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Indenter studies of the swelling, creep and pressure solution of Bure argillite

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Abstract

Experimental deformation of Bure argillites was performed by indenter techniques in order to investigate the kinetics of pressure solution processes. Various parameters included in pressure solution laws were tested: solubility of the mineral in solution (calcite), diameter of the indenter, temperature and stress. None of the observed effects confirm the contribution of pressure solution. In long duration experiments, a stabilized linear relation was found between displacement rate and stress, at least after 2 months. However, wide scattering of the data was observed, associated with unstable microfracturing around the indenter that may be linked to the swelling that always occurs before the indenting and that weakens the strength of the rocks (especially when using water). Argillite deformation is likely to be linked to the deformation of clay minerals acting as potential weak zones. The study does not exclude the possibility that pressure solution mechanisms may be of some importance for the long-term behavior of the natural barrier. This work just underlines the difficulty of studying this mechanism in weak rocks where several mechanism of deformation may competes and the faster strain rate hide the slower one at human time scale, whereas the slower one may be predominant on long-term duration.

Keywords: Argillite; Bure; Pressure solution; Creep; Swelling

1. Introduction

The so-called ‘‘argillite‘‘ rocks of the Bure Site (France) are a complex mixture of quartz, calcite, micas-clays and secondary minerals. The relative proportions of each mineral may vary. However, the typical composition may be as follows: 45–50% of micas and inter-stratified mixed layer minerals, 25–30% of calcite, 20% of quartz, 5% of secondary minerals. As such rocks contain a relatively large amount of minerals that are soluble under stress (such as carbonates and quartz), the effects of pressure solution mechanisms are suspected to be of some importance for the long-term behavior of the natural barrier. In addition to cataclastic deformation, pressure solution is one of the most important mechanisms of rock deformation in the upper crust from 0 to 10–15 km depth (Fig. 1b) (Kerrich, 1978; Rutter, 1983; Wheeler, 1992). Enhancement of pressure solution creep rates by clay minerals have been noticed both in nature (Heald, 1955; Weyl, 1959; Renard et al., 1997) and in experiments (Renard et al., 2001). Evidence of pressure solution mechanisms that occurred during the diagenetic processes is found in the argillite

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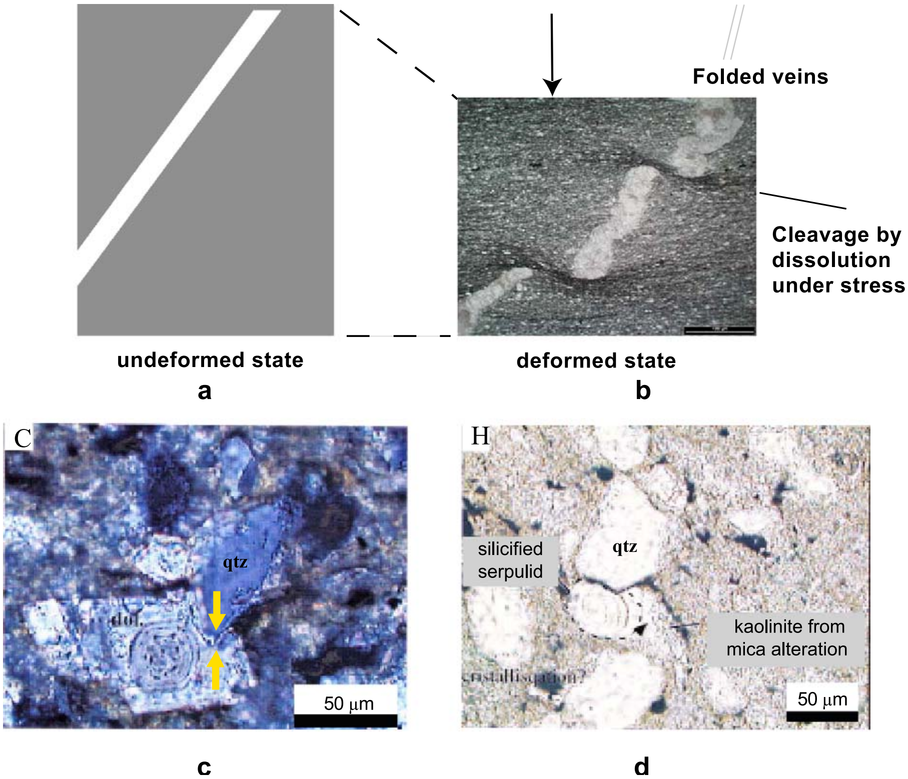

Fig. 1. (a, b) Natural deformation by pressure solution cleavage in silto-clay marl (right) analogous to the argillite of Bure (aggregate of micas-clays, calcite, quartz and secondary minerals) with schematic restoration to its initial undeformed state (left). (c, d) Pressure solution indenting processes (quartz, calcite and dolomite) in the argillites of Bure in Coquinot (2000).

rocks of Bure (Fig. 1c and d) (Coquinot, 2000). The idea behind our work was to try to reproduce such a mechanism in the laboratory in order to evaluate the kinetics of the processes and consequently the possible long-term behavior of the natural argillite barrier.

The main problem with the pressure solution creep mechanism is that its strain rate is always so slow that experiments are very difficult to carry out. The reason for this very low rate of deformation is linked to the basic processes of the mass transfer deformation, which characterize pressure solution, due to complex interaction between mechanical and chemical processes. The driving forces for mass transfer from the dissolution site to the deposition site are a function of the difference in normal stress, strain energy (elastic and plastic) and surface energy between the two sites (Paterson, 1973; Robin, 1978; Dewers and Ortoleva, 1990). The pressure solution strain rate is dependent on the kinetics of three successive steps: kinetics of dissolution, rate of mass transfer (either by diffusion or/and by infiltration) and kinetics of re-deposition. If one of these processes is much slower than the others, the strain rate is controlled by the slowest step. Various models of pressure solution creep have been proposed based on different driving forces and different rate-limiting processes (Weyl, 1959; Rutter, 1976; Raj, 1982; Dewers and Ortoleva, 1990). Relations between the displacement rate of an indenter and the various pressure solution parameters may be derived from theoretical creep laws. For polymineralic rocks (Fig. 2a), the limiting process is either:

