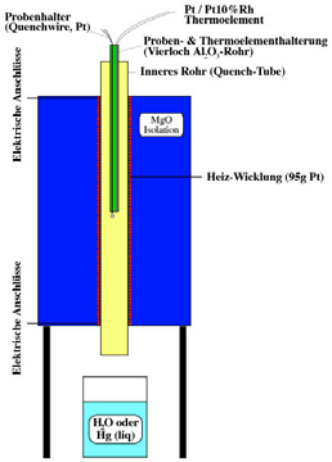
1) An experimental project must have
- a bulk composition to investigate
  - either a given bulk - phase relations
  - or a given reaction or process - how to investigate ?

what about volatile components (H2O, CO2), oxygen fugacity etc. ?

2) Which state does one want his composition in ?
oxide-mixes, glasses, gels, mixtures of seeds and gel/glass, minerals, H2O as a hydroxide, how CO2

Preparation: weighting, grinding, grinding and grinding

weighting and grinding + grinding



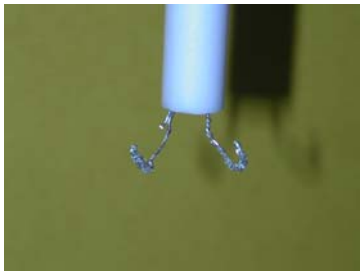
1-Athmosphärenofen
für Experimente bis 1600°C

mostly used to make glass
starting materials

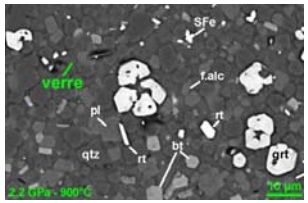
Geophysical Laboratory
Design (Bowen, 1906)

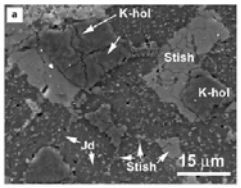
Probenhalter (Korund)
mit Quenchdraht (Pt)
und Thermoelement (Pt-Rh)

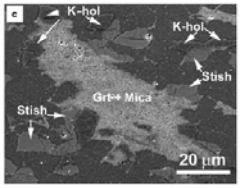
Quenchdraht (Pt-Draht)

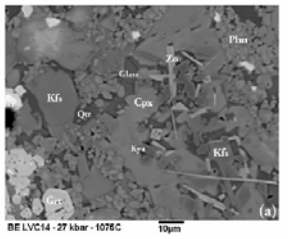


equilibrium textures - disequilibrium textures







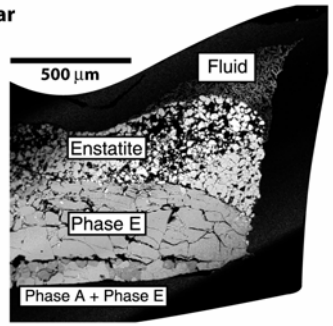


phase segregation

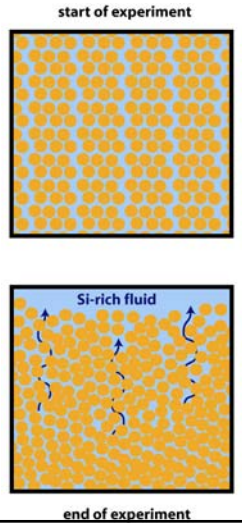
or: disaster strikes

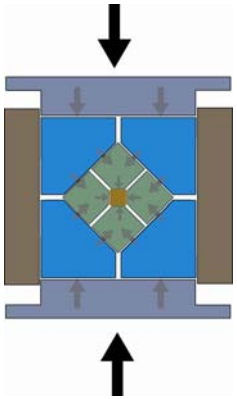
The starting point.....

