

A CONSTITUTIVE STUDY OF THERMO-ELASTO-PLASTICITY OF DEEP CARBONATIC CLAYS

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SUMMARY

Argillaceous masses considered for potential nuclear waste repositories may exhibit significant space variability in their carbonate content. This may affect mechanical clay properties, such as strength or maximum apparent preconsolidation stress known to strongly depend on carbonate content. This paper investigates experimentally the dependence of thermo-hydro-mechanical behaviour of clays on carbonate content. The properties investigated are thermal strains, thermally induced over-consolidation, strength changes, destructuration, and thermally developed water pressure in undrained conditions. The experimental data are analysed in terms of a thermo-elasto-plasticity theory for clays, being an extension of Cam-clay model, modified to include the variability of the carbonatic content. © 1998 John Wiley & Sons, Ltd.

Key words: carbonatic clays; thermo-plasticity; nuclear waste disposal; thermo-mechanical tests

INTRODUCTION

This paper brings together two issues of importance to the design of nuclear waste repositories in natural clay formations: thermo-mechanical behaviour of clay and the effect of carbonates on it. Carbonate content is one of the most important mineralogical characteristics of clay soils known to affect their mechanical properties. This may be observed within the same formation, when samples retrieved from different depths or locations, exhibit correlated variations in carbonate content and in basic mechanical properties.¹

Classically, for medium to strongly overconsolidated clays, the peak strength and the maximum oedometric pre-compression vertical stress are empirically linked to soil specific volume, or void ratio. On the other hand, the same characteristics are linked to the content of carbonates.^{2,3} The dependence of the pre-compression stress on the void ratio is exponential, as implied by most volumetric hardening theories. The maximum pre-compression stress dependence on carbonates content is also non-linear, but no consensus exists on the applicable relationship. McKown and Ladd⁴ suggest the existence of an upper threshold value of carbonates content, above which an

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increase of carbonates does not seemingly affect the strength of interparticle cement bridges, but rather results in the filling of the interparticle voids.

The presence of carbonates is one of the elements contributing to the phenomenon known as destructuration. Destructuration is a term describing a reduction of strength and increase in deformability of natural clays as a result of deformation beyond the initial yield limit in the hardening range, as proposed by Leroueil *et al.*⁵ According to this concept most natural materials develop a 'structure' as a result of suspension aggregation, sedimentation, primary consolidation, secondary compression (or creep), thixotropy, cementation, periodical wetting and drying, freeze and thaw, etc. This 'structure' may be destroyed not only through a dilatant softening but also during a compressive hardening. Typically, the triaxial undrained strength decreases as a result of destructuration over about 20–40 per cent, the modulus by 50 per cent or more, the strain at failure increases by 50 per cent, and the stress–strain curve markedly flattens at the peak. It is often emphasized that destructuration is not limited to cemented clays but is characteristic for most natural clays of low to medium plasticity.⁶ Extensive analysis of the mechanical behaviour of natural clays with the emphasis on destructuration has been given by Leroueil and Vaughan⁷ and Burland.⁸ Models for destructuring soils and weak rocks were discussed by Desai *et al.*⁹ using a disturbed state concept, Gens and Nova¹⁰ and Lagioia and Nova¹¹ using a kind of isotropic kinematic softening, and by Adachi and Oka¹² using a reduced stress concept. On the other hand, when cemented clays are subjected to flow of water solutions of acids their apparent maximum pre-compression stress may substantially be reduced⁴ and their permeability increased.¹³ Thus, cementation may be perceived as a process with a rate driven either through a mechanical or chemical variable.

Clay formations of the size required for a repository of long-lived radioactive waste may often be characterized by a pronounced inhomogeneity within the formation. The inhomogeneity has an influence on both the interpretation of laboratory results and on modelling. The thermo-mechanical behaviour of deep natural clays considered for radioactive waste repositories, is assessed on the basis of a relatively low number of experiments, due to the limited number of specimens that can be extracted. The specimens often come from sites, tens to hundreds of meters distant in depth, or from boreholes drilled kilometers apart horizontally. Mineralogical content of the specimens may be different what may result in a substantial variability of physical properties such as void ratio, water content, plastic and liquid limits. The variability in physical parameters may lead to differences in the mechanical and thermo-mechanical properties of clay. In order to interpret the results of the thermo-mechanical tests, the mechanical parameters of each specimen have to be assessed on the basis of the physical properties of the specific samples. A procedure to include the content of carbonatic minerals into mechanical model has been proposed by Del Olmo *et al.*¹ The procedure is based on an experimentally substantiated dependence of uniaxial undrained strength, maximum apparent preconsolidation stress, as well as the material deformational moduli on two physical properties: content of carbonates and initial void ratio. Using this procedure the apparent maximum pre-compression vertical stress may be calculated for each sample knowing its void ratio and carbonate content.

Thermo-mechanical properties of clays have been studied for the purpose of nuclear waste disposal technologies since later 1960s.¹⁴ The principal design factor in these technologies is the temperature developing within the soil mass. During heating the pore pressure increases, generating a pore pressure gradient which may substantially increase the natural water flow. This may enhance a potential contaminant transport if an early breakdown of the inner engineered

barrier occurs. The spacing of tunnels and/or boreholes, which is a principal design parameter is determined as to control temperature and pore pressure gradients.

Thermal pore pressure excess measured in the lab in undrained isotropic conditions ranges between 1.5 and 5 MPa per 100°C.¹ At deviatoric stress the thermally induced growth of pore water pressure may lead to failure and/or hydraulic fracturation.¹⁵ Formation of such fractures may produce preferential transport paths for radionuclides during the radionuclide release phase, long after the thermal phase is finished. The pore pressure results from the difference between thermal expansion of pore water and thermal volumetric strain of the skeleton. While the pore water expansion is poorly known, the thermal strain during heating depends critically on isotropic effective stress: it is expansive and reversible for very low effective stresses, and it is contractive and partly irreversible for high effective stress.¹⁶ Water content and hydraulic conductivity are additional factors in the process. The conceptual framework for thermo-mechanical analysis is the thermo-plastic version developed earlier¹⁷ of Cam-clay model² to deal with thermal strain, possible water pressure buildup and other thermo-mechanical effects.

In this paper an interrelation between carbonate content of clays and thermo-mechanical effects is discussed for Spanish clays called SS-1 and IC-1. These clays indeed show a strong dependence of strength and maximum pre-compression stress on the initial void ratio and on the carbonate content.¹ The thermo-mechanical properties investigated are thermal strains, thermally induced overconsolidation, strength and destructuration changes with temperature. This study has a character of sensitivity analysis of the above major mechanical and thermo-mechanical properties with respect to carbonate content. The framework for the analysis is the thermo-plasticity model,¹⁷ extended to include carbonate content as briefly outlined in the next section. However, it is believed premature to offer a model for thermo-mechanical behaviour of carbonatic clays on such a small data base.

THERMO-PLASTICITY MODEL FOR CLAYS WITH A CARBONATE CONTENT

In the previous papers Cam-clay² based constitutive equations were proposed for thermo-elasto-plastic behaviour of plastic clays,^{17,16} subsequently extended for clays with cementation sensitive strength and maximum preconsolidation stress.¹ Following this approach the size of the ellipsoidal elastic domain¹⁸ depends on a dead parameter of content of carbonates, in addition to the accumulated plastic volumetric strain and temperature, as follows:

$$f = \left(\frac{p'}{p'_c/2} - 1 \right)^2 + \left(\frac{q}{Mp'_c/2} \right)^2 - 1 = 0 \quad (1)$$

where p'_c is the apparent maximum isotropic pre-compression stress, p' and q are effective mean stress and deviatoric stress invariant, and M is the critical state line coefficient related to ultimate friction angle $\phi'_u = \arcsin[3M/(6 + M)]$. The term *apparent* maximum isotropic stress is employed to reflect the fact that the yield surface was not necessarily generated through a plastic loading up to that stress, but also through cementation or thermal loading history.

The thermo-elastic volumetric strain in clays may be represented as a function of the logarithm of isotropic effective stress p' and temperature, see References 19 and 17.

$$\varepsilon_v^{te} = \left[\frac{\kappa}{1 + e_0} + (\alpha_1 + \alpha_3 \Delta T) \Delta T \right] \ln \frac{p'}{p_g} + (\alpha_0^* + \alpha_2 \Delta T) \Delta T \quad (2)$$

where α_i are constant coefficients and ΔT is temperature difference with respect to the onset of heating; p'_g is the reference isotropic stress *in situ* and κ is isothermal elastic bulk modulus at reference temperature.

Temperature dependence of the yield function is modelled through the dependence of the apparent maximum isotropic stress p'_c on thermal softening function, $A(T)$,

$$p'_c/2 = \frac{p'_{c0}}{1 + \beta} \exp \left\{ \frac{1 + e_0}{\lambda - \kappa_T} \varepsilon_v^{pl} (1 - a_0 \Delta T) \right\} + A(\Delta T) \quad (3)$$

where

$$\kappa_T = \left[\frac{\kappa}{1 + e_0} + (\alpha_1 + \alpha_3 \Delta T) \Delta T \right] (1 + e_0)$$

while ε_v^{pl} is the irreversible thermo-plastic volumetric strain, κ_T and λ are thermo-elastic and elasto-plastic bulk moduli; β (≥ 1) is the yield function shape coefficient; p'_{c0} is the initial isothermal value of the maximum past (initial) isotropic stress. e_0 is the initial void ratio of clay *in situ*. Thermal softening function $A(\Delta T)$ is always negative and constrained by the condition that $p'_c > 0$ during any thermo-mechanical loading process.

$$A(\Delta T) = a_1 \Delta T + a_2 (\Delta T)^2 \quad (4)$$

where a_1 and a_2 are constants. An additional thermal effect is thermal ductilization represented by the multiplier $[1 - a_0 \Delta T]$ in equation (3), which affects the amount of strain hardening at different temperatures. Thermal softening is uncoupled from thermal ductilization. As a result equations (1) and (3) simulate the elastic domain which shrinks with the increasing temperature, and expands during cooling (Figure 1). A database for Boom clay and identification procedures for the determination of the constitutive parameters are presented by Baldi *et al.*¹⁹ and Hueckel and Baldi.¹⁶

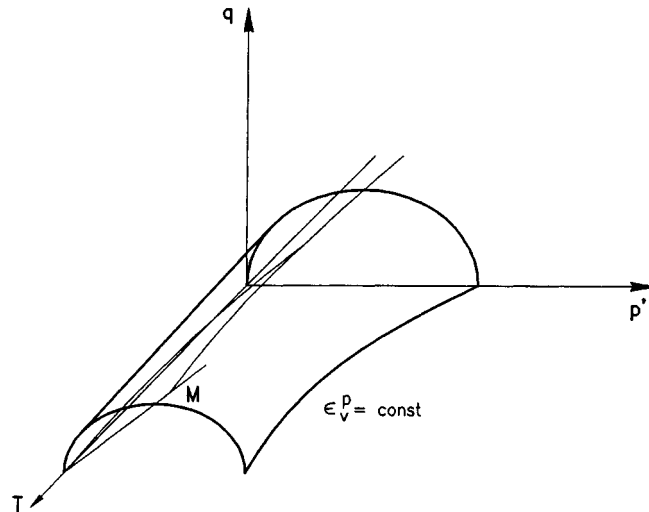


Figure 1. Temperature dependence of the yield function as modeled through the dependence of the apparent maximum isotropic stress p'_c on temperature

Modelling of the carbonate content dependence of mechanical properties of clay is limited here to bulk moduli and apparent preconsolidation stress. The carbonate content will be considered as a dead parameter, which means that it does not evolve. The carbonate content used as a parameter throughout the paper refers directly to the mass of CO_2 released in a test. It is measured by gasometric method on 1 g of oven dried material pulverized to less a # 10 sieve. The specimen is reacted with hydrochloric acid in a closed vessel (2–3 min). The measured pressure (using appropriately calibrated bourdon tube pressure gage) of carbon dioxide is proportional to the mass of gas released. It may be converted to the content of CaCO_3 using the formula: $\text{CO}_2 = 0.44 \times \text{CaCO}_3$.

