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Water-Rock Interaction Mechanisms and Ageing Processes in Chalk

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1. Introduction

The microstructures of geomaterials and their evolution under the effects of applied loading and/or environmental conditions can affect the integrity of the solid skeletons and eventually change the mechanical behaviours of the materials at the macroscopic scale. Analyses of geomaterial microstructure and its 'ageing' are therefore critical to the understanding of their mechanical behaviour and performance in engineering environments.

This process of progressive ageing of the microstructure is mainly related to the interaction between the solid skeleton and the fluids that partially or completely saturate the porous network. Water/rock interaction mechanisms and ageing processes in geomaterials are often slow, leading to structural and textural changes that are generally imperceptible to the naked eye but which can significantly affect matrix integrity and cause sudden collapse. This is a particularly well-known phenomenon in shallow abandoned chalk mines, such as those found in France, where the chalk may remain stable over many tens of years but then suddenly break down, leading to collapse of the openings and the ground above them and subsidence at a macroscopic scale (Sorgi, C. & Watelet, J., 2007).

In this chapter we present results from a research program (led by Institut National de l'Environnement Industriel et des Risques (INERIS), France, in collaboration with Ecole des Ponts ParisTech – CERMES, France) that was carried out to evaluate the mechanical behaviour of chalk in the shallow underground mine of Estreux, located in northern France. The study was conducted at three different scales:

i.   site scale (stability analysis)
ii. laboratory scale (standard core testing)
iii. microscopic scale (electron scanning environmental microscope (ESEM) observations and micro-testing)

In situ characteristics of the Estreux mine are first described, including mine geometry, excavation method, overburden lithology, pillar monitoring, and in situ measurements.

Then, petrophysical properties, microstructural characterisation, retention properties, and mechanical behaviour of the chalk investigated at laboratory scale (oedometric and triaxial tests) are presented.

A microstructural analysis of the Estreux chalk is also presented. This part of the study was conducted using an ESEM, a recent technology that allows the observation of

microstructural changes in geomaterials in their natural state, under controlled conditions of temperature and pressure.

Some other aspects of this technology are discussed, along with suggestions for potential development of this tool for further geomechanical applications and analyses of the Estreux chalk.

2. Site scale: Stability analysis

The study was carried out on Estreux abandoned underground chalk mine in Northern France, which is located 10 km east of the city of Valenciennes. The Estreux chalk formation is of the Late Cretaceous geological period (89 to 94 Ma years ago).

The Estreux mine, which was principally dug for blocks for dimension stone (for buildings and masonry) at the end of the 18th century, covers an area of 10 hectares. The chalk was excavated at a depth of 20 metres using the pillar-and-stall method, where networks of galleries were dug from access shafts, leaving large pillars in place to support the weight of the roof. This prevents the openings caving in, at least during excavation. The width of the galleries in the Estreux mine vary from 2 to 3 metres, and the pillars measure on average between 1.5 and 4.5 metres square. The excavation ratio (relation between the area exploited and the total area) is around 78%.

2.1 Lithology

The lithostratigraphic section of the Estreux mine overburden comprises, from top to bottom:
-     a few tens of centimetres of soil
-     one metre of clay
-     around eleven metres of tuffeau
-     three metres of white chalk
-     two metres of grey glauconitic clay
-     impermeable argillaceous marls

In 1980, Raffoux and Ervel conducted geomechanics laboratory tests on over one hundred core samples from this mine. The mechanical properties obtained are shown in Table 1. From these characteristics and the mining geometry, they estimated a stability factor close to 1 in several zones of the mine. As the mine is not stable in the long term, site monitoring was deemed necessary, and four convergence meters were subsequently installed in a weak zone of the mine.

State	Elastic Limit, Mpa	Breaking limit, Mpa	Young's Modulus, GPa	Poisson's Ratio
Dry	9	10.3	2.83	0.3
Saturated	4.6	5.3	1.61	0.3

Table 1. Mechanical characteristics of the Estreux chalk (Raffoux & Ervel, 1980)

Since August 2003, new instrumentation has been installed (Fig. 1) to analyse geomechanics phenomena of the rock mass over time. The galleries were monitored and a pillar of 1.4 m square and 1.8 m high was instrumented. The instrumentation comprises:
-     an ambient hygrometer
-     a water-level sensor

- a convergence meter between the wall and the roof of the mine
- three pore-pressure sensors together with rock temperature sensors, fixed at different points within the pillar (15 cm, 35 cm, and 70 cm from the surface)
- an extension meter measuring lateral displacement from the pillar surface at two points within the pillar

Data were recorded from February 2004 to February 2008, and the following were observed:
- ambient hygrometry was varying between 85% and 100%
- there was (very slight) seasonal variation in the water level in the galleries, with an overall decreasing trend (Fig. 2)
- seasonal variation in pore pressure and rock temperature within the instrumented pillar was occurring (Figs. 3 and 4), indicating cycles of alternating saturation/desaturation
- pore pressure and rock temperature were generally decreasing from the surface to the core of the pillar (Figs. 3 and 4 respectively)
- during each rise in water level there was increasing pore pressure within the pillar (Fig. 3) and lateral extension (swelling) of the pillar (Fig. 5)
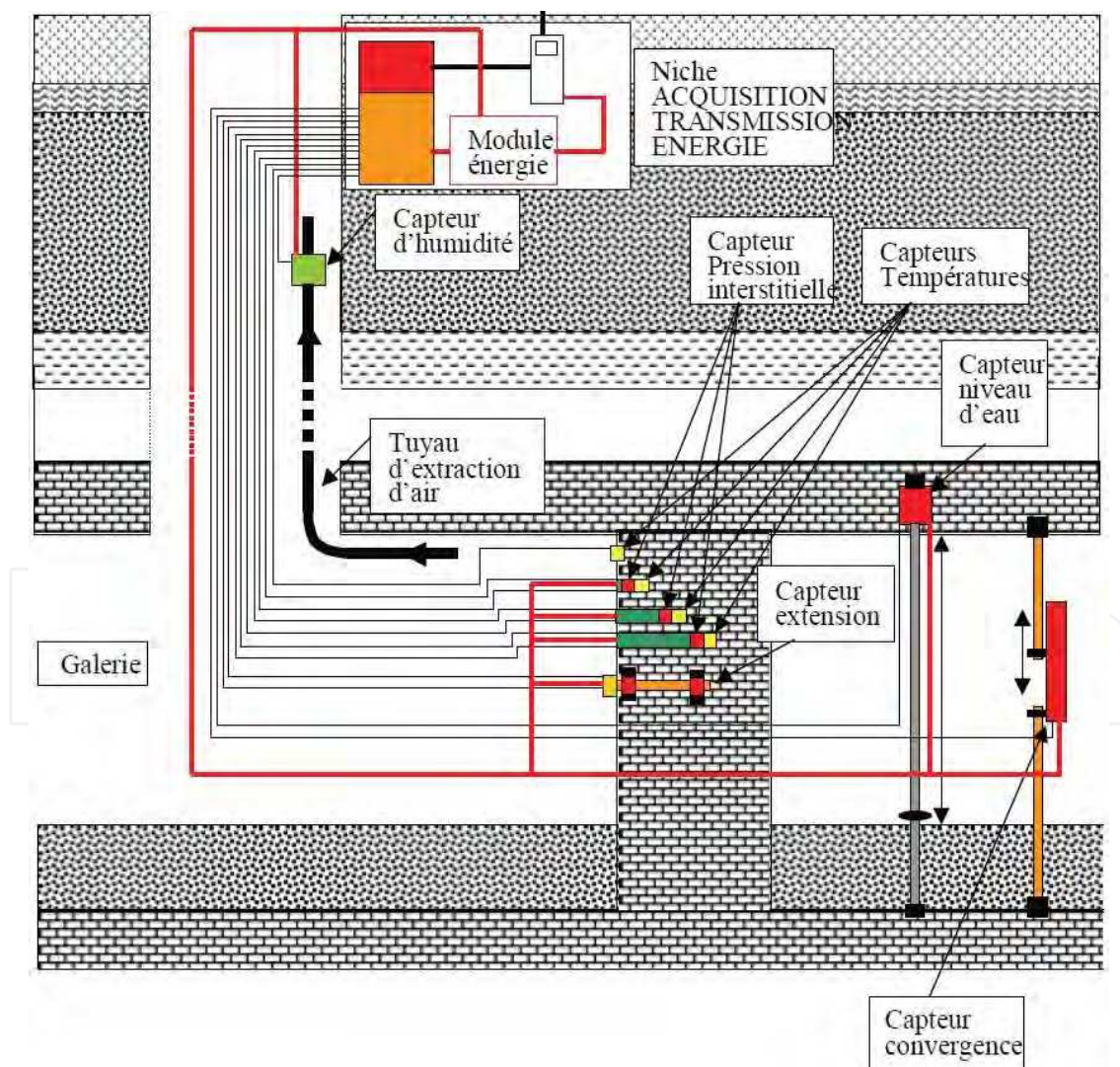- convergence of the wall and roof over time (Fig. 6)

