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To cite this version:

Eleni Stavropoulou, Matthieu Briffaut, Frédéric Dufour, Guillaume Camps. Time-dependent behaviour of the Callovo-Oxfordian claystone-concrete interface. Journal of Rock Mechanics and Geotechnical Engineering, 2020, 12 (1), pp.89-101. 10.1016/j.jrmge.2019.09.001 . hal-02506037

HAL Id: hal-02506037

https://hal.science/hal-02506037v1

Submitted on 12 Mar 2020

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Journal of Rock Mechanics and Geotechnical Engineering xxx (xxxx) xxx

Contents lists available at ScienceDirect

Journal of Rock Mechanics and Geotechnical Engineering

journal homepage: www.rockgeotech.org

Full Length Article

Time-dependent behaviour of the Callovo-Oxfordian claystone-concrete interface

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ARTICLE INFO

Article history:
Received 4 February 2019
Received in revised form
6 May 2019
Accepted 12 June 2019
Available online xxx

Keywords:
Callovo-Oxfordian (COx) claystone
Interfaces
Time-dependent behaviour

ABSTRACT

In the context of the Cigéo project, the French National Radioactive Waste Management Agency (Andra) is studying the behaviour of a deep geological facility for radioactive waste deposit in the Callovo-Oxfordian (COx) claystone. The assessment of durability of this project requires the prediction of irreversible strain over a large time scale. The mechanical interaction of the host rock and the concrete support of tunnels must be investigated to ensure the long-term sustainability of the structure. The instantaneous and time-dependent behaviour of the claystone-concrete interface is experimentally investigated with direct shear tests and long-duration shear tests of a few months. The mechanical and structural state of the claystone which is affected after interaction with concrete reflects to the response of the claystone-concrete interface, and thus different types of COx claystone-concrete interfaces are tested. The delayed deformation of the interface is found to be linked to the level of the normal loading and the loading history, while a different response of the interface was observed from the short- and long-duration tests, indicating a possible progressive modification of interface under long-duration loadings.

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1. Introduction

In France, the French National Radioactive Waste Management Agency (Andra) is studying the long-term performance and safety of a deep geological repository in Callovo-Oxfordian (COx) claystone. This claystone has been considered as a potential host formation due to its favourable properties such as geological stability, low hydraulic conductivity, self-sealing properties and significant radionuclide retention capacity. The safety of the excavated claystone tunnels during the operation phase is reinforced with construction of concrete supports. The sealing of the underground repository tunnels will be ensured by a bentonite barrier mechanically maintained by a concrete plug, either directly cast on the host rock or with prefabricated concrete blocks. Bentonite has been chosen for its swelling behaviour upon resaturation, which will apply a radial pressure closing the voids in the excavation damaged zone (EDZ) around the tunnel in the COx claystone. In order to develop this radial pressure, the bentonite will be confined by two low pH concrete plugs, as illustrated in Fig. 1 (La Vaissière et al., 2015; Armand et al., 2017a). The choice of a low pH concrete for the lining and retaining of the tunnels ensued from the need of a highly resistant concrete in a long-term scale, with a reduced thermo-chemical reaction with the hosting claystone, low heat production and elimination of the alkaline attack to the COx claystone (Codina, 2007).

The design of the tunnels support and their sealing requires consideration of complex interactions and hydro-mechanical processes between different materials, i.e. swelling pressure of the sealing, resistance of the concrete plugs/linings, creep, or chemical interactions. The application of an axial force with the swelling of bentonite plug will activate the host rock-tunnel concrete interface in shear, the short- and long-term response of which this work aims to investigate.

The presence of a discontinuity can affect in a significant way the mechanical behaviour of a structure. The comprehension of the joint behaviour starts with laboratory-scale experiments, most commonly, with shear tests on "single-jointed" samples. The laboratory analysis of the behaviour of rock joints started in the 1960s.

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Peer review under responsibility of Institute of Rock and Soil Mechanics, Chinese Academy of Sciences.

https://doi.org/10.1016/j.jrmge.2019.09.001
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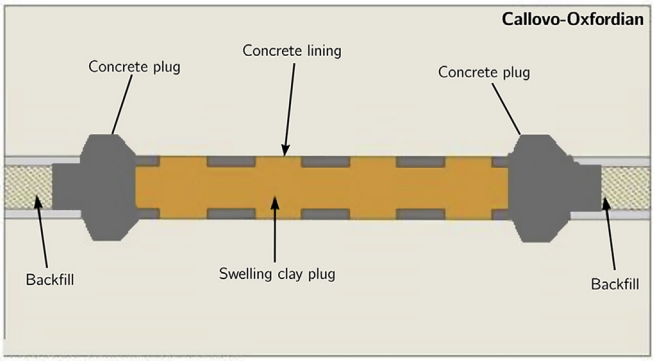

Fig. 1. Concept of disposal cell for high level waste-long lived (HLW-LL).

Authors like Patton (1966) and Ladanyi and Archambault (1969) determined proposed criteria which link the shear strength and the applied normal stress. Barton (1973) was of the first who determined a criterion to connect shear strength with normal stress, involving explicitly a coefficient related to the morphology of the joint.

Since Barton et al. (1985), several constitutive models have been developed in order to describe the behaviour of joints in geomaterials on various direct shear paths (Leichnitz, 1985; Gens et al., 1990; Samadhiya et al., 2008). However, the predictability of any model depends on the quality of tests performed in laboratory, taking into account possible scale effects (Pinto da Cunha, 1993). In the literature, several experimental approaches have been developed for the short-term study of various types of interfaces under different loading conditions (static, dynamic, cyclic). Desai et al. (1985) employed a direct shear apparatus for displacement-controlled cyclic testing of concrete-sand interfaces. A modified direct shear apparatus was used by Fioravante et al. (1999), allowing shear tests under constant normal stiffness which can be considered as an intermediate situation between traditional constant normal load (CNL) and constant normal displacement (CND) tests. Boulon (1995) introduced a three-dimensional (3D) testing device (BCR-3D). This testing device consists of three independent orthogonal axes, i.e. one normal to the joint and two in shearing directions, which is able to apply shearing under static or dynamic loads.

However, for predicting a tentative life-time of nuclear waste storage facilities, investigation of the delayed response is of equal importance. The different interfaces between the structure and the hosting formation can greatly affect the durability of such structure, even more given the complex couplings that govern the interacting materials. This paper aims to investigate the delayed behaviour of the claystone-concrete interface through a proposed new methodology including a series of short- and long-duration shear tests. Three different types of COx claystone-concrete interface have been studied, in an effort to depict the possible in situ types of such interfaces: (i) liquid concrete cast directly on the claystone – lining concrete, (ii) prefabricated saturated concrete (solid phase) in contact with the claystone – prefabricated concrete plugs, and (iii) prefabricated dry concrete (solid phase) in contact with the claystone – not directly representative of the in situ conditions, but considered herein in order to examine the decoupled response of two materials without water interaction.

For an accurate interpretation of the response of the interface, one should take into account that the mechanical behaviour of an interface obviously depends on the materials composing it. Therefore, before the presentation of the current experimental work, a section is devoted to a short bibliographic review on the different literature findings on the properties of the two interacting materials, i.e. the COx claystone and the low pH concrete.

2. Mechanical behaviour of the interface materials

Andra has been working over the last two decades on the characterisation of the hosting claystone and different materials involved in the construction of the underground repository, through a large number of experimental and numerical studies in cooperation with different research laboratories. In the following paragraphs, some of the most important findings on both short- and long-term behaviour of the claystone and the low pH concrete are listed, taking into account different couplings governing the response of each material.

2.1. Short-term behaviour

The thermo-hydro-mechanical (THM) behaviour of the COx claystone has been investigated with numerous in situ large-scale experiments and laboratory tests on core samples and its different properties have been detailed (e.g. Lenoir et al., 2007; Mohajerani et al., 2011; Armand et al., 2014, 2017b; Wang et al., 2014; Menaceur et al., 2016). The mechanical response of COx claystone is strongly related to its water content, exhibiting lower macroscopic strength and more ductile response for higher water content. Escoffier (2002) observed a higher Young's modulus not only for lower water content, but also for lower carbonate content. Other authors (e.g. Robinet et al., 2012) related the effect of water content, and more precisely drying and wetting processes, to the modification of microstructure in clayey rocks. Wang et al. (2014) studied the evolution of clayey rocks under hydric cycles and observed nonlinear deformation at high relative humidity, related not only to damage, but also to the nonlinear swelling of the clay mineral. A number of experimental works (Chiarelli et al., 2003; Homand et al., 2006) have been performed on partially saturated conditions, taking into account the presence of inclusions in the clay structure, such as smectite, which is very sensitive to water content and triggers swelling at higher relative humidity. An increase of temperature leads to thermal expansion of the clay matrix under drained conditions (Mohajerani et al., 2014), while under undrained conditions, temperature increase leads to more ductile response of lower strength (Zhang, 2018).

