

# **From Mud to Shale: the Role of Microquartz Cementation\***

**Jens Jahren,<sup>1</sup> Brit Thyberg,<sup>1</sup> Øyvind Marcussen,<sup>1</sup> Turid Winje,<sup>1</sup> Knut Bjørlykke,<sup>1</sup> and Jan Inge Faleide<sup>1</sup>**

Search and Discovery Article #50206 (2009)

Posted September 23, 2009

\*Adapted from oral presentation at AAPG Annual Convention, June 7-10, 2009

<sup>1</sup>Department of Geosciences, University of Oslo, Oslo, Norway ([jens.jahren@geo.uio.no](mailto:jens.jahren@geo.uio.no))

## **Abstract**

The important mud to shale transformation is well known in sedimentary basins and is identified by changes in physical rock properties observed in well log velocity and density measurements. The transformation processes is, however, poorly understood. New discoveries of fine-grained micropore filling quartz cement found in Upper Cretaceous mudstones offshore Norway containing reactive silica releasing phases like opal-CT and smectite reveal the importance of microquartz cementation and its impact on petrophysical properties. Based on direct petrographic evidence of microquartz crystals with CL-responses indicating authigenic origin a microquartz cementation process that may explain how mudstones originally containing smectite stiffen to shale is proposed. The fine-grained quartz released in the clay mineral reaction smectite to illite within the micropores of the shale precipitate as 1-3  $\mu\text{m}$  sub-spherical discrete grains, short chains, and small clusters interpreted to be parts of larger interconnected microquartz networks and interlocking aggregates of several microquartz and authigenic clay (illite-smectite and illite) crystals. A significant increase in the velocity is recorded at a burial depth around 2500 m /80-85°C, reflecting formation of a pervasive microquartz cement network at this depth in smectite rich mudstones. The smectite to illite reaction will commence between 60 and 70°C in mudstones, indicating that the temperature (80-85°C) where the velocity increase takes place reflects formation of a critical amount of interlocking complexes of interconnected microquartz networks and aggregates stiffening and strengthening the mudstones. The sluggish nature of the illitization process in mudstones reflected by the wide temperature range (60-100°C) that smectite is found in mudstones result in a progressive formation of microquartz crystals. This will, in most mudstones with less smectite than the ones studied herein, most probably only result in a slow continuous progressive stiffening of the mudstone framework. This may explain why this important cementing process in smectite containing mudstones has been overlooked in the past.

## **Selected References**

Bjørlykke, K., 1998, Clay mineral diagenesis in sedimentary basins; a key to the prediction of rock properties, examples from the North Sea Basin: *Clay Minerals*, v. 33/1, p. 15-34.

Boles, J.R., and S.G. Frank, 1979, Clay diagenesis in Wilcox Sandstones of southwest Texas: Implications of smectite diagenesis on sandstone cementation: *Journal of Sedimentary Petrology*, v. 49, p. 55-70.

Foster, W.R., and H.C. Custard, 1980, Smectite-illite transformation--role in generating and maintaining geopressure (abstract): *AAPG Bulletin*, v. 64, p. 708.

Götze, J., M. Plötze, and D. Habermann, 2001, Origin, spectral characteristics and practical applications of the cathodoluminescence in quartz- a review: *Mineralogy and Petrology*, v. 71, p. 225-250.

Hower, J., E.V. Eslinger, M.E. Hower, and E.A. Perry, 1976, Mechanism of burial metamorphism of argillaceous sediment: Mineralogical and chemical evidence: *GSA Bulletin*, v. 87, p. 725–737.

Marcussen, Ø., B.I. Thyberg, C. Peltonen, J. Jahren, K. Bjørlykke, and J.I. Faleide, 2009, Physical properties of Cenozoic mudstones from the northern North Sea: Impact of clay mineralogy on compaction trends, *AAPG Bulletin*, v. 93, p. 127-150.

Moe, P., 2006, Velocity-depth trends. A comparison between laboratory and log derived measurements for wells from the Norwegian Shelf: Master's thesis: University of Oslo, Oslo, 155 p.

Mondol, N.H., K. Bjørlykke, J. Jahren, and K. Høeg, 2007, Experimental mechanical compaction of clay mineral aggregates - changes in physical properties of mudstones during burial: *Marine and Petroleum Geology*, v. 24, p. 289-311.