Fig. 1. Diagram of the full instrumentation employed in the Estreux mine
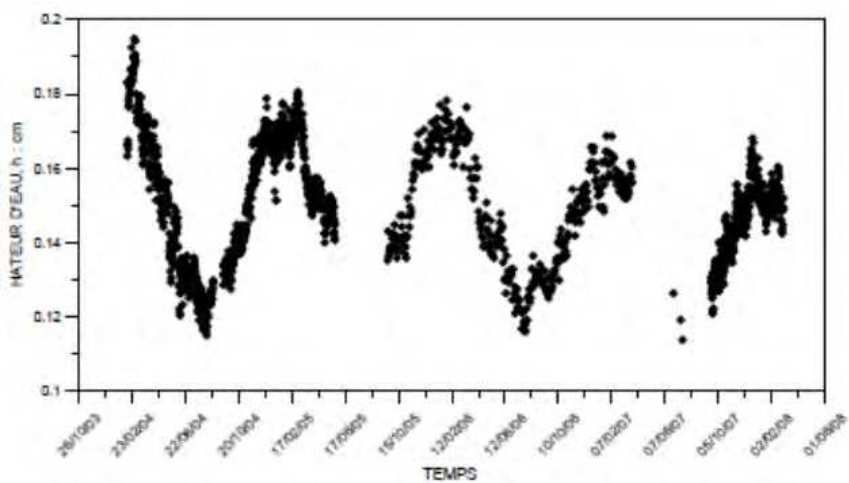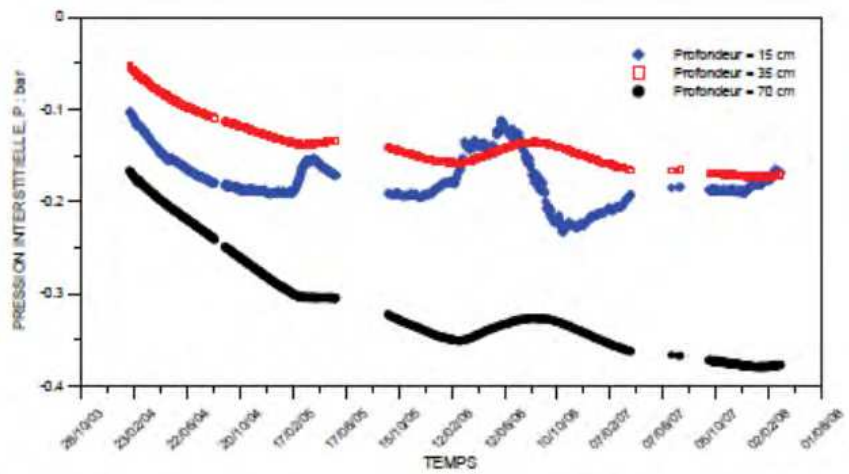
Fig. 2. Evolution of the water level over time

Fig. 3. Evolution of pore pressure at different points within the pillar
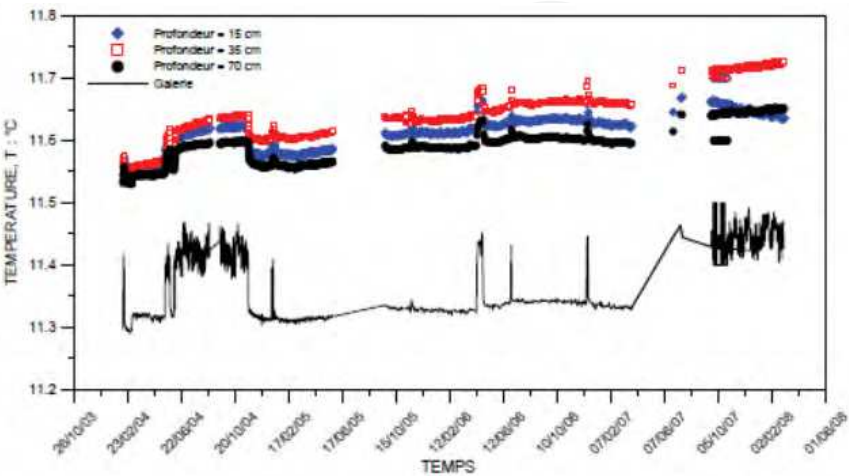

