M 10 Methods of Conservation and Restoration of Built Heritage

Microscopy to evaluate microstructural defects and consolidants in porous mineral materials

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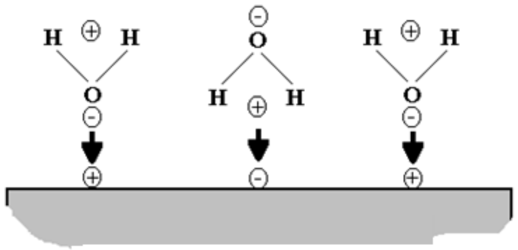
Heritage Train

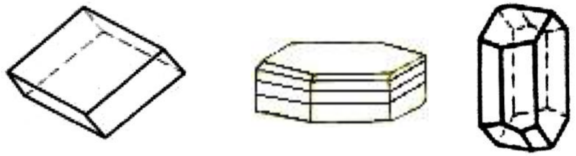
Funded by the European Union

Basic properties of minerals:

- solid
- non-metallic, non-organic
- usually polar → affinity to polar liquids (e.g. water)



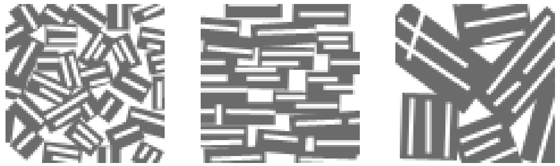
- either covalent or ionic bonds, or combinations of both → varying hardness and chemical stability
- usually crystalline → often anisotropic



Mineral materials = composites of minerals

Natural stone – mortar – ceramics - ….

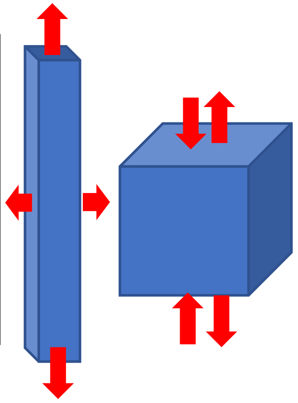
- consist of numerous single mineral grains, usually of different nature, shape and size
- are often porous, i.e. they have empty spaces between the mineral grains
- are characterized by their mineral content and their specific textures



Mineral (geo-)materials possess many important „**macroproperties**“ …..
…. they can be measured for test bodies (cubes, prisms, cylinders) by means of standardized protocols

- Chemical & mineralogical bulk composition
- Petrophysical properties
    - Porosity and poresize distribution
    - Water absorption coefficient
    - Water vapour permeability
    - Drying rate
- Mechanical properties
    - Compression strength
    - Tensile & bending tensile strength
    - Modulus of elasticity
    - Hydric & hygric dilatation
    - Thermal dilitation
    - Surface hardness
    - Drill restistance
    - Peeling test
    - Ultrasound transmission (on drill cores)
    - etc.

Values refer to the bulk of the test bodies, usually across several centimeters - they assume homogeneous materials accross the diameter

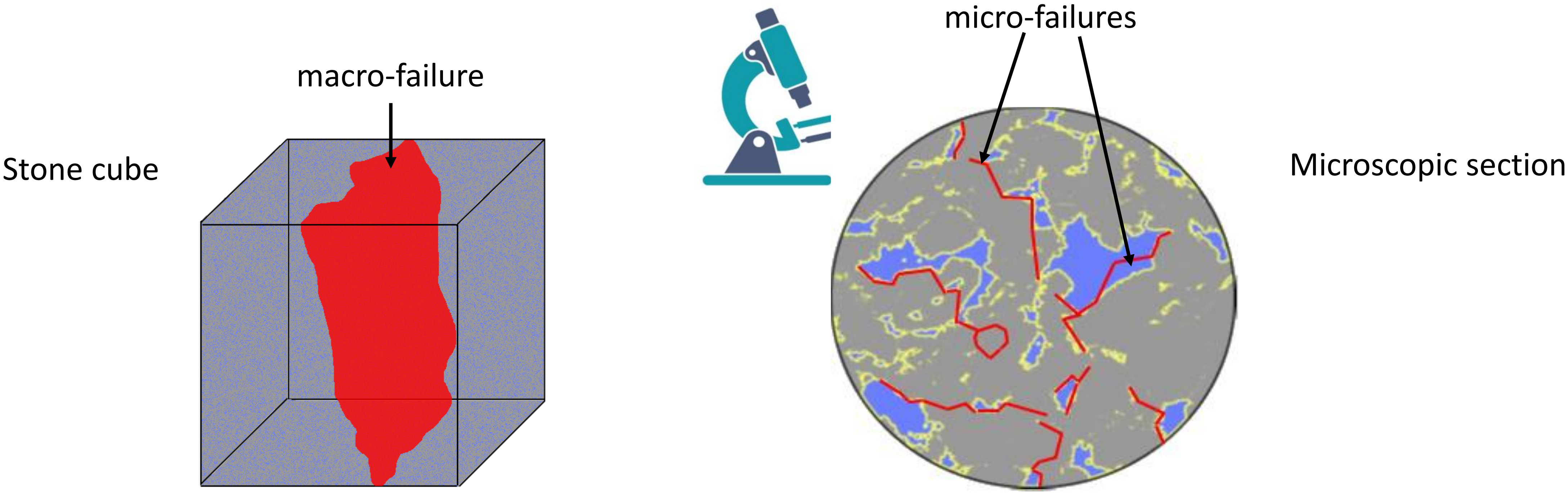


Values describe depth profiles – they consider material gradients

BUT: Mineral (geo-)materials are **never homogeneous** as they consist of different minerals and empty spaces (pores) arranged in a specific way (microstructure, texture)

→ their macroproperties are related to the microstructure
→ Studying the microstructure is useful to understand macroproperties and predict failure

AND: Weathering often forms **steep gradients of properties** which require high enough spatial resolution of investigative methods – the same holds for **conservation treatments**



Weathering of rocks in the nature



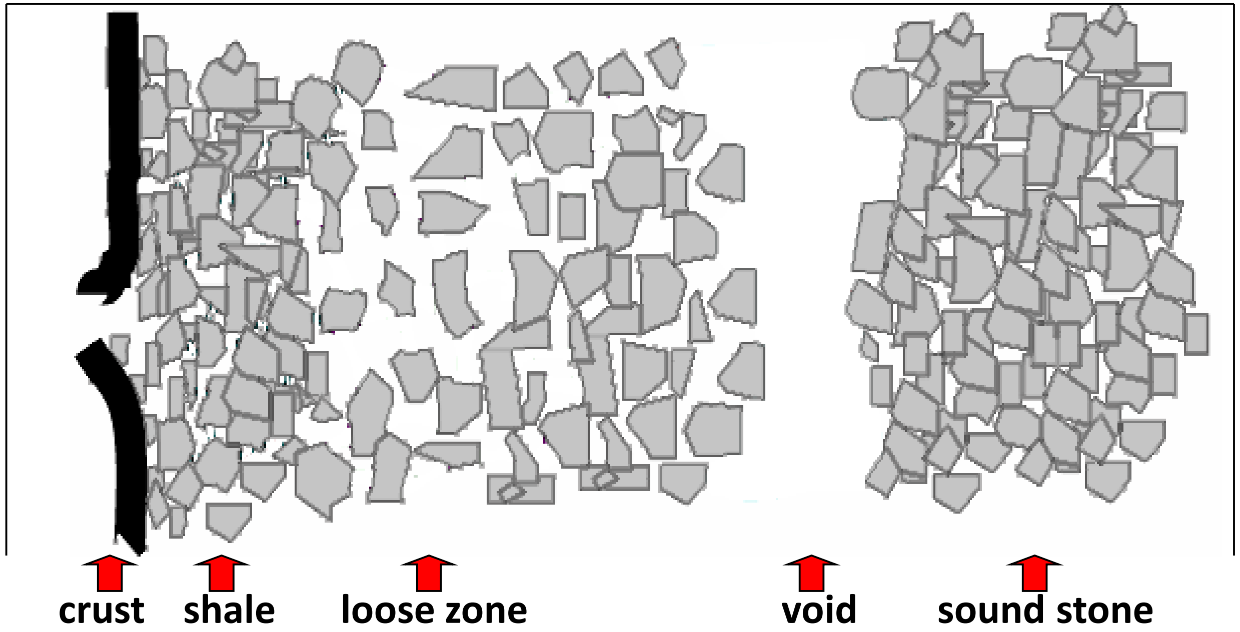



Weathering of stone in architectural and sculptural context

What is specific?

Tolerance: losses of small volumes or losses of a few tens of millimeters from the surface may result in a total loss of artistic or historic value !