P = 138 kbar
T = 950 °C

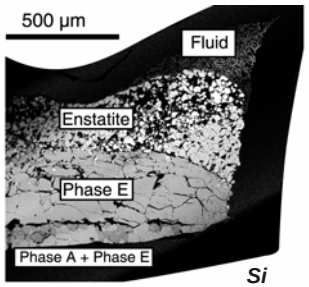


Si-distribution



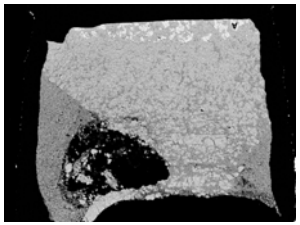


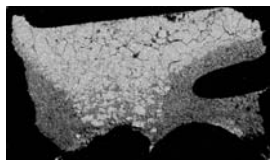
static



Si

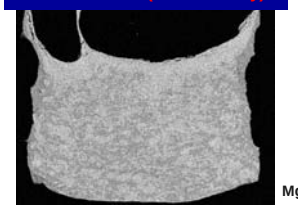
1 turn/ 15 minutes



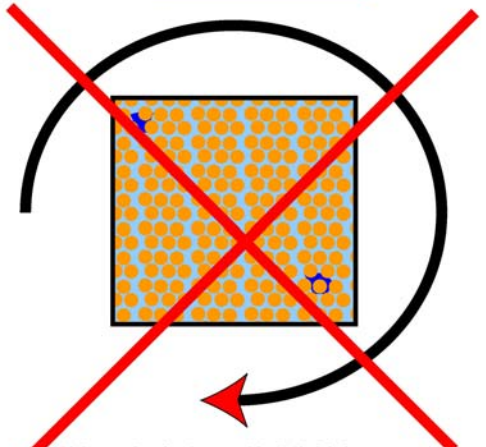


1 turn/ 1 minute

1 turn/ 30 sec (continuously)

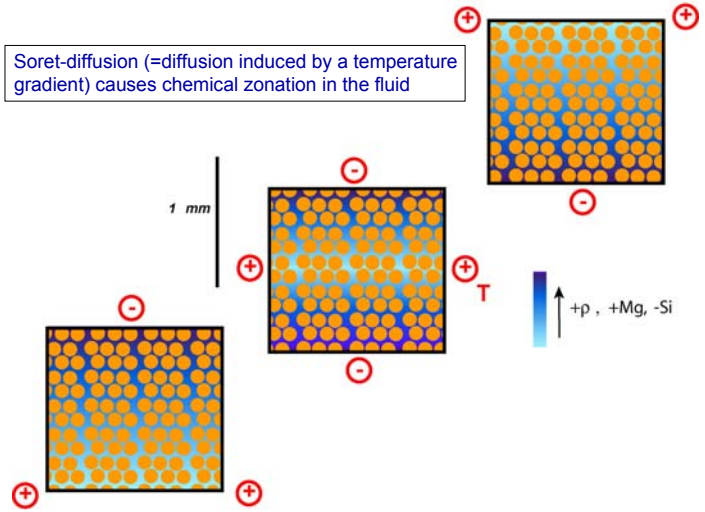


~~INERTIA~~

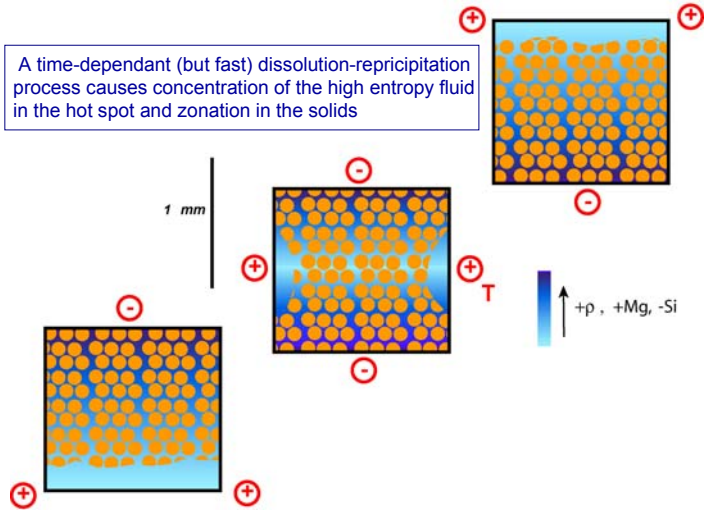


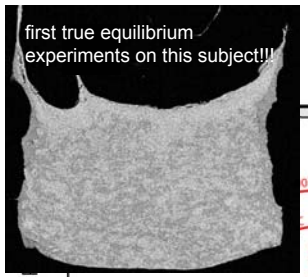
ca. 11 vol% hypothetical pure H_2O-fluid, i.e. >11 vol% real fluid

Soret-diffusion (=diffusion induced by a temperature gradient) causes chemical zonation in the fluid