Fig. 4. Changes in rock temperature over time at different points within the pillar and ambient temperature
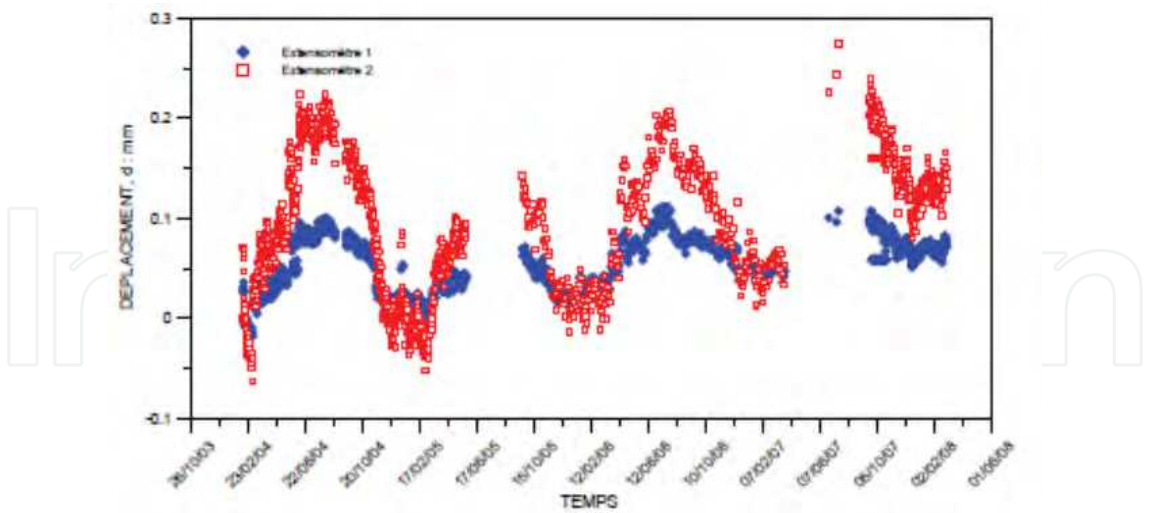
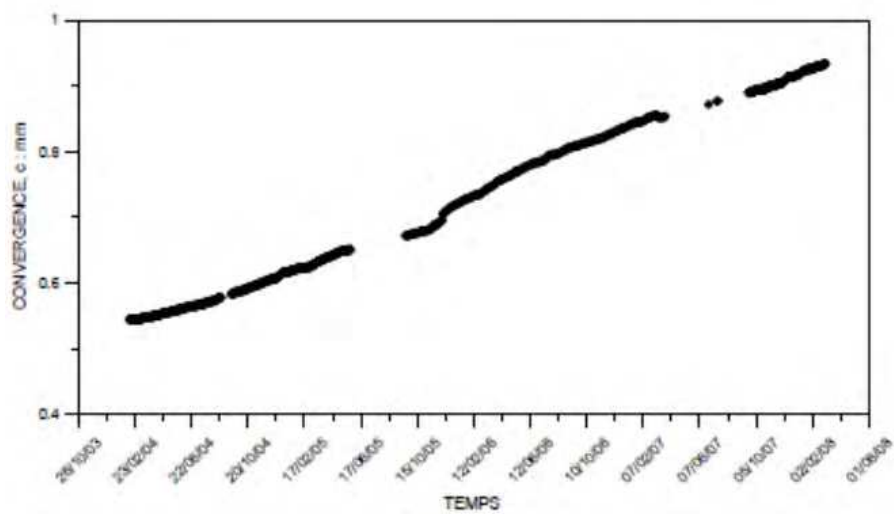Fig. 5. Changes over time in lateral extension at two points within the pillar

Fig. 6. Convergence over time between the mine wall and roof

In a mine where the water-level shows a generally decreasing trend, the influence of ambient hygrometry can have a major impact on the evolution of the pillars. This has been observed in some gypsum mines (Auvray et al., 2004), where scanning electron microscopy analysis of rock sections from different distances from the surface of the pillar has shown extensive traces of dissolution at the surface of the pillar, reducing towards the centre. This dissolution can induce rock matrix degradation that can lead to progressive spalling (flake-off) of the pillar (Fig. 7). As a direct consequence, the section of the pillar reduces over time, as does the pillar's ability to sustain the stability of the rock mass.

The instrumentation of the underground mine in Estreux shows significant seasonal changes in the water level and ambient hygrometry. These changes induce variations in pore pressure, leading to alternating cycles of saturation/desaturation that can contribute considerably to the degradation of the pillars over time.

To study the consequences of this type of phenomenon on the rock matrix in detail, laboratory tests were carried out reproducing the environmental conditions in the mine.

Fig. 7. Progressive spalling of a pillar in a gypsum mine. From left to right, photos from 1996, 2000, and 2004 (Sorgi & Watelet, 2007)

3. Laboratory scale: Core testing

Estreux chalk is a glauconite-rich chalk. Glauconite is an alumino-silicate of iron, potassium, and sodium. Its mineral composition is close to that of illite, although glauconite is not hydrated, with the additional presence of sodium and strong isomorphism by substitution of aluminium atoms with Fe^{2+} and Fe^{3+} iron atoms. Glauconite is often present in chalk deposits in northern France (Masson, 1973).

The porosity of Estreux chalk is about 37%, its specific gravity is G_s = 2.74, and the average water content is equal to 20.7% when the rock is water-saturated. At the microstructural level, the solid matrix is made up of micrometric grains that are principally fragments of coccolithes. Sometimes intact coccolithes also occur. The chalk is then principally made up of calcite (calcium carbonate, $CaCO_3$), which often also constitutes the cementing agent at the intergranular contacts. Microfossils and mineral impurities are also frequently observed.

3.1 Retention properties of Estreux chalk

The Estreux chalk samples were completely saturated when extracted; the mine temperature was 11°C and the relative humidity, h_r, was $\cong$ 100% (with 2% accuracy of the hygrometry resistive sensors). Based on Kelvin's law, the change in relative humidity modifies the total air/water suction, s_t, the difference between the water vapour pressure (assumed equal to the atmospheric pressure, p_a), and the water pressure, p_w, according to the following relation:

$$s_t = p_a - p_w = -\frac{\rho_w}{M_v} RT \ln \frac{p_v}{p_{vs}} \tag{1}$$

where ρ_w is the water density, M_v the molar mass of the water vapour, R the universal constant of an ideal gas (8.314 Jmol-1K-1), T the absolute temperature, p_v the vapour pressure and p_{vs} the pressure of the saturating vapour at temperature T ($h_r = p_v/p_{vs}$).

It is well known that any change in total suction induces a change in the degree of water saturation, S_{rw}, which can be quantified via the water retention curve (WRC) of the material. The WRC of Estreux chalk is presented in Fig. 8 (De Gennaro et al., 2006). As it can be observed, significant changes in S_{rw} occur when suction varies between 1 and 2 MPa, causing near-total desaturation.
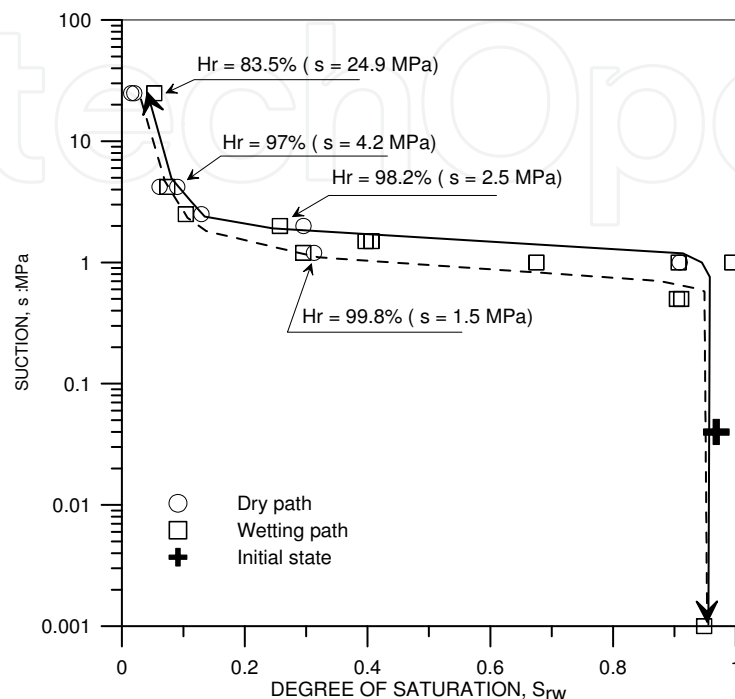