„Short distance effects“: due to the small dimensions of most artefacts, opposite sides of exposed objects are subjected to very different conditions and develop different material properties (e.g. Dissolution/precipitation horizons are close to each other)



typical weathering profile of porous carbonate stones

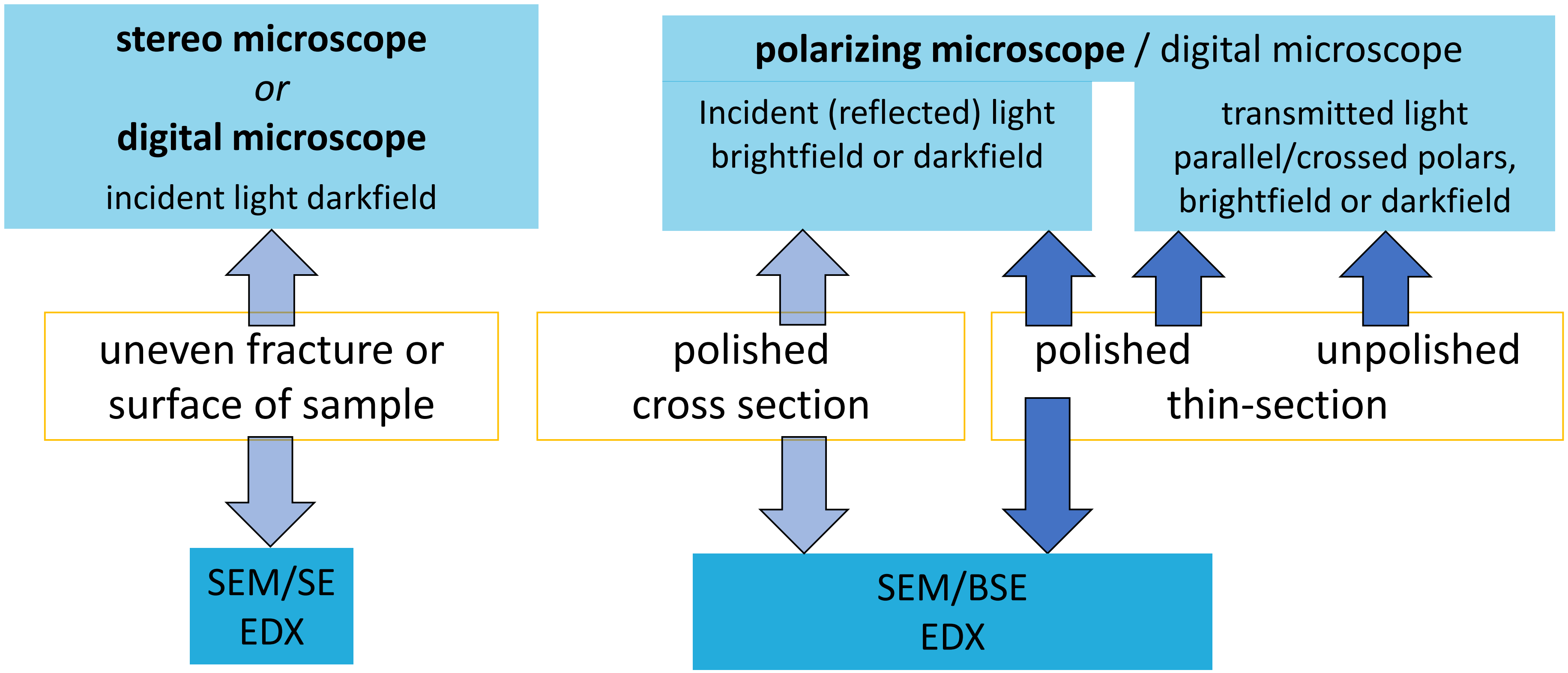
ICOMOS- Illustrated glossary on stone deterioration patterns

https://www.icomos.org/publications/monuments_and_sites/15/pdf/Monuments_and_Sites_15_ISCS_Glossary_Stone.pdf

Main categories	detailed decay patterns
Crack and deformation	fracture – star crack – hair crack – craquele – splitting
Detachment	blistering – bursting – delamination – disintegration– fragmentation – peeling – scaling
Features induced by material loss	alveolisation – erosion – mechanical damage
Discoloration and deposit	crust – discolouration – efflorescence – encrustation
Biological colonisation	

Most of these patterns can be observed under the microscope!

Ways of sample preparation and possible methods of microscopy

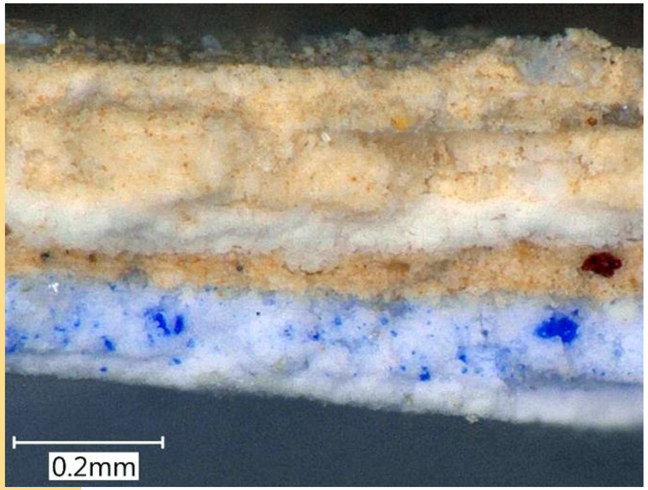


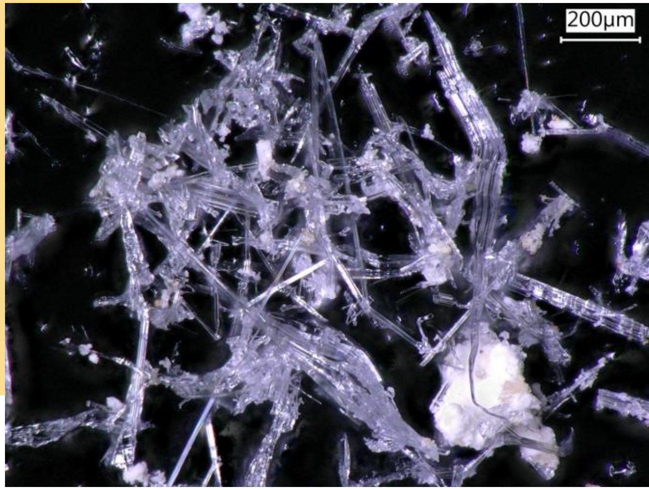
Abbreviations:
SEM=scanning electron microscope (SE=secondary electron detector, BSE=back-scattered electron detector); EDX=energy-dispersive X-ray spectrocopy

stereo microscopy
5 x to ca. 100 x

digital microscopy
ca. 30 x to 1000 x

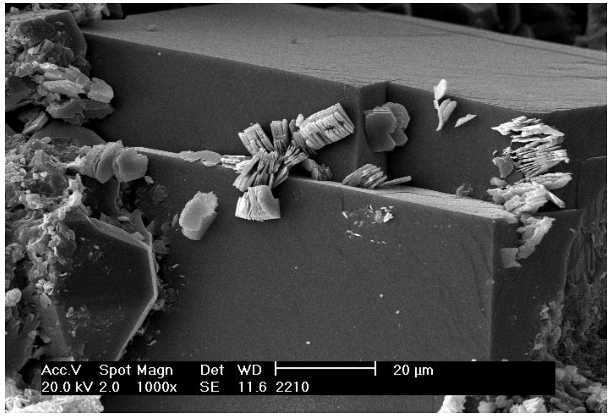
incident/transmitted light, (incl, darkfield) and polarizing facilities

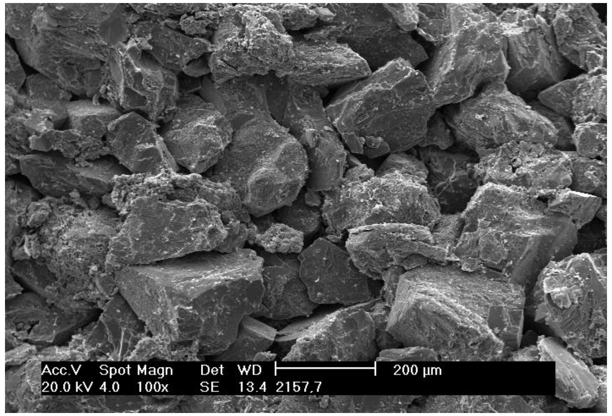




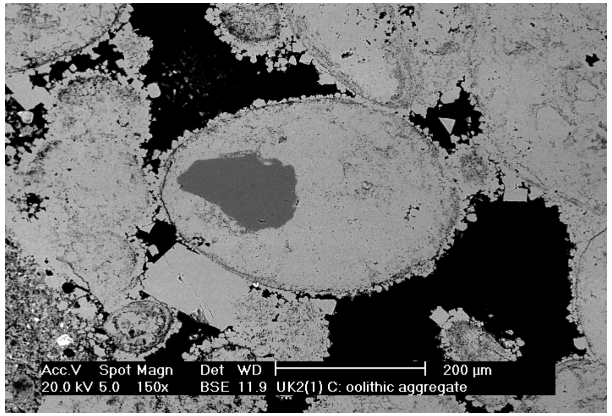
Scanning electron microscopy (SEM)

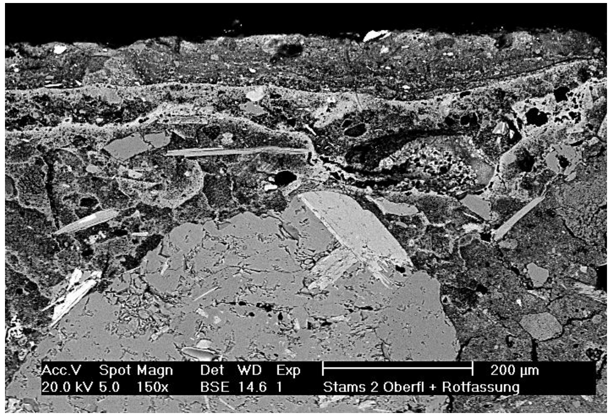
- Secondary electrons (SE): uneven surfaces with high depth of focus, up to about 10.000x magnification)



