DILATOMETRY OF POROUS LIMESTONES UNDERGOING FREEZING AND THAWING.

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The dilatometrical behaviour of calcarous rocks cylinders was measured in diameter and in length during freeze-thaw cycles. These measurements have shown intricate and non-isotropic variations in the dimensions of the rocks which are in connection with migrations of unfrozen water within the samples at negative temperatures. These migrations can be regarded as responsible for contractions instead of the global expansion which was previously expected. These experiments stress the importance of factors which have been acknowledged by many authors, ie the water content in the sample, its lithology, the rate of cooling and the slight alterations of the experimental device.

INTRODUCTION

A very important dilatometric research on materials undergoing freezing and thawing was published by Thomas (1938). He has observed complicated curves of variations in length of some rocks, bricks and tiles samples when they are freezing. He tried to explain these variations but had some difficulties to understand why some wet materials undergo contraction under freezing. He explained that inside these samples, the pressure given by the newly-formed ice induces liquefaction for the ice in favourable position, and that this process promotes intrusion of water into unfilled pores.

Lehmann (1955) has described a similar contraction of wet bricks under freezing. His observations became explicable in the papers of Powers and Helmuth (1953) and Powers (1958) on the dilatometric variations of cement pastes (figure 1). They have shown that, if microscopic bubbles are inside the cement pastes (the bubbles must be so numerous that they are separated by layers of paste of only a few thousandths of an inch thick), the freezing produces shrinkage rather than dilatation. And they proposed to explain this shrinkage by transfer of water from the paste to the air bubbles.

This migration of water by cryosuction gives on explanation for some part of the dilatometric curves recorded in experiments carried out with porous limestone. With these explanations, the dilatometric curves enable one to see what happens during the freezing of wet material and the process of gelifraction.

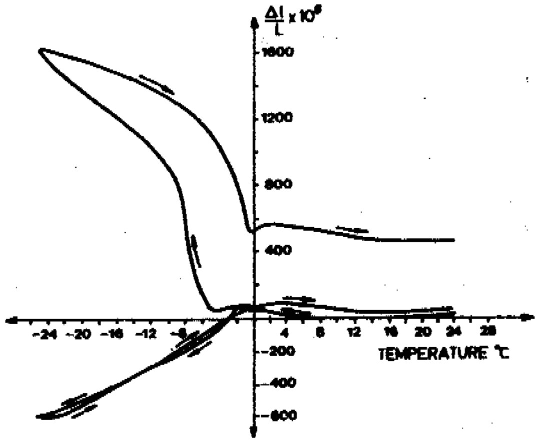

Figure 1 : Dilatometric curves of cement pastes - Upper curve shows dilatation produced in paste containing no air bubbles. Lower curve shows same paste with entrained air. DL/L = length change in millionths (Powers, 1958).

OUR MEASUREMENTS

Figure 2 shows the experimental device with which we have made dilatometric measurements. The experiment samples are cylinders of limestone ± 10 cm in length and 4 cm in diameter. The main peculiarity of our system is that we not only measure the variations in length of cylindric sample in one place but in several locations : in the central point of the circular base, near the border of the same base, and also on two points which are facing each other on the sides of the cylinder. With all these measurements, we register not only the variations in length of the cylinder but also its variations in diameter.

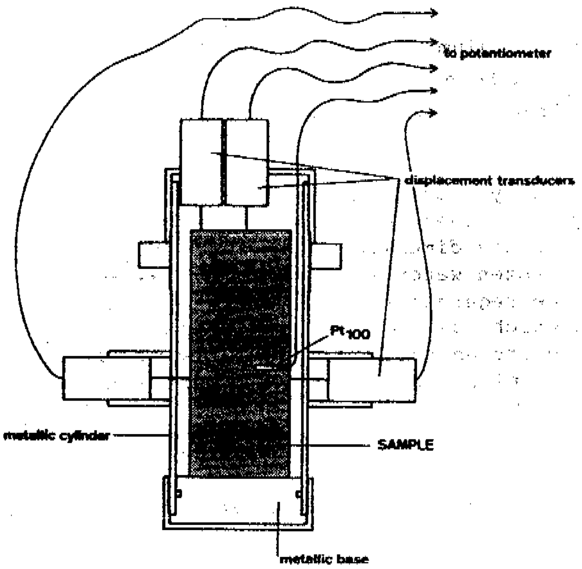

Figure 2 : Experimental device used in our experiments.

The experiments that we present below were made with two kinds of rock : the stone of Caen which is a bathonian bioclastic limestone and a turonian "Tuffeau" called Brézé. The porosity of the stone of Caen is between 32 and 33,2 %; for the Brézé, the value is between 46,6 and 48 %.