(1) the kinetics of the reaction rate (k) for dissolution or deposition,
(2) the diffusion rate (D_m) of mass transfer through the entire rock formation,

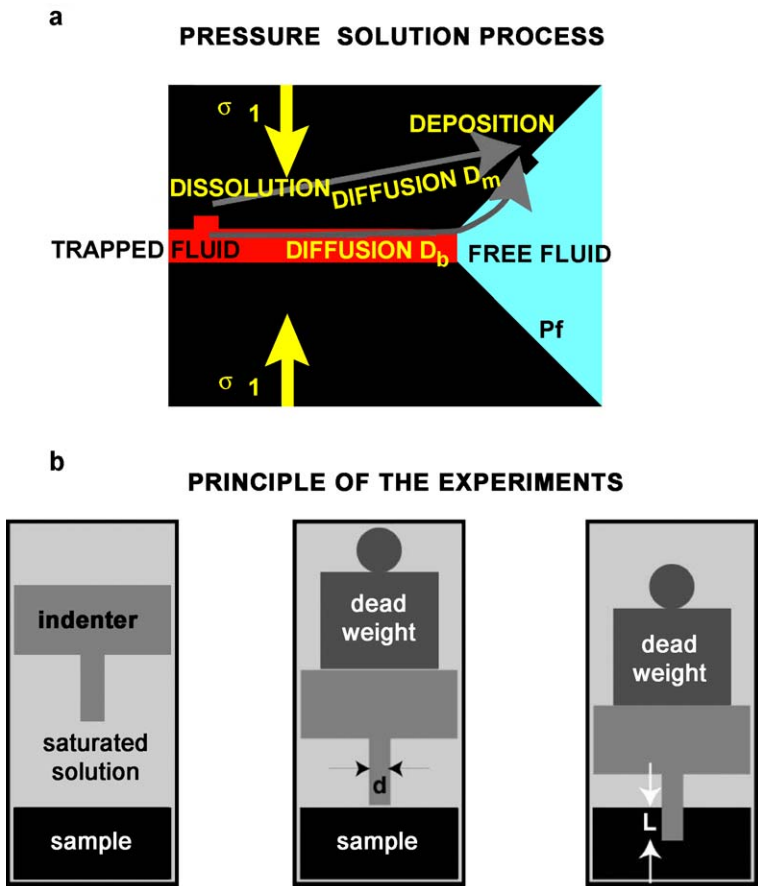

Fig. 2. (a) Schematic view of pressure solution process indicating the three successive steps:

- dissolution at sites with the highest chemical potential (= sites with the highest normal stress = σ_1);
- mass transfer either by diffusion (D_b) along trapped water film between non-porous solids, or by diffusion (D_m) through porous aggregate;
- deposition at sites with the lowest chemical potential (= sites with the lowest normal stress = free-fluid pressure p).

(b) Principle of the experiment: from left to right: a cylindrical stainless steel indenter is mounted under a free-moving Teflon piston. Before the indenter is brought into contact with the rock, the sample is immersed in the fluid in order to trap a saturated fluid phase under the indenter (fluid saturated with rock powder). The piston is loaded by a dead weight and the indenter is kept in contact with the rock sample in the presence of its solution. The device is maintained within a furnace at constant temperature for several months (6–7).

(3) the diffusion-rate (D_b) of mass transfer along the indenter–rock interface (along a trapped water-film).

$$\gamma_k = A\ k\ c\ \Delta\mu/RT \quad (1)$$

$$\gamma_{Dm} = B\ D_m\ c\ \Delta\mu/RT\ d \quad (2)$$

$$\gamma_{Db} = C\ D_b\ c\ w\ \Delta\mu/RT\ d^2 \quad (3)$$

where R is the gas constant, T is the temperature, c is the solubility of the solid in solution, w is the width of the transport path along the indenter–rock interface (thickness of the trapped water film), d is the diameter of the indenter and $\Delta\mu$ is the difference in chemical potential between the dissolution and the deposition site, mostly the effect of the difference between the normal stress under the indenter and the fluid pressure around the indenter. A, B and C are numerical coefficients that depend on the geometry of the system. For low stress values, which is the case considered here, a linear relation may be assumed between the driving force (normal stress difference) and the strain rate (Rutter, 1976). The inverse relation between the displacement rate and the size of the indenter (models 2 and 3) is a key characteristic of creep laws by mass transfer. Such inverse relation is also found in Nabarro-Herring and Coble creep laws at higher temperatures.

Evidence of pressure solution may be found by carefully investigating the following parameters.

– The effect of the solubility of the solid in solution (thus the effect of the nature of the fluid) is of crucial interest. Using high solubility fluid/mineral systems is the best way to activate experimental pressure solution strain rate. This was done either by using salts (Rutter, 1976; Raj, 1982; Urai et al., 1986; Spiers et al., 1990; Gratier, 1993) or by using natural mineral with very good solvents, i.e. quartz + NaOH M (Gratier and Guiguet, 1986), calcite + NH_4Cl 5% (Gratier, 1984).

– The effect of the temperature may discriminate between model (1) with high activation energy (90 kJ mol/K) (Gratier and Jenatton, 1984), and models (2) and (3) with low activation energy (15 kJ/mol/K) (Rutter, 1976, 1983).

– The effect of the size of the indenter allows one to discriminate between the three models. However, it has been shown that the development of radial fractures under the indenter may drastically activate the strain rate by reducing the distance of diffusion along the trapped water film (Gratier et al., 1999). In deformation modeling of natural structures, when diffusion is the limiting process of the pressure solution strain-rate (models 2 and 3), the parameter d is most often taken as the mean distance between the fractures that cross cut the solution cleavage surface (Renard et al., 2000).

– Finally, the effect of stress linked to the difference between the normal stress (σ_1) under the indenter and the free-fluid pressure (p) must, of course, be tested.