The mechanical response of concrete depends similarly on various factors, such as mineralogy (both in terms of granulates and cement), water content or curing period. Taking into account the coupled behaviour of the COx claystone, Andra launched a series of experimental campaigns for high long-term resistance concrete (uniaxial compressive strength (UCS), $R_c > 70$ MPa) that would exhibit low heat production and shrinkage (<500 μm/m), but also minimise the chemical interactions with the hosting rock (Leung Pah Hang, 2015). Brue et al. (2012) performed a series of uniaxial compression tests on the Andra concretes (CEM I and CEM V base) showing a clear dependency of the measured initial value of Young's modulus with respect to the degree of saturation. Vu et al. (2009) showed the influence of the water/cement ratio on the obtained volumetric strain, on samples with similar granular consistency. In order to minimise the effect of the presence of concrete and in particular the high pH value of the pore solution of a Portland-based cement on the confining properties of bentonite, a study of "low pH" concretes has been undertaken (Codina, 2007). A low pH pore solution is chosen in order to eliminate the alkaline attack to the clayey components; a swelling reaction occurs over time between highly alkaline cement paste and non-silica of aggregates, given sufficient moisture. Xi et al. (1997) observed, after 14 d of hydration, a decrease of the pH value of a studied binder

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consisting of CEM I cement, slag and silica fume, due to the capacity of the slag binders to decrease the alkali of the interstitial solution. The finally selected low pH concrete was of a TL formulation – with "L" standing for Laitier in French – based on ternary (T) binders (cement, silica fumes and blast furnace slag), exhibiting a compressive strength higher than 70 MPa after 90 d of casting.

2.2. Long-term behaviour

The time-dependent behaviour of the two geomaterials, i.e. claystone and concrete, has been studied using creep tests. Similarly to the instantaneous response, creep depends not only on the material properties (intrinsic parameters: mineralogy, porosity, water content), but also on the applied test conditions (extrinsic parameters: applied stress, strain rate, temperature, relative humidity).

In the case of the COx claystone, Zhang and Rothfuchs (2004) showed that creep behaviour under an increasing compressive load is characterised, first, by a transient phase with decreasing rates governed by strain hardening, followed by a second phase with a constant rate after strain recovery. The same authors pointed out that in uniaxial creep tests, the creep rate varies very slowly and linearly with stress at low stress levels (below 13–15 MPa), whereas above this stress level, the creep rate increases in a nonlinear form, involving possible damage. The importance of the initial water content on the long-term behaviour of the COx was highlighted already by Ghoreychi (1997). Gasc-Barbier et al. (2004) performed a series of long-duration triaxial tests on COx claystone samples, under different deviatoric stresses (from 2 MPa to 20 MPa), and found that after 2 years of constant loading, the strains did not stabilise and the strain rate seemed to remain constant. Several long-term triaxial tests were performed under different levels of applied axial stress; the measured volumetric strain from all creep tests revealed a contractant response of the claystone, while no failure (tertiary creep) occurred to any of the performed tests (Armand et al., 2017b).

Fewer studies have been performed on the long-term characterisation of the low pH concrete. Leung Pah Hang (2015) performed long-duration tests on low pH concrete samples 90 d after casting, and the results were compared to those obtained from creep tests on high-performance concrete conducted by Ladaoui (2010). The tested samples underwent two types of curing: endogenous (no water exchange with the environment), and humid (eliminating auto-desiccation effects). The different curing modes seem to have a low impact on the delayed response of the two low pH formulations, in favour of the desired in situ requirements.

The prediction of long-term performance of radioactive waste disposal facilities is of great importance; however, the study of the different individual materials involving in its construction is not enough. Even though the mechanical properties of the materials involved (claystone and low pH concrete) are reasonably known, there have been only a few studies on the mechanical behaviour of the contact interfaces between these materials. The time-dependent behaviour of these interfaces is a subject that has rarely been reported in the literature. The simulation of the long-term response in large-scale problems requires the study of the behaviour with tests during at least some months or even years. The production of experimental results is thus the first step towards large-scale modelling. In this paper, a new methodology is presented, involving a series of laboratory-scale tests on the interfaces between the underground concrete gallery and the hosting claystone (COx): an initial series of direct shear tests, the results of which are used for the design of a series of long-term shear tests with the recently developed creep device SInC Box (Stavropoulou et al., 2017). The creep mechanisms of the interface are investigated with shear tests under different loadings and the results are discussed.

3. Short-term behaviour of the interface

The short-term mechanical behaviour of the claystone-concrete interface is studied with a series of direct shear tests under different boundary conditions and different interface samples. The objective of this campaign is to determine the failure response of the interface taking into account possible influence factors, depending on either mechanical (boundary conditions) or physical (type of interface) parameters. The definition of a failure envelope is indispensable for the design of the later long-duration shear tests.

Taking into account the coupled hydro-mechanical behaviour of the COx claystone, three types of interface samples were prepared and tested in shear, by varying the hydric state of the *in contact* low pH concrete. Fig. 2 shows the three types of claystone/concrete interfaces that have been considered:

(1) COx-fresh concrete interface: low pH concrete has been cast directly on top of a cylindrical claystone sample;
(2) COx-prefabricated saturated concrete interface: a cylindrical concrete sample, after 28 d of curing under humid conditions, has been placed on top of a cylindrical claystone sample of the same diameter; and
(3) COx-prefabricated dry concrete interface: a cylindrical dried concrete sample has been placed on top of a cylindrical claystone sample of the same diameter.

The claystone cores delivered by Andra are sealed into special packaging cells. The so-called T1 cells are intended to maintain a state of stress on the core to avoid core desaturation and micro-cracking (Conil et al., 2018). The claystone core (diameter $d = 78$ mm) is cut into cylindrical samples ($d = 78$ mm, and height $h = 50$ mm) of a smooth surface with a circular saw, shortly after its opening in order to avoid water loss to the possible extent. The water content of the claystone cores has been measured in the range of 6.15%–7.14%, i.e. within the range of total saturation (Armand et al., 2014). However, the sawing of the samples causes some water loss at least at the claystone's surface. Given the sensitivity of the claystone to drying or wetting (Wang et al., 2014), a partial drying of the claystone sample during preparation might affect its mechanical response. However, this condition can be considered as a representative in situ case where ventilation takes place in some parts of the excavated tunnels, causing drying at the surface of the exposed claystone face.

The interface sample is sealed in two metallic half shear boxes, as described in Stavropoulou et al. (2017). The claystone sample is placed in one half shear box (lower) with a 5 mm exceeding surface. This exceeding surface is laterally protected by two plexiglas plates of 10 mm in thickness, on top of which the second half shear box (upper) is then fixed. According to the type of interface, the next step where the concrete is introduced might vary. In the case of a COx-fresh concrete interface, the fresh concrete is simply poured on top of the free COx surface, filling up the upper half shear box. When a prefabricated concrete is used (either saturated or dry), the concrete sample (same dimensions as the claystone sample) is placed on top of the free COx surface and is then sealed in the upper half shear box with mortar. In all cases, the claystone sample is then similarly sealed in its half shear box. The interface samples are left to curing for 28 d, a typical curing time period for concrete, and more in specific, the minimum amount of time that the used low pH concrete needs in order to reach a 70 MPa compressive strength. During the 28 d of curing, no humidity exchange with the

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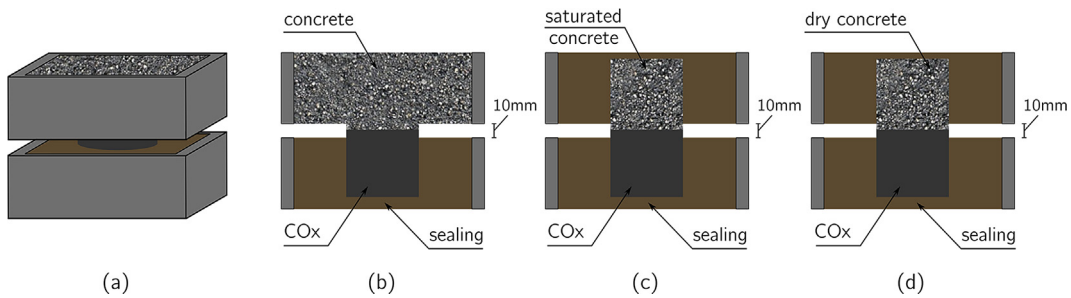

Fig. 2. (a) 3D aspect of the interface sample; (b) COx-fresh concrete interface sample; (c) COx-saturated concrete interface sample; and (d) COx-dry concrete interface sample.

environment is supposed to take place, since the interface core is sealed laterally with the plexiglas plates. Finally, the removal of the plexiglas plates just before the beginning of the shear test leaves free claystone-concrete interface of 10 mm in height.