The carbonate content dependence of uniaxial undrained strength of Spanish clays SS1 and IC1²⁰ manifested by its 5–7 fold variation with the carbonate content was studied by Del Olmo *et al.*¹ Assuming that a relation exists between the uniaxial strength and apparent maximum pre-compression isotropic stress through the initial yield condition (equation (1)) a relationship is found between the apparent maximum isotropic stress p'_c and the content of carbonates expressed through CO_2 content and *in situ* void ratio e_0

$$p'_{c0} = p'_0 \exp \left(\frac{-[e_0 - e_{10} \exp(-\chi C)]}{\Lambda_0 \exp(-\xi C)} \right) \quad (5)$$

where C is the mass of CO_2 released from 1 g of clay, as described above. Λ_0 and e_{10} are the values of instantaneous secant moduli $\bar{\lambda} - \bar{\kappa}$ of a non-linear behaviour during geological process and of void ratio at zero carbonate content and at p'_0 (unit pressure) taken as 0.1 MPa; ξ and χ are constants. The values of these constant for the Spanish clays have been calculated on the data base of 28 samples¹ as: $\Lambda_0 = 0.211$, $e_{10} = 1.246$, $\xi = 1.27$, $\chi = 0.0798$.

As far as the strain in thermally affected clays is concerned, it needs first to be stated that the classical Terzaghi effective stress principle for thermo-mechanically loaded clay may still be considered as valid.²¹ However, the total strain rate $\dot{\epsilon}_{ij}$ (positive in compression) needs to include a stress rate-dependent part and a temperature rate-dependent part, divided in turn into their respective reversible and irreversible parts as follows:

$$\begin{aligned} \dot{\epsilon}_{ij} = & C_{ijkl}(\Delta T) \dot{\sigma}'_{kl} - \frac{1}{3} \alpha(\sigma'_{kl}, \Delta T) \dot{T} \delta_{ij} \\ & + \frac{1}{H} \left(\frac{\partial f(\sigma'_{kl}, \Delta T)}{\partial \sigma'_{mn}} \dot{\sigma}'_{mn} + \frac{\partial f(\sigma'_{kl}, \Delta T)}{\partial T} \dot{T} \right) \frac{\partial f(\sigma'_{kl}, \Delta T)}{\partial \sigma'_{ij}} \end{aligned} \quad (6)$$

where σ'_{ij} is the effective stress. The first term on the right-hand side is the reversible (elastic) mechanical strain, and C_{ijkl} is a tensor of thermally sensitive elastic incremental compliance constants. The second term on the right-hand side is reversible thermal expansivity strain, assumed to be isotropic. It is characterized by an incremental cubic thermal expansivity coefficient α . The coefficient depends on effective stress and temperature.¹⁹ The third term on the right-hand side is the plastic strain rate. It also composes of separate stress and temperature driven parts. Associative plastic flow rule is assumed. Thus, while the elastic thermal strain is always isotropic, the irreversible thermal strain may have also a deviatoric component, depending on the stress state. H is temperature and accumulated plastic strain-dependent plastic hardening modulus

$$H = - \frac{\partial f}{\partial \epsilon_v^p} \frac{\partial f}{\partial p'} (\sigma'_{ij}, \Delta T). \quad (7)$$

General rules of such system of constitutive equations for a generic clay, as well as specific forms of constitutive functions for Boom clay and Pontida clays were described by Hueckel and Borsetto¹⁷ and Hueckel and Baldi.¹⁶ In the next sections, the mechanical and thermo-mechanical behaviour of Spanish clays SS-1 and IC-1 will be examined in the framework of the above model.

It should be noted that in the present formulation, carbonate content parameter C is considered as a function of space, but not of time. This means that while it is important to assess exactly the initial size of the elastic domain for each point in the clay mass, or for each sample, it is assumed above that the effect of carbonates does not change during any further loading (chemical or mechanical). As it will be seen in what follows, this assumption appears to be valid only in a limited class of applications.

MECHANICAL BEHAVIOUR

Two particular aspects of the mechanical behaviour of Spanish clays are addressed in this analysis, in view of their impact on thermo-mechanical behaviour. These are: the relationship between carbonate content and the peak strength or apparent maximum precompression isotropic stress, and destructuration. The purpose of this analysis is to validate the previously established relationship (equation (5)) for monotonic (peak strength) and complex (destructuration) stress paths.

The investigated samples came from various depths between 295 and 566 m of boreholes SS-I and IC-I. The samples have very scattered physical and engineering index properties: carbonate content, $C(\text{CO}_2) = 2\text{--}42$ per cent (average 13 per cent), void ratio $0.34\text{--}0.64$, and friction angle $\phi' = 25\text{--}33$ (average 29). The clay fraction is about 50 per cent, consisting of smectite (25–100 per cent) and illite (0–75 per cent); the plasticity index varies between 18 and 28 per cent.

It should be stated at the beginning that the material tested did not exhibit clear pattern of anisotropy. Data of isotropic compression both in elastic and plastic range on 10 specimens have shown axial to lateral strain ratio between 0.49 and 1.78, the latter strains calculated from the measured volumetric and axial strain components. Also, undrained triaxial compression effective stress paths were typical of isotropic clays.

Peak strength and apparent maximum pre-compression isotropic stress

Because the peak strength and apparent maximum precompression isotropic stress and fundamental characteristics in the modelling of carbonatic clays the predictions of the former based on equation (5) are compared to the experimental values. Triaxial tests have been performed at various constant confining stress values. For these test conditions predictions were made of the initial yield surface using equations (1) and (5) based on the measured values of void ratio and carbonate content for each specimen (Table I), and taking an average value of the critical state coefficient $M = 1.18$ and p'_0 (unit pressure) as 0.1 MPa. In particular, the peak strength and apparent maximum preconsolidation isotropic stress values were calculated. The obtained yield surfaces are correlated to the experimental points in Figure 2. Overall, the tested specimens vary quite visibly in terms of the initial size of the yield surfaces. Both factors: porosity and carbonate content equally affect the initial yield surface.

The general trend of the strength variation is well predicted. The strength values themselves are predicted with an accuracy of 20–30 per cent. All together, the carbonatic Spanish clays have strength of the same order of magnitude as much more plastic Belgian Boom clays, with low to

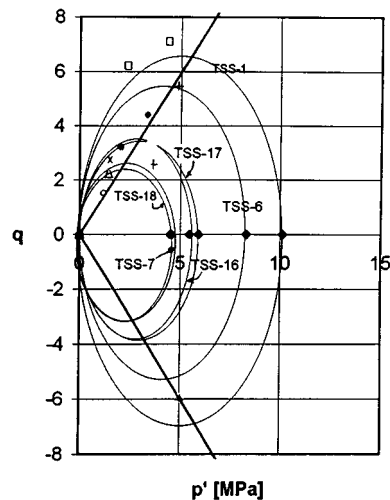


Figure 2. Initial yield surfaces determined on the basis of the measured void ratio and carbonate content using equation (5) and correlated to experimentally determined peak strength for a number of specimens: (\square) TSS-1, (\bullet) TSS-6, (+) TSS-7 (initial yielding and ultimate strength), (\times) TSS-16, (\triangle) TSS-17, (\circ) TSS-18, (\blacklozenge) denotes predicted apparent maximum pre-compression isotropic stress for each specimen

none content of carbonates and visibly weaker than highly carbonatic (30 per cent) Italian Pasquasia clay from Sicily. On the other hand, as far as deformability is concerned, the bulk moduli for mid- to high carbonatic Spanish clay specimens are much lower in the plastic range (0.042 for SS-4 and 0.048 for SS-15) than for Boom clay (plastic modulus $\lambda = 0.112\text{--}0.129$) and comparable in the elastic range ($\kappa = 0.016$ for both SS-4 and SS-15), against $\kappa = 0.0123\text{--}0.023$ for Boom clay. It is thus concluded that the modification of Cam clay model through equation (5) describes satisfactorily the strength dependence on carbonates in monotonic loading tests.

Destructuration

Destructuration is a prominent feature of natural clays. The considered clays are cemented, therefore strong reasons exist to test them for destructuration. The main purpose of the following test was to examine the departures of the clay behaviour from that of plastic clays as predicted by the Cam-clay model. It is also conceived as a background for the study of thermal effect on destructuration. A procedure classically used to evaluate the destructuration consists of loading a specimen in K_0 conditions about 10 per cent beyond the maximum precompression vertical stress, followed by unloading and a loading until failure in triaxial test in a low confining stress range.

