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## Long term behavior of a chalk: effect of relative humidity and flooding

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ABSTRACT: The shallow underground room-and-pillar mines where chalk rocks were extracted are now abandoned and are enduring the effects of time and weathering which increase their risk of collapse. The Parisian Basin in France contains many abandoned underground chalk mines and number of them were already instrumented or subjected to a feedback analysis aimed to aid the development of prevention strategies. Campaigns of laboratory characterization are often conducted to complete the understanding of the hydromechanical behavior of these chalks. For instance, laboratory characterization was conducted to understand the long-term behavior, among other, of Saint-Martin-le-Noeud chalk with respect to the relative humidity for levels ranging between 90% and 100%, the most representative of the *in situ* conditions of abandoned underground openings. The main conclusions are: (a) the existence of stress and relative humidity thresholds from which the creep of this chalk starts, (b) the flooding at the last levels of stress and hygrometry leads to an accelerated creep causing the ruin of samples without transient phase.

#### 1 INTRODUCTION

Chalk rocks have been used for centuries as building materials and for the lime production. The shallow underground room-and-pillar mines where chalk rocks were extracted have since been abandoned and now endure the effects of time and weathering, increasing potentially their risk of collapse because of frequent flooding/unflooding cycles. Then, the risk of instability of these underground structures in relation to the time, weathering and flooding or dewatering effects, is a key issue for more than 10 000 towns in France.

To prevent the risk of collapse, Ineris (Institut national de l'environnement industriel et des risques, France) and GeoRessources have been studying the rock behavior of partially flooded underground chalk mines in northern France, through both *in situ* and laboratory characterizations. The objectives are to determine the impact of groundwater-table fluctuations on the stability of these mines: this is particularly the case of the Saint-Martin-le-Noeud underground mine (Fig. 1) located on a hillside to the south of Beauvais (Picardie, France). This is one of the three underground chalk mines of Parisian Basin already instrumented or subjected to a feedback analysis by Ineris. The main goal of the *in situ* characterization was to determine the impact of groundwater-table fluctuations on the stability of the mines. These fluctuations lead to chemical water-chalk interactions that have an impact on the mechanical behavior of chalks, as has been demonstrated by numerous studies (Risnes et al. 2005, Duperret et al. 2005, Talesnick & Shehadeh 2007, Gombert et al. 2013, Lafrance 2016).

The effect of time on the behavior of an unlined underground structure encompasses various phenomena responsible for the variations in the material properties: (i) degradation of minerals in the rock as a result of physico-chemical action, leading to a reduction in the mechanical characteristics (Risnes et al. 2005, Duperret et al. 2005, Talesnick & Shehadeh 2007); (ii) differed deformation due to constant loading (Dahou et al. 1995, Gutierrez & Hickman 2010); (iii) variations in relative humidity (often linked to the airing conditions in the underground mine) and

thus in the saturation and suction conditions that affect the rock mass, over time (Priol et al. 2007, Lafrance 2016). The monotonic and/or cyclical variations in the hydric boundary conditions may generate damage due to hydro-mechanical couplings. The influence of all of these phenomena must be taken into account when studying the temporal evolution of abandoned chalk underground mines, with the aim of being able to provide optimum remediation strategies for ensuring the long-term stability of the quarries with regards to public safety and the protection of built resources.

A large laboratory characterization was conducted to firstly understand the short-term behavior of the Saint-Martin-le-Noeud chalk with respect to the relative humidity (RH), by limiting ourselves to the RH levels ranging between 90% and 100% which are the most representative of the *in situ* conditions of abandoned underground mines (Lafrance 2016). The ageing aspects as well as the impact of weathering on the macro-mechanical properties are described in Lafrance et al. (2016). These laboratory tests clearly indicate an elastoplastic behavior with strain-hardening/softening, the RH-dependent strength, the pore collapse of chalk, as well as time-dependent shear and volumetric strains. More precisely, for the short-term behavior: (a) a linear behavior and an appearance of plastic strains at low deviatoric stress (the elastic limit is low); (b) under low confining pressures, the samples failure corresponds to the formation of a shear band inclined with respect to the sample axis. There is a dependence of the mechanical behavior on the relative humidity. Finally, what can be found in the extensive literature of the short-term behavior of chalks in particular, or highly porous rocks in general.