All shear tests were performed using the BCR-3D shear box in 3SR Laboratory in Grenoble (Hans and Boulon, 2003). This device is composed of three orthogonal loading axes, each one independent of the others: a normal (Z) and two parallel (X and Y) to the plane axes of the interface (Fig. 3a). All three axes can be controlled in force or displacement, allowing application of shear under various boundary conditions. In all cases, shearing is applied as a result of symmetrical and opposite displacement of the two walls of the interface (upper and lower), reassuring a normal loading always centred on the joint's active part and at the same time preventing any relative rotation (Fig. 3b).

For characterisation of the interface, shear tests under CND or CNL are performed. In this work, "CNL" corresponds to constant normal stress conditions, however, a "CNS" test refers to a "constant normal stiffness" test and thus, the term "load" is used. For each type of interface and each type of boundaries, three samples are tested under a different initial normal stress, which either is maintained constant during shearing in the case of CNL ($\sigma =$ constant) or can vary when a surface is not perfectly smooth with breakage of existing asperities in the case of CND ($u_n = 0$). Once the normal loading is applied, the interface sample is subjected to a cycle of shear ($\pm$4 mm). For each direction of shear, a maximum shear stress (τ_{max}) is measured, allowing calculation of a Mohr-Coulomb failure envelope.

The failure envelopes of a claystone-fresh concrete and a claystone-dry concrete interface under CNL are plotted in Fig. 4, in blue and green colour, respectively. One can immediately notice a significantly lower shear strength response of the claystone with the fresh concrete interface. This result involves the sensitivity of the unconfined claystone to water interaction. As shown by Pellet et al. (2013), a saturated COx–COx interface exhibits a lower shear response than a non-saturated one. In the case of a claystone-fresh concrete interface, free water of poured concrete passes to the unconfined claystone, introducing a large difference in water gradient and creating cracks, and eventually damage throughout its volume (Stavropoulou et al., 2019). This cracking response of the claystone is not directly representative of the in situ conditions from a stress state point of view, where the claystone is under compressive stresses. However, during operation phase where tunnels will be excavated and ventilated until their final closure, cracking might occur at least in the few first centimetres of the exposed claystone. This possible response could occur, not only because of the released stress conditions normal to the tunnel wall, but also due to a difference in water gradient in the ventilated and thus desaturated claystone surface when fresh concrete is cast. In the case of a COx-dry concrete interface, no water interaction between the two materials is involved – if there is an inverse interaction of water from the claystone to the dry concrete, it occurs in a much more moderate way – and thus, no cracking occurs in the claystone, resulting in a higher shear resistance.

The Mohr-Coulomb failure envelopes of a claystone-fresh concrete and a COx-prefabricated saturated concrete interface sample,

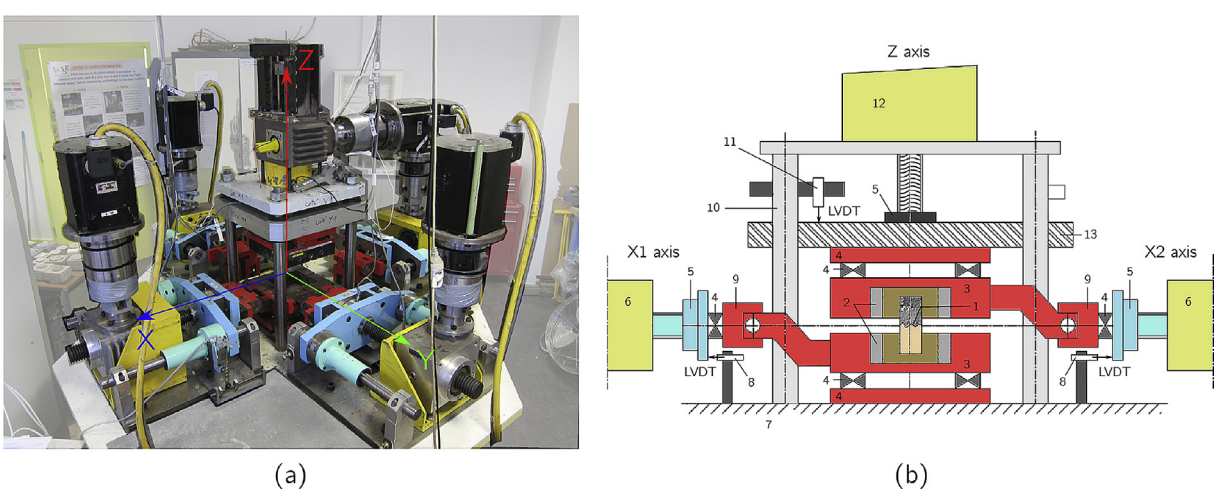

Fig. 3. (a) The BCR-3D shear device with the three principal loading axes (X, Y and Z); and (b) Front view section along one shear axis of the BCR-3D (Armand, 2000). 1 - interface sample; 2 - internal removable shear boxes (sample); 3 - external fixed boxes; 4 - rail allowing translation of the boxes; 5 - load cells; 6 - horizontal actuators; 7 - rigid base; 8 - displacement transducers (LVDT measuring dX1 and dX2); 9 - kneecaps; 10 - rigid fixed columns; 11 - displacement transducer (LVDT measuring dZ); 12 - vertical actuator; 13 - rigid vertical plate free to move along Z axis.

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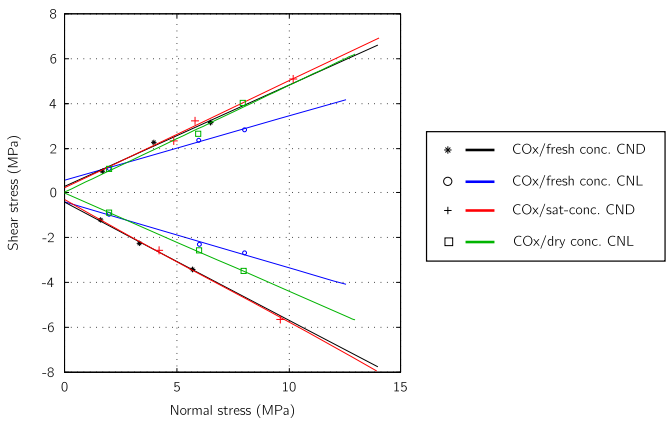

Fig. 4. Mohr-Coulomb failure envelopes for each tested type of claystone-concrete interface during the first cycle of shear, with fresh (COx-fresh concrete), saturated (COx-prefabricated saturated concrete) and dry (COx-dry concrete) interface samples, under CNL or CND conditions.

for shearing under CND, are plotted in Fig. 4, in black and red colour, respectively. It is first of all worthy to notice the almost identical shear response between these two types of interfaces tested under the same boundary conditions. This behaviour reveals the significance of the water presence to the exhibiting resistance of the interface in shear. In both cases, the free water passing from the concrete to the claystone provokes progressive damage to the claystone during the curing period of the entire interface sample and at the end, the two types of interfaces present a similar shear strength. In all cases where water was transferred from the concrete to the claystone, a layer of claystone was found attached on the concrete after the end of each shear test, as shown in Fig. 5.

In the performed tests, the same type of interface – COx-fresh concrete – exhibits a lower shear resistance in terms of friction angle under CNL than that under CND. This is, first, due to a not-any-more smooth failure interface with cracking of the claystone when in contact with the liquid concrete. The creation of cracks, the appearance of which starts very soon after the casting of the concrete, results in not only the creation of roughness and eventually a less "matched" interface from a geometrical point of view, but also a negligence of any apparent cohesion from a mechanical aspect. When a "mismatched" or rough interface is sheared under constant normal stress, the sample is free to dilate or contract. This is, however, not the case when shearing without allowing any normal displacement, where the display of contraction or dilation is restricted and the existence of created roughness adds to the shear response.