The same principle was adopted in the tests performed in the triaxial tester, although the test portion corresponding to K_0 path was performed in steps. The specimen in test TSS-16 had $e_0 = 0.57$, $C(\text{CO}_2) = 0.198$ and thus estimated $p'_{\text{CO}} = 5.46$ MPa and $\sigma_1^f = 2.19$ MPa, which qualify this specimen as average in strength. The test program used the principle of the multiple triaxial test (see References 22–24) with the stress paths shown in Figure 3(a). The test consisted of

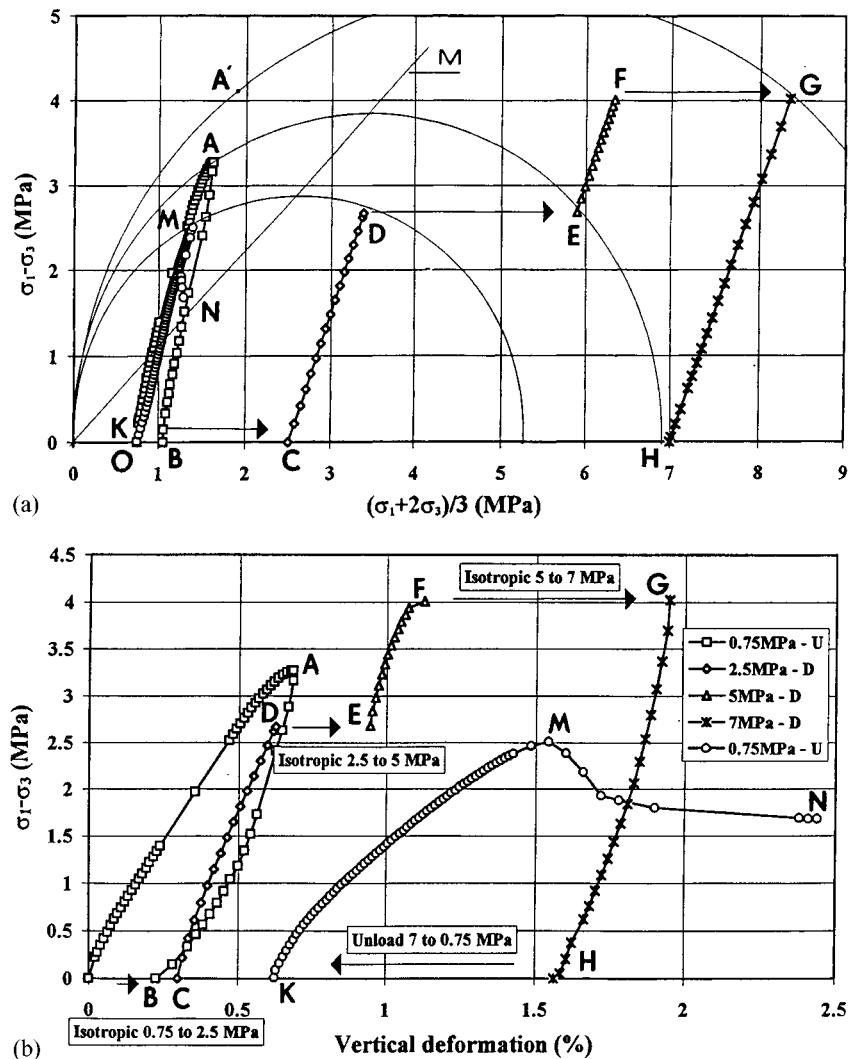


Figure 3. Destructuration tests: (a) stress paths; (b) stress-strain curves

a triaxial undrained loading at 0.75 MPa up to (and not beyond) peak stress, A, unloading to isotropic stress at 0.75 MPa, AB, consolidation to 2.5 MPa, BC, loading CDEFG (step-wise, triaxial and constant stress deviator isotropic loading) to the stress point of $q = 4.0$ MPa, $p' = 8.4$ MPa, expected to be on K_0 line, followed by an unloading, GH, to isotropic state of 7.0 MPa, unloading, HL, back to 0.75 MPa and undrained triaxial loading, LMN, until failure at 0.75 MPa. Destructuring understood as irreversible crushing of the internal structure during loading FG is manifested by a substantial volumetric deformation (0.0125), when compared to that during portions DE (0.0071) and BC (0.0045). While BC is an elastic isotropic loading process, portion DE is clearly plastic.

Comparing for the triaxial loading OA at 0.75 MPa the virgin stress–strain curve and that for the already preloaded material, along LMN (Figure 3b), one may easily see the effects of destructuring, which can be quantified as follows: (i) a 23 per cent lower peak strength in destructured material compared to the virgin material, and 46 per cent lower compared to the theoretically increased strength at A' ; (ii) a 40 per cent lower undrained Young's modulus during triaxial loading (a drop from 464 to 277 MPa) with a visible flattening of the stress–strain curve; (iii) increase by 40 per cent of the strain at failure, with respect to the virgin curve; (iv) unaffected critical stress level; (v) 35 per cent drop in the maximum excess pore pressure during the undrained triaxial portion of the test. The above differences, although not so marked as in Leroueil *et al.*⁵ findings, indicate a strong reduction in soil strength. Clearly, the response to complex loading histories including destructuration is beyond the scope of the model.

THERMO-MECHANICAL BEHAVIOUR

Selected thermo-mechanical properties of Spanish clays SS-1 and IC-1: thermal strains, thermal effect on triaxial peak strength, thermal effect on overconsolidation, thermal effect on destructuration will be now analysed from the point of view of their relation to carbonate content. Because of a limited data base no attempt is made to extend the existing thermo-plastic model to include the effect of carbonates at this time.

Thermal strains: thermal expansion and thermal consolidation

Volumetric thermal strain is one of the basic properties of the clay behaviour in thermo-mechanical conditions. In the context of nuclear waste disposal, volumetric thermal strain contributes to the build-up of pore pressure, its gradient, the rate of water flow and eventually of radioactive contaminant transport. It also affects clay strain hardening and the apparent overconsolidation. In practical terms, it may cause a displacement of the ground surface.

Thermo-mechanical tests were performed in the high temperature (reaching 200°) and high pressure (up to 20 MPa) triaxial tester, HITEP.²⁵ A series of seven drained heating tests performed on SS-1 and IC-1 clays: TSS-4, TS-8, TSS-9, TSS-0, TSS-11, TSS-15, TSS-17 and TSS-18 with different carbonate content and void ratio and in different conditions of effective isotropic stress and strain history are analysed.

The tests consisted in a very slow heating at the rate of $\frac{1}{2}^{\circ}\text{C/h}$ to 2°C/h , under computer controlled zero pore pressure regime, usually reaching 90° or 100°C and subsequent cooling back to room temperature. In some tests additional loading phases were included as discussed case by case below. During heating vertical strain and volume of water expelled from the specimen were recorded. The collection of the available vertical deformation – temperature curves is presented in Figure 4. First, it may be seen that the vertical strains during heating are small in comparison to strain in mechanical tests. Second, both expansive or contractive vertical heating strains are recorded. The apparatus does not allow for the measurement of lateral strain due to difficulties with the reliability of the gages at high temperatures. However, the volumetric thermal strain may still be estimated using the relationship between the volume of water expelled from the specimen during heating, the water volume increase due to the non-linear thermal expansion and thermal expansion of the solid skeleton.¹⁴ The assumption that clay water expands as bulk water at no pressure has been found inconsistent with the experimental results on dense plastic Boom clay,¹⁹ and leading to an overestimation of the expansive thermal strains.

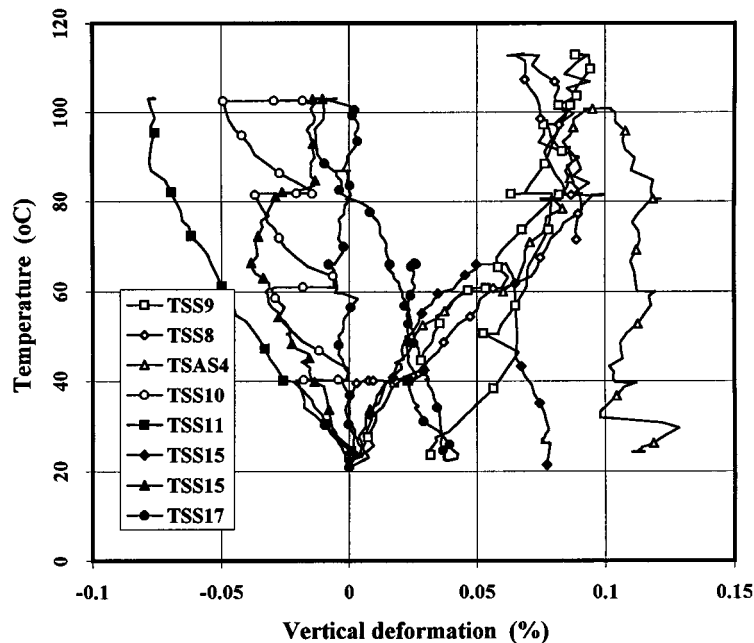


Figure 4. Vertical deformation – temperature curves during heating and cooling in drained conditions at different values of constant effective isotropic stress (during test TSS-4, at 1 MPa the vertical strain measurement recording system malfunctioned, and no relative data are available)

The calculated volumetric strains are either expansive or compressive as shown in Figures 5 and 6, presenting a pattern consistent also with the results obtained for Boom, Pontida and Pasquasia clays.²⁹ The characteristics of the specimens employed and of the tests are given in the appendix (Table II). The differences in the response are due to differences in the mechanical variables such as isotropic confining effective stress, and loading and heating/cooling history. The scatter can be attributed to differences in controlled or measurable physical factors such as initial void ratio, carbonate content, clay (smectite) content, and uncontrollable and unmeasurable physico-chemical variables as: thermal expansion of clay pore water, adsorbed to bulk water mass ratio, adsorbed water dehydration, and residual swelling.

The heating tests during which the vertical strains were expansive were: TSS-10 at 4 MPa, TSS-11 at 7 MPa TSS-18 at 0.75 MPa, TSS-15 at 7 MPa and TSS-17 at 2.5 MPa in its lower temperature part (Figure 4). On the other hand, the tests during which the calculated volumetric strains have shown a similar tendency, at least over a portion of the heating process are TSS-4/1 at 1 MPa, TSS-10, TSS-17 and TSS-18 (Figure 5a).