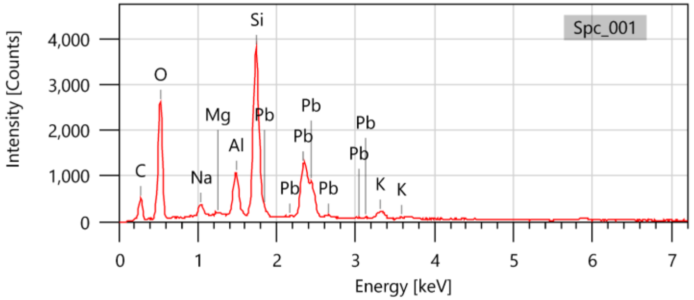


- Back-scattered electrons (BSE): mainly polished surfaces





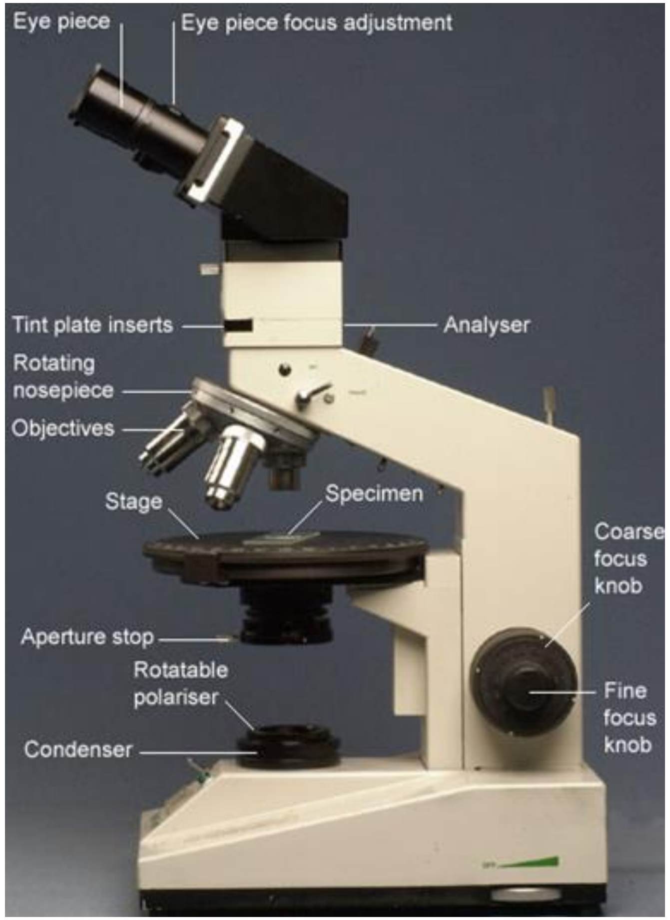
- X-ray spectroscopy (EDX): Spots or defined areas of the sample



Polarizing microscopy (PLM) of thin-sections

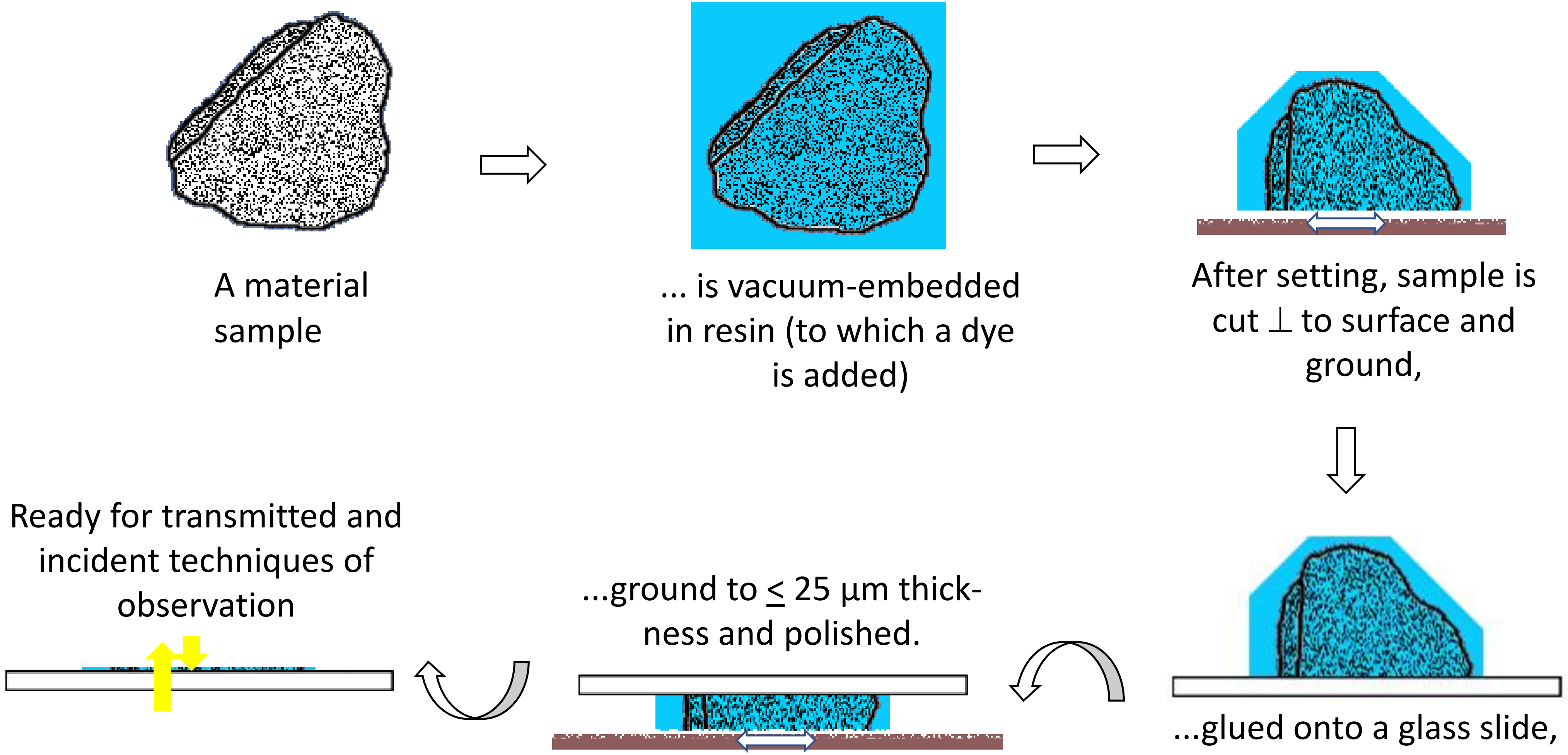
- <u>identification of minerals</u>, *and*
- study of the <u>fabric and texture</u>

Comment: Particularly the texture is linked to the physico-mechanical properties. It thus contains the most important pieces of information…
*• to understand **material properties***
*• to interpret **decay phenomena***
*• to predict **material failure***
*• to understand the **working technique** (especially for mortars)*



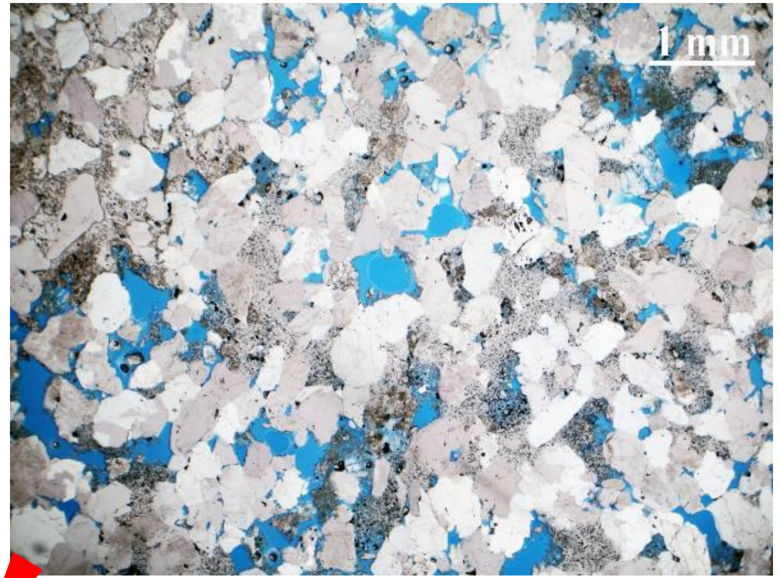
PETROGRAPHIC THIN-SECTION

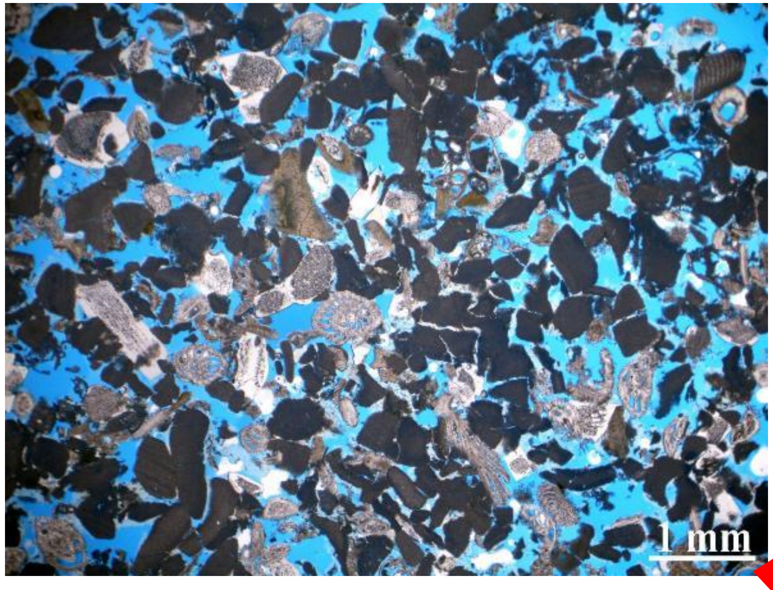
Thin-section: ca. 25 µm = 0,025 mm thick slice which is usually observed in transmitted light (most components are transparent at 25 µm) --- when polished, it can also be observed in incident light or e.g. by electron-optical techniques



Typical textures of clastic sedimentary rocks (e.g. sandstones)