The different failure modes between the two different boundary conditions are further expressed in terms of the shear response along the two opposite directions. In the case of the CNL tests, the shearing response is symmetrical, unlike the tests under CND. When shearing under CND, the more restricting boundary conditions may involve a higher elimination of existing asperities, different parts of which are activated when shearing towards different directions.

The calculated friction angle and apparent cohesion for each type of interface and for each direction of shear are presented in Table 1. Globally, all friction angles have a similar value ($\sim 25°$), except for the case of the claystone-fresh concrete interface under CNL which is found lower for the reasons as explained above. What is surprising, however, is the similar friction angle of the COx-dry concrete interface to the ones obtained from the CND tests. Even though the response is the same, the mechanisms behind are different, where in one case (COx-dry concrete), the claystone is not damaged, and in the other (COx-fresh or saturated concrete), the claystone is damaged but the existence of roughness increases the shear resistance. It thus seems that cracking is compensated in terms of strength by the roughness increase.

Even though one cycle of shear is enough for the definition of failure envelope, each tested sample has been submitted to totally three cycles under an increased normal stress in order to further investigate the response of the interface. Fig. 6 shows the evolution of the normal stress with normal displacement of three COx-fresh concrete samples during three cycles of shear. Fig. 6a illustrates the evolution of the normal stress with normal displacement of all three samples tested under CND. Fig. 6b shows the evolution of three samples tested under constant normal stress. In both cases, there is certain variability during the application of the first normal load. This response brings in mind the representation of the normal stiffness obtained for a non-interlocked joint by Patton (1966). In the present study, roughness is not a variable taken into account since the two materials are smooth during the sample preparation. Some sort of roughness within the damaged claystone is however involved in the initial response of the interface. This can explain the variable and low initial normal stiffness measured during the closure of any voids in between the two materials and the claystone layers. Upon the application of the next two normal stresses (+2 MPa in the second cycle and +4 MPa in the third cycle), the measured normal stiffness is not varying anymore, following the response of a well matched interface.

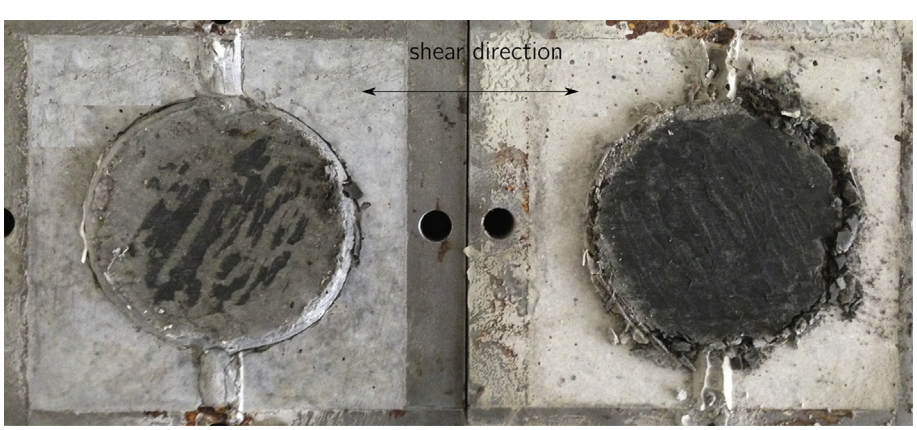

Fig. 5. Claystone-fresh concrete interface sample after a shear test under CNL: concrete (left) and claystone (right).

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Table 1
Friction angle and apparent cohesion for each type of interface in the first cycle of shear while shearing in one direction (+) and the opposite (−).

Test type	Friction angle, ϕ (°)	Apparent cohesion (MPa)
CND fresh	+24.2	+0.44
	−27.6	−0.39
CNL fresh	+17.4	+0.29
	−16.5	−0.24
CND sat.	+25.3	+0.44
	−29.2	−0.39
CNL dry	+25.4	+0.31
	−23.6	−0.38

Fig. 7 shows the evolution of shear stress with shear displacement for two COx-fresh concrete samples, each being tested under different boundary conditions (CND and CNL) and a COx-dry concrete sample tested under CNL. All three samples have been tested in three cycles of shear under a similar normal load: the first cycle under $\sigma_0 = 2$ MPa, the second under $\sigma_0 = 4$ MPa and the third under $\sigma_0 = 6$ MPa.

In all cases, the shear stiffness of the interface is measured lower, during the first application of shear, than that at the following shearing cycles. This increase in shear stiffness is expected for higher applied loadings. What is more important in this graph is the appearance of a shear stress peak at the first cycle of shear under CNL, revealing a dilatant behaviour until failure. This peak is less obvious, or even non-existent, at the following cycles. The elimination of the stress peak indicates a homogenisation of the interface with shearing and elimination of any roughness due to the damaged claystone, what is happening by definition when shearing under CND. In the case of the COx-dry concrete interface, no dilation occurs since the interface is not damaged and the flat surface presents no dilation during shearing.

4. Time-dependent behaviour of the interface

This section aims to analyse the influence of external (e.g. applied loads) or internal (e.g. water presence) variables on the time-dependent behaviour of the interfaces. The long-term behaviour of the claystone-concrete interface is investigated with shear tests of a few months duration, using the SInC box (see Figs. 8 and 9). The SInC box allows application of normal and shear loads of long duration with the aid of a hydraulic pump and a dead weight,

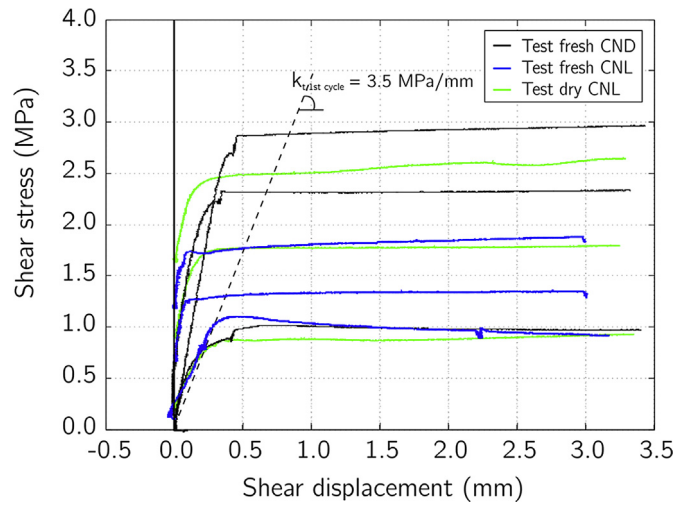

Fig. 7. Evolution of the shear stress with shear displacement for a claystone-fresh concrete interface under different boundary conditions, during the three performed cycles of shear.

respectively (Stavropoulou et al., 2017). The displacement in both directions is measured, allowing investigation of failure mode.

The interface sample is prepared and sealed in two half shear boxes, in the same way as described above for the short-duration shear tests. Three gauges measure the normal relative displacement between the two walls of the interface, each at a different point on the plane of the interface in order to evaluate any relative rotation. The relative normal displacement of the sample is then calculated as the mean value of the three obtained measurements from each gauge, i.e. $u_n = (u_{n1} + u_{n2} + u_{n3})/3$.

In the shear direction, the relative displacement is measured with an LVDT fixed in one of the two half shear boxes and in contact with the other half shear box. Three SInC boxes are operational in Laboratoire 3SR and can perform long-term shear tests on interface samples in parallel (Fig. 10) in a temperature-controlled environment of 20 °C.