Under the assumption that the material is isotropic, one would expect the vertical and volumetric strains from Figures 4 and 5 to differ over the factor of 3. This however is not the case probably because of a possible anisotropy of thermal expansion of clay skeleton and/or an inaccurate evaluation of the thermal expansion of the clay pore water. While the mechanical anisotropy on some samples has been checked and resulted limited, the thermal expansion of clay water cannot be easily determined. The range of the uncertainty in strain related to this factor

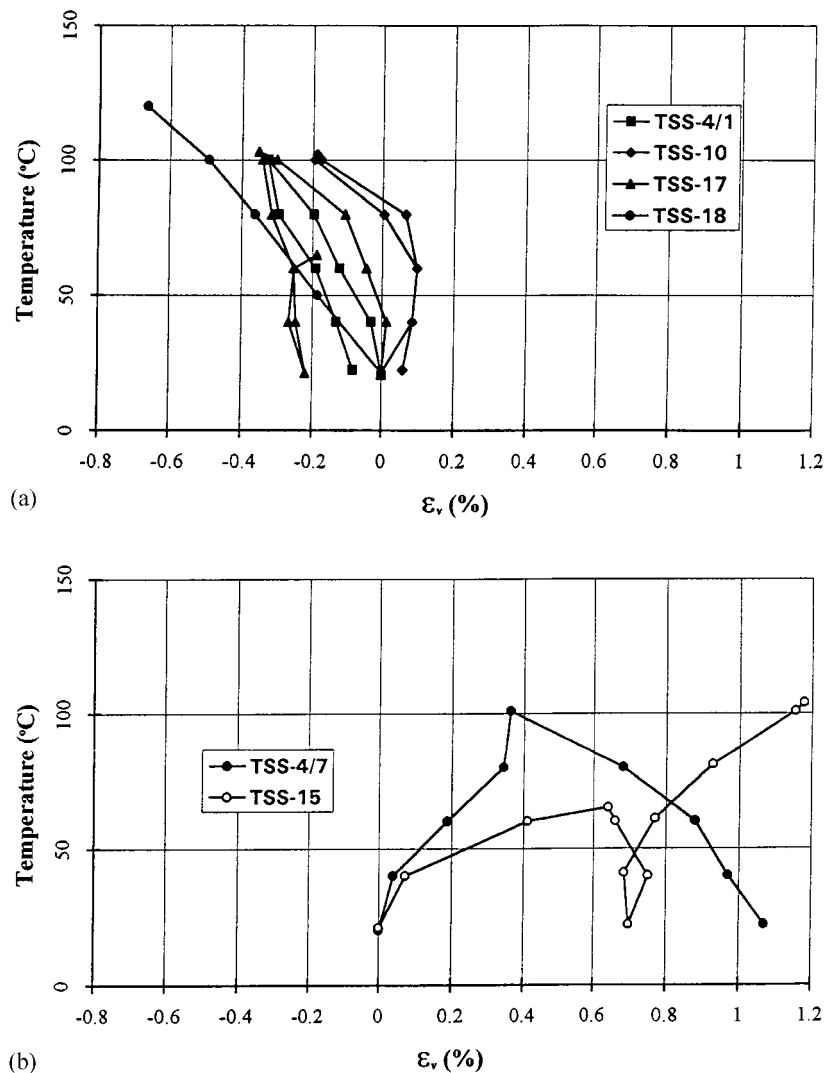


Figure 5. Volumetric strain during heating and cooling: (a) Expansion and contraction during process at low confining stress: for TSS-4 at 1 MPa, TSS-10 at 4 MPa, TSS-17 at 2.5 MPa and TSS-18 at 0.75 MPa; (b) Contractive volumetric strain during heating and during cooling: Tests TSS-4 at 7 MPa and TSS-15 at 7 MPa: Calculated volumetric strain-temperatures curves obtained during heating and cooling cycles in drained conditions at constant effective isotropic stress

depends on water content, and may be quite significant, amounting for example to 0.002 at 100°C for $w = 18$ per cent.

In the tests which included cooling, the strains remaining upon the temperature recovery were in most cases negligible. An exception is test TSS-17, with a net expansive irreversible volumetric strain of 0.2 per cent. Considering the predicted values of p'_{c0} at the onset of heating, the effective stress in all the above tests was initially well inside the yield surface, with $\text{IOCR} = p'_{c0}/p'$ varying

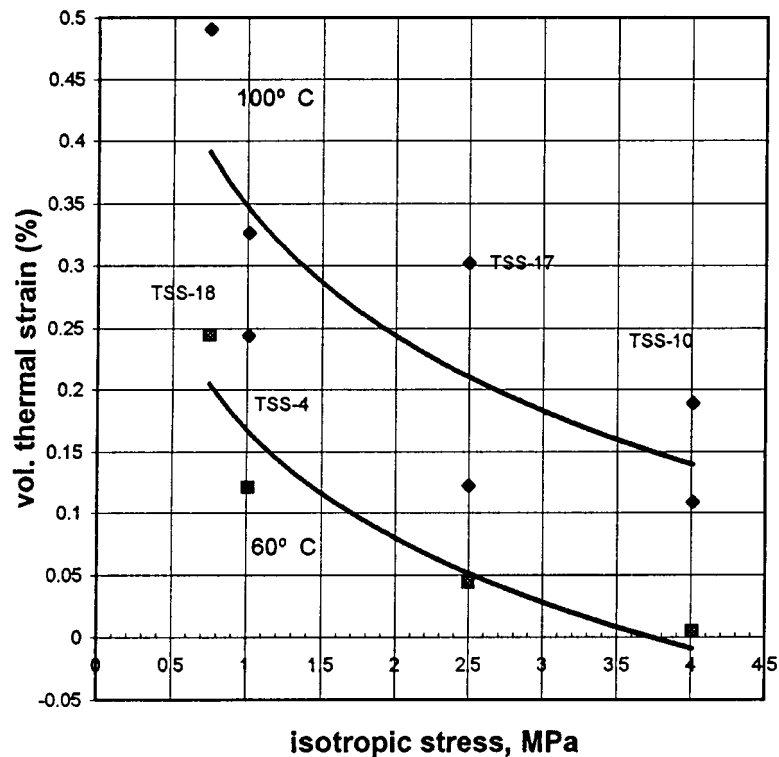


Figure 6. Tests TSS-4 at 1 MPa, TSS-10 at 4 MPa, TSS-17 at 2.5 MPa and TSS-18. Correlation between the volumetric strain at 100, 60°C and isotropic stress

between 6 and 2.36. The lack of the irreversible compressive strain rates during heating confirms that shrinking of the yield surface was such that p'_c was always larger than that the current effective stress. This also means that for instance for TSS-10, the yield surface did not shrink in terms of p'_{c0} more than 1.0 MPa for 100°C. This suggests that the strains in TSS-4, 10, 17 and 18 are essentially all thermo-elastic.

However, the strain values are somewhat scattered. For the reasons elaborated above the scatter cannot be attributed to plastic strain. Excluding that the scatter is random, it may be attributed to the differences in isotropic pressure, IOCR, void ratio or carbonate content. Void ratio is quite uniform in the tested specimens. As for the correlation to carbonatic content, the volumetric thermo-elastic strain does not exhibit any consistent correlation. Pure carbonates expand about 10 per cent less than pure clay mineral solids.²⁷ Thus, the presence of carbonates should have a minor reduction effect.

Most of the large-scale variability of the volumetric strain may be attributed to isotropic stress, following the tendency observed previously in plastic (no or low-carbonatic) Boom clay, Baldi *et al.*¹⁹ and confirmed by the correlation found at 60°C and 100°C (Figure 6). Interpolating the effective stress dependence of the volumetric thermal strain in this figure using a logarithmic function (equation (2)), separately for $T = 100^\circ\text{C}$ and for 60°C , one can calculate the average

coefficients as follows:

$$\begin{aligned}\alpha_0^* &= 4.13 \times 10^{-5} \text{ } 1/^{\circ}\text{C}; \alpha_1 = -4.73 \times 10^{-5} \text{ } 1/^{\circ}\text{C}; \\ \alpha_2 &= 1.1 \times 10^{-8} \text{ } (1/^{\circ}\text{C})^2; \alpha_3 = 3.58 \times 10^{-7} \text{ } (1/^{\circ}\text{C})^2\end{aligned}$$

A set of two drained isotropic heating tests: TSS-4/7 and TSS-15, have yielded compressive thermal strain, in both cases after *a priori* isotropic consolidation to 7 MPa. In both cases the yield surface has been activated to reach the isotropic stress of 7 MPa. Thus, the stress state at 7 MPa is already at yielding at the onset of heating, and the thermal strain qualifies as thermo-plastic. According to the thermo-plasticity theory the yield surface is immobile during heating in a plastic state when the stress is kept constant (see Reference 17). Otherwise, its shrinking would leave the stress point outside the current yield surface, which is statically inadmissible. The generated plastic strain at constant stress induces strain hardening, to compensate for thermal softening. This hypothesis is confirmed in both tests, by the volumetric and vertical strain development, which both are compressive right from the beginning of the heating (see Figures 4 and 6). During cooling, the volumetric strain is again compressive and is similar to that observed during 'elastic' heating/cooling cycles at low effective stress (Figure 5b).

To evaluate the potential influence of carbonatic content on thermo-plastic strain let us consider an infinitesimal increment of temperature dT at a constant effective stress, thus at $d\sigma_j = 0$, at yielding. Theoretically, the consistency condition of Prager during such process implies that

$$\frac{\partial f}{\partial T} dT + \frac{\partial f}{\partial \epsilon_v^{\text{pl}}} d\epsilon_v^{\text{pl}} = 0 \quad (8)$$

Rearranging the above equation and assuming an associative flow rule, the rate of volumetric plastic strain per rate of temperature may be determined as a ratio of the rate of change in p'_c with temperature, to the rate of its change with plastic volumetric strain, as follows:

$$\frac{d\epsilon_v^{\text{pl}}}{dT} = \frac{-\partial p'_c / \partial T}{\partial p'_c / \partial \epsilon_v^{\text{pl}}} \quad (9)$$

Assuming an uncoupled thermal softening and strain hardening, by setting $a_0 = 0$, and $\beta = 1$, one arrives at

$$\frac{d\epsilon_v^{\text{pl}}}{dT} = \frac{-2(a_1 + 2a_2\Delta T)}{[p'_{c0}/(\lambda - \kappa_T)](1 + e_0) \exp[(1 + e_0)/(\lambda - \kappa_T) \epsilon_v^{\text{pl}}]} \quad (10)$$

From the above equation it appears that for the materials with the same e_0 and the same plastic history, a higher carbonate content increases p'_{c0} , (equation (5)) and decreases moduli $\lambda - \kappa$ (with α_1 and α_3 being insensitive to carbonate content as seen above), and reduces the thermal deformation rate at a given temperature difference ΔT . Apparently, this is not the case of the two tests presented in Figure 6. However, it must be realized that the two test had not the same plastic strain history. In fact, the less carbonatic specimen TSS-4 ($C(\text{CO}_2) = 0.0571$) had a lower apparent maximum pre-compression isotropic effective stress, $p'_{c0} = 3.19$ MPa, than specimen TSS-15 with $p'_{c0} = 5.98$ MPa at $C(\text{CO}_2) = 0.176$. Thus during the consolidation between their respective p'_{c0} and $p' = p'_c = 7.0$ MPa, specimen TSS-4 was subjected to a considerably higher plastic volumetric strain. This lead to a higher hardening modulus. To compensate for the same amount of thermal softening which depends solely on temperature, the specimen TSS-4 would

require more plastic volumetric strain, being the strain hardening an exponential function of ε_v^{pl} . To quantify the difference, the hardening function (4) may be written for two states: (1): at the onset, and (2): at the end of heating:

$$\begin{aligned}\frac{p'_c}{p'_{c0}} &= \exp\left(\frac{1+e_0}{\lambda-\kappa} \varepsilon_v^{pl(1)}\right); \\ \frac{p'_c}{p'_{c0}} &= \exp\left(\frac{1+e_0}{\lambda-\kappa_T} \varepsilon_v^{pl(2)} (1+a_0 \Delta T^{(2)})\right) + 2A(\Delta T^{(2)})\end{aligned}\quad (11)$$

Given the fact that the effective stress remains constant all the time at yielding at isotropic effective stress p'_c , the amount of the volumetric plastic strain produced at the end of heating between the reference temperature and $T^{(2)}$ may be calculated as follows:

$$\varepsilon_v^{pl(2)} = \frac{1}{1+a_0 \Delta T} \left[\frac{\lambda-\kappa_T^{(2)}}{\lambda-\kappa} \varepsilon_v^{pl(1)} + \frac{\lambda-\kappa_T^{(2)}}{1+e_0} \ln \left\{ 1 - 2A(\Delta T^{(2)}) p'_{c0} \exp\left(-\frac{1+e_0}{\lambda-\kappa} \varepsilon_v^{pl(1)}\right) \right\} \right] \quad (12)$$

Considering the fact that the thermal softening function A is negative valued, at least for the uncoupled case ($a_0 = 0$) the volumetric plastic strain produced during heating, $\varepsilon_v^{pl(2)} - \varepsilon_v^{pl(1)}$, is smaller for a larger prior hardening, $\varepsilon_v^{pl(1)}$, at all other variables and parameters equal.