A second campaign of laboratory tests focused on the characterization of the time-dependent behavior of this chalk, through multi-stage uniaxial creep tests with controlled humidity (ranging between 90% and 100%) and samples flooding during the last stress and relative humidity levels. The resulting the effects of water-rock interactions on the mechanical long-term behavior of this chalk is discussed in this paper.

#### 2 METHODOLOGY

#### 2.1 The Saint-Martin-le-Noeud underground chalk mine

The Saint-Martin-le-Noeud underground mine is located on a hillside to the south of Beauvais (Picardie, France). The material extracted from this mine (closed in 1830) is a white to grey homogenous chalk containing dolomite (14%) and was used to build Beauvais Cathedral in the  $12^{th}$  century The chalk was exploited by the room and pillar method, leaving behind 3 to 5 m wide galleries, originally 4 m in height, and irregular pillars of between 2 and 4 m width. The estimated ratio of extraction is 50% to 67% (Gombert et al. 2013), leads to an average of vertical stress in the pillars about 1 to 1.5 MPa. The stratigraphy of Saint-Martin-le-Noeud has a near-tabular profile with a dip of  $\approx$ 4–10°. Due to this shallow dip, the deepest parts of the underground mine reach the shallow aquifer, resulting in the formation of several "underground lakes" (Fig. 1).







Figure 1. View of the studied underground mine of Saint-Martin-le-Noeud: (a) June 2010; (b) May 2011; (c) May 2012.

Chalk samples were collected from the mine by drilling 120-mm diameter, 2 to 4 m long boreholes horizontally into the pillars. It was not possible to determine the Discontinuity Spacing (DS) index (Bieniawski 1984) in these core samples due to signs of artificially-induced fracturing. Cylindrical specimens (50 mm diameter, 100 mm height) were sampled from each of the previous cores. The longitudinal axis of each specimen is therefore parallel to the axis of the pillar and perpendicular to the stratigraphy. These specimens were then cut and rectified using a double circular saw in order to ensure flat, smooth and parallel surfaces.

The samples were then instrumented in order to measure axial and lateral strains. Strain gauges were glued in pairs to the sample using the same adhesive material each time. The lateral or axial gauges were positioned opposite each other, mid-height of the sample. Partially saturated samples were obtained using the vapor equilibrium technique, by placing the dried samples under imposed hygrometry using saline solutions to control the RH (Blatz et al. 2008). The vapor equilibrium technique allows the relative humidity in a sealed environment to be controlled with saline solutions at a given temperature. By using different types of salts, different levels of RH can be imposed. All tests were performed at ambient temperature (average 20 °C).

A series of creep tests under specific controlled relative humidity were conducted over long period (2 years). Through these tests, we aimed to characterize the time-dependent behavior of this chalk according to its microstructure-mineralogy and to the (deviatoric stress, hygrometry) pairs. There are therefore uniaxial multi-stage creep tests in terms of stress (0.5, 1, 2 and 3.5 MPa) and hygrometry (RH = 85, 90 and 98%).

Each sample is placed in a sealed cell under 85%, 90% and 98% RH controlled using saline solutions, and the equipment provides also the possibility of introducing a subsequent flooding phase (Fig. 2). Once the RH samples have stabilized, the test is initiated. The first step corresponds to an axial stress of 0.5 MPa. After loading at a rate of 0.05 MPa/s, the stress level is maintained until the strain rate becomes constant. Two additional stages of stress (1 and 2 MPa) are then imposed before reaching the final level of 3.5 MPa. The samples at 85 and 90% RH are then brought up to a hygrometry of 98% for a hydric stage. Finally, samples at 98% RH and 3.5 MPa are rapidly and completely flooded with a water chemically balanced with (and at the same temperature as) the corresponding chalks, while maintaining the axial stress at 3.5 MPa and the strain rate constant. The experimental conditions of two of the three initial relative humidity (85, 90 and 98%) are summarized in Table 1. The realization of these creep tests by increasing axial stress ( $\sigma_1$ ) allows to assess the threshold of creep for each initial RH; the amplitude and the rate of time-dependent strains for each pair of ( $\sigma_1$ , RH); the creep acceleration threshold corresponding to the long-term strength.