Similarly to the short-term study, the interface consists of a 10 mm high free interface, 5 mm for each material (claystone and concrete). The study of the behaviour of the interface with time involves thus the mechanical response of the materials constituting it, together with a zone where their mechanical properties are

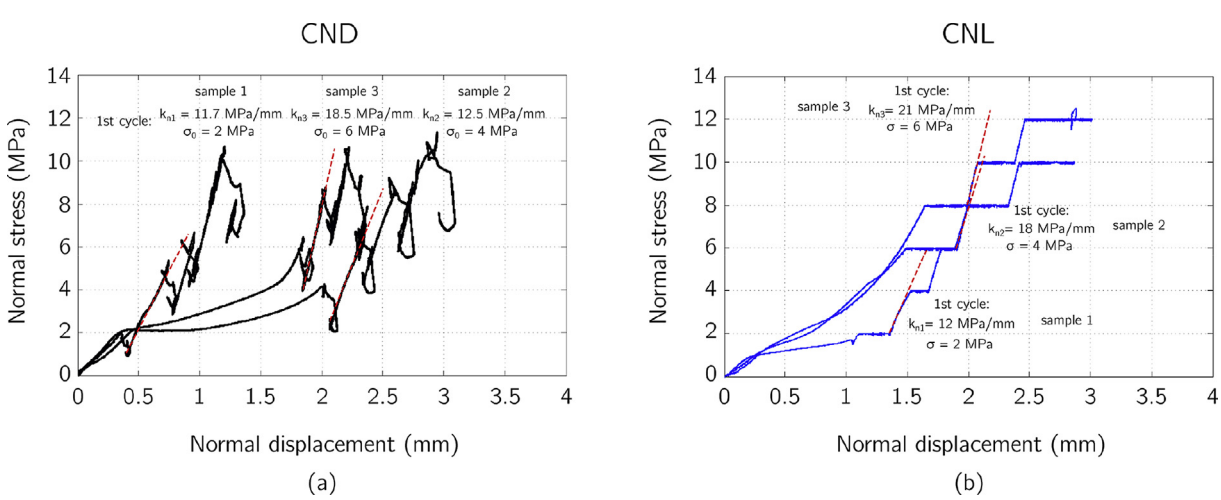

Fig. 6. Evolution of normal stress with normal displacement for three claystone-fresh interface samples tested under CND and CNL conditions.

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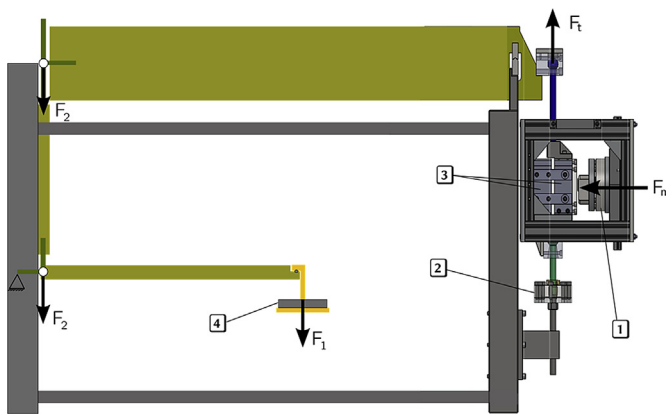

Fig. 8. Two-dimensional (2D) concept of creep apparatus. 1 - normal axis load (F_n) cell; 2 - shear axis load (F_t) cell; 3 - shear box; 4 - dead weight applying the shear load (Stavropoulou et al., 2017).

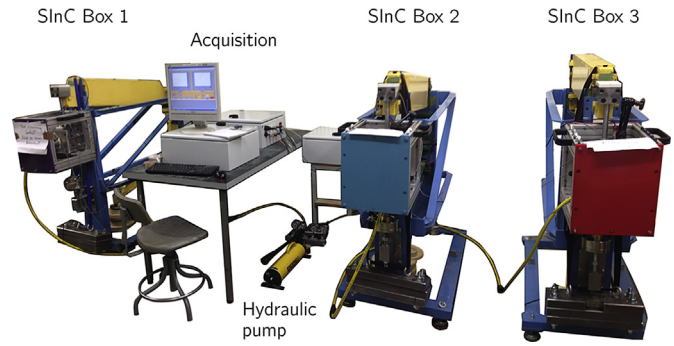

Fig. 10. Experimental room in 3SR with the three SInC boxes.

modified because of its presence. Consequently, the term "interfacial zone" is preferred. The sets of long-term experiments for characterisation of the mechanical behaviour of the claystone-concrete interface are presented and analysed as follows.

4.1. Experimental campaign and methodology

In this study, three sets of long-duration shear tests under normal stress were carried out, which are categorised according to the type of the tested interface and the applied loading. The first set of tests consists of three tests on COx-fresh concrete interface samples under a high initial normal stress ($\sigma = 12$ MPa), whereas in the second set, two tests are performed on the same type of interface under a lower initial normal stress ($\sigma = 4$ MPa). Given the similar short-term response of the claystone-fresh concrete with the claystone-saturated prefabricated concrete interface samples, only two different types are investigated in this campaign, i.e. claystone-fresh concrete and claystone-dry prefabricated concrete interface samples. The third set includes two tests on a COx-dry concrete interface under a high normal stress, similar to the first set ($\sigma = 12$ MPa). The different tests were performed in pair in order to evaluate the repeatability of the procedure. The performed long-duration tests were designed aiming to study the delayed response under the maximum expected in situ loadings developed from the swelling of the sealing bentonite (first set of tests), evaluate the influence of the level of the applied loading on the time-dependent response of the interface (second set of tests), and investigate the delayed strain of an interface possibly without any hydro-chemical interaction between the two materials constituting it (third set of tests).

All samples are submitted to an initial compression of one week before the application of shear. The aim of this procedure is to decouple the delayed response between the two orthogonal directions and facilitate the analysis of the shear behaviour. The details of each performed test are summed up in Table 2, including the type of interface, the applied stresses and the duration of each loading.

The investigation of the delayed strain of the interfacial zone in shear implies the isolation of different components constituting the interface sample. The measurement of the interface's displacements requires the subtraction of the system's displacements, including different components of the experimental setup and sealing. For this purpose, a solid intact cylindrical sample of dry concrete has been sealed in two half shear boxes, in the same way as the interface samples, as shown in Fig. 11, after protection of the sample from any water interaction with sealing. A dry intact concrete sample is assumed to present very low, almost negligible viscous strain (Ulm et al., 1999; Tamtsia and Beaudoin, 2000), especially when compared to the delayed strain when an interface exists. The removal of the interface removes the creep due to its presence (no interfacial zone), and the contribution of sealing to the delayed strain can be examined. This measurement is thus used in

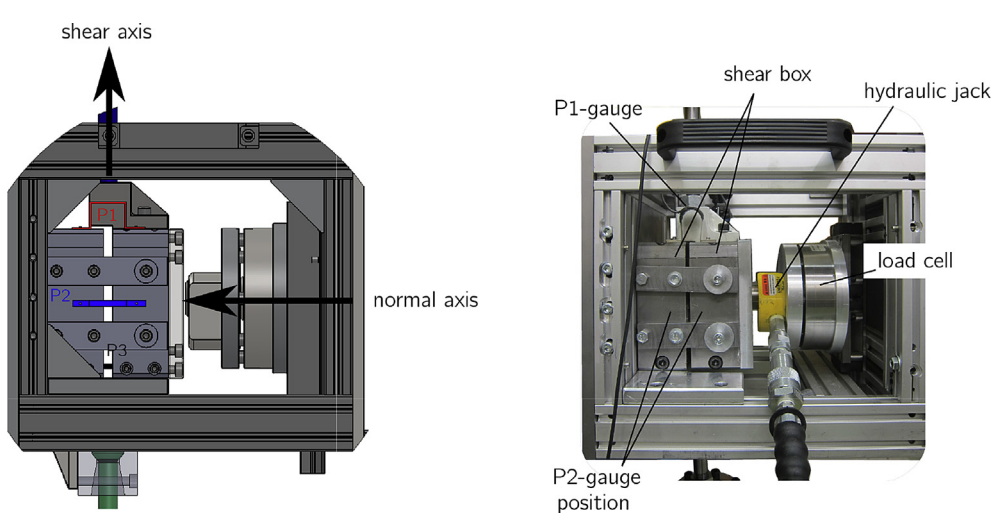

Fig. 9. Zoomed-in setup of SInC box: Digital and real illustration of the shear box (interface), the pi-shape displacement transducers, the hydraulic jack applying the normal load, and the load cell measuring it.

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order to correct the delayed strain of the sealing from the total delayed shear strain of the interface sample. Considering the test on sample 3 as reference test (the longest duration and applied stresses representative of the expected in situ stress state), the same set of stresses is applied on the intact concrete sample, i.e. $\sigma = 12$ MPa and $\tau = 7$ MPa.