2. Principles of the experiments

Indenter techniques have been developed in order to study pressure solution (Tada and Siever, 1986; Hickman, 1989; Gratier, 1993). These techniques allow one to test the key parameters for pressure solution creep, such as the stress values ($\sigma_1 - p$), the nature of the solution, the temperature and the diameter of the indenter. The principle of the indenter technique specifically developed at the Grenoble laboratory for pressure solution creep studies is shown in Fig. 2b. Cylindrical stainless steel indenters were used in all the experiments that lead to the measurement of the displacement rate. As with pressure solution creep laws the displacement rate is dependant on the diameter of the indenter, mostly as an inverse dependence, these diameters must be as low as possible. We choose a range of 200–600 μm that was already used for studying halite pressure solution (Gratier, 1993). The indenter mounted under a free moving Teflon piston and loaded by a dead weight is kept in contact with the rock sample in the presence of its solution. Before the indenter is put in contact with the rock, the sample is immersed in the fluid in order to trap a saturated fluid phase under the indenter. In all cases, this fluid is a solution that was already saturated with rock powder. This device was maintained within a furnace at constant temperature. Two types of experiments were performed. In some cases the displacement of the indenter was monitored (by inductive axial movement gauges), for the entire duration of the experiments (several months). In other cases, the depths of the holes developing during the experiments were measured under a microscope at the end of the experiments (6–7 months).

Indentation experiments were performed on two types of samples drilled from the Bure site. The first one, referenced as: Est 104–458, here named "sample 1", was left in an open bag (so it was free to dry slowly). The second one, referenced Est 104–480, here named "sample 2", was kept confined in a sealed bag (so it was kept wet). This sample 2 was also used in the experiments of the LMS Polytechnique team (Vales et al., 2002). The size of the sample (about 1 cm) was chosen in order to be very large versus the size of the indenters.

As the experiments were performed at relatively low temperature (maximum 80 °C) calcite is the only major mineral presumed to be mobile under stress. The slow kinetics of dissolution of quartz at low temperature prevent pressure solution from happening for this mineral at such temperatures (Oelkers et al., 1996). However, at higher temperatures, quartz is more mobile especially when put in contact with highly alkaline solutions (Gratier and Guiguet, 1986). Hyperalkaline solutions may result from natural water leaching concrete from the building material of site (Berner, 1992).

3. Results

3.1. Swelling

Firstly, all the experiments were performed on small cylinders (1 cm diameter, 0.5 cm thickness) drilled from the two types of samples. All the experi-

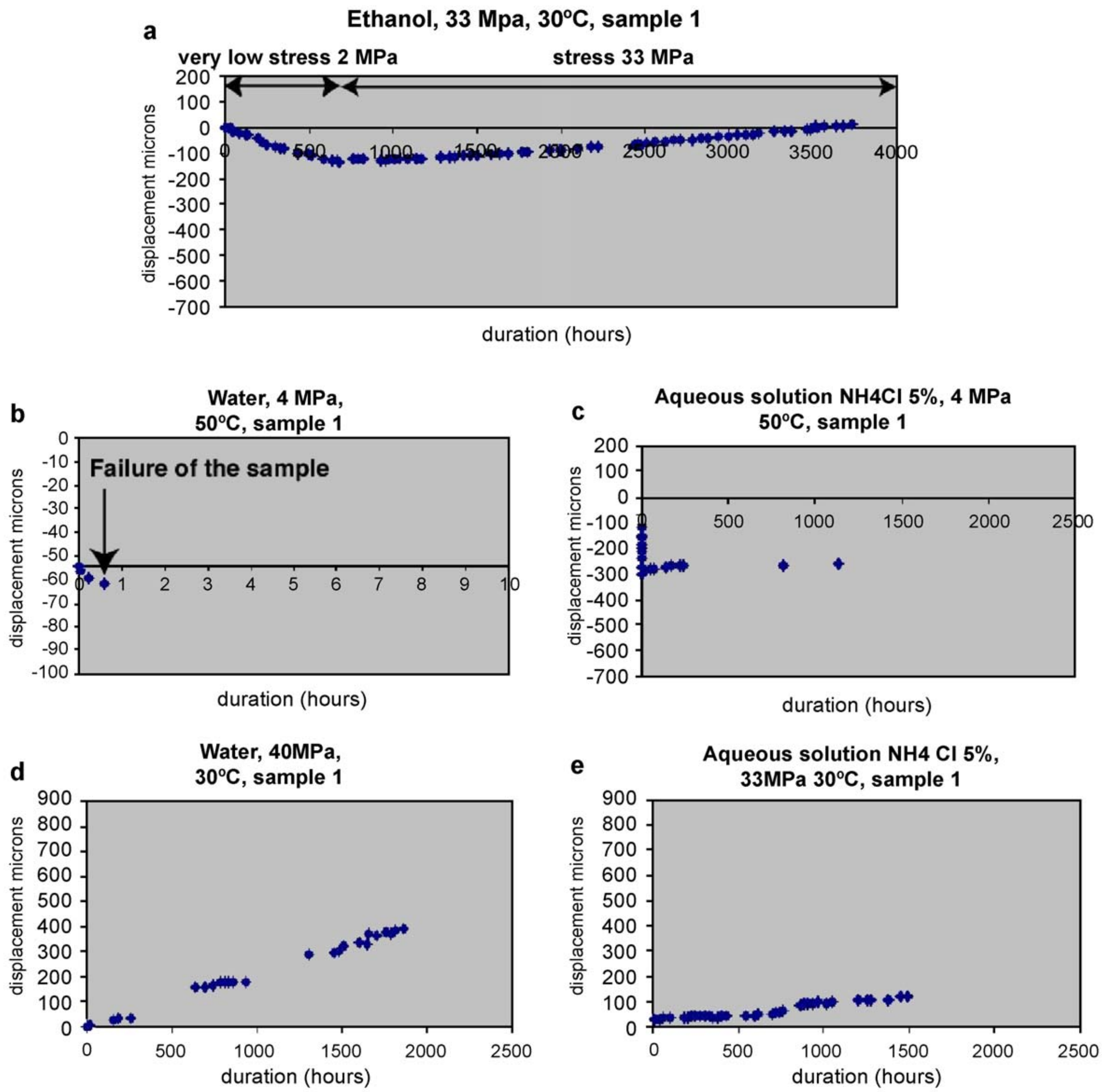

Fig. 3. Experiments performed with displacement of the indenter monitored throughout. The displacement rate (μm) is plotted versus the duration (h). Indications of the nature of the saturated fluid of the stress values on the indenter and of the temperature are given in the caption of each diagram. All these experiments were performed on sample 1.