Table 1. Experimental conditions for multistage creep tests: controlled relative humidity (RH) series.

RH(%)	σ <sub>1</sub> (MPa)	% of $\sigma_c^{dry}$	Duration(d)	RH(%)	$\sigma_1(MPa)$	% of $\sigma_c^{dry}$	Duration(d)
90	0	0	45	98	0	0	45
90	0.5	5	70	98	0.5	5	70
90	1	9	22	98	1	9	22
90	2	18	101	98	2	18	101
90	3.5	32	331	98	3.5	32	326
98	3.5	32	159	Saturated	3.5	32	5*
Saturated	3.5	32	1*				

<sup>\*</sup> sudden failure of sample;  $\sigma_1(MPa)$  = uniaxial stress;  $\sigma_c^{dry}$  = uniaxial compressive strength (UCS) under dry conditions.







Figure 2. Experimental device used for creep tests with controlled relative humidity.

#### 3 RESULTS AND DISCUSSION

#### 3.1 Basic physical and short-term properties of the studied chalk

Physical measurements (porosity, dry and wet density, water content, etc.) were previously performed to characterize the natural state of this chalk (Lafrance 2016). Total porosity and water content in the natural state are respectively 42 and 27(%). The connected porosity, measured at the mercury porosimeter, is 40%. Petrographical analysis (Fig. 3) indicated that the chalk of Saint Martin-le-Noeud is a micritic calcareous rock containing micrite (68%), sparite (17%) as well as the presence of dolomite (13%) evidenced by the observation of lozenge cavities (void) and having retained their shape (indicating that any stress was applied to the rock after the disappearance of these minerals) as shown in the 2D image analysis from SEM (Scanning Electron Microscopy) observations (Fig. 3c). The fact that there are voids and rhombuses with perfect shapes means that the dolomite has been dissolved preferentially compared to the calcite minerals without any chalk compaction after dissolution.

The gas permeability is in the order of  $10^{-15}$  m<sup>2</sup>, while the water permeability is one order of magnitude smaller. Uniaxial compression and Brazilian tests carried out on this chalk provide the values of its strengths under dry and saturated conditions (Lafrance 2016). The average values of uniaxial compressive strength (UCS) are 11.2 and 2.6 MPa, respectively under dry and saturated conditions. In the latter case, the measured isolated value of 7.8 MPa was not considered in the average. Under dry conditions, the average tensile strengths were 1.2 and 0.6 MPa, respectively under dry and saturated conditions.

#### 3.2 Long term response according to controlled RH

Figure 4 shows the evolution of the axial viscoplastic strain  $\epsilon_{ax}^{vp}$  and lateral viscoplastic strain  $\epsilon_{lat}^{vp}$  with respect to the time, measured during the multistage  $(\sigma_1, RH)$  creep tests. These are time-dependent deformations in which the instantaneous strains induced by the sudden application of stresses and hygrometry, have been subtracted. Based on these measured deformations, the volumetric viscoplastic strain  $\epsilon_v^{vp}$  and viscoplastic distortion  $\epsilon_{eq}^{vp}$  (time-dependent shear strain) were computed, as follows:

$$\varepsilon_{v}^{vp} = \varepsilon_{ax}^{vp} + 2\varepsilon_{lat}^{vp} \qquad \varepsilon_{eq}^{vp} = \sqrt{\frac{2}{3}(\varepsilon_{ax}^{vp} - \varepsilon_{v}^{vp}/3)^{2} + \frac{4}{3}(\varepsilon_{lat}^{vp} - \varepsilon_{v}^{vp}/3)^{2}}$$
(1)

The positive values of the volumetric strain correspond to the time-dependent contraction of the chalk, while the negative values reflect its dilatancy.