Fig. 8. Water retention curve of Estreux chalk

The slight differences observed between the drying and wetting paths denote a moderate hysteresis effect, also observed in other chalks (Priol, 2005). A possible effect of the glauconite fraction in reducing the hysteresis effect is suspected, although a clear explanation of the slight hysteresis is not straightforward. The drying curve shows that the air entry value of Estreux chalk can be estimated at approximately 1.5 MPa. Following the drying path, the degree of saturation exhibits a dramatic reduction, with a value as low as 30% at 2.5 MPa. At the highest suction (s = 24.9 MPa, h_r = 83.5%) the degree of saturation is as low as 2% to 5%, showing that the chalk is nearly completely desaturated. Based on the water retention curve, the suction of a dry sample can be estimated at 30 MPa. The shape of the water-retention curve of Estreux chalk and the sudden decrease in saturation above 1.5 MPa show that changing values of the ambient relative humidity in the mine (between 80% and 100%) can definitely lead to significantly unsaturated states, at least at the surface of the pillar, directly in contact with the ambient relative humidity. As a consequence, the mechanical properties of the chalk in unsaturated states have to be considered when addressing the long-term stability of the pillars. As a first step, the compressibility properties of the chalk under various controlled suctions are investigated.

3.2 Oedometer tests
Oedometer tests involve uniaxial compression of samples that are prevented from expanding laterally. The two independent stress variables commonly used in the

investigation of the mechanical behaviour of unsaturated soils are the suction, $s = u_a - u_w$ (where u_a and u_w are the air and water pressure respectively), and mean net stress, $p_{net} = p - u_a$ (where p is the total mean stress).

The loading paths followed during the oedometric tests are presented in Fig. 9. The resulting compressibility curves in the $[\log \sigma_v : e]$ diagrams are presented in Fig. 10, where e is the void ratio ($e = V_v/V_s$, where V_v is the volume of void and V_s is the volume of solid skeleton).

The testing program comprises four compression tests carried out as follows:

- Test T1 ($e_i = 0.575$): dry compression ($s \cong 30$ MPa) up to 39.7 MPa, unload down to 0.44 MPa, water injection under 0.44 MPa, and subsequent loading up to 39.7 MPa.
- Test T2 ($e_i = 0.61$): dry compression ($s \cong 30$ MPa) up to 22.41 MPa, unload down to 10.19 MPa, reload to 29.28 MPa, and water injection.
- Test T3 ($e_i = 0.602$): suction controlled compression ($s = 4.2$ MPa) up to 39.7 MPa, unload down to 8.82 MPa, and reload to 39.7 MPa.
- Test T4: ($e_i = 0.581$): saturated compression up to 20.38 MPa, stress release at 0.26 MPa, and reload at 40.76 MPa.
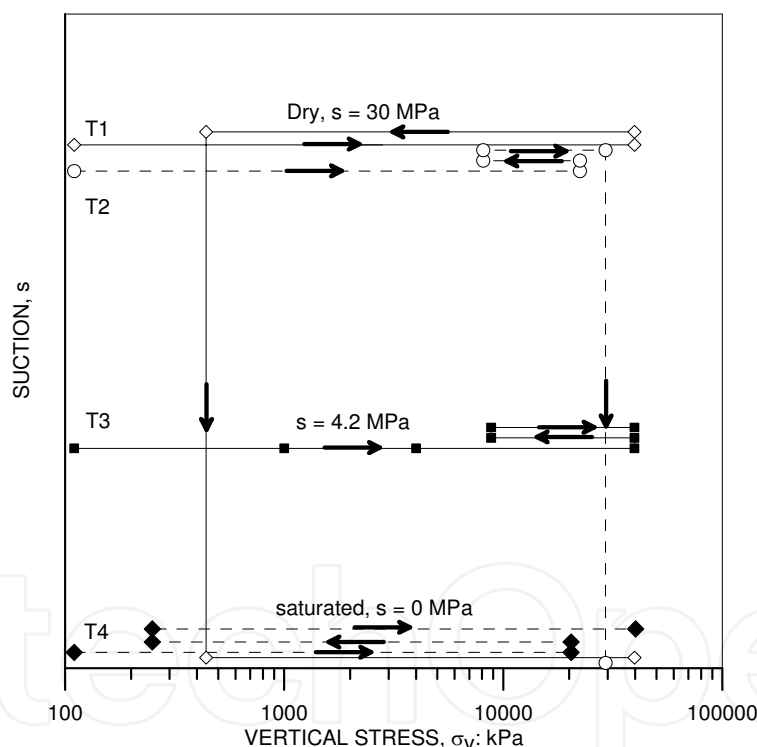

Fig. 9. Loading paths

The compressibility curves in Fig. 10 show some responses typical of unsaturated soils:

- increase in yield stress with increased suction
- increase in compressibility with decreased suction
- slight suction dependency of the pseudo-elastic compressibility module
- slight swelling due to suction release in the elastic zone
- significant collapse during water injection under high constant applied vertical load (pore collapse). Interestingly, the void ratio of the collapsed sample (i.e., after water injection) is close to the saturated compression curves of tests T1 and T4

State	Compressibility		Yield Stress, MPa
	Elastic	Plastic	
Dry (T1)	0.0022	0.1082	16.0
Dry (T2)	0.0055	0.0940	13.5
Suction Controlled (T3)	0.0095	0.1137	1.4
Saturated (T4)	0.0039	0.1350	7.5

Table 2. Compressibility and yield stress of Estreux chalk derived from oedometer tests.

The values of the mechanical parameters are given in Table 2. These illustrate the sensitivity of the mechanical response of the Estreux chalk to changes in suction. They are in good agreement with the water-weakening effects described by Matthews and Clayton (1993), and with earlier observations on reservoir chalks (with water and oil as pore fluids) by De Gennaro et al. (2004) and Priol (2005).