The measurements were made with linear electronic displacement transducers made by Schlumberger (Sangamo) which give variations in length with an accuracy of 1 mm or less. The bases of the studied samples were stuck inside a steel cylinder on which the displacement transducers were set. The dilatation curves that we present below were corrected for the coefficient of expansion of the steel cylinder.

Results of our Measurements

The interpretations of dilatometric curves obtained during freezing and thawing were illuminated by the results of dilatometric measurements made on the same calcareous rocks during dessiccation experiments (Pissart and Lautridou, 1984; Hamès et al., 1987). In these papers we have shown that important changes in the length of cylinders of the same rocks occur when the water content fluctuates. Variations in length are at the highest when the water moves in or out of the thinner fissures of the rocks. This result was demonstrated when we observed that the loss of a very small amount of water gives in samples with very little water an important dilatometric shrinkage. This contraction in length was by far less important when the sample held more water and lost the same weight by dessiccation.

The presentation of dilatometric curves that we give below will demonstrate the influence of the water content, of the porosity of the rock, of the slight alterations of the experimental device and the rate of cooling, all factors which are well known as controlling gelifraction processes.

Influence of the Water Content inside the Sample

We shall see the effect of variations in the water content of Brézé samples through the comparison of dilatometric curves recorded for a cylinder which contains 56,8 g of water (which represents 98 % of the full saturation) with a cylinder which held 42,1 g of water that is to say 72 % of its full saturation. The rate of change in temperature during both experiments was 2 °C/h.

Figure 3 gives a view of the variation in length and in diameter observed for the sample with 98 % of his full saturation. On this graph, we recognize the successive phases :

1. Contraction of 20 mm in length and dilatation of 10 mm in diameter when the sample goes from 20 °C to 0 °C.
2. Great increase in length and in diameter when the sample remains at 0 °C (zero curtain).
3. Small increase in length when the temperature of the sample goes down until -10.7 °C; the diameter increases during a longer time.
4. Low shrinkage in length and in diameter during the 16 hours when the sample remains at a temperature of -12,5 °C.
5. Important contraction during the warming until 0 °C and the zero curtain.
6. Increase in length and in diameter when the warming occurs above 0 °C.

These different parts of the curve may probably be explained by the processes described now, taking into account the numbers given above :

1. Thermic contraction and migration of water inside the sample by the formation of a gradient of temperature inside the cylinder.
2. Freezing of the free water in the rock almost at the full saturation.
3. The adsorbed water freezes at temperatures below 0 °C.
4. Unfrozen water remains at -12.5 °C. Some of this water is moving inside of the sample to pores which are void.

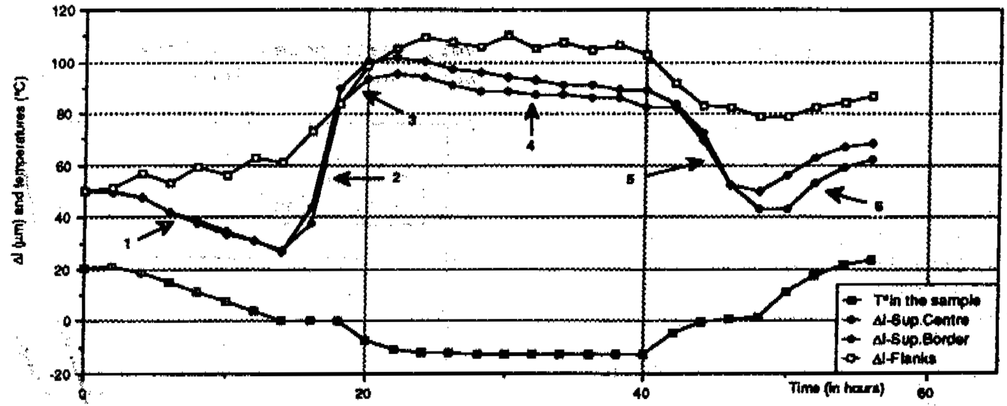

Figure 3 : Dilatometric curves recorded for a cylinder of "tuffeau de Brézé" with 98 % of the full saturation. Rate of cooling 2 °C/h. The numbers indicate the different phases described in the text.
Legend : T° in the sample = temperature measured with Pt100 inside the sample; Δl = dilatometric variations in µm; Sup.Centre = measure on the central point of the superior face of the cylinder; Sup.Border = measure on the border of the superior face of the cylindre; Flanks = sum of the measures made on the sides of the cylinder.
All our dilatometrical measurements are put to the initial value of 50 in order to facilitate the comparision between curves.