ments that were run without a fluid phase on sample 1 failed to show any measurable deformation. No experiment was run without a fluid phase on sample 2. All the other experiments were run in the presence of a saturated fluid phase on both samples 1 and 2. Swelling was observed at the beginning of all these experiments, which were monitored for the entire duration of the deformation. When using water as fluid phase (distilled water saturated with argillites + calcite powder), the swelling is sometimes so considerable, after the sample has been immersed in the solution that the indenter under stress can fracture the argillite and lead to the collapse of the sample (Fig. 3b). When using other fluids such as ethanol or aqueous solution with 5% NH_4Cl, swelling also occurs, but the argillite remains strong enough to withstand the stress. The first experiment was conducted with a minimum stress value on the indenter (2 MPa) and then after a month of constant swelling (Fig. 3a) a dead weight was placed on the piston (33 MPa) and the indenting process began almost immediately. All the other experiments started directly with stress on the indenter (as understanding swelling mechanisms was not the main aim of the study). The same swelling is observed (Figs. 3 and 4) whatever are the fluid phases. It should be noted that part of this swelling may be related to the heating of the sample, at the very beginning of the experiment, since it was not possible properly to pre-heat all the deformation and sample system (experiments were designed to study long-term pressure solution, not specifically the swelling). When using various fluids (except water) in parallel, at the same temperature (60 °C), and with nearly the same stress onto the indenter, the swelling appears to be nearly the same for all the fluids (Fig. 4). Predictably, the swelling is inversely proportional to the stress value.

3.2. Indenting

After the swelling step and in the presence of a fluid phase, indentations were always observed (Figs. 3–5).

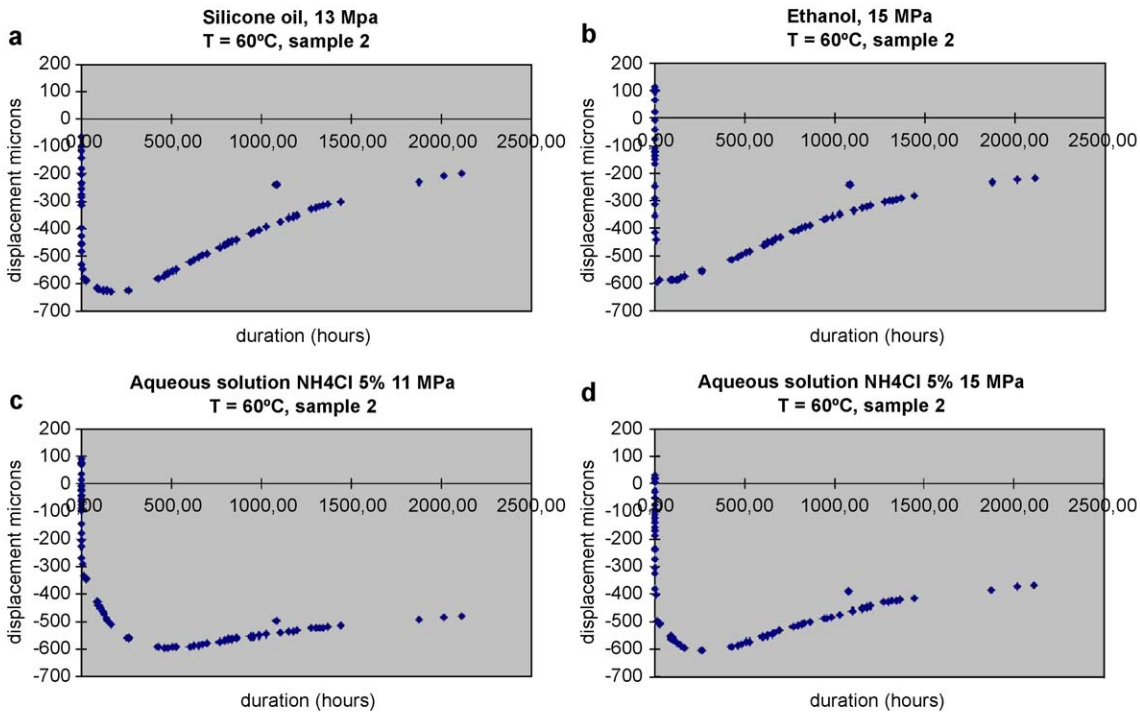

Fig. 4. Experiments performed with the displacement of the indenter monitored throughout. The displacement rate (μm) is plotted versus the duration (h). Indications of the nature of the saturated fluid of the stress values on the indenter and of the temperature are given in the caption of each diagram. All these experiments were performed on sample 2.