Fig. 11 shows the evolution of the total delayed shear strain with time, measured on sample 3 (blue continuous curve), together with the delayed shear strain due to sealing (red continuous curve), which is fitted with a nonlinear function of time according to the Lemaitre model (Lemaitre et al., 2009). The black curve results from the subtraction of the two other, and corresponds to the corrected shear strain of the interface with time.

The fit of the measured sealing creep is expressed as

$$\varepsilon_{\text{seal.}} = aq^b t^\alpha \tag{1}$$

where q is the applied deviatoric stress; t is the time; and a, b and α are the fitted constants ($a = 0.00093$, $b = 0.85$, and $\alpha = 0.12$).

The same procedure is followed for correction of the delayed response in shear for all tests. In order to take into account the difference in applied stresses, Eq. (1) is multiplied by $(q_i/q_{\text{conc}})^b$, where q_i is the actual value of deviatoric stress and q_{conc} is the value of the deviatoric stress applied on the concrete sample. The estimated delayed strain of sealing is then expressed as

$$\varepsilon_{\text{seal.},\,i} = (q_i/q_{\text{conc}})^b \varepsilon_{\text{seal.}} \tag{2}$$

In the following, the procedure of the experimental analysis is explained in detail for the first set, considered as the reference set in this study.

Table 2
Type of interface and applied levels of stress and test duration for each creep test.

Test	Sample	Interface type	Normal stress, σ (MPa)	Shear stress, τ (MPa)	Duration (d)
1st set	1	COx-fresh concrete	12	4	28
				7	50
	2	COx-fresh concrete	12	4	41
				7	43
	3	COx-fresh concrete	12	7	122
2nd	4	COx-fresh concrete	4	2	60
	5	COx-fresh concrete	4	2	58
3rd	6	COx-dry concrete	12	4	25
	7	COx-dry concrete	12	4	37

4.2. Analysis of the reference set

The results of the first set of creep tests are listed in Table 2. Three long-duration tests have been performed on COx-fresh concrete interface samples under a normal stress $\sigma = 12$ MPa, representative of the expected underground in situ stress. The application of the shear load is based on the results of the short-term response of the interface. Taking into account the previously calculated failure envelopes, an initial low shear stress is chosen, which will later be increased towards a value approaching the maximum instantaneous shear strength.

Samples 1 and 2 are submitted to an initial shear stress $\tau_1 = 4$ MPa ($= 67\%\tau_{\max}$) for about a month, which is then increased to $\tau_2 = 7$ MPa ($= 112\%\tau_{\max}$). The application of a shear stress exceeding the previously calculated Mohr-Coulomb envelope comes after the 10-d consolidation phase, where the two parts of the interface have been undergoing a geometrical readjustment, possibly modifying its shear resistance. The variation of applied shear stress will allow the investigation of the dependence of the interfacial zone's delayed deformation on the stress level, but more importantly aims to exacerbate the creep mechanism. The third interface sample is submitted directly to a shear stress $\tau = 7$ MPa and it is left to creep in shear for four months. This last test which is of the longest duration will be used as a reference test. It is subjected to a level of normal and shear stresses which correspond to the in situ stress state.

Fig. 12a and b presents the applied normal and shear stresses on the three interface samples. As it can be seen, the applied loads decrease as the sample creeps in both directions. This is due to the fact that in the normal direction, the jack applying the load is volume controlled, and in shear direction, as the interface slides, the dead load applying the shear force changes position modifying slightly the applied force. In order to maintain the stress on the desired levels, the sample is frequently manually reloaded. Table 3 shows the values of the coefficient of variation (CV) and the mean values of the applied normal ($\overline{\sigma}$) and shear stresses ($\overline{\tau}$), from the application of the given load until the end of the test.

The measured normal and shear displacements for all three tests are presented in Fig. 13. The application of a normal load followed by a shear one 7 d after results for both directions in a first instantaneous part and then a delayed nonlinear one, the rate of the last decreasing with time. The instantaneous response due to the applied normal stress ($\sigma = 12$ MPa) leads to both a normal and a low shear displacement. The measurement of some shear

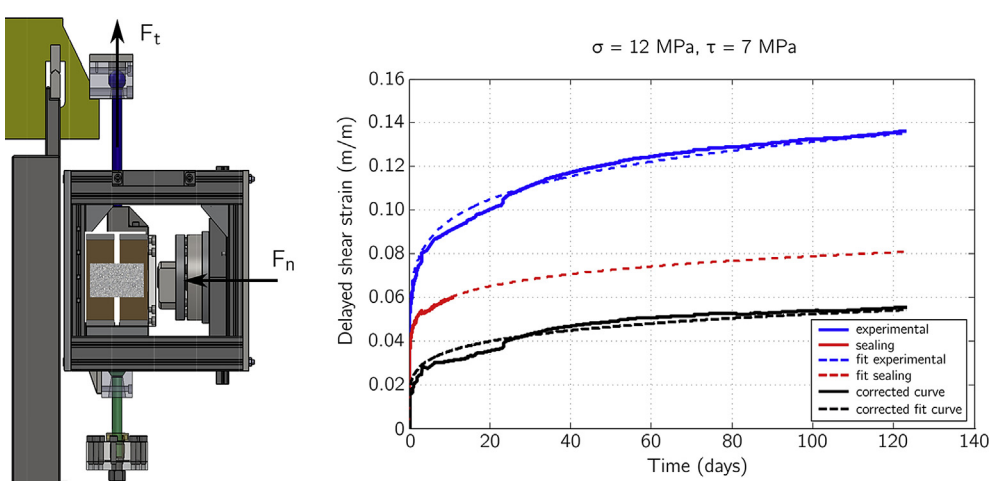

Fig. 11. Correction of the delayed shear strain for sample 3 (COx-fresh concrete interface).

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displacement during compression only can be the result of a mismatched interface, the two walls of which close with the applied normal load and the contact surface is rearranged. This small instantaneous shear displacement cannot be directly compared with the results from the short-duration tests, since the two types of tests are controlled in a different way. While the short-duration tests (BCR-3D) are performed in electronical displacement-control along the shear axis, the long-duration shear tests (SInC box) are manually controlled in force along the shear axis. Regarding the instantaneous behaviour with compression itself, the response presents in both normal and shear directions a certain variability between the three samples. This variability can be explained again by the variable state of the claystone volume, containing cracks of a different amount and orientation.

The applied shear load on samples 1 and 2 leads similarly to an initial instantaneous displacement, followed by a nonlinear part. With the application of the first shear stress ($\tau_1 = 4$ MPa), the instantaneous response in terms of shear displacement is variable between the two samples. After a month of shear loading, the two samples are submitted to a higher shear load ($\tau_2 = 7$ MPa), and the instantaneous response during this second loading is no more variable. This result reveals that the phase of consolidation is not fully developed on both normal and shear directions during the initial application of the normal load. The application of the first shear load results in shear strain and further consolidation along the shear axis, involving closure of possibly still existing voids in the claystone – this time normal to the shear direction which had not been activated during compression. The first applied normal and shear loads contribute to the increase of uniformity of the interfacial zone, from a possibly "mismatched" one to a better matched, reducing the variability of the instantaneous response when the second shear load is applied.

4.3. Delayed behaviour of the interface

In this section, the delayed response of all three sets of tests (Table 2) is exploited, separate from the instantaneous one. Similarly to the first set of tests, the second set is performed on two COx-fresh concrete interface samples, i.e. samples 4 and 5. In order to examine the influence of the applied loading, a lower normal stress $\sigma = 4$ MPa is used, followed by the shear stress $\tau = 2$ MPa, and the samples are left to creep for a couple of months. The value of the applied shear stress is based on the calculated Mohr-Coulomb envelopes, and corresponds to $95\%\tau_{max}$. The last set of shear creep tests is performed on a COx-dry concrete interface, aiming to investigate the effect of the damaged claystone on the delayed response of the interface in shear. Samples 6 and 7 are submitted to a normal stress $\sigma = 12$ MPa and a shear stress $\tau = 4$ MPa, similar to samples 1 and 2, for a period of one month.