Müller, Axel, 2000, Cathodoluminescence and characterisation of defect structures in quartz with applications to the study of granitic rocks: Ph.D. Dissertation, Universität zu Göttingen, Dresden, Göttingen, 229 p.

Peltonen, C., O. Marcussen, K. Bjørlykke, and J. Jahren, 2008a, Mineralogical control on mudstone compaction; A study of Late Cretaceous to Early Tertiary mudstones of the Voring and More Basins, Norwegian Sea: *Petroleum Geoscience*, v. 14/2, p. 127-138.

Peltonen, C.B., O. Marcussen, K. Bjørlykke, and J. Jahren, 2008b, Clay mineral diagenesis of Late Cretaceous to early Tertiary mudstones from the Norwegian Sea: The effects of smectite to illite transformation and quartz cementation on rock properties: *Marine and Petroleum Geology*, v. 26/6, p. 887-898.

Small, J.S., 1994, Fluid composition, mineralogy and morphological changes associated with the smectite-to-illite reaction: An experimental investigation of the effect of organic acid anions: *Clay Minerals*, v. 29, p. 539-554.



UNIVERSITY  
OF OSLO

# From Mud to Shale: The Role of Microquartz Cementation

Jahren Jens, Brit Thyberg, Øyvind Marcussen, Turid Winje,  
Knut Bjørlykke and Jan Inge Faleide

Department of Geosciences, University of Oslo



Department of Geosciences



# Outline

- Introduction
- Methods/materials
- Direct petrographic evidence of (fine-grained) quartz in mudstones
- Microquartz cementation/rock stiffening
- Summary





## Diagenesis

Mud



shale

### Why?

- Mechanical compaction?
- Early cementation - carbonates, etc?
- Chemical compaction – clay minerals?, quartz cement?





# Mud to shale

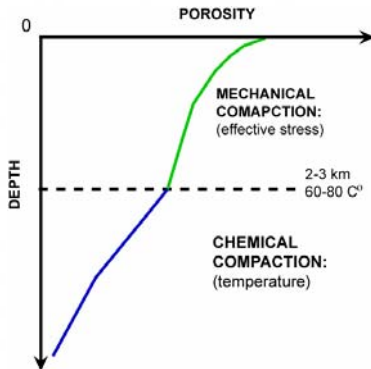
- The processes of transforming soft mud to hard shales have not been studied extensively.
- Compaction of soft mud to hard shale involves both mechanical and chemical processes changing the rock physical properties significantly.
- Experimental mechanical compaction (Mondol et al., 2007) document gradual changes in petrophysical properties as a function of increased stress.
- These changes are easily identified in well logs as velocity and density increase.



## Controlling factors on porosity reduction in both sandstones and shales



UNIVERSITY  
OF OSLO



Mechanical compaction is a function of increased stress.

Chemical compaction is a function of thermodynamics and kinetics and is independent of the confining stress (e.g., Bjørlykke, 1998).



Department of Geosciences



## Redistribution of silica during chemical compaction of mudstones

- Closed system diagenesis (diffusion, convection and advection unimportant)
- Temperature control (thermodynamics and kinetics)
- Cement sources
  - biogenic amorphous silica,
  - smectite to illite or chlorite reactions,
  - kaolinite to illite reaction,
  - clay-induced dissolution and/or pressure dissolution of detrital silt particles,
  - other sources?