The hardening function (4) can be used also to quantify the effect of carbonate content on thermal softening. The thermal softening function, again for the uncoupled case, $a_0 = 0$, and $\beta = 1$, may be obtained for the constant effective stress heating, knowing the resulting volumetric plastic strain, as follows:

$$2A(\Delta T) = p'_c/p'_{c0} - \exp\left\{\frac{1+e_0}{\lambda-\kappa_T} \varepsilon_v^{pl}\right\} \quad (13)$$

It must be realized that the current plastic volumetric strain in the above equation includes the plastic strain attained during loading between p'_{c0} and p'_c and the plastic heating strain. The pre-consolidation strain and thermo-plastic strain at 100°C in the considered cases were, respectively, for TSS-4: 0.017 and 0.011, and for TSS-15: 0.0015 and 0.016. The moduli $\lambda-\kappa$ were estimated on the basis of the isotropic compression tests and were found for TSS-4 as 0.026 and for TSS-15 as 0.03. To obtain κ_T , coefficients α_1 and α_3 determined above were used. The measured elasticity modulus for the specimen TSS-4, $\kappa = 0.016$ was used. As a result, the thermal softening function yields the following values at 100°C

for TSS-4, $A(100^\circ\text{C}) = -4.48$ MPa and for TSS-15, $A(100^\circ\text{C}) = -1.86$ MPa

This indicates that thermal softening in the more carbonatic clay, SS-15 was lower than in the less carbonatic clay, as physically expected, despite the opposite tendency exhibited by the respective thermo-plastic strain.

Temperature effect on overconsolidated peak strength

The effect of temperature on peak stress has been studied on a specimen (SS-18) with $e_0 = 0.51$ and $C(\text{CO}_2) = 0.07$, with the predicted apparent pre-consolidation isotropic stress of $p'_{c0} = 4.50$ MPa, and the predicted uniaxial strength of 1.81 MPa. The strength was investigated on

a single specimen for the reason of heterogeneity of the specimen population, as explained earlier. The test was executed at a single value of confining stress of 0.75 MPa, using a multiple triaxial compression technique adapted as follows: at first the specimen at temperature of 22°C was compressed up to the peak stress, and not beyond, and unloaded. Next, the specimen was heated to 50°C at isotropic stress in drained conditions. At this temperature kept constant, subsequent triaxial drained compression was performed and interrupted at peak. The interruption at the peak prevents the development of any strain softening, as understood in Cam-clay theory, and thus any strength drop is attributed to thermal softening. Subsequently, cycles of unloading, further increase in temperature and triaxial compression were performed at 80, 100 and 120°C (Figure 7).

The results in Figures 8(a)–(c) show that the triaxial peak strength is nearly linearly decreasing with temperature. The overall reduction of strength over 100°C amounts to about 25 per cent. This is slightly more than it has been reported for Pontida clay, but less than for Boom clay.¹⁶ No minimum in strength drops, reported by Rousset²⁶ around 80°C, is observed. There is no change as well in the residual strength (the critical state) or ultimate friction angle.

According to thermo-plastic version of Cam-clay model¹⁷ the peak strength $\tilde{q} = (\sigma'_1 - \sigma'_3)^f$ for a constant lateral stress test decreases with temperature as long as the stress remains at the softening side, $\tilde{q} > \tilde{q}_{cr}$, where \tilde{q}_{cr} is the value of stress deviator at the intersection of the stress path and the critical state line, as shown in Figure 9. For the temperatures for which the yield surface shrunk below \tilde{q}_{cr} the ultimate strength is reached through a hardening process, starting at a yield point below \tilde{q}_{cr} , and ending at the same asymptotic value $\tilde{q} = \tilde{q}_{cr}$. Thus, the value \tilde{q}_{cr} is the lower bound of strength. Thus, in general, a plateau of strength – temperature relationship is expected. In this experiment, it may not occur until strength drops to $\sigma_1 - \sigma_3 = 0.65$ MPa, which probably takes place above the range of applied temperatures.

Triaxial strength and apparent maximum pre-compression isotropic effective stress are linked by a one-to-one relationship resulting from the equation of the yield condition (1) at

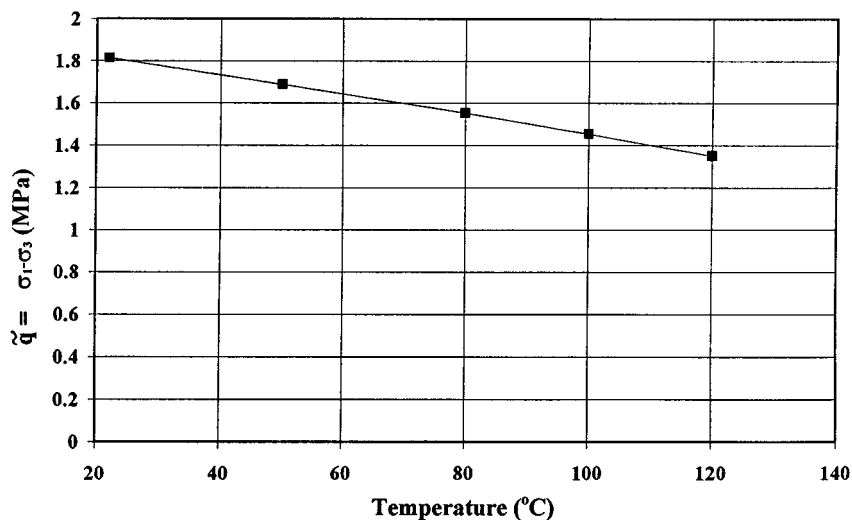


Figure 7. Triaxial peak strength vs. temperature at low confinement stress. Test TSS-18

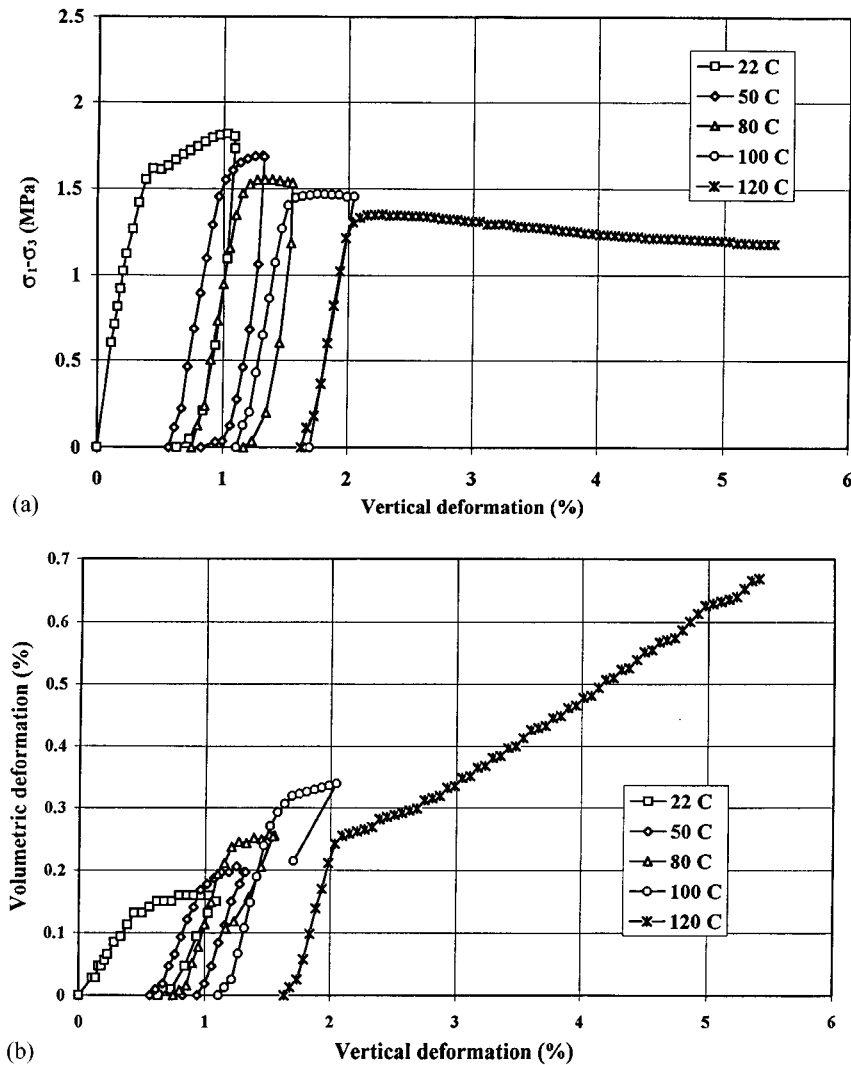


Figure 8. Temperature dependence of response to multiple triaxial compression test, at 22°, 50°, 80°, 100° and 120°C; Test TSS-18: (a) deviatoric stress vs. vertical deformation; (b) volumetric vs. vertical strain

$\sigma'_3 = 0.75$ MPa. The experimental value of triaxial strength at 22°C is 2.57 MPa corresponding to the apparent maximum pre-compression isotropic effective stress of $p'_{c0} = 3.11$ MPa, while at 100°C the triaxial strength is 2.25 MPa corresponding to the apparent maximum pre-compression isotropic effective stress of $p'_c = 2.46$ MPa. The value of thermal softening function $A(T)$ at 100°C is obtained by calculating

$$A(\Delta T) = \frac{1}{2} [p'_c(100^\circ\text{C}) - p'_{c0}(22^\circ\text{C})] \quad (14)$$

The value obtained for $A(100^\circ\text{C})$ is -0.65 MPa. This value is clearly lower than the ones obtained in the previous Section through the measurement of the volumetric thermo-plastic

strain in isotropic heating tests which yielded $A(100^\circ\text{C}) = -4.48$ MPa for TSS-4, and $A(100^\circ\text{C}) = -1.855$ MPa for TSS-15. This result indicates that thermal softening affects differently different portions of the yield surface. It may also suggest that the thermal changes of yield surface shape are not necessarily homothetic.