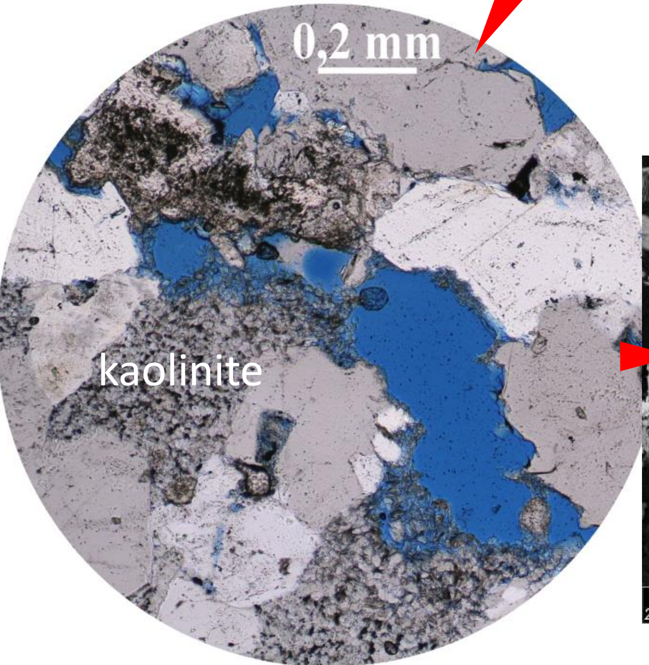
quartz arenite

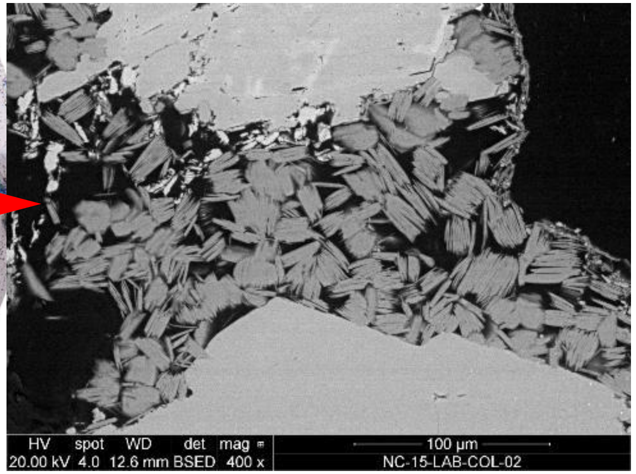


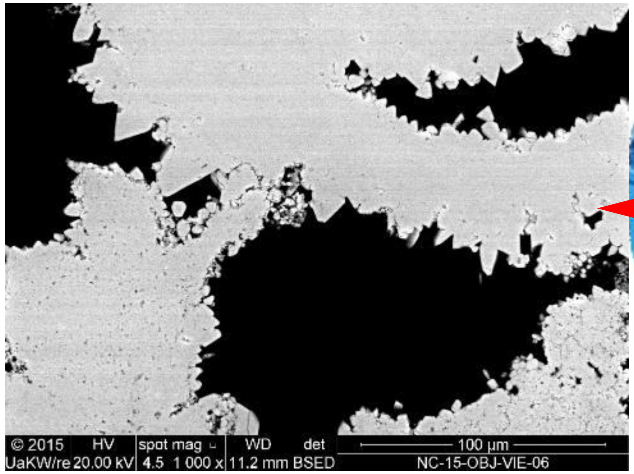


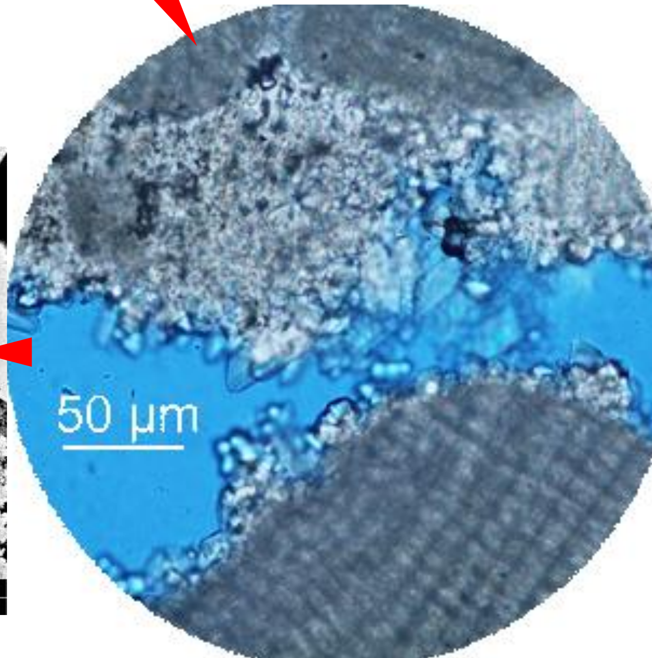
(bio)calcarenite

The binder minerals are of great importance for technical properties and durability



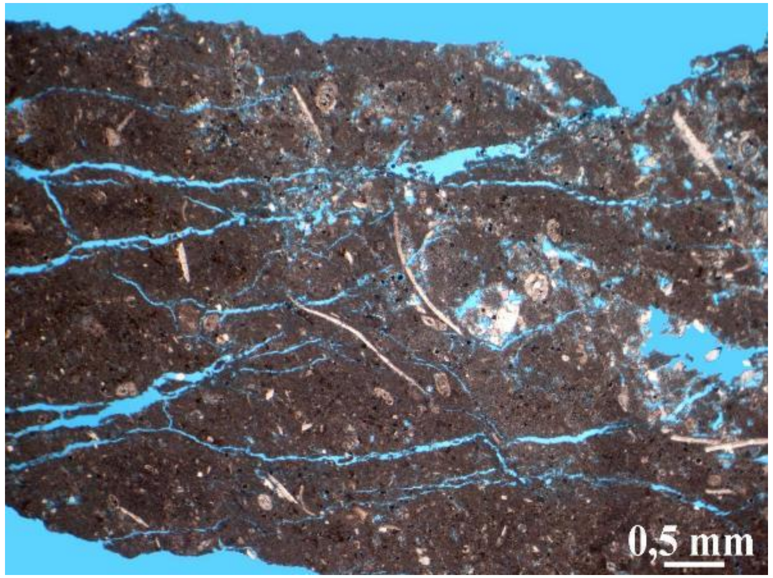


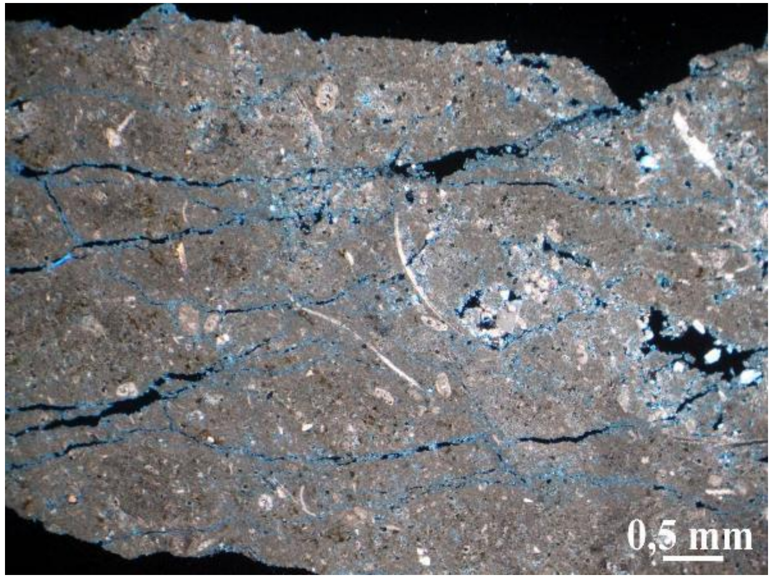




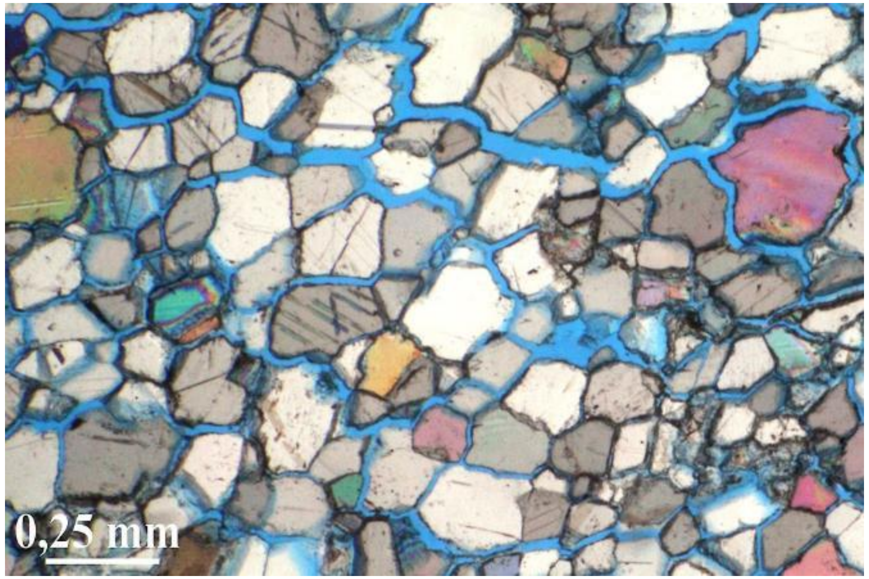
microstructural defects caused by weathering