Figure 5 plots the evolution of the volumetric viscoplastic strain and viscoplastic distorsion as a function of the time. The evolution of the volumetric viscoplastic strain as a function of the viscoplastic distorsion is represented in figure 6: this allows to characterize the volumetric creep behavior of the studied chalk.

On these figures where a zoom was performed at the beginning of the tests, each starting hygrometry is denoted by a given color: red for RH equals 85%, orange for RH equals 90% and blue for RH equals 98%. The vertical dashed lines separate the different stages of applied stress (green) and/or hygrometry (violet) summarized in Table 1. The end of the curves corresponds to the total failure of sample for each test.



Figure 3. Microscopic analysis: (a) natural light; (b) polarized light; (c) SEM image.

It can be observed that for the test initiated with a quasi-dry sample (i.e. RH = 85%), the failure occurs at the third stress stage after 50 days. For the other two tests started at 90 and 98% of RH, the failure of the samples occurs at 1 and 5 days, respectively, after the total flooding without the establishment of transient, stationary and tertiary creep phases. One of the first observations concerns the magnitude of the viscoplastic strains measured for the test initially placed at 85% of RH. These amplitudes before sample failure are low and the sample abruptly breaks in the form of columns. Its behavior is rather contractant and the creep occurs at constant volume for the last level of stress (before the failure).

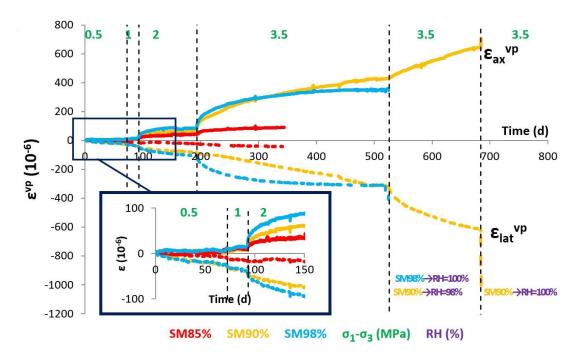


Figure 4. Creep curves of Saint-Martin-le-Noeud underground chalk mine. Axial  $(\epsilon_{ax}^{vp})$  and lateral  $(\epsilon_{lat}^{vp})$  strains as a function of time.

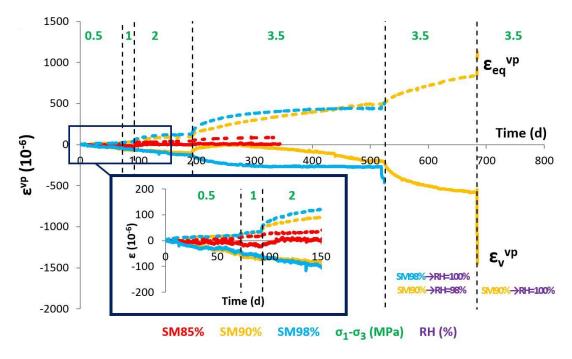


Figure 5. Creep curves of Saint-Martin-le-Noeud underground chalk mine. Volumetric  $(\varepsilon_v^{vp})$  and deviatoric  $(\varepsilon_{eq}^{vp})$  strains as a function of time.

#### 3.3 Discussion

A more thorough analysis is herein performed on the other two partially saturated sample creep tests (initially at RH = 90% and RH = 98%), followed by the flooding phase. The time-dependent behavior of the Saint-Martin-le-Noeud chalk (Fig. 4-6) suggests involvement of the characteristics and mechanisms discussed below.