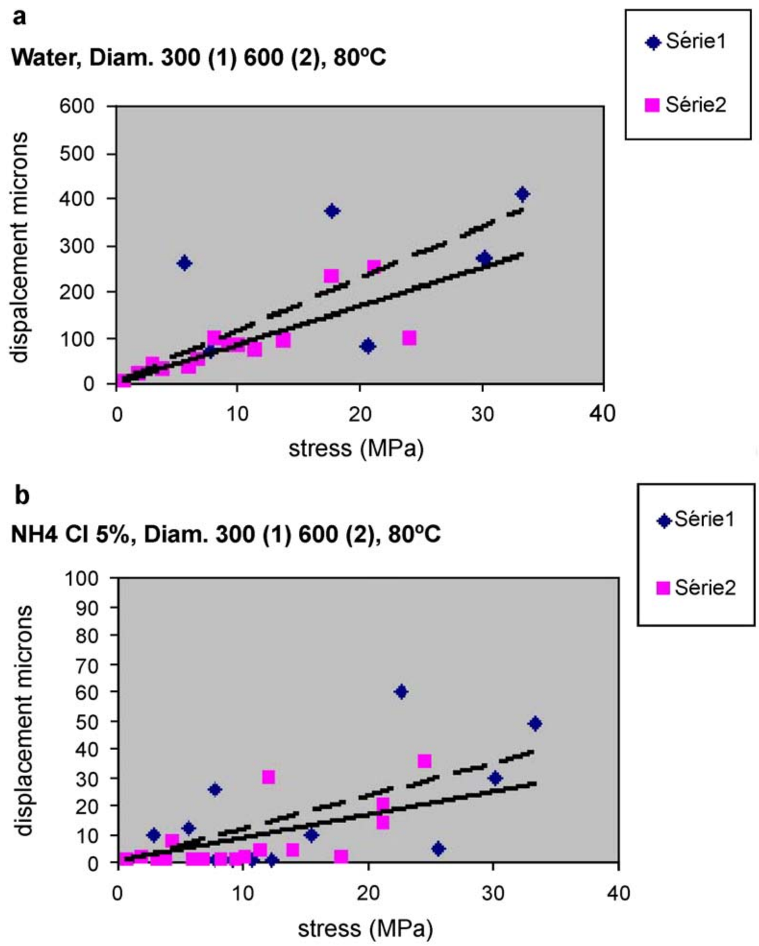

Fig. 5. Experiments performed without continuous monitoring: the depths of the holes formed during the experiment were measured under a microscope at the end of the experiments. The displacement rate (μm) is plotted versus the stress values (MPa). Indications of the nature of the saturated fluid (water or NH_4Cl 5% solution) and of the diameter of the indenter (300 or 600 μm) are given in the caption of each diagram. All these experiments were performed on sample 1 for the same period (186 days) and with the same temperature (80 °C).

In continuous measurement experiments (Figs. 3 and 4), the displacement versus time relations always show the same changes over time. After the swelling process (negative displacement in all the figures), indentation is observed, by a positive displacement. The rates of this indenting displacement change with time, with progressively decreasing values that stabilize after at least 2 months under stress. In order to compare the two types of experiments, we tried to standardize the data as follows.

For type 1 experiments, with continuous measurements, we used the mean slope of the displacement versus time relations obtained after at least 2 months under stress (1500 h).

For type 2 experiments with measurements of the depth of the hole at the end of the experiment, we always imposed very long duration experiments (lasting 6 or 7 months) in order to minimize the effect of the relatively fast indenting rate during the first month.

The effect of the solubility of the soluble species in solution (calcite) is studied by testing the effect of the nature of the fluid, since the solubility of calcite is very different depending on the fluid phase. The content in soluble minerals of the argillite rocks, at 60 °C, is 38 mg/l for pure water, 2435 mg/l for aqueous solution with NH_4Cl 5%, and negligible for ethanol and silicone oil. If pressure solution mechanisms control the deformation rate, as displacement rates are linearly proportional to the solubility (Eqs. (1)–

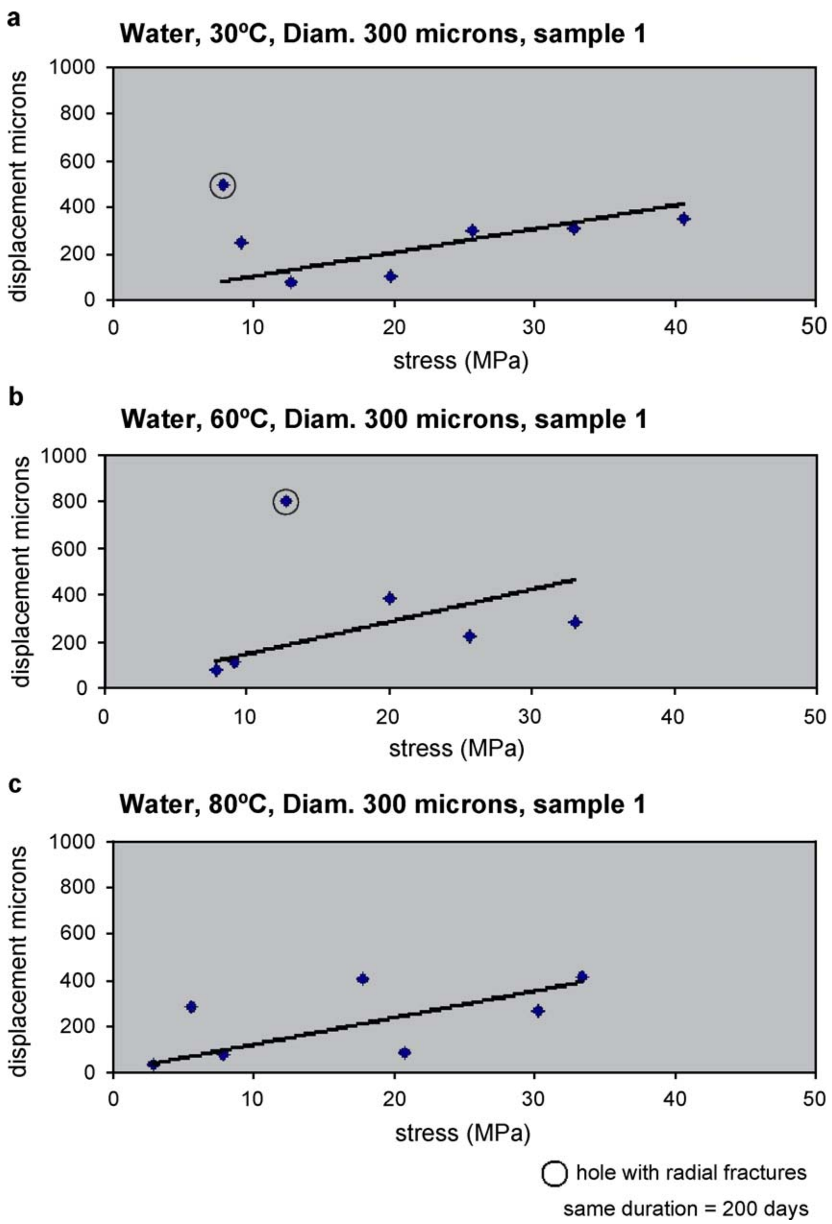

Fig. 6. Experiments performed without continuous monitoring: the depths of the holes formed during the experiment were measured under a microscope at the end of the experiments. The displacement rate (μm) is plotted versus the stress values (MPa). Indications of the temperature are given in the caption of each diagram. All these experiments were performed on sample 1, with the same nature type of the saturated fluid (water), same diameter of the indenter (300 μm) and for the same period (200 days). The experiments marked with a circle involved particularly extensive development of radial fractures (see Fig. 8).