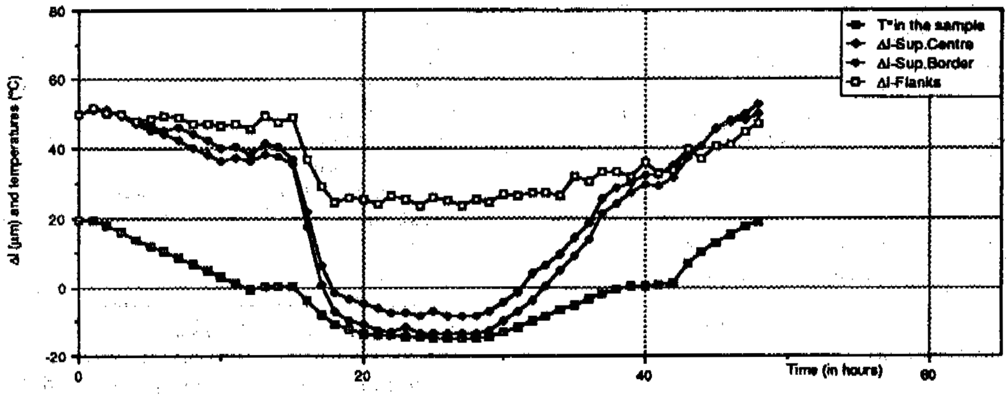

Figure 4 : Dilatometric curves recorded for a cylinder of "tuffeau de Brézé" with 72 % of the full saturation. Rate of cooling -2 °C/h. See legend below figure 3.

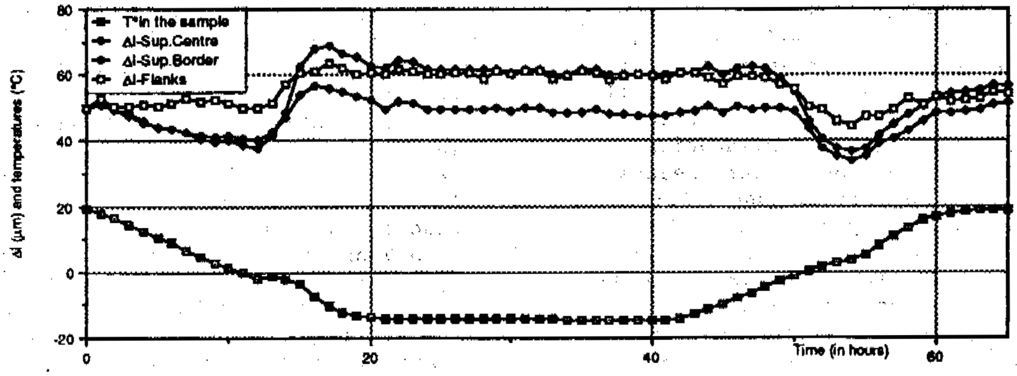

Figure 5 : Dilatometric curves recorded for a cylinder of stone of Caen with 77 % of the full saturation. Rate of cooling -2 °C/h.
See legend below figure 3.

5. During the warming when the temperature remains below 0 °C, the ice which is under pressure and which is in contact with some salts begins to melt or allows a shrinkage of the rock.

6. The water which comes from the melting of the ice goes back into the thinner pores of the rock and gives a dilatation. At the same time a thermal dilatation occurs.

With 72 % of the full saturation, below 0 °C, the dilatometric curve becomes completely different (figure 4) :

The retraction of the sample above 0 °C is related mainly to the thermal coefficient of dilatation. The freezing of the free water which occurs at 0 °C (zero curtain) did not give any dilatation because there were enough voids without water inside the sample to accept the change in volume of the freezing water without a significant increase in pressure. After the zero curtain, the sample undergoes a very important retraction which is the same phenomenon as the one described by Thomas (1938) that we have mentioned in the introduction of this paper. The variation in diameter is similar to the variation in length if it is remembered that the length of the studied cylinder is 10 cm and its diameter 4 cm. By comparison with the interpretation of Powers (1958) we believe that this retraction results from the migration of water from the very thin fissures present in the rock to the nearest empty larger pores where ice crystals are growing.

A slow migration of water goes on when the temperature oscillates around -15 °C.

During the warming, the movement of the water going back into the thin fissures is the main reason why dilatation occurs.