At a RH of 85%, the creep threshold (i.e. level of stress at which creep strains are initiated) is 1 MPa (in agreement with dry creep tests, Lafrance 2016), whereas at higher levels of RH, time-dependent strains appear from the moment where the smallest stress is applied (0.5 MPa). This mean that the creep threshold of this chalk depends both on the level of the applied stress and the imposed relative humidity, that is to say a function of  $\sigma_1$  and RH. More precisely, the more important the sample hygrometry, the less mechanical stress threshold to initiate the creep will be

For most all levels of RH and axial stress, lateral deformations stay close to the axial ones, leading to deferred dilatancy of the material ( $\varepsilon_v^{vp} = \varepsilon_{ax}^{vp} + 2 \varepsilon_{lat}^{vp} \approx \varepsilon_{lat}^{vp}$ ), whereas the short-term response of this chalk remains contractant in the range of stresses and hygrometry applied during these creep tests (Lafrance 2016). The same behavior (i.e. contractant instantaneous response and dilatancy during creep tests) was observed on chalks by Dahou et al. (1995), Xie (2005). In addition, dilatant character of the time-dependent behavior as reported by Maranini & Brignoli (1999) is consistent with results obtained on most geomaterials and also with the fact that most creep models are written only in terms of deviatoric stress and not the mean stress. The relationship between volumetric strain and viscoplastic distortion given in figure 6, depends only on the pair ( $\sigma_1$ , RH) and not on the loading history. By neglecting amplitudes of volumetric strains below 50  $\mu$ m/m (or 50  $\mu$ def), thresholds of 2 MPa stress and 98% RH are obtained for the initiation of volumetric time-dependent deformations that remain dilatant.

As most of geomaterials, the creep curves indicate that the time-dependent strains increase nonlinearly with the applied stress level. This is also the case of the studied chalk. For a given stage of deviatoric stress, the viscoplastic strains also increase nonlinearly with time. In addition, the strain rates depend on the magnitude of the viscoplastic deformation. Finally, figures 4 and 5 clearly indicate that the viscoplastic strains also increase non-linearly with the moisture hygrometry for a given deviatoric stress, particularly when the relative humidity is increased from 85% and 90% to 98%.

Water flooding also significantly increases viscoplastic strain. The more saturated the sample (higher levels of relative humidity), the greater the amount of deformation observed in the sample.

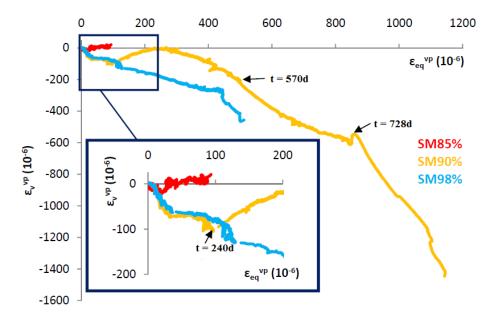


Figure 6. Creep curves of Saint-Martin-le-Noeud underground chalk mine. Viscoplastic dilatancy  $(\varepsilon_v^{vp})$  versus deviatoric viscoplastic strain  $(\varepsilon_{eq}^{vp})$ .

The mechanisms concluded to be at the origin of creep deformations are physico-mechanical in nature and involve the breaking and restoring of electrostatic bonds between grains. Flooding with water destroys the menisci, causing the chalk to become weaker and significantly increases the time-dependent strain rate. From published literature, flooding and hydric loading significantly increase the volumetric viscoplastic strain (Risnes et al. 2005). At levels of 3.5 MPa deviatoric stress and 98-100% RH, the volumetric viscoplastic strains develop in the same manner as reported by numerous studies (Andersen et al. 2012, Megawati et al. 2013).

#### 3.4 Towards a rheological model

In recent years, some models of creep have been developed for chalks or softs rocks, based on: (a) the microstructure of the material according to the work of Pietruszczak et al. (2002) or (b) as part of a phenomenological approach using the formalism of Perzyna (1966).

The time dependent behavior is governed by deformation processes corresponding to different physical mechanisms for change in material microstructure. Then, using the formalism proposed by Perzyna (1966), it is possible to express the total viscoplastic strain rate.