Thermal effect on overconsolidation

Earlier experiments with illite²⁸ and with Boom clay¹⁶ indicate that a heating and cooling cycle may induce in a normally consolidated clay a state of apparent overconsolidation. To study this effect in carbonatic clays, an influence a heating/cooling cycle has on undrained shear behaviour has been examined in test TSS-15. The test consisted in two cycles both performed at isotropic stress of 7 MPa. The first cycle was composed of drained heating to 65°C and cooling back to 22°C , followed by an undrained triaxial compression up to $q = 2.5$ MPa and unloading to the isotropic stress. The second cycle consisted in drained heating up to 103°C followed by an undrained triaxial compression up to failure. Theoretically, the response to the triaxial compression in the first cycle should be elastic, and in the second cycle should be plastic (Figure 10).

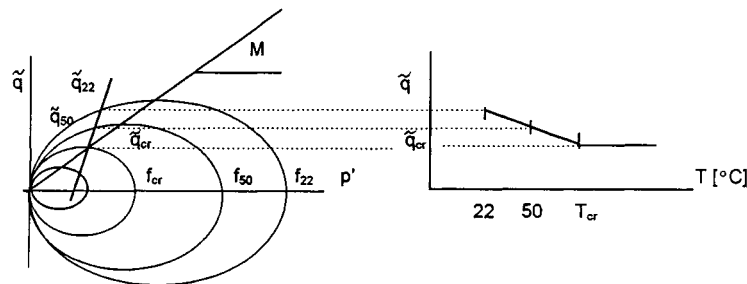


Figure 9. Evolution of peak strength \tilde{q} for constant lateral stress test with temperature: (a) for the peak points for the stress path at the softening part, $\tilde{q} > \tilde{q}_{cr}$, the strength can decrease with temperature, for the yield surfaces shrunk below \tilde{q}_{cr} , the strength is reached through a hardening process always at the same value $\tilde{q} = \tilde{q}_{cr}$, (b) variation of the strength with temperature

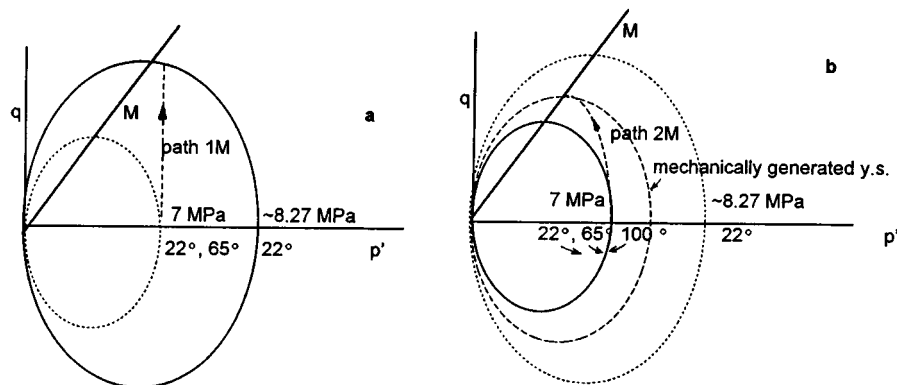


Figure 10. Test TSS-15: the evolution of the yield surface and the stress paths

The apparent preconsolidation isotropic stress for specimen SS-15 has been predicted for 5.98 MPa. Thus, the consolidation to 7 MPa should induce a state of normal consolidation. Therefore, the subsequent heating to 65°C, should generate irreversible volumetric compactive strain. In fact, from the consistency equation (8) at isotropic, constant effective stress it follows that (for $\beta = 1$, $a_0 = 0$)

$$\varepsilon_{ij}^{pl} = \frac{1}{H} \frac{\partial f}{\partial T} \frac{\partial f}{\partial \sigma_{ij}} \dot{T} = - \frac{1}{(\partial f / \partial \varepsilon_v^{pl})} \frac{\partial f}{\partial T} \dot{T} \delta_{ij} = - \frac{2(a_1 + 2a_2 \Delta T) \dot{T} \delta_{ij}}{[(1 + e_0)/(\lambda - \kappa_T)] p'_c \exp[(1 + e_0)/(\lambda - \kappa_T) \varepsilon_v^{pl}]} \quad (15)$$

During heating the yield surface should not change and thermal softening have to be compensated by strain hardening. The experiment confirms this hypothesis, as seen in Figure 5b, where a significant irreversible compressive volume strain can be observed. The same is true for vertical strain (Figure 11) (Path 1T). However, during cooling from 65 to 21°C as the yield surface grows back, a state of thermally induced overconsolidation is generated. Thus, the subsequent undrained triaxial compression at 7 MPa, should produce a stress path typical of lightly overconsolidated clays. Figure 12(a) shows an obtained effective stress path (Path 1 M) which is purely deviatoric. The lack of virtually any irreversible vertical strain upon unloading and the linear stress-strain curve (Figure 11), as well as the linearity and reversibility of pore pressure (Figure 12(b)) confirm the hypothesis that the behaviour was elastic and the stress path did not leave the elastic domain.

Taking the end of the stress Path 1 M as a limit of elastic behaviour, theoretically the yield surface could have been expanded during cooling to $p'_c = 8.27$ MPa. Thus, at the onset of second heating the estimated overconsolidation was $\text{IOCR} = 1.21$. The hypothesis of thermally induced overconsolidation is also very convincingly confirmed by the development of thermal strain. During the second heating (Path 2T) the vertical strain rate is expansive up to 65°C, and above that value it changes the sign and becomes compressive (Figure 11). According to the

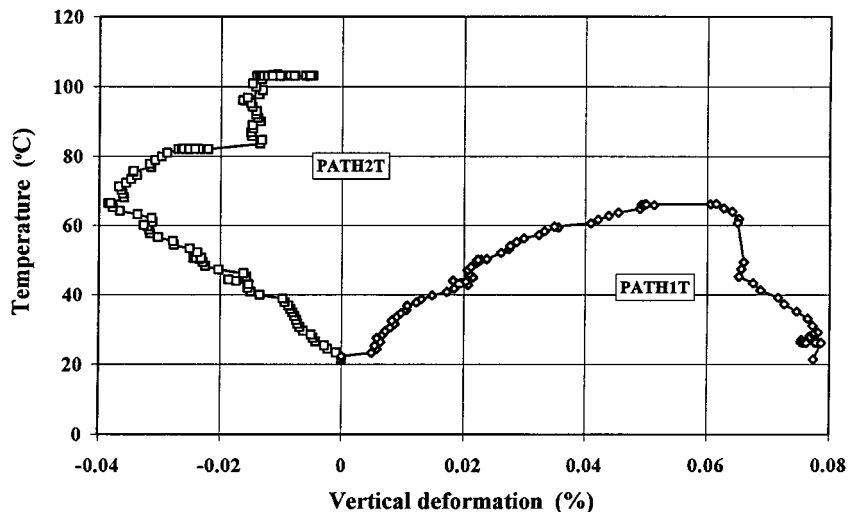


Figure 11. Test TSS-15: vertical strain development during drained heating to 65°C and cooling after isotropic consolidation to 7 MPa (Path 1T) and during drained heating to 103°C

thermoplasticity theory, this means that up to 65°C the stress state was elastic and thus overconsolidated, and the strain was elastic, while above that temperature the response became thermo-plastic. The volumetric strain exhibits hysteresis, with expansion between 21° and 40°C, and compression above the latter temperature value, with a gradually increasing strain rate (Figure 5b). However, the strain rate is lower than during the previous heating.

The above behaviour seems to be a sufficient corroboration of the claim that at about 65°C the yield surface have shrunk back to $p'_{c0} = 7$ MPa, what prompted plastic yielding above this temperature. Thus, the subsequent undrained triaxial compression at 103°C should theoretically

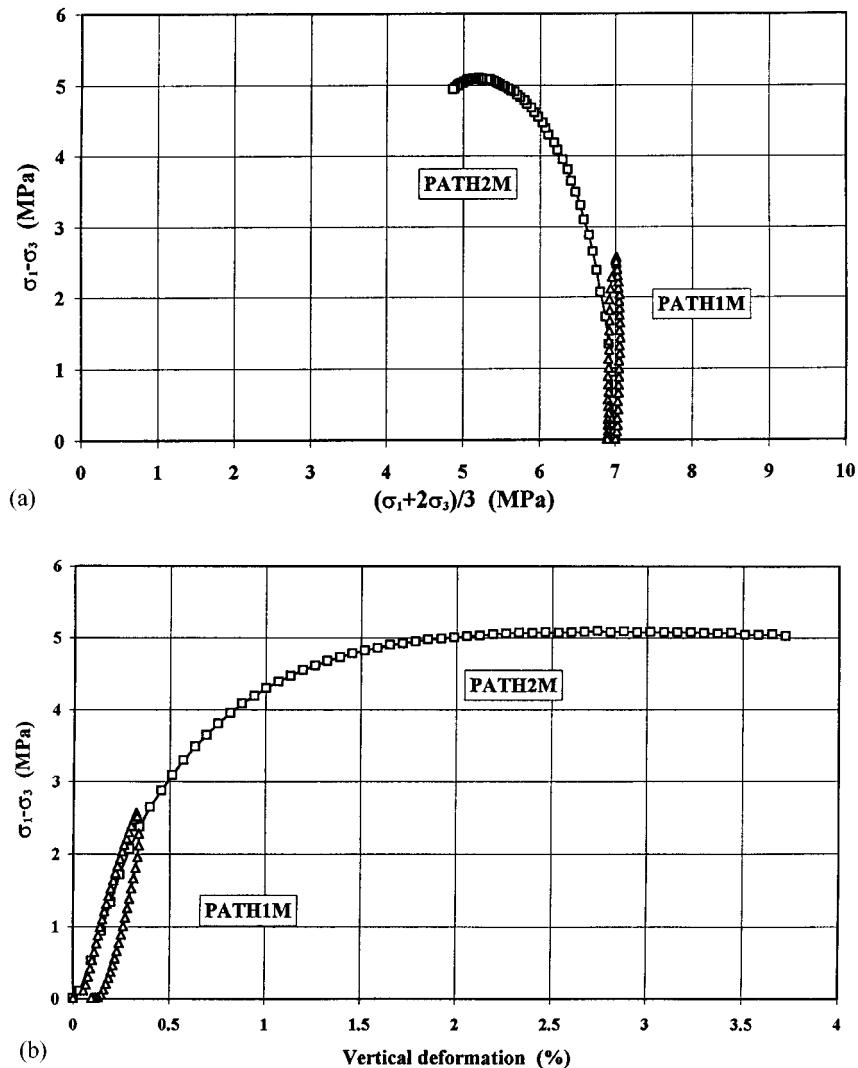


Figure 12. Test TSS-15: behaviour during triaxial undrained compression: (a) mechanical effective stress paths; (b) stress deviator vs. vertical strain; (c) pore pressure vs. vertical strain