Influence of the Porosity of the Rock

With a similar amount of water (77 % of the full saturation), the curves recorded with a cylinder of stone de Caen (figure 5) do not show the shrinkage we have seen under the same conditions in the sample of Brézé (figure 4). As soon as the freezing begins (moment clearly visible on the temperature curve by the small increase in temperature which occurs inside the sample), we observe a small dilatation which continues until the temperature reaches -1 °C. Below this temperature a contraction occurs and this probably appears when the migration of water from the smaller pores and fissures exceeds the dilatation related to the increase in volume by water freezing.

The different responses of the Caen and Brézé stones must be seen in connection with the characteristics of porosity of the two rocks (figure 6). The Brézé stone has a great number of pores smaller than 0,05 mm. These very small pores are known to induce important shrinkage by dessiccation.

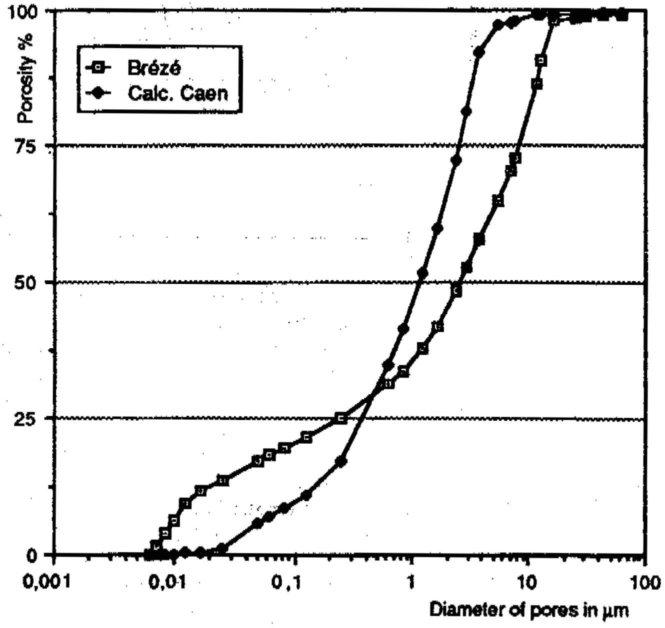

Figure 6 : Distribution of the pore size in stones of Caen and in "tuffeaux de Brézé" as they are measured with a mercury porosimeter. The percentage of the pores under 0,15 mm is higher in the "Brézé" than in the stone of Caen. The difference between these two limestones is greater for the pores below 0,025 mm.

Significant Influence of Slight Alterations of the Experimental Device

The experiment presented in figure 5 like the other ones we have presented till now had occured in our refrigerator in which there is no ventilation system. For the experiment shown in figure 7, an electric fan was set inside the cold room to mix the air during the entire experiment. These cooling conditions, with a cold air current which blows horizontally, are different from the conditions used for the previous experiments, and consequently we notice differences in the dilatometrical behaviour.

We had seen on figure 5 a pronounced dilatation in length and in diameter in connection with the freezing of free water. Figure 7 shows a dilatometrical response in length which is different from that in diameter, if we remember that the length of the cylinder is 10 cm, and its diameter, 4 cm. The behaviour shown on this figure is very different from the one observed on figure 5. This seems normal since it is known that these behaviours are related to different water

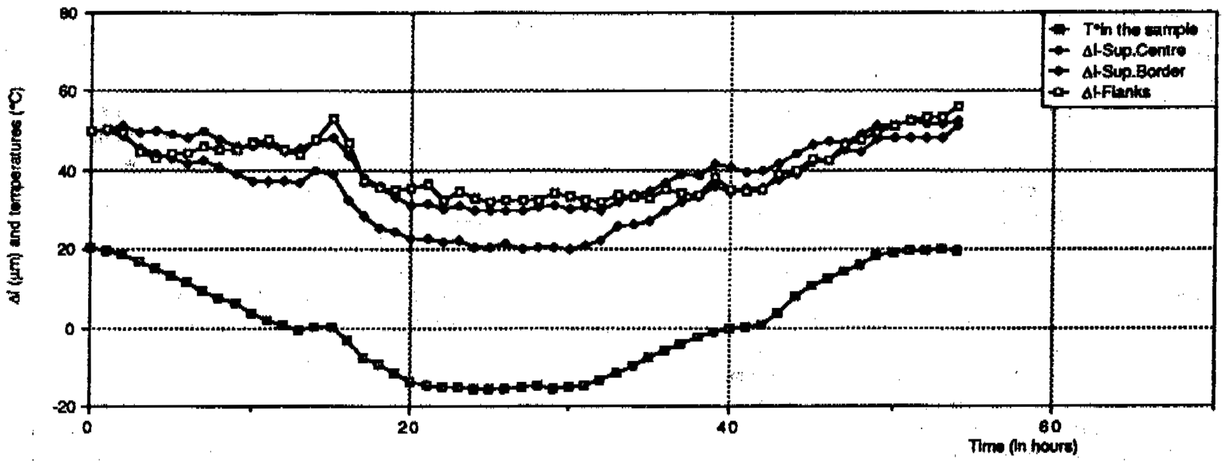

Figure 7 : Dilatometric curves for a stone of Caen with 77 % of the full saturation. Rate of cooling -2 °C/h. An electric fan has mixed the air in the refrigerator during the entire experiment. See legend below figure 3.