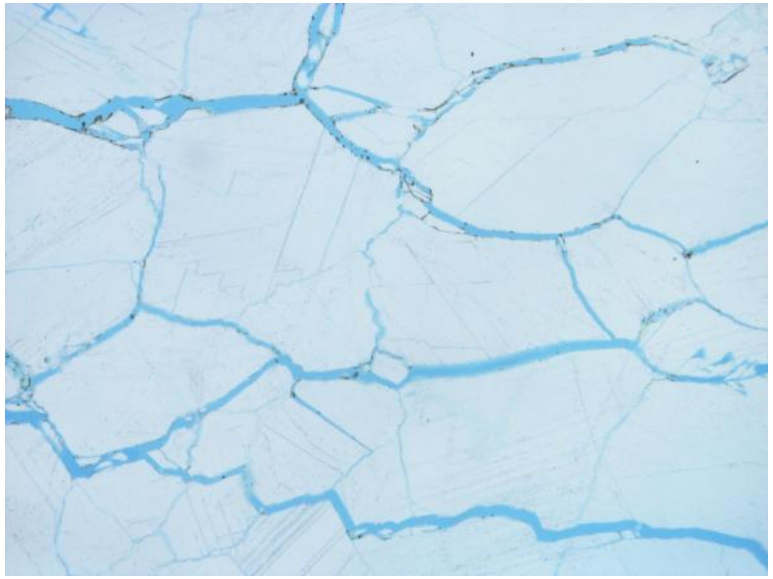
Delamination
(slate)





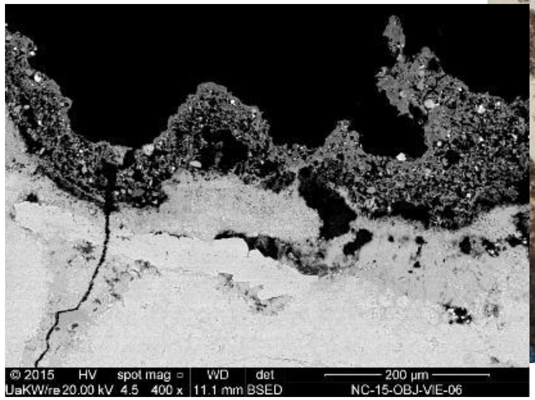
Granular
disintegration
(marble)

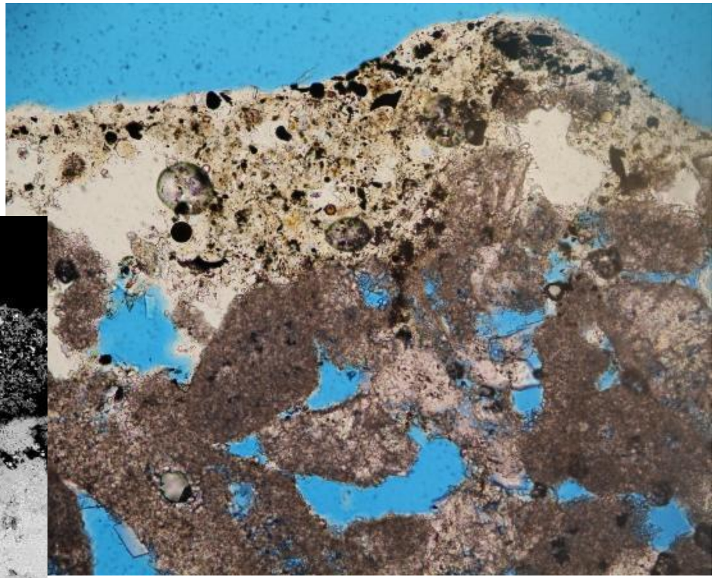


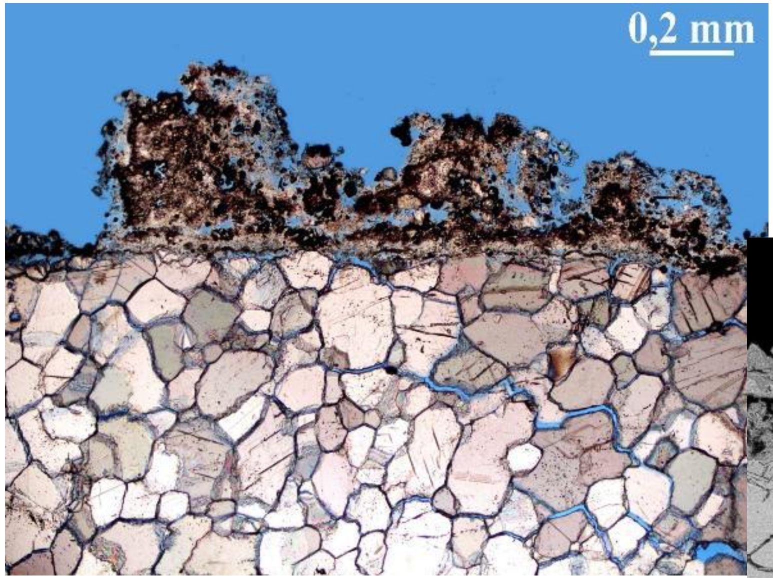


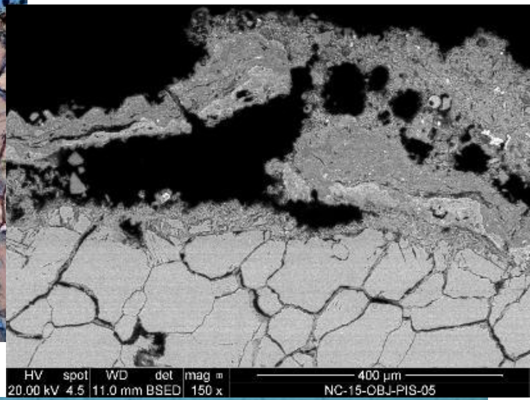
The texture of weathered stone as a key to understand microstructural defects

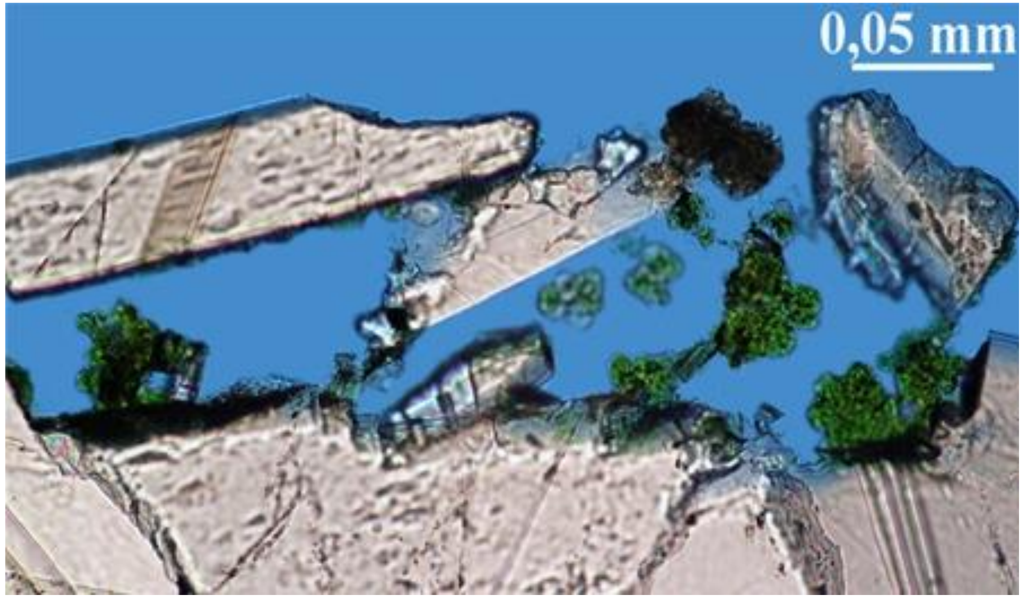
Chemical weathering – black crusts (gypsum)