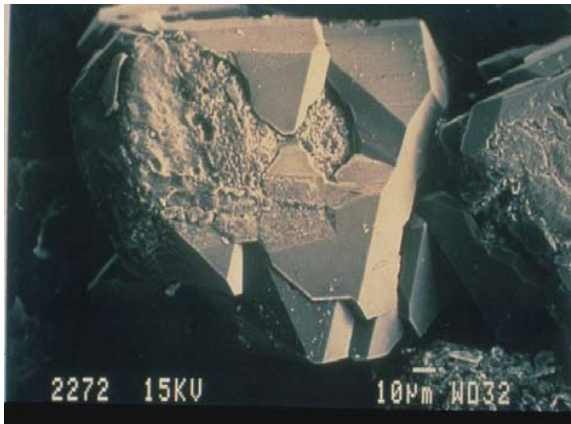




# What is quartz cement



UNIVERSITY  
OF OSLO



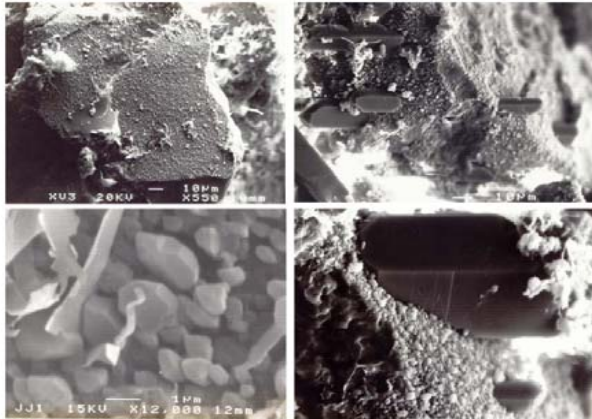
Department of Geosciences

Quartz cement in  
sandstone from  
Haltenbanken, KB

# Quartz cement – Microquartz in sandstones at about 4500m burial depth



UNIVERSITY  
OF OSLO



From Jahren and Ramm 2000

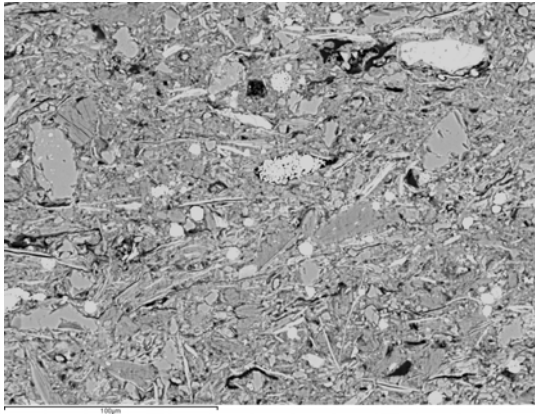


Department of Geosciences

What about quartz cement ?? in mudstones – here  
buried to more than 4000m from Haltenbanken. From  
Mo, 2006



UNIVERSITY  
OF OSLO



Department of Geosciences

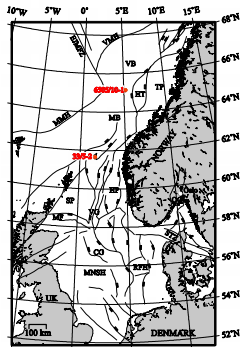


## Quartz from the smectite to illite reaction

- Chemical compaction processes in mudstones include the clay mineral reaction smectite to illite through a mixed layer illite-smectite (IS) taking place between about 60-100°C
- It has been known for a long time that smectite releases significant amounts of silica when it is dissolved and replaced by illite.
- Quartz cement sourced by dissolved smectite has a extremely fine-grained nature (e.g., Hower et al., 1976; Foster and Custard, 1980; Small, 1994).
- Despite indirect evidence (mostly from XRD – “clay-sized quartz”) direct petrographic evidence of finely dispersed quartz cement in mudstones is sparse from previous studies of mudstones.
- This could be because many authors have concluded that silica released as a result of clay-mineral reactions in mudstones and shales is transported and precipitated as quartz cement in associated sandstones (e.g., Hower et al., 1976)



# Late Cretaceous mudstone sequences



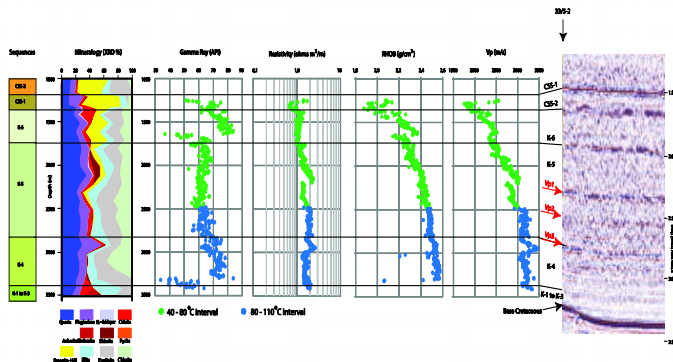
In the present study (**red wells** on the map) we have undertaken detailed high resolution petrographic examination of Upper Cretaceous mudstones at depth/temperatures corresponding to the smectite-illite transformation



# Mineralogical and Petrophysical well log data – well 33/5-2



UNIVERSITY  
OF OSLO

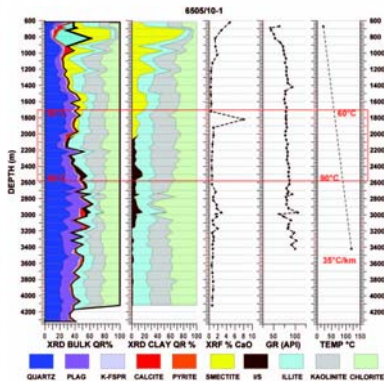


Department of Geosciences

# Bulk and clay mineralogy – well 6505/10-1



UNIVERSITY  
OF OSLO



Department of Geosciences

Peltonen et al, 2006

## Velocity and density

The study target is the interval close to and below a sharp velocity increase close to 2500 m (80/85°C) associated with a more gradual increase in density.