(3)), they must be, for example, 64 times faster with NH_4Cl solution than with pure water and almost nil with ethanol and silicone oil.

This effect is compared in Figs. 3–5. Fig. 5, which present the displacement versus stress relations, clearly shows both the effect of the nature of the fluid and that of the stress values, since the other parameters are identical (temperature, diameter of the indenter and duration). Firstly, the effect of stress is clear. Apparently, there may be a linear relation between the displacement rate and the stress value. However, a wide large scatter of the results should be noted. This will be discussed later. Secondly, the displacement rate is always faster with water than with NH_4Cl solution, and this is not dependent on the temperature. The same behavior is observed when comparing the two experiments of Fig. 3d and e. As the solubility of calcite in solution is 64 times higher in NH_4Cl solution than in water, this means that the solubility of the solid in solution does not have the crucial effect predicted by pressure solution laws (see Eqs. (1)–(3)). Comparison between experiments that run with the best solvent (NH_4Cl 5%) and the worst (ethanol and silicone oil) show the same behavior (Figs. 3 and 4, respectively, for the two different samples).

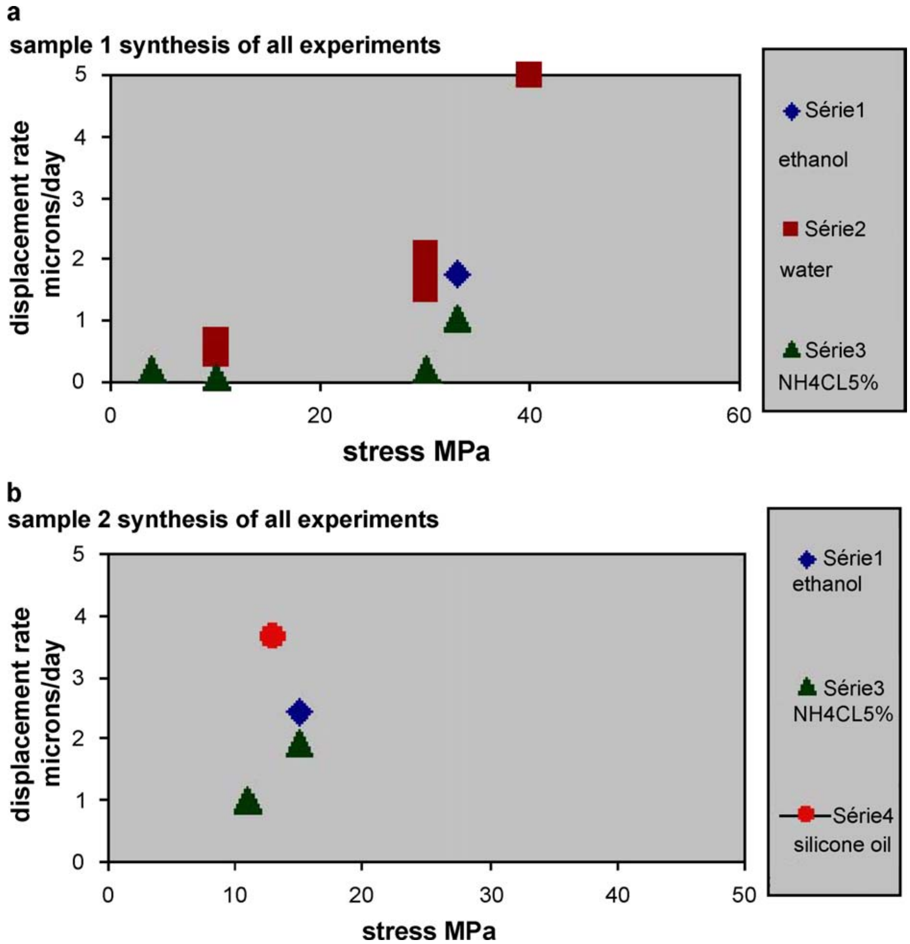

Fig. 7. Synthesis of all the results. For type 1 experiments, with continuous measurements, we used the mean slope of the displacement versus time relations obtained after at least 2 months under stress (1500 h). For type 2 experiments with measurements of the depth of the hole at the end of the experiment, we used the mean values given by least square fitting of the data. Indications of the nature of the fluids are given in the caption of each diagram.