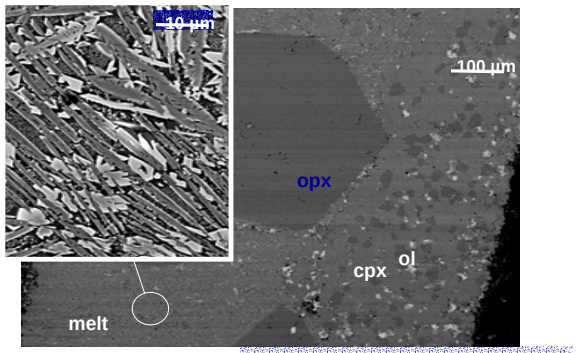
A time-dependant (but fast) dissolution-repricipitation process causes concentration of the high entropy fluid in the hot spot and zonation in the solids

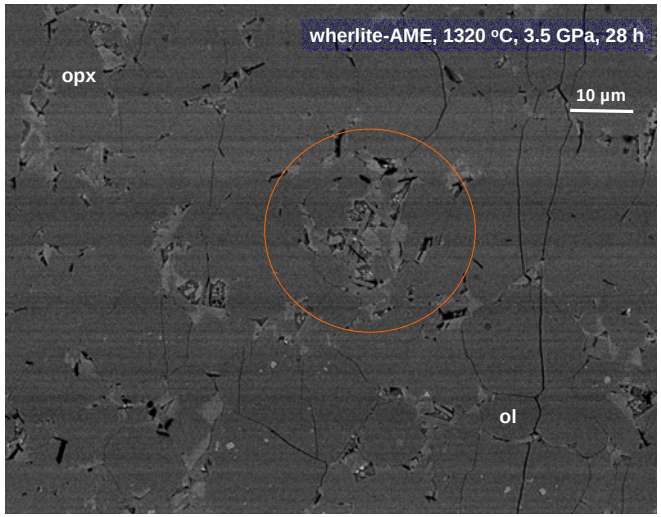




Major problem: melts and fluids are often not-quenchable

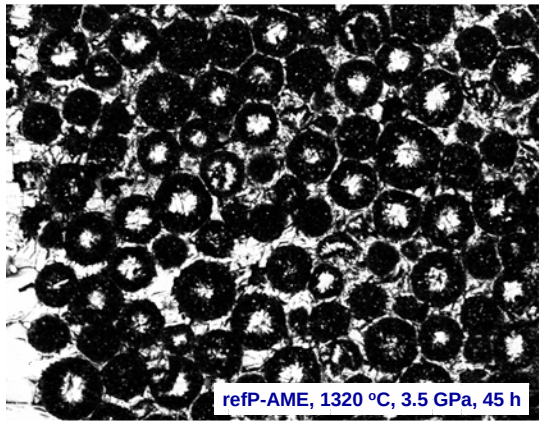
needs extraction techniques if amounts are small (which is the situation normally relevant in nature)





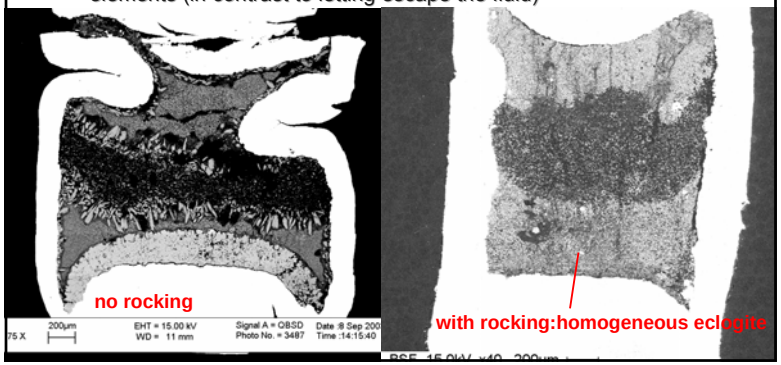
ol+opx+cpx, quench (mainly dp+phl) is only interstitial

melt in vitreous carbon sphere layer (30 µm Ø)

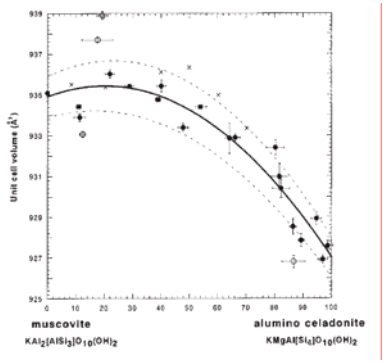


diamond-trap experiments - strategy:

- rigid diamonds keep “open space” which is then filled by supercritical melt during the experiment
- after quench and depressurization, capsule is frozen before opening and kept frozen during analyses
- we measure the composition of solidified mud/ ice, that retains all elements (in contrast to letting escape the fluid)