These changes in physical properties take place in the depth/temperature interval where smectite is transformed to illite/smectite and illite.

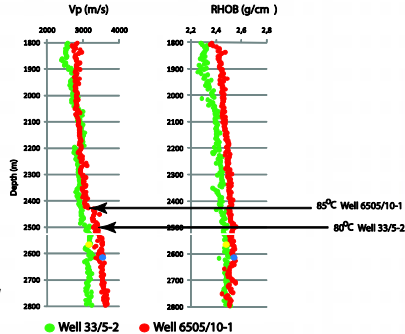
The interval associated with the velocity contrast is relatively homogeneous with respect to clay-mineral content and has low carbonate content, initiating a high-resolution SEM/CL investigation with respect to quartz cement.



Department of Geosciences



UNIVERSITY  
OF OSLO







## Methods – finding fine-grained micropore filling quartz cement

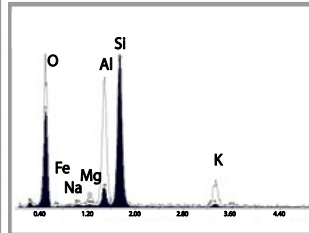
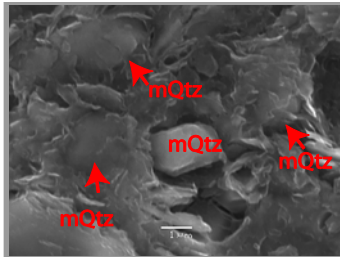
- Selected samples in the sample depth interval 2370 – 2870 m corresponding to 80-90°C (assuming a temperature gradient of 30-35°C/km (Evans et al., 2003)) from the North Sea well 33/5-2 as well as 6 samples below 2520 m (Peltonen et al., 2008) from the revisited Norwegian Sea well 6505/10-1 were studied.
- Thin sections from cuttings
- Dried material from the clay fraction ( $< 2 \mu\text{m}$ ) mixed with ethanol was disintegrated in an ultrasonic bath for a few minutes. The finest fraction of the disintegrated clay fraction material was taken out by a pipette and transferred to a polished carbon-slab, dried and carbon-coated.
- Authigenic quartz cement found in these samples was investigated petrographically by Scanning electron microscopy (SEM) linked with energy dispersive X-ray spectroscopy (EDS) and equipped with monochromatic wavelength dispersive cathode luminescence system (Gatan MonoCL).



# Petrographic evidence of microquartz cement

Microquartz crystals embedded in  
the fine-grained illitized clay matrix

The characteristic EDS spectrum  
of the chemistry of the illitized clay  
matrix (unfilled) and the  
microquartz crystals (filled).



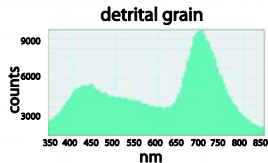
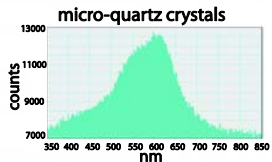
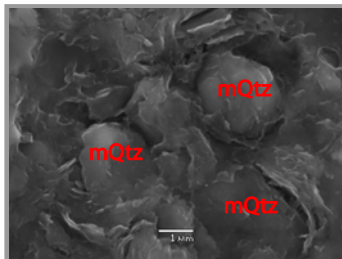
# Petrographic evidence of microquartz cement



UNIVERSITY  
OF OSLO

Microquartz crystals embedded in the fine-grained clay matrix

Most common CL-response of the microquartz crystals indicating an authigenic origin



Department of Geosciences

## Petrographic evidence of quartz cement from CL



UNIVERSITY  
OF OSLO

The CL-emission of low temperature authigenic quartz may be due to intrinsic effects related to a combination of lattice defects of oxygen vacancies around 580 nm and/or from non-bridging oxygen hole centers around 620 nm (e.g., Muller, 2000; Gotze et al., 2001).