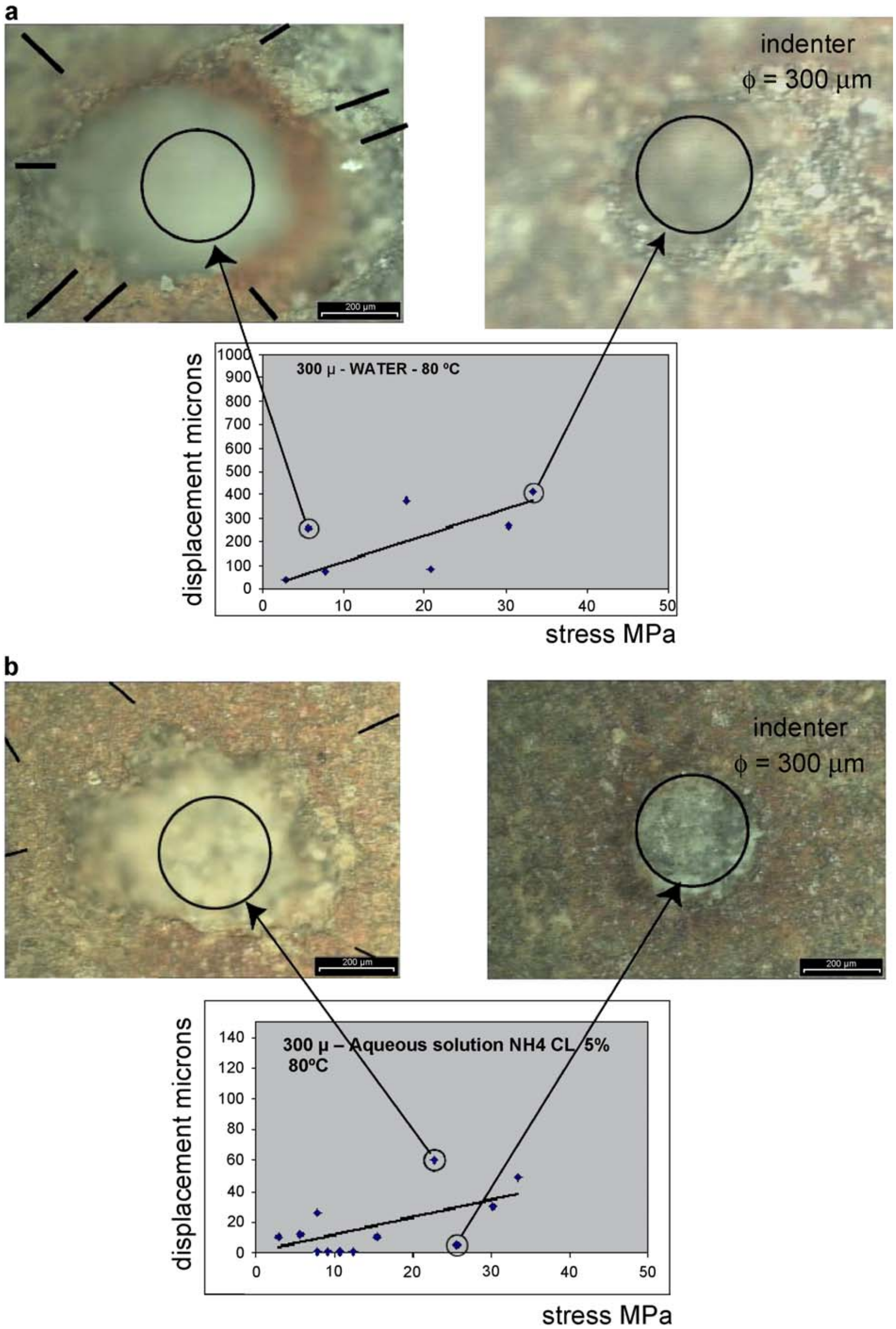

Fig. 8. Photographs of the holes obtained by experimental indenting: the shape of the holes and the associated radial fractures are compared for experiments that give values of the displacement rates above the mean square line and experiments that give values of the displacement rates near or below the mean square line. It is clear that the development of radial fractures is associated with the fastest displacement rates (photographs on the left).

The effect of the diameter of the indenter was also tested using indenters of various diameters comprise between 200 and 600 μm. The effect of the difference between experiments using 300 and 600 μm indenters is shown in Fig. 5 and does not seem significant.

The effect of temperature was tested by using saturated water between 30 and 80 °C. The difference does not seem to be significant here either (Fig. 6). This means that the activation energy of the process of deformation must be very low.

As temperature does not seem to play a major role, all the experiments obtained using 300 μm diameter indenters were plotted on the same diagram showing the relation between the displacement rate (μm/day) and the stress values (Fig. 7). For the continuous measurement experiments, the displacement rate values stabilized after 1500 h were used (see Figs. 3 and 4). For the experiment where the depth of the hole was measured at the end of the run, a mean displacement value was determined using the linear least square linear relations (see Figs. 5 and 6). The effect of stress remains clear. For a given sample, for example sample 1, displacement rate values are the fastest with water, the slowest with NH_4Cl 5% and at an intermediate level with ethanol. With sample 2, the ranking for displacement rate fluid activation is the effect of silicone oil, ethanol and NH_4Cl 5%, respectively, from the faster to the slower.

The difference between the two samples should also be noticed. There are two explanations. The two samples do not have not exactly the same chemical composition (sample 2 has a few higher clay mineral