Influence of water sensitivity is also denoted by the swelling observed in test T1 (during water injection under 441 kPa of applied vertical load) and by the collapse observed in test T2 under water injection at 29.28 MPa of applied vertical stress. The increase in compressibility and decrease in yield stress with increased degree of saturation (decreased suction) are two other manifestations of the water-weakening effect.
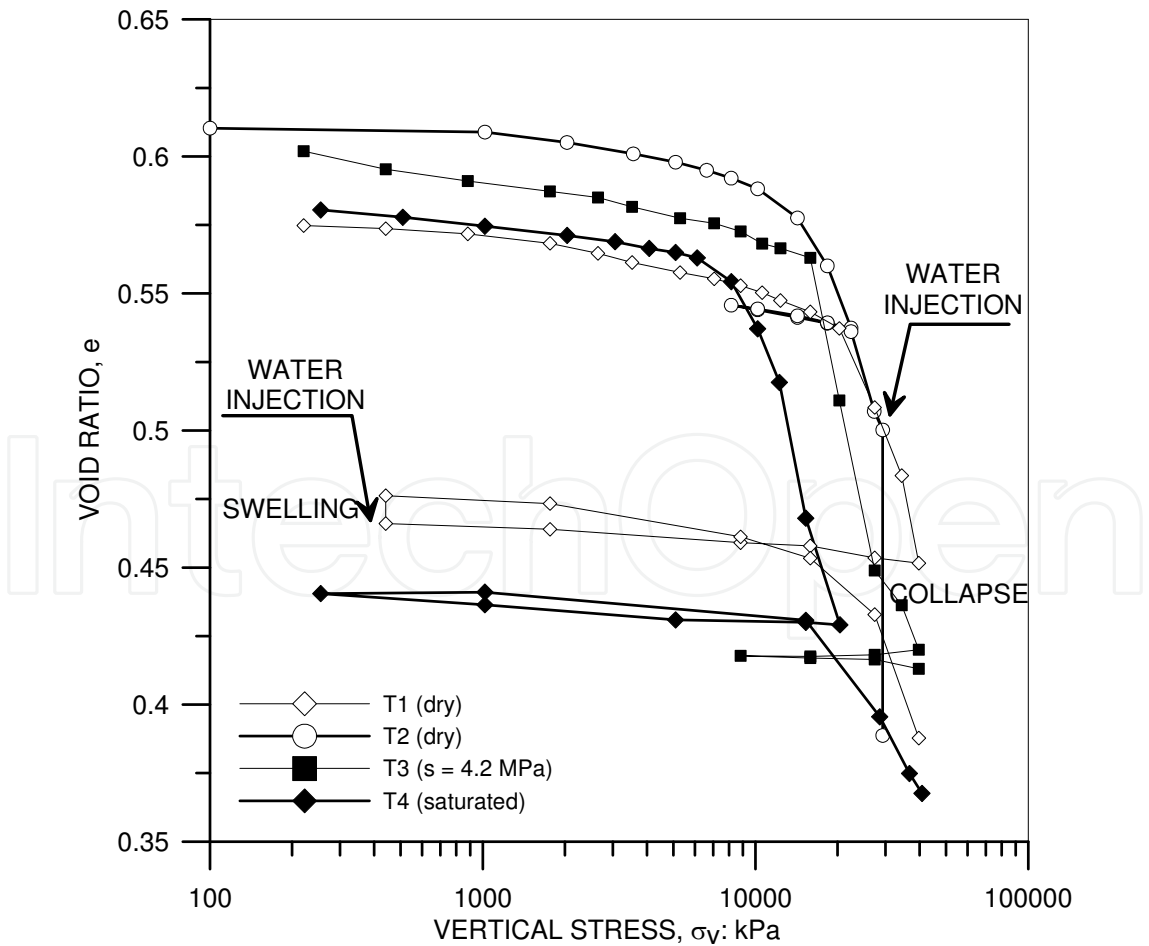

Fig. 10. Compressibility curves obtained with oedometers

4. Microscopic scale: Electron Scanning Environmental Microscope (ESEM) observations and micro-testing

The electron scanning environmental microscope (ESEM) allows the observation of microstructural changes of geomaterials in their natural state, under controlled conditions of temperature and pressure. Unlike the traditional scanning electron microscopy (SEM), ESEM technology does not require any preliminary treatment of the observed samples (such as dehydration or conductive coating). This has undeniable advantages in the analysis of the microstructure of geomaterials. In this study, a FEI Quanta 400® ESEM equipped with a Deben® microtesting facility has been used as a tool for the microstructural and micromechanical characterisation of Estreux chalk.

Changes in S_{rw} were reproduced by controlling sample temperature and pressure following the state diagram of water (Fig. 11), being simultaneously correlated to the corresponding microstructural evolutions. A further step of the analysis involved the investigation of the microstructure while the material was subjected to a micromechanical loading, under constant or variable relative humidity, by means of ESEM micromechanical in situ tests.
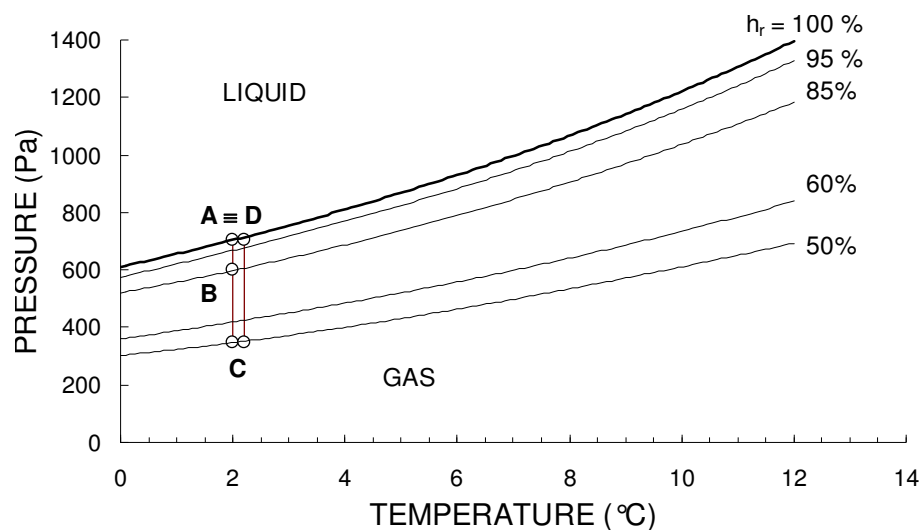

Fig. 11. State diagram of water and imposed changes during observation (ii)

Three types of observations were conducted: (i) changes in microstructure under wetting, (ii) samples submitted to saturation/de-saturation cycles starting from their natural state of saturation (path Fig. 11), and (iii) samples submitted to unconfined axial compression microtests under variable states of water saturation (Sorgi & De Gennaro, 2007).

4.1 Sample preparation

Sub-samples were extracted from available blocks of Estreux chalk retrieved from the underground mine, and these were sealed and stored in a thermo-regulated chamber to ensure the preservation of in situ conditions in terms of water content.

Observations (i) and (ii) were conducted on sub-samples having a square section (about 10 mm/side) and a thickness varying from 2 mm to 4 mm. These were fixed on the observation plate inside the ESEM chamber using a carbon conductive glue. Reduced plug thicknesses ensured a more uniform temperature distribution within the samples, and temperature was controlled using a thermo-electric cooler (based on Peltier's effect). The corresponding value

of the pressure in the observation chamber was used to define the level of hygrometry, h_r, based on the state diagram of water (Fig. 11).