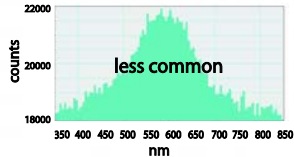
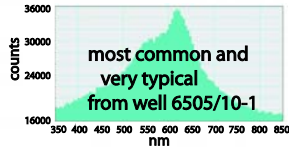
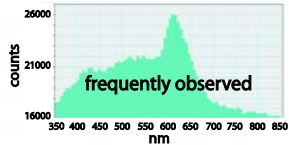
These intrinsic effects are giving rise to the typical Gaussian shaped CL-emission spectra.

The 580 nm peak is frequently missing or has less intensity, giving rise to a more irregular shape of the CL-response.

Reasons: complete or partial healing of the oxygen vacation lattice defects during the electron exposure, or may represent the CL-properties of microquartz crystals sourced from smectite dissolution



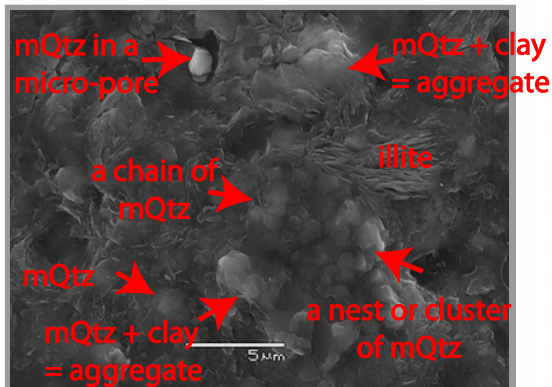
Department of Geosciences



# Microquartz cement embedded in the fine-grained matrix - Well 33/5-2

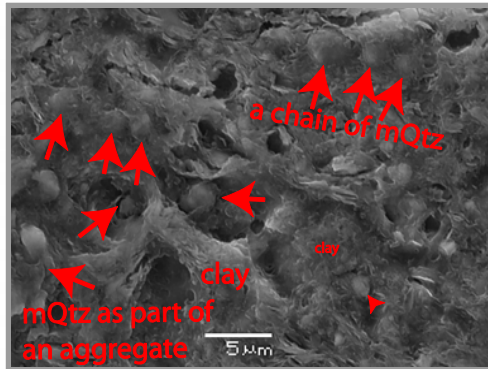


UNIVERSITY  
OF OSLO



Department of Geosciences

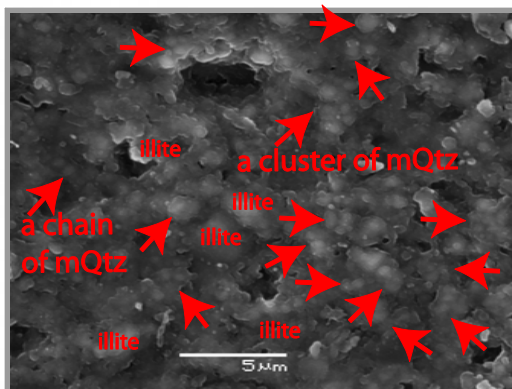
## Microquartz embedded in the fine-grained clay matrix - well 33/5-2



# Microquartz embedded in the fine-grained clay matrix - well 6505/10-1



UNIVERSITY  
OF OSLO

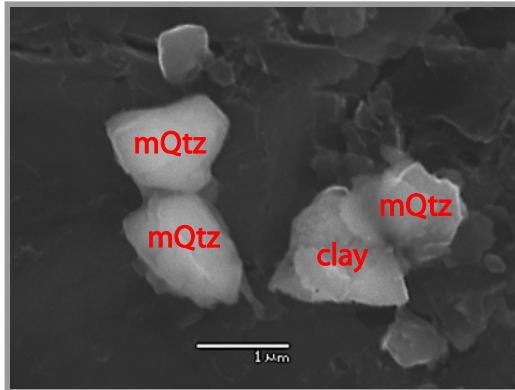


Department of Geosciences

## Aggregates (ultrasonic treated) – interlocking cementing relationships



UNIVERSITY  
OF OSLO



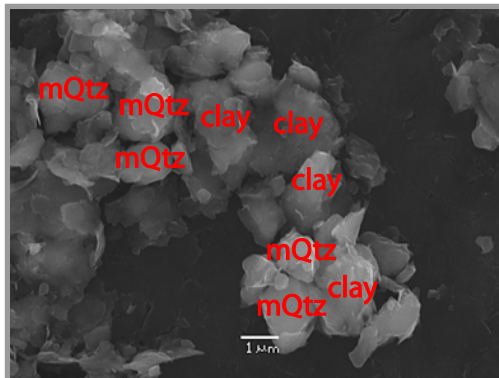
Department of Geosciences



# Aggregates (ultrasonic treated) – interlocking cementing relationships



UNIVERSITY  
OF OSLO



Department of Geosciences

**Presenter's Notes:** The silica released produces a subtle interconnected microquartz network interlocked with aggregates of microquartz and authigenic clay crystals.