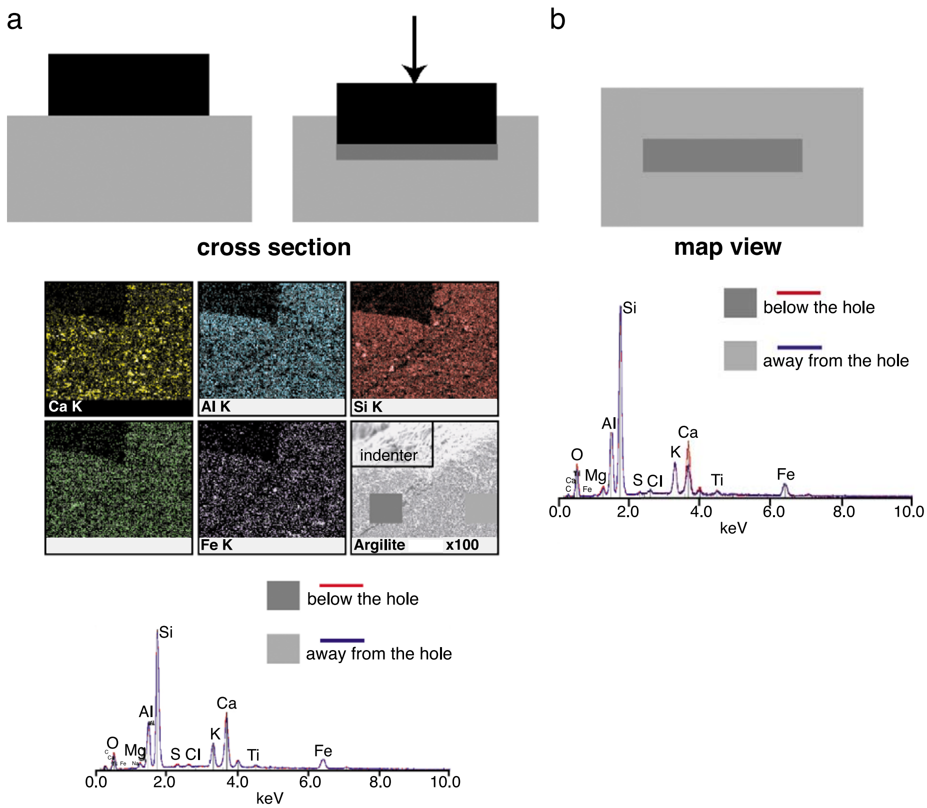

Fig. 9. Principle of the study of passive concentration of insoluble species (black points) below a rectangular indenter (black) during pressure solution process: if pressure solution mechanisms develop, comparative chemical analyses should reveal a higher content in the insoluble species below the indenter (bottom of the hole) than around the hole. This is not the case: when comparing reactive surfaces perpendicular to the indenter (Fig. 9b), the content in soluble species (calcite) is higher below the hole than away from it and when considering cross sections parallel to the indenter (Fig. 9a) no difference of composition is found between areas below the indenter and away from it.

content than sample 1). Another difference is that sample 1 was kept in an open bag and so was allowed to dry slowly, whereas sample 2 was kept in a sealed bag and consequently was prevented from drying. This sample 2 was the same one used by the LMS Polytechnique team (Vales et al., 2002) in their experiments concerning long duration and low stress indenting processes.

Finally, it should be noted that when many experiments are performed in parallel and in the same conditions, most of the results show a rather wide scatter (Figs. 5 and 6). In order to understand such behavior, microscope studies were performed, the results of which are given in Fig. 8. Optical microscope views show the crucial effect of the development of radial fractures that activated the displacement rate. All the displacement rates of experiments that are above the mean least-square line, either for water or for NH_4Cl, are associated with a large array of radial fractures. In contrast, displacement rates of experiments that are below the mean least-square line (or on the line) show almost cylindrical holes without any radial fracture.

A final test was performed in order to identify pressure solution processes that could be hidden behind the clay-related deformation of the rock. Pressure solution, if it occurs, should lead to passive concentration of the insoluble species below the indenter. So we compared the chemical compositions at the bottom of the hole with the undeformed part of the sample (Fig. 9a and b). A rectangular ceramic indenter was used in order to performed chemical analyses of argillite at the bottom of the hole, since a minimum length is needed for the beam reflection. An experiment was done with NH_4Cl 5% and it led to a rectangular hole of 1 mm depth that was pertinent for chemical analysis. Comparative analyses between the areas below and away from the hole show the same content for all the major elements except for Ca, which has a higher content below the hole than away from it. This is just the reverse that could be thought if pressure solution was the main mechanism of deformation. It happened that calcite crystals crystallized at the bottom of the hole. This is probably an effect of fluid being expelled from the argillites. This effect prevented us from making an accurate direct comparative analysis between deformed and undeformed argillites. To solve this problem, we cut the sample and we made a cross section parallel to the indenter. On this cross section, we were able to compare the chemical compositions of the sample below the reactive surface. No clear change of chemical composition was found when comparing the rock below the indenter and away from the effect of this indenter (Fig. 9a).

4. Discussion

Swelling mechanisms were not the main aims of the experiments and will not be discussed here. The indenting-creeping process may be discussed in the light of the theoretical pressure solution laws: none of the observed effects confirm the pressure solution process.

The effect of the solubility of the solid in solution, tested by using various fluids associated with various values of calcite solubility of in solution, does not support a mechanism of pressure solution creep. In such a mechanism (see Eqs. (1)–(3)), the displacement rate of the indenter is theoretically linearly dependent on the solubility of the solid in solution. This is clearly not the case here.

The effect of the diameter of the indenter could have allowed us to discriminate between the three types of creep relation (see Eqs. (1)–(3)). However, this cannot help here in the absence of the effect of solubility in solution.

The effect of temperature is almost negligible. Due to the wide scatter of the data, it is not realistic to try to evaluate an activation energy value.

Consequently, the observed deformation is likely to be linked to the deformation of the clay minerals, which could act as potential weak zones in the polymineralic argillite aggregate. The associated microstructural mechanisms were not investigated here.

The effect of microfracture development, which was not planned at first, appears to be very significant and must be emphasized. An explanation of this unstable behavior is not easy to draw from the data. As fracturing processes seems to be more developed with water than with NH_4Cl solution, it may be related to the swelling process, given that polar molecules of water may amplify swelling and consequently enhance the development of radial fractures (Katti and Shanmugasundaram, 2001; Cui et al., 2002). The true relation between the effects of clay mineral deformation, which dominate the experiments and microfracturing processes remains to be studied.

We also conclude that mass transfer does not occur at the scale of the indenter. However this does not rule out the possibility of mass transfer at the scale of the grains but this was not investigated here.

5. Conclusion

The indenter technique allowed us to investigate the slow deformation of the Bure argillites. The effects of various parameters were tested in order to investigate the kinetics of pressure solution processes: solubility of the solid in solution, diameter of the indenter and temperature. None of the observed effects confirms such a pressure solution process.

In long duration experiments, stabilized linear relations were found between the displacement rates and the constant stress values imposed on the indenter, at least after 2 months. A wide scatter of the data was observed, linked to the unstable development of radial microfractures around the indenter. Such fracturing processes may be linked to the swelling processes that always occur after immersing the samples in fluids before the indenting. When using pure water as fluid phase, this swelling is sometimes so considerable that it lead to the collapse of the sample.

Argillite deformation in the conditions studied here is likely to be linked to the deformation of clay minerals acting as potential weak zones in the polymineralic argillite aggregate.

The study does not exclude the possibility that pressure solution mechanisms may be of some importance for the long-term behavior of the natural barrier. This work just underlines the difficulty of studying such a mechanism in weak rocks where the strain rate imposed by the weakest minerals (clays) may hide the other slowest mechanisms of deformation at human scale whereas this last ones may be predominant on long-term duration.

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