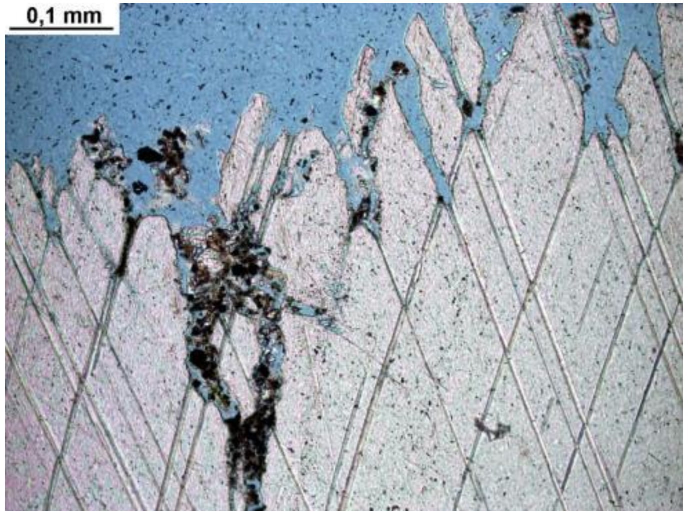




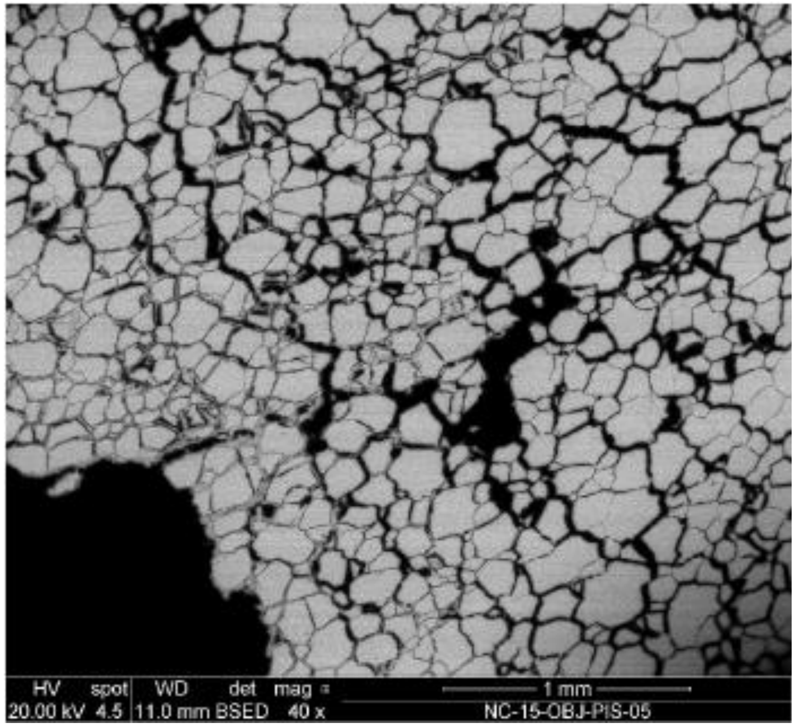


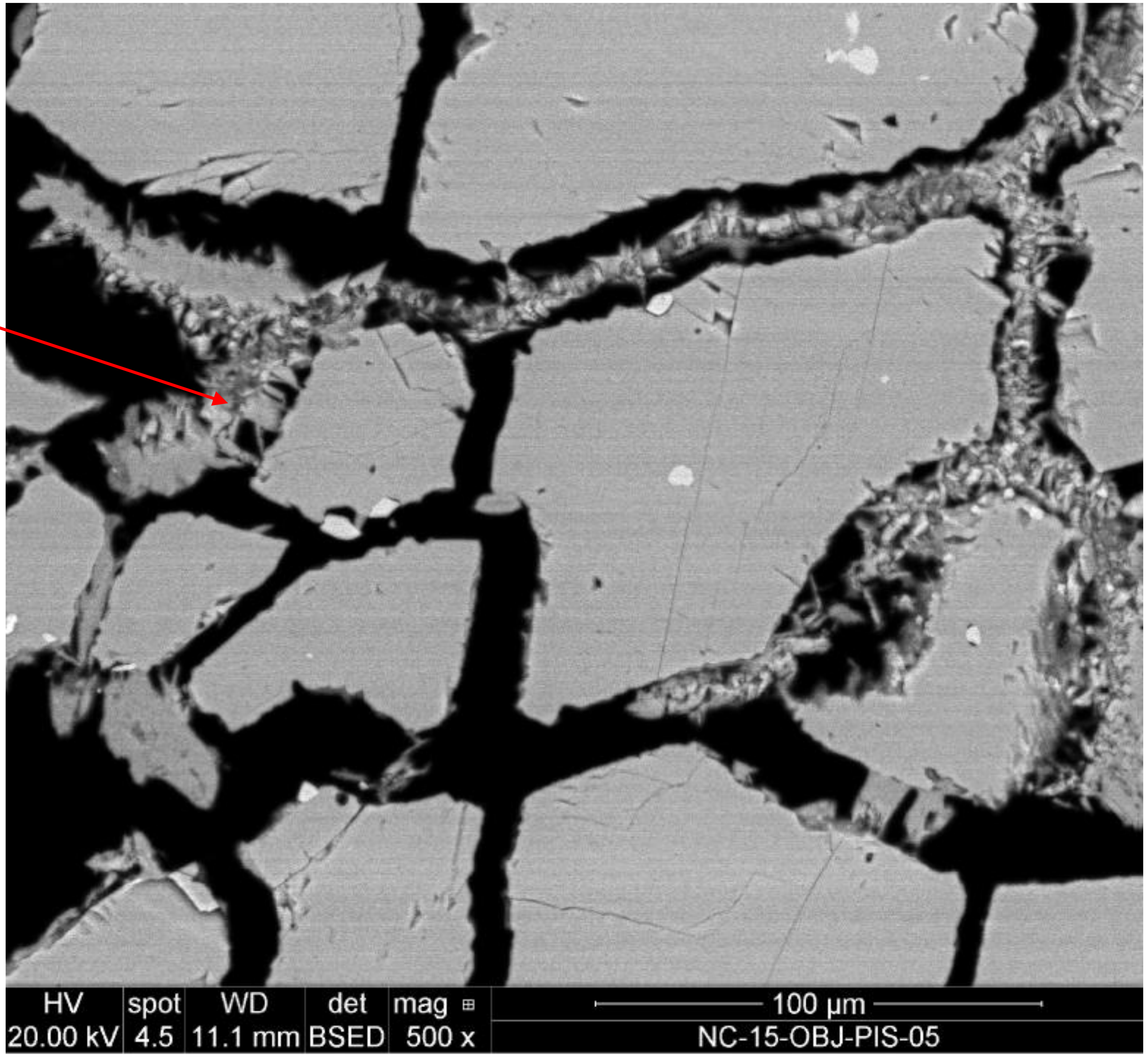


Microbiology +/- etching



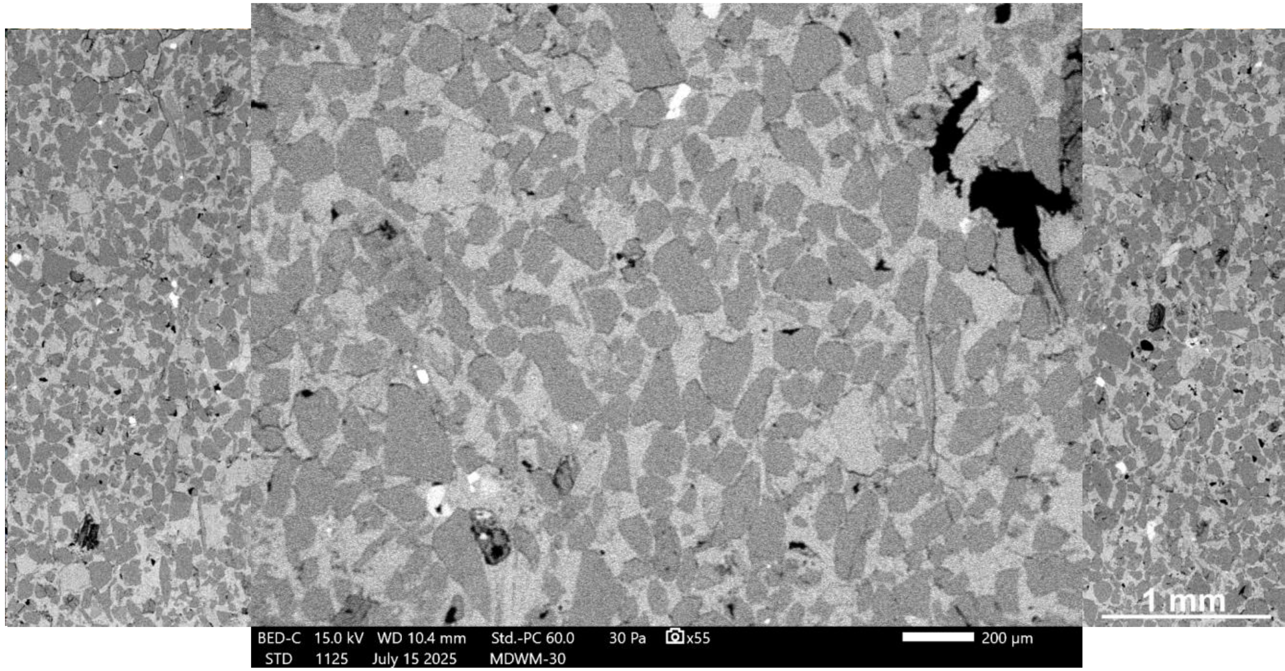
Marble with strong granular disintergration & gypsum as secondary product