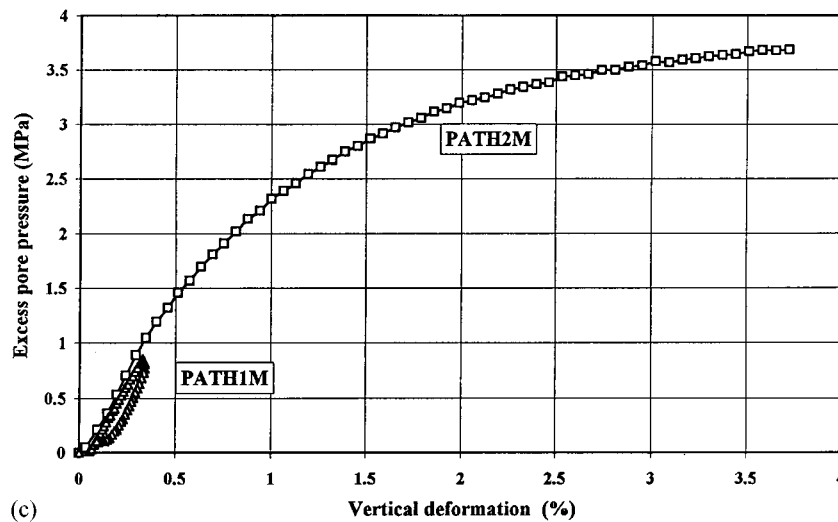


Figure 12. Continued

be plastic from the very beginning of the loading (Figure 10). The deviation of the effective stress path from the vertical course, Path 2 M (Figure 12(a)), and the non-linearity of the stress–strain and pore pressure–temperature curves confirm this hypothesis (Figures 12(a) and (c)).

This test is a very compelling, even if indirect demonstration that heating induces normal consolidation in an overconsolidated clay, and that a heating/cooling cycle induces overconsolidation in a normally consolidated clay. Note that a direct demonstration involving changes in p'_c is difficult, because of a highly conventional character of this variable.

Thermal effect on destructuration

The test dedicated to investigate the influence of heating on destructuration was performed on sample TSS-17 with $e_0 = 0.557$ and $C(\text{CO}_2) = 0.179$ (unfortunately taken as an average for the whole sample) with a predicted moderate $p'_{c0} = 5.9$ MPa. The test program was similar to that in isothermal conditions (Figure 3) and included (Figure 13(a)): isothermal undrained triaxial compression at $p' = 0.75$ MPa, interrupted at the peak stress, A, unloading to isotropic stress, B, isotropic loading, C, to 2.5 MPa, followed by a drained heating cycle 21–100–21–65°C at C, and a destructuration cycle consisting of: drained triaxial constant temperature compression, D, to deviatoric stress (2.6 MPa), isotropic stress increase of 2.5–4.5 MPa at constant stress deviator, DE, further triaxial compression, EF, to a higher deviatoric stress (3.5 MPa), isotropic compression at a constant stress deviator, FG, from 4.5 to 7 MPa, mechanical isothermal drained triaxial unloading to isotropic stress, H; isotropic unloading 7–0.75 MPa, HK; strength change check: mechanical undrained triaxial compression up to failure, KMN, and cooling. The objective of the heating at isotropic stress of 2.5 MPa was to subject clay to thermal changes without introducing thermo-plastic strain, and to see whether a pre-loading DEFG which in test TSS-16 resulted in clear destructuration, would show at 65°C any deviation from the results obtained in the former test.

The results of test TSS-17 are presented in Figure 13(b). It should be realized that the strength in the virgin unheated material in test TSS-17 (2.7 MPa of stress difference) is visibly lower than in

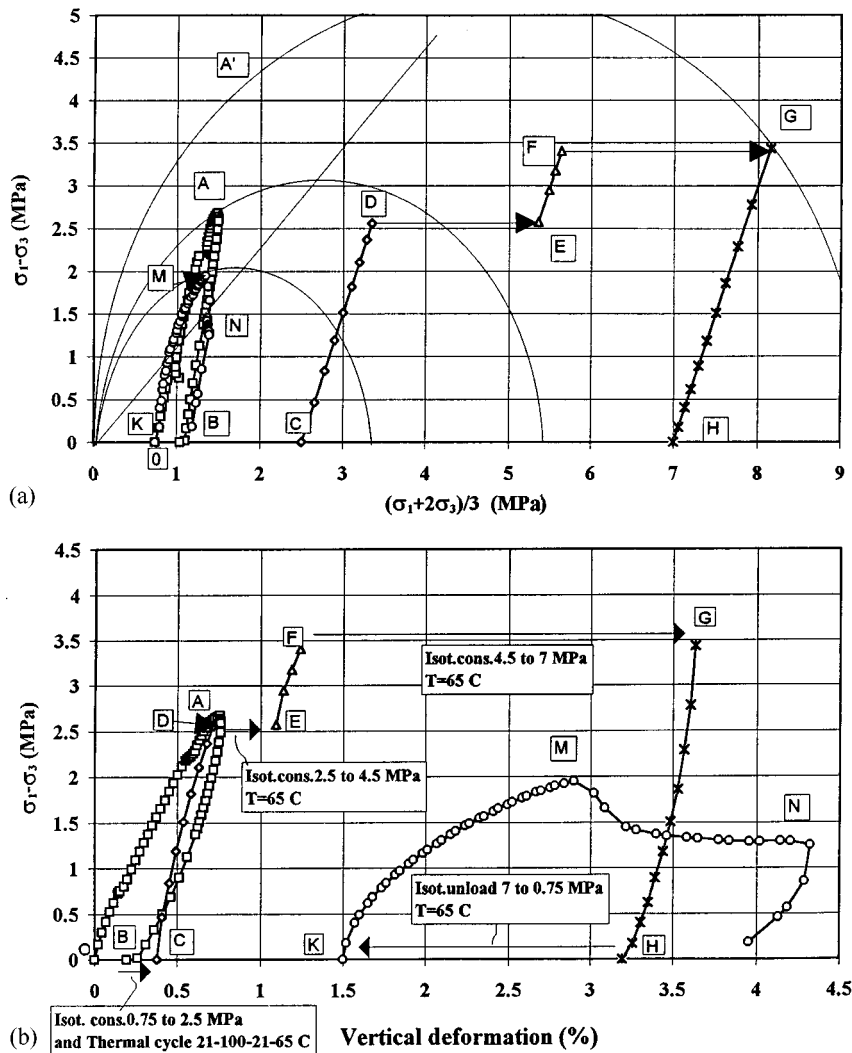


Figure 13. Destructuration test on a heated specimen: (a) stress paths; (b) stress-strain curves

TSS-16 (3.3 MPa). Also, Young's modulus was lower (113 MPa against 157 MPa). This might suggest that material in TSS-17 is per se weaker than the material in TSS-16. However, if the results are considered on a non-dimensional basis, the effect of difference may have a limited impact. The vertical strain during the main test portion inducing destructuration, i.e. during compression from 4.5 to 7 MPa at constant stress deviator, was in TSS-17 at constant temperature (65°C) almost three times larger than during the corresponding loading at room temperature in TSS-16 (2.4 vs. 0.8 per cent). However, the destructuration had a very similar effect as in TSS-16. In particular, comparing the virgin stress-strain curve for the triaxial loading with that for the mechanically and thermally preloaded material the effects of destructuring can be quantified as follows: (i) a 27 per cent reduction of peak strength in destructured material

compared to the virgin strength against 23 per cent drop in the cold material, and a 61 per cent reduction compared to the material which theoretically should have undergone hardening, to A', against 46 per cent in the cold clay. Note however that strength is expected to be somewhat lower for the purely thermal effect. Judging from the data obtained in test TSS-18, in a non-destructured specimen heating to 65°C should reduce strength over 12 per cent (see Figure 9(c)); (ii) a reduction of the undrained Young's modulus during loading, from 113.04 to 44.87 MPa, thus amounting to a 60 per cent drop, against 44 per cent in cold material; (iii) a visible flattening of the stress-strain curve at the peak; with an increase of the strain at failure by 93 per cent, against 40 per cent in cold

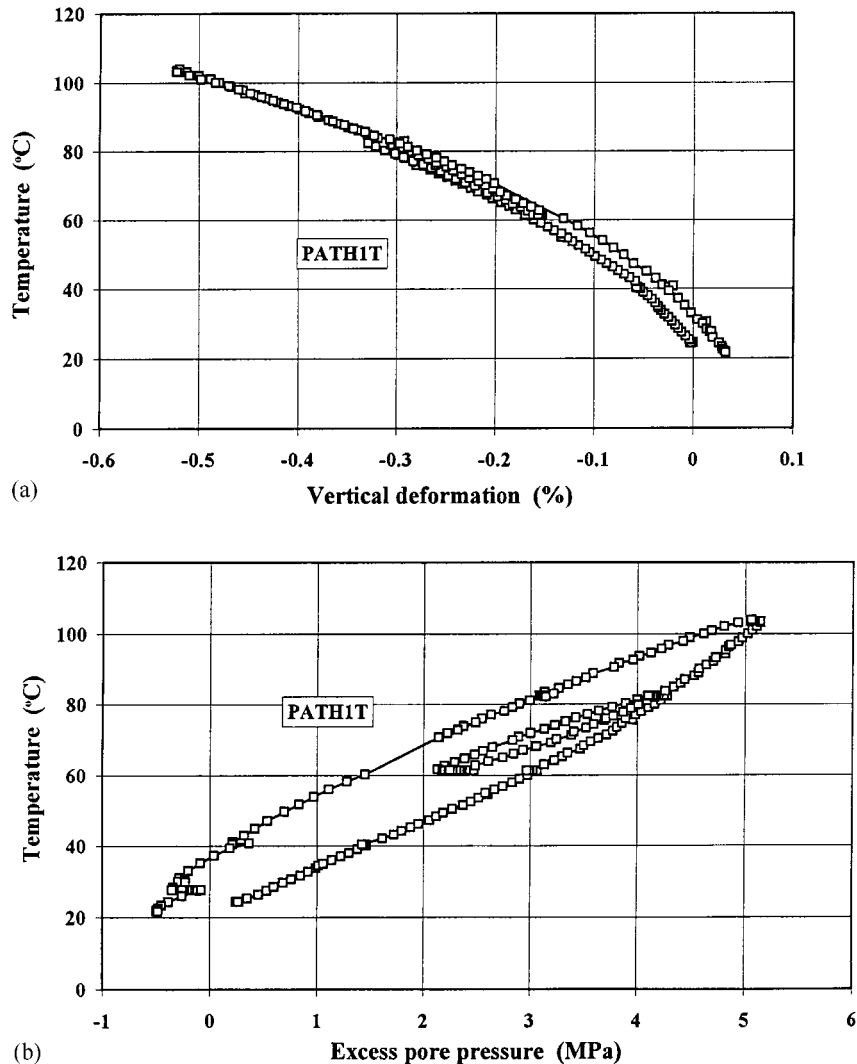


Figure 14. Pore pressure development during heating at constant total isotropic stress of 7 MPa in undrained conditions: Test TSS-14: variation of (a) vertical strain and (b) water pressure versus temperature, and (c) of water pressure vs. vertical strain