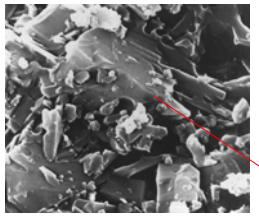


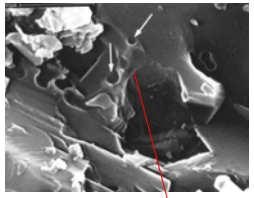
The making of ..... thermodynamic data

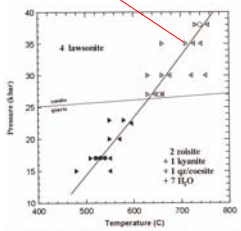


composition - volume

The making of ..... thermodynamic data







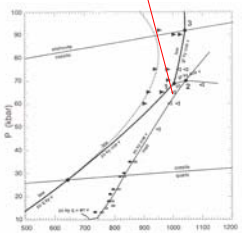


Fig. 4. Experimentally determined phase diagram for the lawsonite-out reactions in the SiO_2 + H_2O-saturated system

The making of ..... thermodynamic data

C_P-function, H_0, S_0, α, K, dK/dT

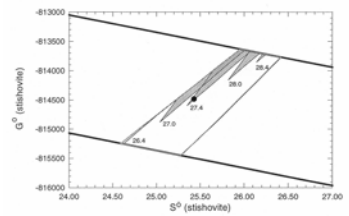


FIGURE 2. Feasible region of $S°_{(1298)}$ and $G°_{(1298)}$ of stishovite for different values of $(dK/dT)_P$ of kyanite (numbers at the lower left corner of shaded areas, in megapascals per kelvin). The thick lines represent limits on $G°_{stish}$ from calorimetry measurements of Akaogi et al. (1995). The area enclosed by the thick and thin lines represent feasible solutions if only calorimetric results and experiments on coesite = stishovite from Zhang et al. (1993, 1996) are considered. The shaded polygons represent feasible solutions for $G°_{stish}$-$S°_{stish}$ pairs calculated from calorimetric results, experiments on coesite = stishovite and experiments on kyanite = stishovite + corundum for feasible values of $(dK/dT)_P$ of kyanite.

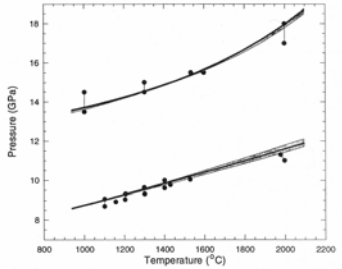
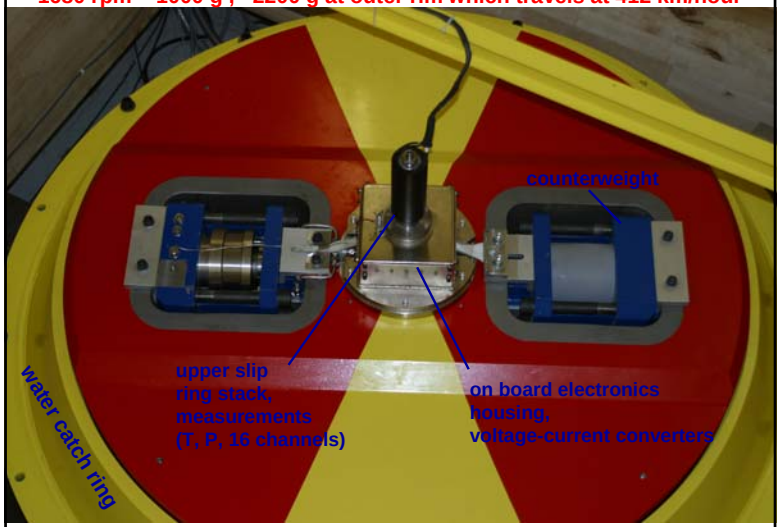


FIGURE 3. Experimental brackets on kyanite = stishovite + corundum from this study and on coesite = stishovite from Zhang et al. (1993, 1996). Thick lines represent the calculated reaction boundaries from our best fit (see Table 3). Shaded areas represent all possible P-T-loc of reaction boundaries as calculated from feasible thermodynamic properties (see text and Fig. 2).