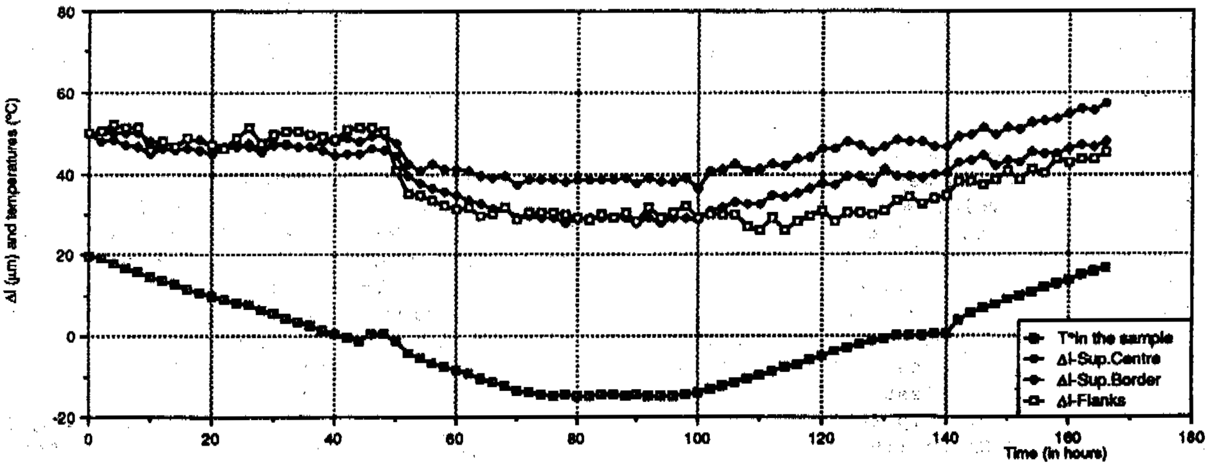

Figure 8 : Dilatometric curves for a stone of Caen with 72 % of the full saturation. Rate of cooling -0,5 °C/h. See legend below figure 3.

migrations induced by different directions of the freezing front propagation.

Measurements of the distribution of H_2O inside a cylinder of Brézé frozen under the same conditions have shown that some water was moving from the center of the cylinder to the sides. This migration of water, induced by the location of a lateral gradient of temperature during the freezing explains the anisotropic character of the dilatation of the sample. These observations show clearly that it is impossible to understand the dilatometric response of a rock if measurements are not made in different directions.

Influence of the Rate of Cooling

The comparison of the dilatometric curves given on the figures 5 and 8 illustrates the influences of the rate of cooling of the samples. With a rate of 2 °C/h a dilatation was observed during the freezing. With an amount of water that is not very different (72 % against 77 %), the experiment shown on figure 8 with a rate of cooling of 0,5 °C/h provokes only a contraction of the sample. It seems that with a very slow freezing, the gradient of temperature was not important enough to give migrations of water and a dilatation. However, a slow freezing allows the migration of water from the narrow fissures to the larges pores where it freezes.

CONCLUSIONS

These experiments show the complexity of the dilatometric variations of wet samples of rocks undergoing freezing and thawing. The increase in volume related to the transformation of water in ice is the main mechanism when the sample is close to saturation; it becomes less important when the voids in the rocks are not filled with water. In this case, the migration of water becomes the main process. Transformation of water in ice gives a dilatation of the rock; migration of water from the small pores and fissures to larger empty pores gives a contraction of the rock.

It has been known for many years that the factors we have discussed here, i.e. the content of water in the sample, the distribution of the porosity in the rock, the influence of slight alterations of the experimental device and the rate of freezing are important parameters in the gelifraction of the rocks.

Here, we have shown that these influences may be seen on dilatometric curves and that their role in gelifraction may be approached in a single freezing cycle. We believe that the dilatometric research will become an important method to analyze the gelifraction process in different materials. Because it clearly shows the influence of the displacement of water inside the rocks which correspond to a process of dessiccation and hydration in the smallest fissures, the dilatometric research may help answer the aggressive question of White (1976): "Is frost action really only hydration shattering?".

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