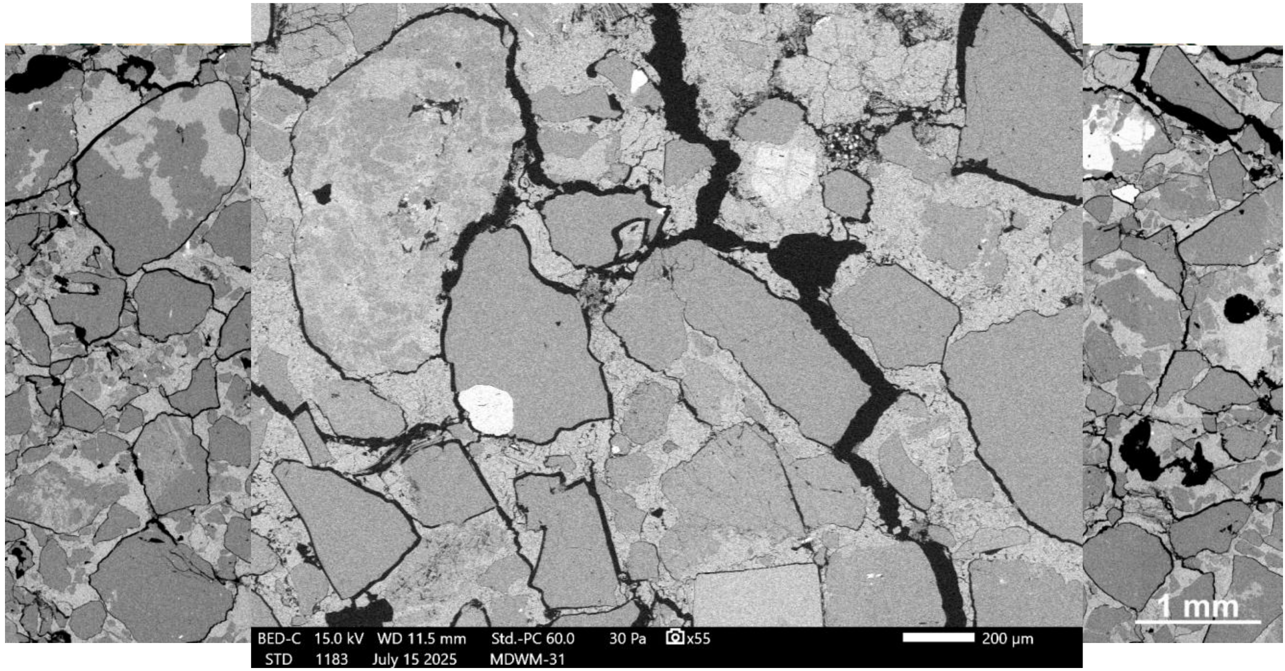


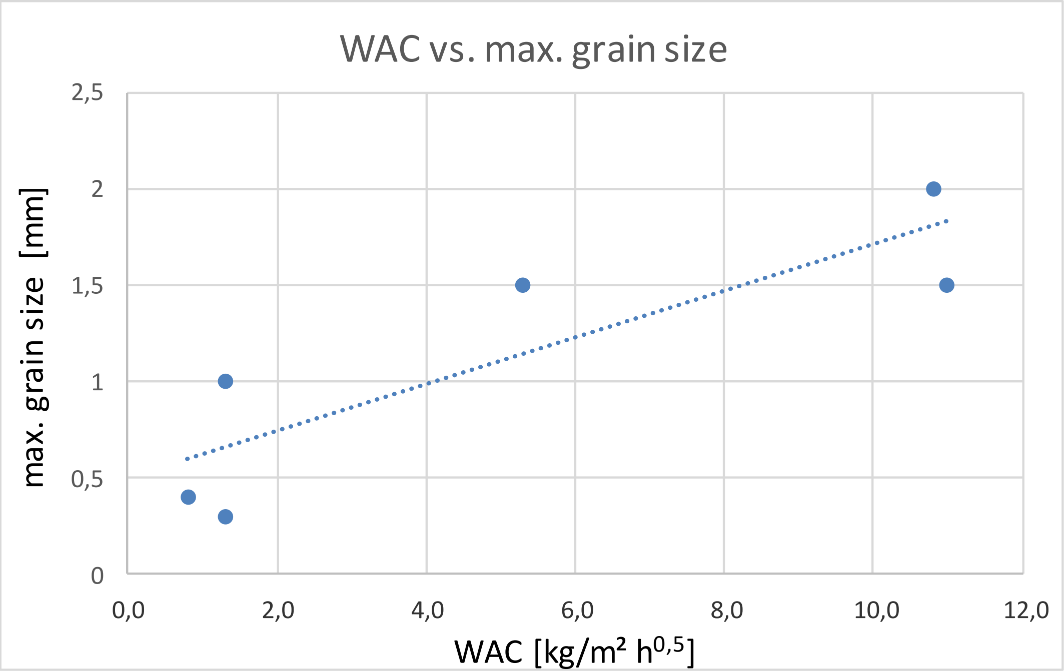
Sandstone of varying grain size and WAC

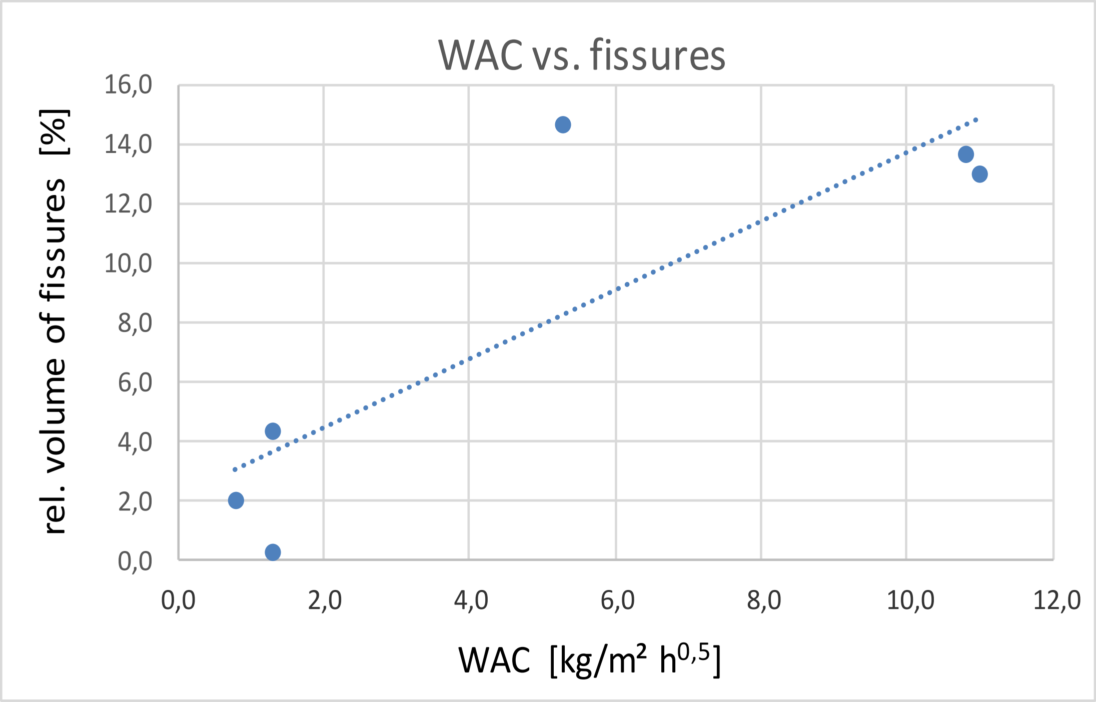
WAC = 1,3 kg/m² $h^{0,5}$



WAC = 10,8 kg/m² $h^{0,5}$





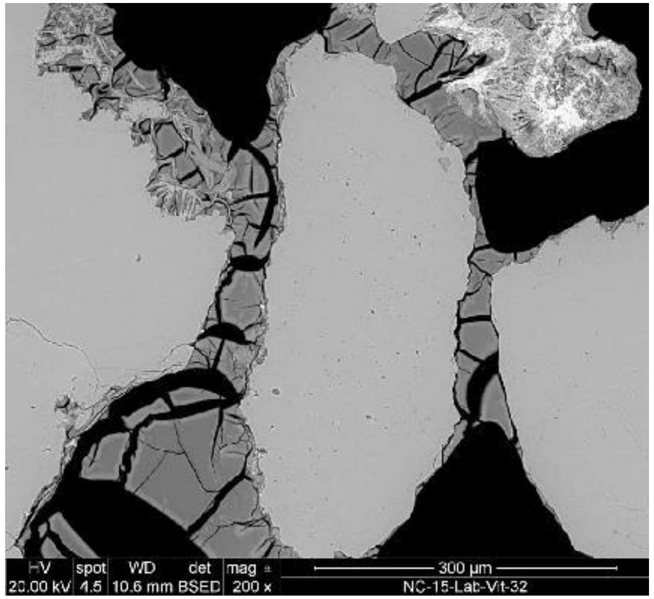


Through point counting and digital image granulometry, quantitative data can be achieved for microscopic images

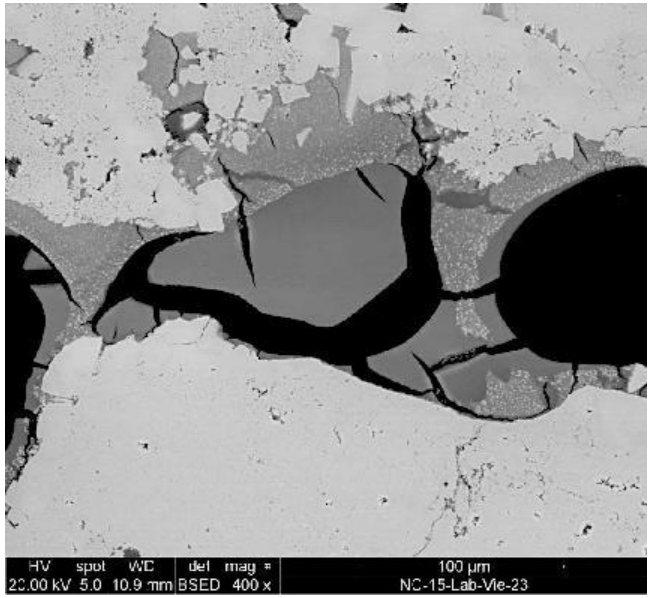
The distribution and microstructure of consolidants in pore systems

The depth of penetration by a consolidant solution yields no sufficient information about the distribution of its solid component after reaction/drying

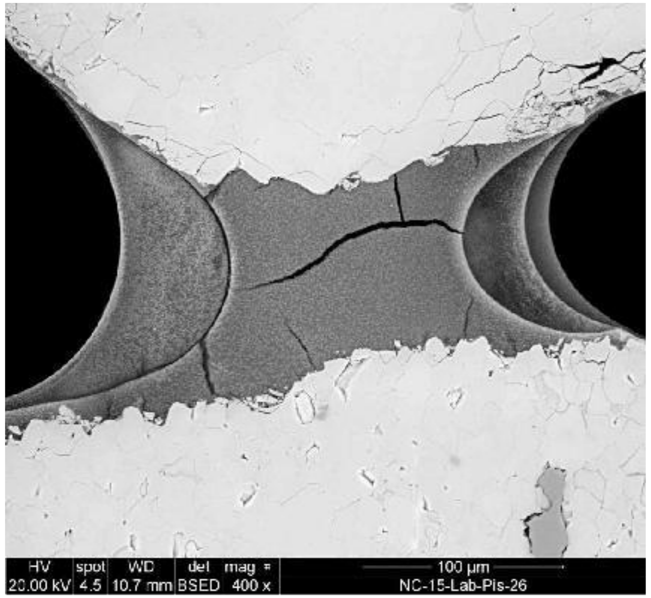
- The majority of consolidants for weathered stone and mortar are of silicate nature – either they belong to the family of TEOS – **ethyl silicates,** or **silica sols** -> both form **silica gel**.
- Other products successfully used are from the group of **nano lime** -> they form **Ca-carbonate.**
- Amongst the key factors of success are (1) the **depth** to which a consolidant is deposited, and (2) the capacity to **bridge voids** of varying dimensions.
- The above parameters can be best assessed on **polished (thin) sections by SEM** – PLM has no high enough resolution.