More precisely, based on the main observations from creep tests, a creep model is proposed by introducing a creep threshold function depending on the relative humidity in the well-known Lemaitre's law. By taking into account all the observed phenomena (previously discussed), the following expression was proposed for the viscoplastic shear strain rate tensor:

$$\underline{\dot{\varepsilon}}^{vp} = A \left(\frac{q - q_0}{\sigma_0}\right)^n \left(\varepsilon_{eq}^{vp}\right)^m \frac{\partial q}{\partial \sigma} \tag{2}$$

where A (RH-dependent) is the material viscosity depending on the relative humidity,  $\sigma_0$  the reference stress, n a dimensionless exponent corresponding to the deviatoric stress power factor, m the exponent of hardening work,  $\varepsilon_{eq}^{vp}$  the viscoplastic strain (or viscoplastic distortion),  $q_0$  (RH-dependent) the deviatoric stress threshold, q the current deviatoric stress,  $\underline{\sigma}$  the stress tensor.

Equation (2) was successfully used to describe the time and RH -dependent behavior of the Estreux chalk, another chalk from the Parisian Basin (Souley et al. 2016).

Finally, we have just seen that delayed deformations (especially lateral strain) impact the volumetric behavior. From a constitutive point of view, this can result in a relationship between the delayed dilatancy rate and the shear deformation. Based on figure 6, we propose a power-type relationship between the volumetric strain rate and the shear ones:

$$\frac{\dot{\varepsilon}_v^{\ vp}}{\dot{\varepsilon}_{eq}^{\ vp}} = \beta = a_\beta \left(\varepsilon_{eq}^{\ vp}\right)^{e_\beta} \tag{3}$$

where  $a_{\beta}$  (RH-dependent) and  $e_{\beta}$  are material parameters; the dilatancy rate  $\beta$  must be limited by its minimum and maximum values ( $\beta_0$ ,  $\beta_m$ ).

Next step will be to consider the short-term response: failure is based on a combination of a cut-off in tensile, a yield function based on Drucker-Prager criterion in shear - including hardening and softening - and an elliptical yield surface for the volumetric cap within which both pore collapse and compaction will be incorporated. In addition, the viscoplastic contractance observed on chalks during hydrostatic creep tests in the literature (Dahou et al. 1995, De Gennaro et al. 2003, Xie 2005) will also be considered regarding the Perzyna's formalism in the final version of the model.

#### 4 CONCLUSION

Accidents due to the collapse of underground chalk mines are among the most destructive in terms of both human and material losses. The main objective of this research is to study, the effects of water-rock interactions on the mechanical behavior of chalk sampled from the abandoned Saint-Martin-le-Nœud underground mine located in the Paris Basin.

Laboratory characterizations were conducted to understand the short and long terms behavior of these three chalks as a function of the degree of relative humidity (RH) or saturation. Tests

focused on relative humidity levels ranged between 90% and 100%, the most representative of *in situ* conditions of abandoned underground openings. The main mechanisms and factors describing the long-term behavior of the chalk were examined with a focus on RH values between 90 and 100% corresponding to conditions prevailing *in situ*, and the main conclusions are: (a) the existence of stress and hygrometry thresholds from which the creep of this chalk starts, (b) the time-dependent increase non-linearly with the deviatoric stress and the relative humidity, (c) the strain rates depend on the magnitude of the viscoplastic deformation, (d) very large volumetric strain deformations develop when the pair (deviatoric stress, relative humidity) is significant.

Water flooding at the last levels of stress and hygrometry considerably increase the lateral strains, inducing further dilatancy of this chalk, and then the complete failure of samples before the establishment of the three different phases (transient, stationary and tertiary).

Based on these observations, the ingredients necessary in writing the long-term constitutive equations with respect to the relative humidity encountered *in situ*, are presented. The next step will be to complete the proposed constitutive equations by integrating the instantaneous elastoplastic behavior, for applications to real cases of chalk underground mines in the Parisian Basin.

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