# The microquartz cementation process



UNIVERSITY  
OF OSLO

- The authigenic microquartz crystals are interpreted to be sourced from the local release of Si from the dissolution of smectite and precipitation of illite.
- The simplified reaction equation below (taken from Boles and Franks, 1979) illustrate the process:
- $\text{Smectite} + \text{K}^+ = \text{Illite} + \text{Silica (aq)} + \text{H}_2\text{O}$
- Dissolution of smectite and precipitation of illite requires:
  - That silica is removed for the reaction to proceed
  - A potassium source (K-feldspar, etc)
- The smectite to illite reaction results in the release of significant amounts of silica.
- The excess silica (aq) will quickly precipitate as microquartz near and together with the neoformed illite-smectite and illite crystals.



Department of Geosciences

# The microquartz cementation process



UNIVERSITY  
OF OSLO

- In a relatively low temperature closed system like the one studied herein the combination of slow quartz growth and high silica saturation creates conditions favoring continuous nucleation and growth of many microquartz crystals within the micropores of the mudstones.
- The microquartz crystals nucleate on suitable substrates like earlier formed microquartz crystals and on illite (not c-faces) leading to formation of subtle microquartz networks and aggregates.
- The microquartz cementing process stiffens the mudstone framework and affects the petrophysical properties of the mudstones
- Formation of aggregates and connected small networks forming longer range stiffening microquartz cement complexes can explain the sharp increase in  $V_p$  seen around 2500 m (80-85°C) in some smectite-rich mudstones.
- The instant change in velocity ( $V_p$ ) well inside the chemical compaction regime may be related to a critical degree of connectivity between the microquartz crystals and the aggregate formation (as nodes?). Hence a "critical mass" of interconnected microquartz and clay crystals may be needed to produce such a significant increase in velocity.



Department of Geosciences

# Summary



UNIVERSITY  
OF OSLO

- This study of Upper Cretaceous mudstones from the northern North Sea present direct petrographic evidence of the existence of fine-grained (1-3  $\mu\text{m}$ ) micropore-filling quartz crystals in drill-bit cuttings found around  $\geq 2500\text{m}/80\text{-}85^\circ\text{C}$ .
- Cathodoluminescence (CL) identifies the microquartz crystals as authigenic.
- The microquartz is most probably sourced from silica released during the smectite to illite dissolution-precipitation reaction.
- The silica released during the smectite to illite reaction is not exported to adjacent sandstones but precipitated locally as microquartz cement.
- The distribution of microquartz, degree of stiffness and thereby petrophysical properties of the shale can be related to the primary content/distribution of the precursor such as smectite (and opal-A, etc).
- This is ultimately linked to provenance, facies and depositional processes determining the seismic properties/facies of mudstones and shales in sedimentary basins

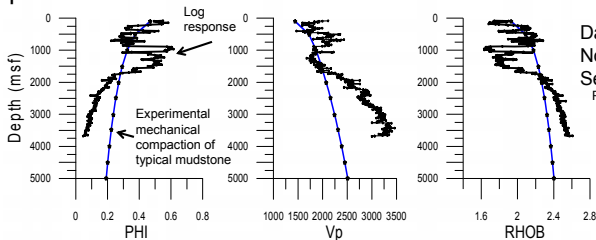


Department of Geosciences

## Chemical compaction – petrophysical log response



UNIVERSITY  
OF OSLO



Data from the  
Norwegian  
Sea  
Peltonen et al 2009

Chemical compaction of mudrocks with reactive clays (smectite, kaolinite) and a source of potassium (k-feldspar) is a dissolution–precipitation process dissolving matrix and filling pore spaces, resulting in compaction and stiffening of the rock. In mudrocks without reactive phases, flattening and redistribution of detrital quartz will take place cementing the rock.



Department of Geosciences The work was funded by NRC/Petromaks and StatoilHydro