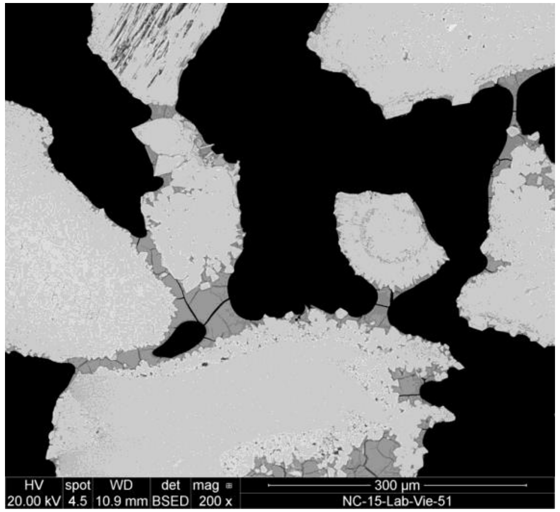
Silica gel in sandstone pores, after a combined treatment with TEOS and silica sol

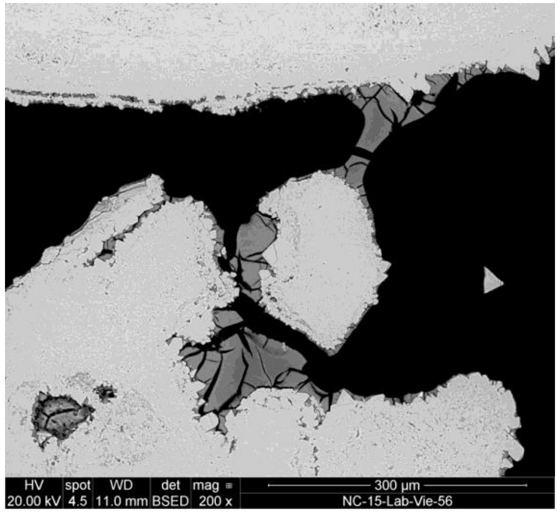


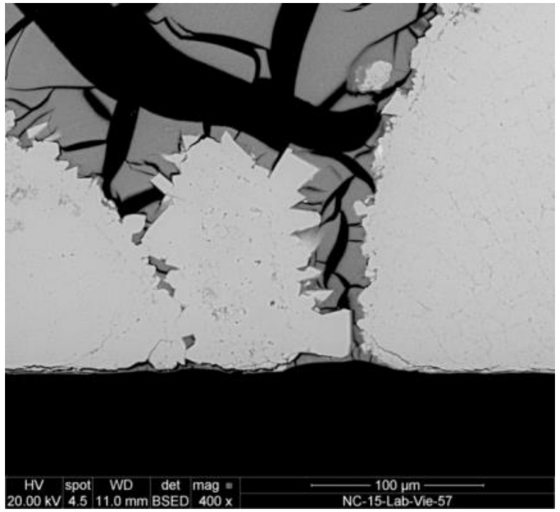
Silica gel with Ca-carbonate in lime-stone pores, after a combined treatment with TEOS and nanolime



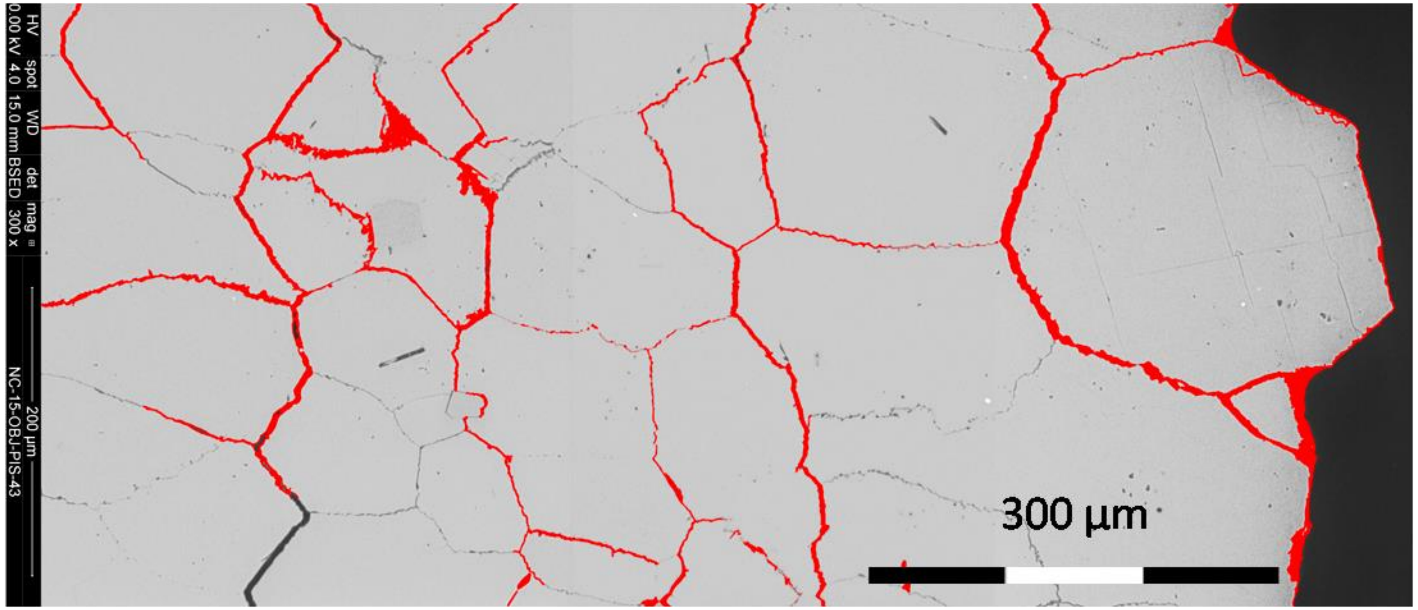
Ca-carbonate in limestone pores, after a 3-fold treatment with nanolime







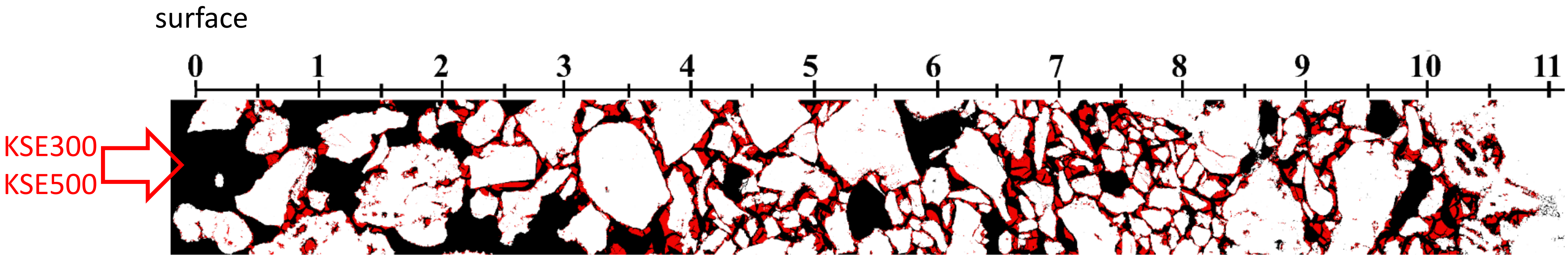
Silica gel – shrinkage and bridging



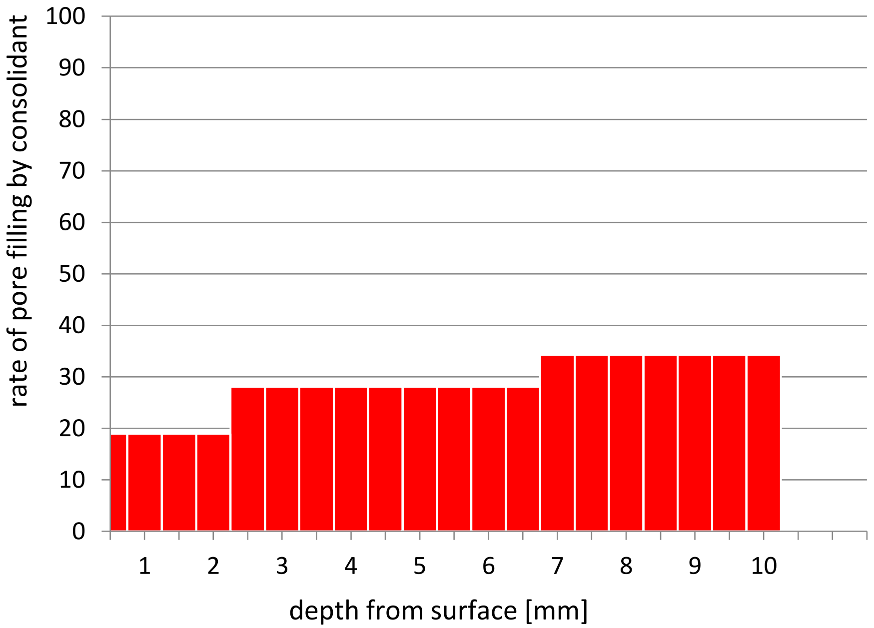
Consolidation of intercrystalline fissures by use of TEOS
Pseudo-colour editing

In-depth distribution of consolidant in laboratory test

SEM/BSE images – semiautomated stitching – pseudocolour editing



Rate of pore filling with silica gel



Ghaffari et al (2012) Methods of polarizing microscopy and SEM to assess the performance of nano-lime consolidants in porous solids

Mascha et al (2020) Assessment of the spatial distribution of consolidating solids in the pore space by SEM

Mascha et al (2020) Silicates for the conservation of stone – nano silica vs. ethyl silicate

Mascha, *Visualization of consolidants in the pore space of mineral materials in conservation*, PhD-thesis, Vienna 2021

Thank you for your attention !

Questions and comments: johannes.weber@uni-ak.ac.at