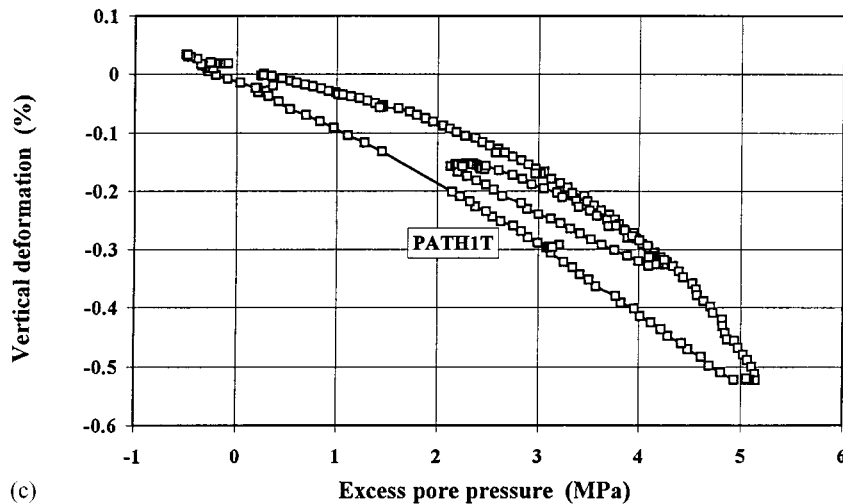


Figure 14. Continued

material; (iv) a lower critical stress level ($M = 0.95$); for intact material taken as an average for the whole sample, $M = 1.18$, the cold destructured material TSS-16 has had $M = 1.36$.

The above comparison does not unfortunately offer enough data for a strong conclusion. With no doubt the effect of destructuration occurs in a heated specimen. All the data gathered in this test indicate that destructuring is enhanced by temperature. However, since the specimen TSS-17 was weaker than TSS-16, it is not possible to state that the enhancement of destructuration has been due to temperature alone.

Undrained heating test

This test has been performed to study the tendency in carbonatic clays towards developing pore water pressure during heating in undrained conditions. The test has been performed on a remarkably porous specimen (SS-14) with $e_0 = 0.557$ and $C(\text{CO}_2) = 0.167$ which yielded a low $p'_{c0} = 1.91$ MPa. Undrained heating/cooling cycles 21–80–60–100–21°C were performed at isotropic stress of 7 MPa. Axial strain and water pressure values versus temperature are presented in Figures 14(a)–(c).

The most important result from this test is that a high water pressure of more than 5.0 MPa develops upon heating to 100°C. It is accompanied by a visible expansive vertical deformation of more than 0.5 per cent. The whole deformation is entirely reversible both upon cooling between 81 and 60.5°C and between the maximum temperature 110 and 21°C. Characteristic slow down of the rate of pore pressure observed at higher temperatures in other clays^{31,33} is also seen above 50°C, with a departure from non-linearity at 100°C of about 1 MPa. The value of the achieved water pressure at 100°C is 4 times higher in an analogous experiment on plastic silty Pontida clay, where 1.5 MPa was reached only. In natural, Orte clays the pore pressure growth was still less than in TSS-14, but it reached 2.5 MPa at 4 MPa of confining isotropic pressure at 80°C and was strongly confining stress dependent.

These results confirm independently the result that the thermo-elastic expansion is lower than in other investigated clays. In fact, the water pressure in undrained tests strongly depends on

thermo-elastic expansion of the porous skeleton, which was found to be lower than in other materials. Given that water content in other clays is similar and assuming that thermal expansion coefficient of the mineral and pore water are the same, it is not surprising that the water pressure is much higher in the carbonatic clays.

CONCLUSIONS

Although the population of the tested specimen was limited, some visible trends can be established. The significant scatter of the value of the apparent maximum pre-compression stress obtained from the isotropic tests is attributed to the influence of cementation. The procedure to correlate strength and pre-consolidation stress to carbonate content described earlier¹ within thermo-plasticity model¹⁷ yields good results.

The comparison of the investigated clays to much more plastic and nearly no carbonate Boom and Pontida clays and highly carbonatic Pasquasia clays was made. As for the elastic deformability, SS and IC-1 clays are comparable with Boom clay and more deformable than Pasquasia clay. However, their plastic deformability is almost one order of magnitude smaller than for Boom clay. In relation to strength, the SS-1 and IC-1 clays are comparable to Pasquasia clay, but stronger than Boom clay.

A significant effect of destructuration has been observed on IC-1 specimens both in room and elevated temperature conditions. Destructuration may play an important role in the behaviour of clay used as a natural barrier in nuclear waste disposal. It appears, that effects related to destructuration which further reduce the yield surface during excavation or elastic heating may result to be very important.

As for thermo-mechanical properties, thermal strains in SS-1 and IC-1 show a similar pattern to the three other materials; in the elastic range at low confining stresses they are expansive; in the plastic range at high confining stresses they are contractive. Thermo-elastic strain seems to be not correlated to the carbonate content, and to depend on isotropic stress. Thermo-plastic strain seems to strongly depend on carbonate content. Higher carbonate content leads to a reduced thermal softening. The experiments have revealed a marked, almost linear reduction in peak strength with temperature up to 120°C. The ultimate strength was confirmed to be rather temperature insensitive. A compelling evidence was found that heating induces a state of normal consolidation in a previously overconsolidated material, whereas a heating/cooling cycle, with a plastic consolidation occurring during heating, induces a visible increase in overconsolidation. Water pressure buildup during undrained heating is very strong, with 5 MPa for 100°C, and is visibly higher than that observed in the other materials mentioned in this study. Overall, the thermo-mechanical response of the carbonatic clays is similar, in principle, to the behaviour of the two other materials. The carbonates make the material less deformable and affect the range of thermo-elasticity.

As far as modelling is concerned, the performed sensitivity analysis indicates that the thermo-elasto-plastic model developed for plastic clays¹⁶ may be suitably adapted to deal with variable carbonatic content. However, it seems premature to undertake such effort before collecting more experimental data. As far as the effects of carbonates on thermo-mechanics are concerned, they first of all refer to the initial yield surface. The most challenging is the effect of destructuration and that of temperature on destructuration.

The thermo-elastic expansion seems to be fairly independent on the carbonate content. Plastic thermal consolidation and thermal softening appear to decrease with the carbonate content. This

conclusion is based on two specimens only, and should be confirmed on a larger population of specimens.

Finally, it must be stressed that the need to deal in detail with the carbonate content is not guided by the intent to map its space distribution in the simulation of a geological formation. Rather it stems from the necessity to interpret jointly the results from different specimens with highly scattered mechanical and thermal properties, to be able to model correctly the observed tendencies.

ACKNOWLEDGEMENTS

Mr. Giuseppe Angeloni of ISMES is acknowledged for conducting the experiments quoted in this paper and for helping in their data elaboration.

APPENDIX

Table I. Initial physical properties of the specimens

| | e_0 | C (CO ₂) | p'_{co} (MPa) | σ_1^f (MPa) | | e_0 | C (CO ₂) | p'_{co} (MPa) | σ_1^f (MPa) |
|-------|-------|------------------------|--------------------|-----------------------|-------|-------|------------------------|--------------------|-----------------------|
| SS-1 | 0.49 | 0.237 [†] | 10.13 | 4.07 | SS-11 | 0.36 | 0.255 | 28.1 | 11.3 |
| SS-2 | 0.529 | 0.237 [†] | 8.49 | 3.41 | SS-12 | 0.34 | 0.255 | 32.1 | 12.9 |
| SS-3 | 0.447 | 0.057 [†] | 5.86 | 2.36 | SS-13 | 0.39 | 0.123 | 10.7 | 4.31 |
| SS-4 | 0.561 | 0.057 [†] | 3.19 | 1.28 | SS-14 | 0.57 | 0.167 | 4.76 | 1.91 |
| SS-5 | 0.50 | 0.057 [†] | 4.35 | 1.74 | SS-15 | 0.54 | 0.178 | 5.98 | 2.4 |
| SS-6 | 0.39 | 0.0791 | 8.31 | 3.34 | SS-16 | 0.57 | 0.198 | 5.46 | 2.19 |
| SS-7 | 0.49 | 0.057 [‡] | 4.57 | 1.84 | SS-17 | 0.49 | n.a. | | |
| SS-8 | 0.42 | 0.066 | 5.024 | 2.19 | SS-18 | 0.51 | 0.077 | 4.50 | 1.81 |
| SS-9 | 0.52 | 0.048 | 3.78 | 1.521 | SS-19 | 0.383 | 0.084 | 8.76 | 3.49 |
| SS-10 | 0.59 | 0.124 | 3.54 | 1.42 | SS-20 | 0.551 | 0.075 | 3.60 | 1.45 |

Table II. Characteristics of tests in thermo-elastic domain

| Test | e_0 | C (CO ₂) | p'_{co} (MPa) | p' (MPa) | IOCR | $p'_{co}-p'$ (MPa) |
|---------------------|-------|------------------------|--------------------|---------------|------------------|-----------------------|
| TSS-4/1 | 0.561 | 0.0571 | 3.19 | 1 | 3.19 | 2.19 |
| TSS-10 | 0.59 | 0.123 | 3.54 | 4 | 3.0 [§] | 1.0 |
| TSS-17 [‡] | 0.54 | 0.179 | 5.90 | 2.5 | 2.36 | 2.1 |
| TSS-18 | 0.513 | 0.077 | 4.5 | 0.75 | 6.0 | 3.25 |
| TSS-4/7 | 0.561 | 0.0571 | 3.19 | 7.0 | 1 | -3.81 |
| TSS-15 | 0.54 | 0.176 | 5.98 | 7.0 | 1 | -1.02 |

[†]Carbonate content estimated as an average for the specimens from the same sample.

[‡]Specimen loaded to 12 MPa before heating.

[§]Data uncertainty.

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