Fig. 14a presents the corrected delayed shear strain, for all the creep tests performed on a COx-fresh concrete sample. For better illustration of the results, all curves have been fitted based on the Lemaitre model as shown in Fig. 14b. As expected, the delayed shear strain is measured higher for larger shear stress for the tests under the same normal load $\sigma = 12$ MPa (black and blue continuous curves). However, the delayed response is noticeable after the increasing shear stress from $\tau_1 = 4$ MPa to $\tau_2 = 7$ MPa, in comparison with the delayed response when directly loaded to a shear stress $\tau = 7$ MPa. The application of a high initial load is supposed to accelerate the delayed response of a material; however, the delayed response between the three samples under the same applied stresses ($\sigma = 12$ MPa and $\tau = 7$ MPa) but different loading history is very similar at least for the given test duration.

Finally, if one compares the delayed response of two types of tests submitted to a similar set of stresses σ and τ close to the failure envelope, one can notice a quite different behaviour. More precisely, both tests under $\sigma = 4$ MPa and $\tau = 2$ MPa (red curves) and $\sigma = 12$ MPa and $\tau = 7$ MPa (black and blue dashed curves) correspond to a loading where $\tau = 95\%\tau_{max}$ and $\tau = 112\%\tau_{max}$, respectively, however, their position on the failure envelope is different. In the case of a test under a higher applied normal load, the measurement of a higher delayed shear strain can be explained by the appearance of phenomena involving damage of the interface materials, which does not occur, or it is less significant, under a lower normal load.

Similarly to the monotonic response, the type of sample influences also the delayed response of the COx-concrete interface. Fig. 15 shows the corrected delayed shear strain of two tests, for two different types of interface sample, i.e. COx-fresh concrete and COx-dry concrete. It is clear that the delayed response of the COx-dry concrete samples is much lower. This response is the result of the combination of two facts: first, the presence of water, which is one of the main reasons of delayed phenomena, is much lower in a COx-

Table 3
Coefficients of variation and mean values of the applied normal and shear stresses for samples 1, 2 and 3.

Sample	Normal stress, σ		Shear stress, τ_1		Shear stress, τ_2	
	CV (%)	$\bar{\sigma}$ (MPa)	CV (%)	$\bar{\tau}$ (MPa)	CV (%)	$\bar{\tau}$ (MPa)
1	2.02	12.53	1.06	4.17	5	6.86
2	1.58	11.77	1.14	4.07	4.1	6.62
3	3.8	11.83	3.9	7.15		

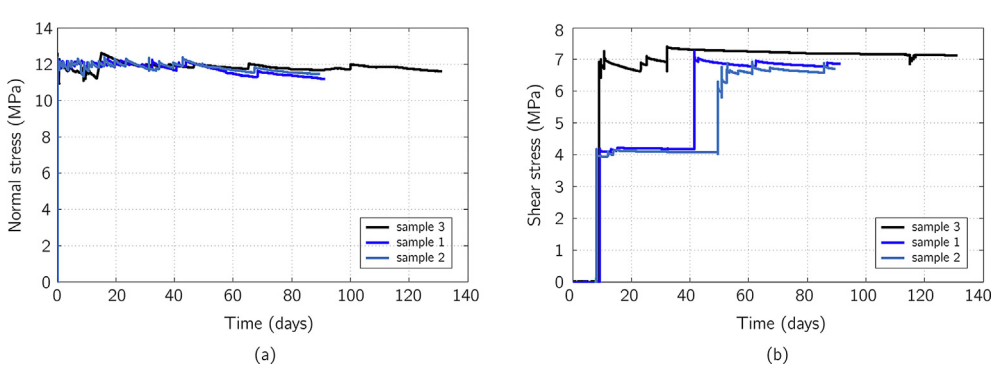

Fig. 12. Applied (a) normal stress and (b) shear stress on samples 1, 2 and 3 (COx-fresh concrete interface).

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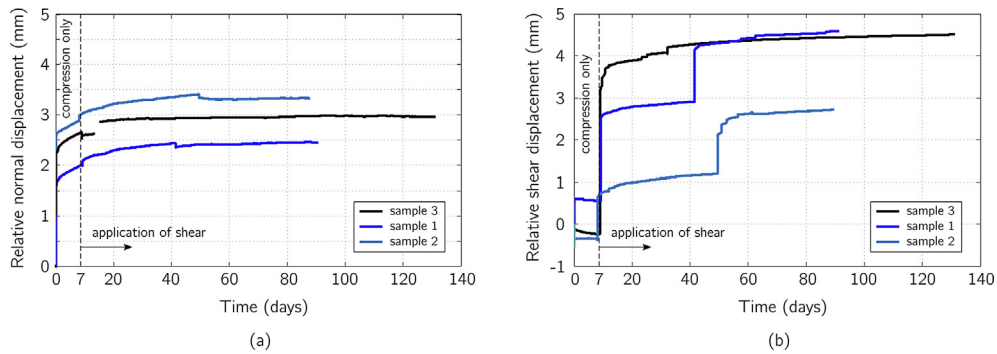

Fig. 13. (a) Normal and (b) shear displacements, measured for samples 1, 2 and 3, under $\sigma = 12$ MPa and $\tau = 4$ MPa and 7 MPa.

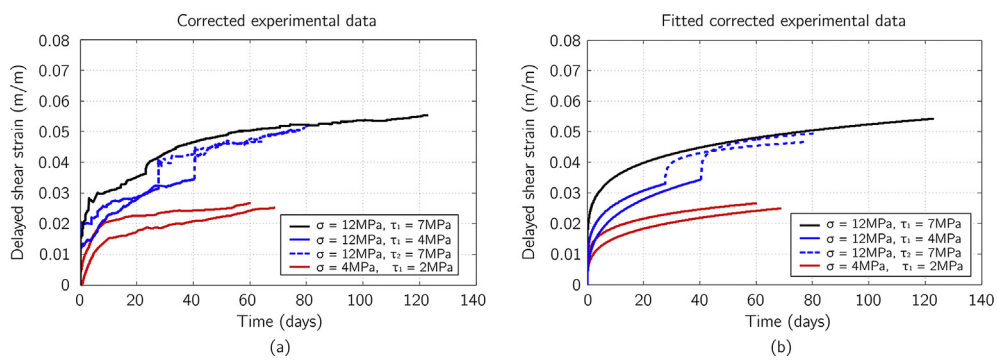

Fig. 14. Delayed shear strain of COx-fresh concrete interface samples: (a) Raw experimental data, and (b) Fitted experimental data.

dry concrete sample, but also the claystone is expected to be less damaged in the absence of its interaction with water. A damaged claystone involves the notion of localised stress at the level of the cracks, which adapts with time and adds to the total measured delayed strain.

Both types of samples deform nonlinearly with decreasing rate. However, the measured data (Figs. 14a and 15a) do not allow a clear distinction of any creep rate variation, due to periodical reloading of the samples during the test. In Fig. 16, both the non-fitted and fitted experimental results of the measured creep rate in shear are plotted for two tests on COx-fresh concrete and two on COx-dry concrete interface samples under the same stress state ($\sigma = 12$ MPa and $\tau = 4$ MPa). While the non-fitted results show a similar creep rate for the two types of samples, the fitted curves reveal a clear rate difference. More precisely, the COx-dry concrete samples creep under lower rates – of around 10 times lower – revealing a dependence not only of the actual delayed strain, but also the creep rate in the presence of water and/or the damage of COx claystone. Other authors have previously suggested that the pore water may be a key factor controlling creep of clay (Rutter, 1983; Zhang and Rothfuchs, 2004), which results from the deformation of intergranular water films rather than solid grains. This claystone microscale assumption could contribute to the interpretation of the delayed response of the COx-fresh concrete interface. However, the measured delayed strain of the interface cannot be deduced as the sum of the delayed response of the two materials constituting it. Indeed, the delayed strain of concrete has been measured lower by an order of magnitude 10^3 (Leung Pah Hang, 2015) and that of the claystone lower by an order of magnitude 10^2 (Armand et al., 2017b). In the case of the interface, the role of the water coming from the concrete is more importantly related to the creation of cracks in the claystone, the existence of which

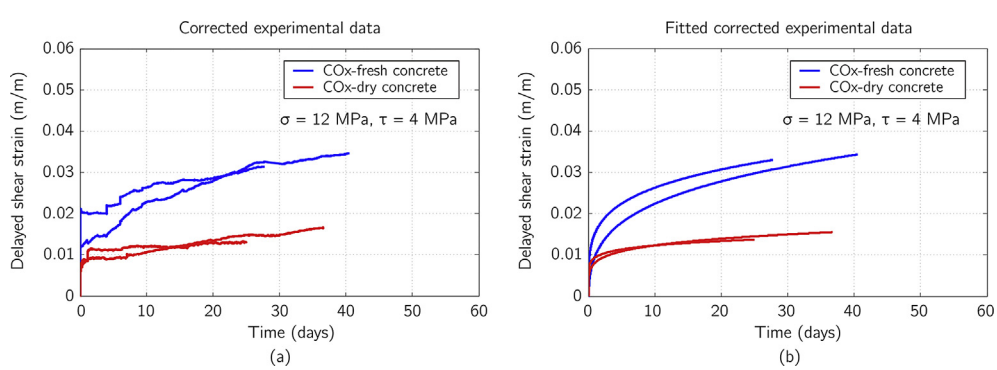

Fig. 15. Delayed shear strain of COx-fresh concrete and COx-dry concrete interface samples under the same applied stress: (a) Raw experimental data, and (b) Fitted experimental data.