1680 rpm = 1000 g , 2200 g at outer rim which travels at 412 km/hour



2890 rpm = 3000 g , 6700 g at outer rim which travels at 860 km/hour

The Centrifuging piston cylinder: 18 kbar – 1800 °C – 3000 *g*

(or the use and abuse of gravity for understanding physical and geochemical processes in magmas)

- crystal settling in magma chambers
- **viscosity of hydrous magmas**
- **melt extraction processes / compaction**
- **chemistry of immiscible liquids**
- migration of fluid inclusions in a gravity field

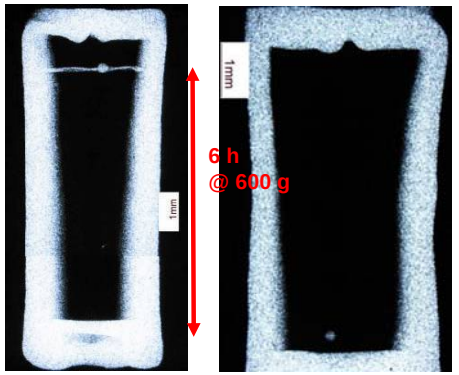
Viscosity Determination by Falling Sphere Method:

$$\text{viscosity} \sim \frac{\text{time} * g}{\text{sinking distance}}$$

PhD Paola Ardia

Pt-sphere

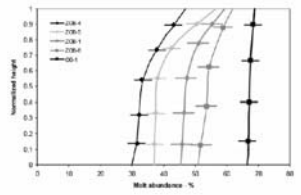
first viscosity determinations of hydrous silicic magmas In the magmatic temperature range

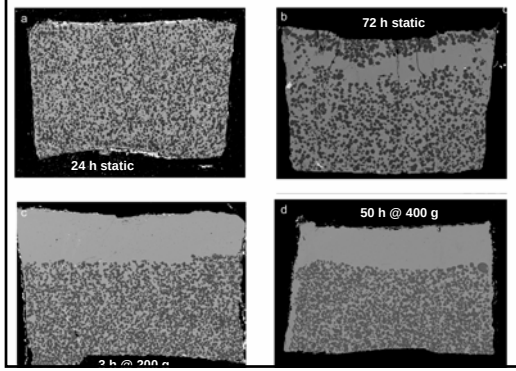


radiography in dental clinic Uni-ZH

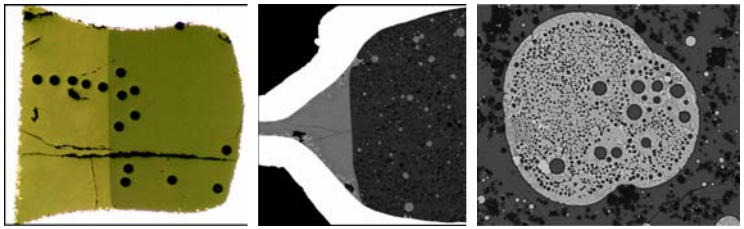
crystal settling in a magma chamber

system olivine+MORB @ 1 GPa, 1270 °C

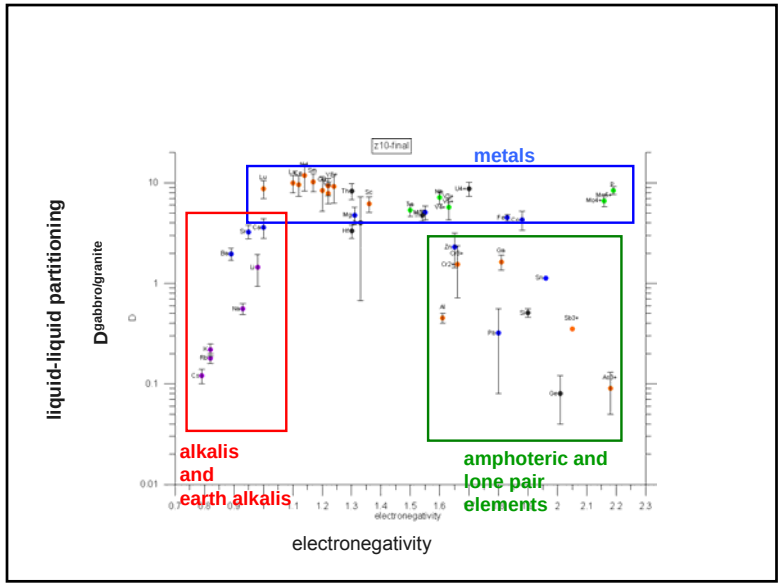




Centrifuged immiscible melts



→ **trace element partitioning as a function of melt composition**


liquid-liquid partitioning
Dgabbro/granite
z10-final
metals
alkalis
and
earth alkalis
amphoteric and
lone pair
elements
electronegativity