4.2 Microstructural changes under wetting

The changes in microstructure under wetting when passing from h_r = 97% (chalk in its natural state at sampling with w = 20.7%) to h_r = 100% are evident when comparing Figs. 12a and 12b. A reference network has been superposed to the micrograph and the boundary of one characteristic pore has been plotted. Since conditions in the chamber correspond to h_r = 100% (p = 705 Pa, T = 2°C), hydration takes place as time passes. In Fig. 12b, the same pore is visualised after the in-situ hydration. As is seen from the two images, hydration produced a progressive enlargement of the pore boundaries due probably, but not exclusively, to the loss of capillary bridges between the grains. Progressive saturation of smaller pores is also observed on the left side of the photo in Fig. 12b. This observation still remains rather qualitative, though it provides an initial picture of the ongoing phenomena. It should be emphasised that pore enlargement is certainly amplified by the specific condition reproduced in the ESEM environment, namely the absence of any external loading and the observation of the external surface of the sample. It is expected that the extent of this phenomenon could be less for the inner non-visible pores. It is worth mentioning that the occurrence of pore enlargement during saturation at zero external applied load is consistent with the swelling shown in Fig. 10 for test T1 during water injection under low applied vertical stress.
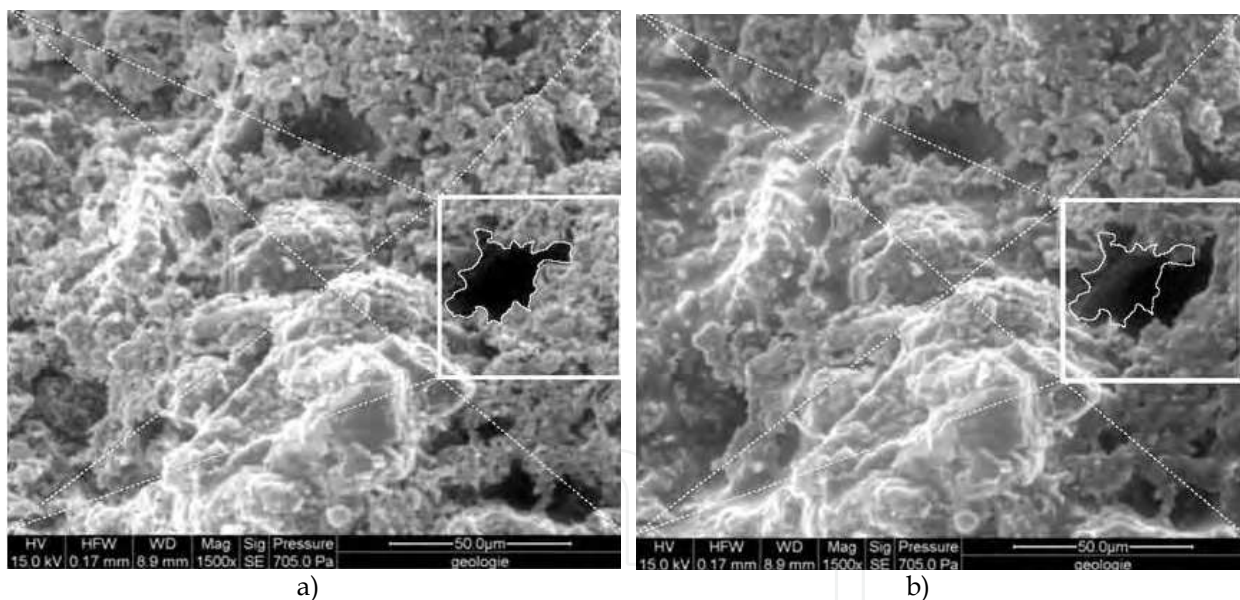

a)　　　　　　　　　　　　　b)

Fig. 12. Modifications of the porous network in chalk during wetting: a) initial state, b) intermediate state before complete saturation

4.3 Saturation/desaturation cycles with ESEM

A series of tests was carried out on samples submitted to saturation/desaturation cycles following the path indicated in Fig. 11. During these tests a constant temperature condition was chosen (T = 2°C). Relative humidity was then modified, changing the level of vacuum inside the chamber between 705 Pa and 346 Pa, corresponding to an h_r varying between 100% and 50% (path A-B-C-D in Fig. 11). Observations were conducted at 1500

magnifications starting from the saturated state (h_r = 100%). During the pressure changes, images were captured every 2 minutes and later mounted as a video clip. The observed zone was characterised by the presence of a rigid inclusion (crystal) embedded in the chalk porous matrix (Fig. 13a). The analysed cycles included:

- Phase 1: saturation and stabilisation; sample was left 90 minutes at T = 2°C and p = 705 Pa, hence h_r = 100% (Fig. 11). The reference image is captured after 90 minutes of elapsed time.
- Phase 2: desaturation; pressure is decreased instantaneously down to 599 Pa (h_r = 85%, path A-B-C in Fig. 11). Sample is left to stabilise for 60 minutes.
- Phase 3: Second saturation; the pressure inside the chamber is increased up to 705 Pa (Fig. 11, path C-D) and sample is left to stabilise for 60 minutes at h_r = 100 %.

During the first phase of saturation (Phase 1), the initial condition corresponding to full water saturation was reproduced inside the samples (Fig. 13a). The successive drying process (Phase 2) induced a fracture opening at the contact between the crystal and the chalk matrix (indicated by an arrow in Fig. 13b). This fracture was not evident at the beginning of the test (Fig. 13a), and its creation would seem to be associated with the changes in suction induced by wetting and drying cycles, athough it is recognised that capillary effects could also be a cause of this microstructural modification (swelling/shrinkage of the material).

In other words, wetting would have brought on fracture closing whereas drying would cause chalk matrix shrinkage around the crystal, inducing fracture opening. Fracture opening could then be the consequence of increasing capillary bridges (hence air/water interfaces) inside the chalk matrix during drying. In contrast, wetting would decrease the number of air/water menisci between the chalk matrix and the crystal, leading to a progressive fracture sealing (Figs. 13c, 13d). If related to material ageing, the evolution of this phenomenon with time following consecutive wetting and drying cycles could contribute to microstructral features associated with material degradation. The observations made here could also be supplemented and improved by advanced techniques of 2D and 3D image analysis, allowing for a more quantitative characterisation of the morphological modifications induced by changes in water saturation (see Sorgi & De Gennaro, 2007).

4.4 Micromechanical in situ testing

The combined use of the ESEM technique with unconfined compression tests was achieved using a micromechanical testing apparatus.

A Deben MICROTEST® loading module allowed the application of a maximal compression load of 5000 N at a constant strain rate of 1x10-5 s-1. A specific setup was developed to carry out micromechanical tests under controlled total suction (controlling the level of relative humidity during the tests). Cylindrical samples of about 8 mm in diameter and 15 mm in height were used. Samples were obtained by means of high-precision coring. End-face parallelism was ensured by means of a high-precision slicer having accuracy of the order of 1 μm. A series of preliminary micromechanical tests was conducted on saturated, partially saturated, and dry samples in order to verify the agreement between the micromechanical test results and the earlier laboratory test results performed on samples with larger (standard) dimensions.