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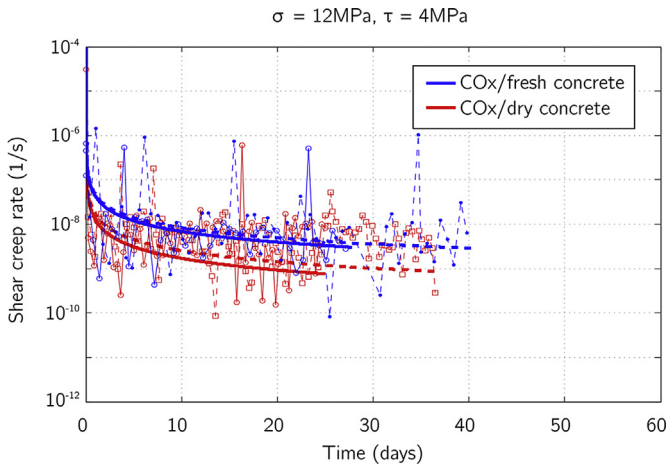

Fig. 16. Delayed shear strain of COx-fresh concrete and COx-dry concrete interface samples under the same applied stress.

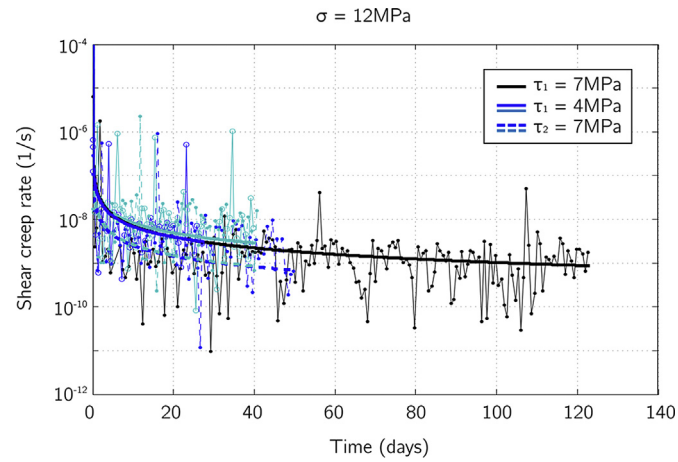

Fig. 17. Delayed shear strain of COx-fresh concrete interface samples.

dominates the delayed response in comparison to the undergoing microscale phenomena within the structure of the intact material. On the other hand, a COx-dry concrete interface does not involve water interaction and thus any undergoing viscous mechanisms are not overlapped by the delayed strains due to cracking closure or rearrangement.

Fig. 17 shows the measured shear creep rate for the COx-fresh concrete samples, loaded under the same normal stress but different shear stresses. The delayed response between the COx-fresh concrete samples in terms of creep rate does not seem to be affected noticeably by the difference in the initial applied shear stress. All tests, under $\sigma = 12$ MPa, exhibit creep under a similar rate for $\tau = \tau_1 = 4$ MPa or 7 MPa. On the other hand, it is likely that the loading history affects the creep rate, i.e. lower creep rates are measured for the same applied shear stress when $\tau = \tau_2$. This is, however, to be confirmed with more results.

During a constant stress test, three phases may be observed along one direction: a primary creep phase, during which the strain rate decreases over a long period; a secondary phase, during which the strain rate remains constant; and a tertiary phase, which is described by an increase in strain rate due to the occurrence of progressive damage. During all tests, the delayed shear strain has a nonlinear evolution with a decreasing rate. This indicates a creep behaviour within the primary phase for the given test duration. This is interesting, if one considers that the applied stresses in some creep tests are higher than the measured short-term shear strength. The sample should normally fail (secondary and tertiary phases) under such high applied stress state, but it does not. On the contrary, the creep mechanism, even though it does not stabilise during the entire duration of each test, is always of a decreasing rate with time, with no indication of potential failure for the given test duration.

This peculiar response indicates a mechanical and even a physical evolution of the loaded interface with time. The reason behind this behaviour can be stiffening and strengthening of the interface while the existing cracks of the damaged claystone close and water equilibrium between the two materials is reached with time. The compaction occurred at the application of a normal load during a long time period might be responsible for the creation of more and better bondings between the two geomaterials, introducing more contact points and a better interlocking of the joint before the application of shear. This hypothesis suggests that the response of the interface after a first initial compaction period is modified, approving the contrast between the short- and long-term results.

5. Conclusions

The long-term behaviour of the COx-concrete interface has been studied using a new proposed methodology. A first series of instantaneous shear tests allowed the definition of a failure envelope of the interface, which has been taken into account for the design of the later performed long-duration tests.

The type of COx-concrete interface and thus the interaction of the claystone with free water of the concrete have been found to have a significant impact on both short- and long-term deformation and strength of the interface. As expected, the level of the applied stress influences the delayed shear strain of the interface samples significantly. For the given test duration, this is not the case with the loading history on the shear direction; even though it has a clear impact on the instantaneous response, its significance is less strong on the delayed shear strain. However, the influence of loading history is indeed not negligible in the time-dependent response of the interface, since it is depicted by the delayed strain rates, expressed by higher rates on samples previously submitted to long-duration loadings.

No failure in shear has been obtained during the test period of each sample (1–4 months), even though several samples have been submitted to loads exceeding the calculated instantaneous shear strength. The obtained results suggest different responses when sheared under long-duration loadings. The application of an initial long-duration load seems to have an effect on the mechanical response of the interfacial zone which undergoes an initial compaction and eventually a better bonding before the application of shear. This is not the case in the short-term direct shear tests where the shear load is applied immediately after the application of the normal load, and an interface sample consisting of a cracked claystone will present lower strength.

The observed behaviour of the interface samples indicates a mechanical and/or geometrical evolution of the interfacial zone. During the application of long-duration loads, the interfacial zone is likely to evolve not only with closure of voids within the damaged claystone, but also with the creation of more contact points between the two geomaterials, and consequently, with the modification of the interfacial zone to a better inter-locked one. However, this behaviour does not imply a stabilisation of the delayed response. A potential homogenisation of the interfacial zone without remaining open cracks might involve an initial strengthening, although the geomaterials constituting it should continue to present delayed deformation. Longer duration tests are necessary for identification of this long-term shear strength, which is of high

importance for predicting the long-term behaviour of a tunnel support or seal system in an underground repository.

The creep rate in shear decreases with time after application of the shear stress, indicating a response within the primary phase of creep for the given test duration. This geometrical evolution is most likely to be correlated to the mechanical evolution of the contact surface, which is initially submitted to high stresses locally at the walls of the cracks of the damaged claystone. The adaptation of the contact surface and thus of the applied stress could be the principal reason accounting for the decreasing measured strain rates.

The concept of the delayed behaviour of an interface is not developed in the literature where numerous studies are reported on intact materials, and thus any conclusions should be carefully drawn. In this work, the delayed phenomena occurring in the limited tested samples have been exploited from a macroscopic point of view, while the precise mechanisms behind the kinematics of the studied interface are still not clear, and their understanding could be improved with microscale studies.

Conflict of interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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Please cite this article as: Stavropoulou E et al., Time-dependent behaviour of the Callovo-Oxfordian claystone-concrete interface, Journal of Rock Mechanics and Geotechnical Engineering, https://doi.org/10.1016/j.jrmge.2019.09.001

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Please cite this article as: Stavropoulou E et al., Time-dependent behaviour of the Callovo-Oxfordian claystone-concrete interface, Journal of Rock Mechanics and Geotechnical Engineering, https://doi.org/10.1016/j.jrmge.2019.09.001