The preliminary results from the unconfined compression microtests are presented in Fig. 14. It can be seen from the test results for the dry samples that there was good reproducibility. The linear slopes of the compression curves after a first tightening phase
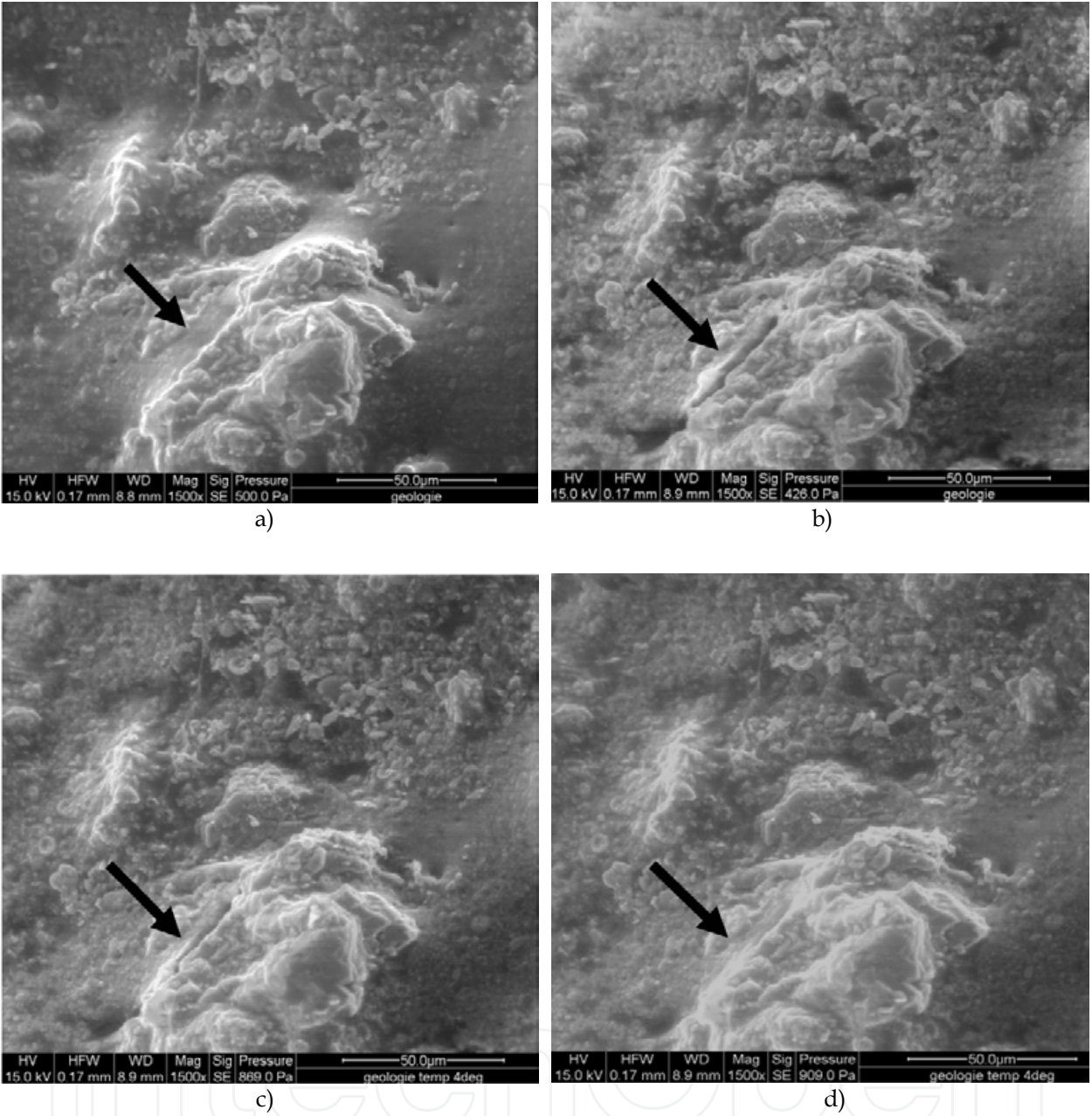
Fig. 13. a) and b) fracture opening in chalk specimen during drying, c) and d) fracture closing following the second saturation

allow the quantification of the Young's modulus. The latter is clearly influenced by the various states of saturation that follow; Young's modulus for dry chalk was E_{dry} = 1.1 GPa, as compared with that of saturated chalk, where E_{sat} = 0.71 GPa. The ratio E_{dry} / E_{sat} = 1.6 is of the same order as that obtained by other researchers by means of standard laboratory unconfined compression tests (e.g. Raffoux & Ervel, 1980). At a suction level, s_o, of 4.2 MPa the value of Young's modulus, E_o, is between E_{dry} and E_{sat}, with a value of 0.78 GPa.

In relation to material strength, the comparison between the unconfined compression strength (UCS) values obtained at saturated and dry states gives a ratio $UCS_{dry}/UCS_{sat} \cong 2$ in

agreement with available data on northern French chalk (e.g. Bonvallet, 1979). Results from the sample tested under constant suction equal to 4.2 MPa ($S_r \cong 97\%$, Fig. 10) show that higher suctions strengthen the rock. This is likely to be associated with additional bonding due to capillary effects. The observed behaviour during unconfined compression microtests seems in good agreement with the general behavioural features observed for this chalk in oedometric compression tests under controlled suction conditions (Nguyen et al., 2008).

Fig. 14. ESEM in situ unconfined compression tests on dry and water saturated chalk

Note that Nguyen et al. (2008) also found a ratio of 2.1 between the yield stress in oedometric tests in dry and saturated conditions, close to the ratio $UCS_{dry}/UCS_{sat} \cong 2$ found during the ESEM micro-testing performed here. The ratio between the yield stress at a suction level of 4.2 MPa and that at saturated and dry state was 1.5 and 0.7, respectively. Similar ratios obtained by micromechanical testing using ESEM were equal to 1.5 and 0.75, showing a notable agreement with the oedometric test results.

Finally, Fig. 15 shows some preliminary results of ESEM in situ testing with simultaneous visualisation of the deformation pattern and the failure mode. The direction of compression is vertical, as indicated on the ESEM image (A). At peak strength (image B), the sample surface is still apparently unchanged. At about 0.9% axial strain, in the softening regime, a pseudo-vertical fracture is visible (image C), followed by a progressive opening in the post-peak phase (images D and E).

The aim of these preliminary tests was to explore the possibility of obtaining a characterisation of the local strain field during hydro-mechanical loading using ESEM. Some

possible developments, like Digital Image Correlation (DIC) (e.g. Vales et al., 2008), might also be considered in order to obtain a quantitative characterisation of the local deformation at microstructural (few hundreds of µm) and mesostructural (some mm) levels.
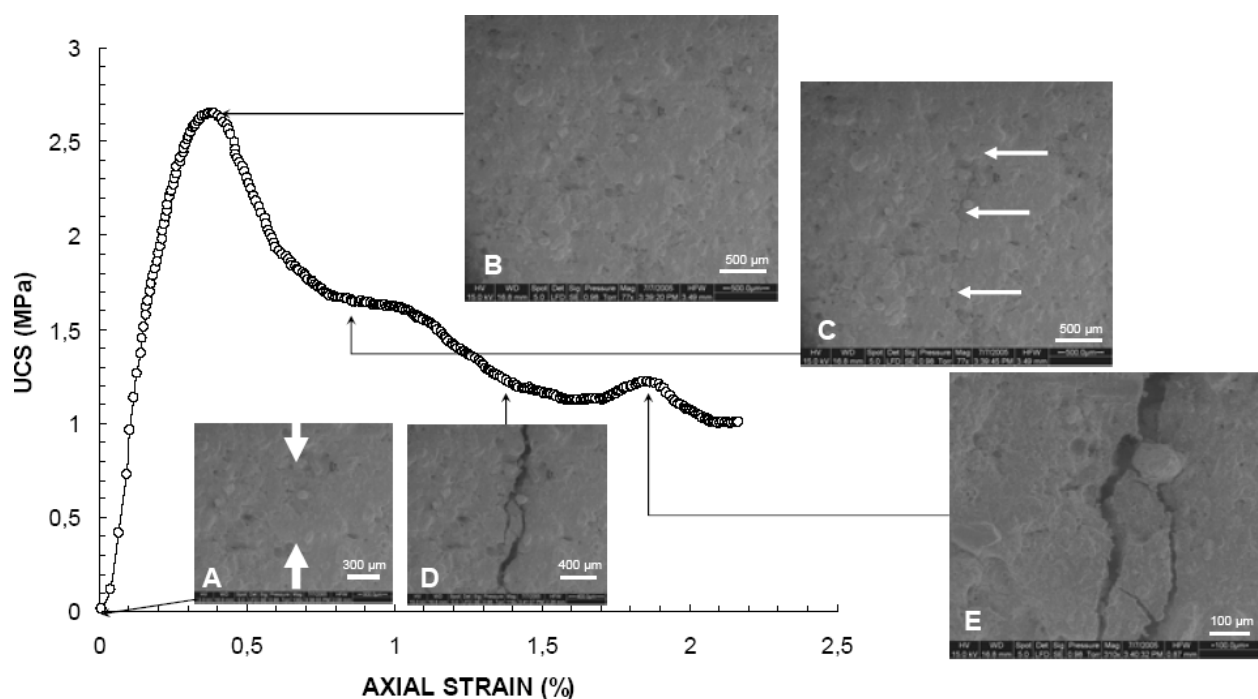

Fig. 15. ESEM failure pattern during ESEM in situ unconfined compression test on water saturated chalk

5. Conclusion

A preliminary investigation of the behaviour of chalk samples retrieved from the pillars of the abandoned Estreux mine (northern France), conducted as part of an assessment of the stability of underground chalk mines, has been presented. Due to environmental changes occurring within the mine (such as hygrometry and water table), the pillars are regularly subjected to variations in the degree of water saturation. The potential impact of the evolution of the water saturation on the mechanical behaviour of the chalk has been assessed based on the methods and concepts of the mechanics of unsaturated soils at different scales: site scale, laboratory scale, and microstructural scale.

At site scale, observations conducted during a four-year period (from February 2004 to February 2008) have highlighted that relative humidity in the mine can vary between 85% and 100%. The seasonal changes in the water table have been of reduced extent during the monitoring period, showing a general tendency towards decreasing water table level. However, as the water table rose, both pillar expansion and roof convergence were observed. Pressure and temperature measurements inside the chalk pillars have shown that both parameters vary on a seasonal basis, causing alternating states of saturation/desaturation. As expected, both pressure and temperature are not constant

inside the pillars, but rather depends on the position of the point considered (altitude and distance from the pillar surface), on the liquid water inflow at the base of the pillar (which depends on the water table level), and on the evaporation rate at the surface of the pillar. It has been shown that, moving inwards from the free surface to the core of the pillar, both pressure and temperature decrease. The observations at site scale confirmed that water saturation in the chalk, controlled by the evolution of relative humidity and water table, influences the deformation of mine pillars and roof.

At laboratory scale, a detailed experimental program was carried out to better quantify the influence of water saturation on the mechanical behaviour of the Estreux chalk. A significant phenomena identified by the water retention properties is that significant desaturation occurs when the suction increases from 1 to 2.5 MPa, corresponding to a change in relative humidity from 99.3% to 98.2%. In such situations, water saturation decreases from almost full saturation (higher than 90%) down to $S_{rw} = 12\%$. The degree of water saturation of the chalk at equilibrium under a relative humidity of 83.5% (suction of 24.9 MPa) was about 7%. Considering that conditions of relative humidity, h_r, of around 80% can occur in the mine, and that for this situation S_{rw} at equilibrium is as low as 5%, the significance of the chalk behaviour in unsaturated conditions is confirmed. Similarly to what has been shown in oil reservoir chalks (De Gennaro et al., 2003, 2004), the methods and concepts of unsaturated soils have been successfully adapted to Estreux chalk. Controlled suction oedometer tests provided more details on the water-weakening effects in chalk, showing increasing compaction during water injection (pore collapse). As observed in unsaturated soils, the yield stress determined in the oedometer increases when suction is increased (i.e. when the degree of saturation is decreased), confirming in more detail the suction-hardening effect as is observed in unsaturated soils.

For the microstructural scale, some applications of the ESEM for the microstructural characterisation of the partially saturated chalk have been presented. ESEM allows the observation of microstructural changes of geomaterials in their natural state under controlled conditions of temperature and pressure. Change in saturation can be easily reproduced in the observation chamber by means of a thermo-electric cooler based on the Peltier effect. This provided a preliminary analysis of the microstructural modifications of the Estreux chalk induced by the saturation/desaturation cycles in the absence of mechanical loading. Suction-controlled micromechanical in situ tests are also feasible. The validation of a specific experimental technique has been presented and results from micromechanical uniaxial unconfined compression tests have been compared with available results from laboratory-scale tests in terms of isotropic Young's moduli and UCS values. Good agreement has been observed between the different tests, confirming the reliability of this technique for further investigation of the micromechanical behaviour of geomaterials under controlled saturation states (controlled suction).

Further developments using the ESEM are expected to all quantitatively characterise the effects of the mechanical and physico-chemical processes associated with the water-rock interaction. In the specific case of carbonate rocks these developments could provide a better understanding of some fundamental processes, such as dissolution, precipitation, crystallisation, and solid transport under stress, that lie at the onset and cause the degradation mechanisms of these rocks under the effect of environmental and mechanical agents.
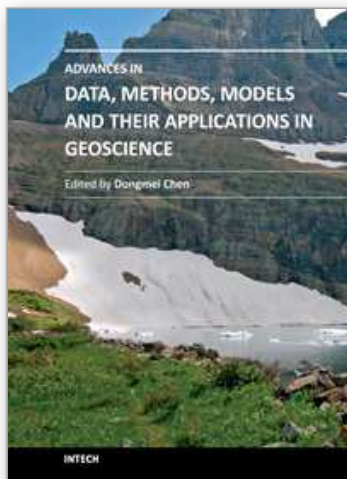
6. Acknowledgment

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