

Characterizing Curing-Cement Slurries by Permeability, Tensile Strength, and Shrinkage

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Summary

This work was carried out to obtain more knowledge about the transition period of curing oilwell cements. The results show that the curing characteristics are a function of temperature and that there is a correlation between shrinkage and cement content. The paper also introduces a new mechanism for gas migration and discusses how the studied parameters can be used to predict gas migration.

Introduction

The setting process of cement slurries in oil wells is very complex. Many parameters contribute to the final result, such as gelling, shrinkage, temperature, pressure, filter loss, cement structure and strength buildup, slurry permeability, entry pressure, capillary pressure, mud and mud cake, formation properties, well history, and possibly other parameters as well. Some of these parameters are next to impossible to characterize. Others are simple to measure in a laboratory setup, but may not reflect downhole conditions. The cement setting process has been investigated extensively, but there are still many factors not fully understood. We have tried to extend the knowledge by monitoring temperature evolution, hydrostatic pressure, permeability, tensile strength, and total chemical shrinkage during hydration. We will in turn discuss the importance of these parameters.

Cement hydration is an exothermic reaction which can be observed as a temperature increase. Temperature is easy to measure, and the shape and peak of the temperature curve give valuable information on the hydration process, i.e., hydration onset and rate. The hydrostatic pressure is important as gas flow into the cement will be initiated when the pressure of the cement column falls below that of a gas bearing formation.¹⁻³ This pressure drop is due to cement shrinkage at the same time as the shear strength develops, enabling the cement to hang onto the wellbore and casing.

But what mechanism will govern inflow of gas when the cement pressure has dropped far enough? We think the capillary entry pressure of the cement pore structure is important. When exposing gas to the water-saturated cement, the nonwetting gas phase has to overcome the entry pressure of the cement pore system due to the interfacial tension between cement pore fluid and gas. The entry pressure is high when the pores are small while the permeability on the other hand is low, and entry pressure is, in general, inversely proportional to permeability. After having overcome the entry pressure, the relative permeability and the differential pressure between the formation gas and the cement column

control how much cement pore water will be displaced by gas. This is a complicated process where the permeability and pressure change continuously.

Considering how important the permeability is for governing flow into and through the cement pores, surprisingly little work has been carried out on permeability during setting. Sutton and Ravi⁴ state that low fluid loss slurries exhibit a permeability of less than 100 md at a static gel strength of 200 lb/100 ft² and that it approaches 5 md at 500 lb/100 ft². Plee *et al.*⁵ have studied the permeability of bentonite-cement slurries and found a typical value of 50 to 100 md for fresh slurries and that decreasing permeability correlates linearly with increasing surface area. Only Appleby and Wilson⁶ have monitored the permeability at several points of time up till and past final set. Their results show an initial permeability about 1 darcy falling down to around 1 md at the temperature peak.

When the cement slurry is in the process of losing its hydraulic (liquid) properties, the strength of the cement matrix is still low. The pressure difference between the formation gas and the hydrostatic pressure of the cement slurry may overcome the strength of the matrix. In this situation it is the compressional strength of a confined cement which is of importance and this strength parameter is the highest one. Therefore, we think that it is not very probable that gas will break the matrix from outside and migrate through the created microfractures. However, if gas has entered the cement through its pores, it is the tensile strength of the cement which has to be overcome in order to break the cement matrix. The tensile strength is the lowest strength parameter, and we believe that this is a likely mechanism of fracturing the cement, leading to gas migration.

We, therefore, propose a new mechanism for initiating gas migration. After the gas has entered the pore system of the cement, the gas inside may overcome the tensile strength of the cement structure, break the cement matrix, and migrate through the microfractures. During the hydration process all the studied parameters change continuously. The hydrostatic pressure inside the cement slurry column drops and the gas bubbles already inside the cement will, therefore, try to expand. The decreasing pore radii of the cement matrix stop this expansion and the bubbles may maintain their pressure while the cement pressure will continue to decrease. This leads to a pressure difference which may be large enough to overcome the tensile strength of the cement structure and thus creating fractures.

The hydrostatic pressure drop of the cement column is mainly governed by the chemical shrinkage of the cement. Before initial set, a low shrinkage rate is preferable because the resulting hydrostatic pressure decline will be slower than for a slurry with a higher shrinkage rate. Slow shrinkage has two advantages: pressure equilibrium between formation and slurry column is reached at a later point of time and afterwards the pressure difference between formation and slurry, the driving force behind flow of pore fluid into the cement, will be lower. Both factors should reduce the risk of early time gas migration. After initial set, how-

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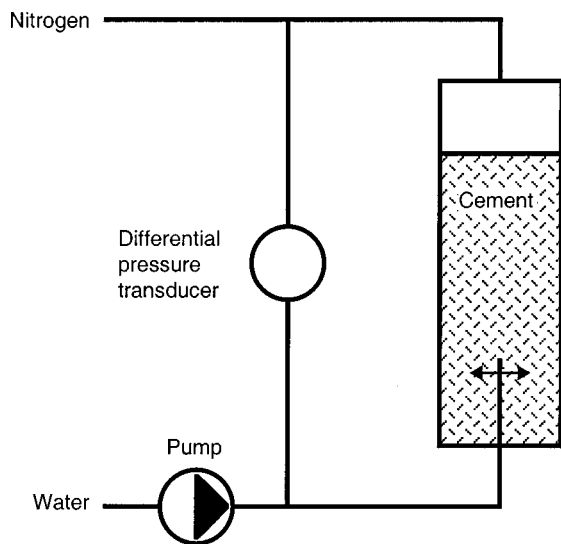


Fig. 1—Principle of the permeability and fracturing setup: water is pumped into the cement causing a pressure increase.

ever, when high shrinkage and pressure loss are inevitable, a short transition time will reduce the time during which gas migration can be initiated.

The chemical shrinkage may be divided in two parts, external and internal. The external shrinkage is the bulk or external dimensional volume change of the slurry leading to a possible microannulus between the cement and the wellbore. The internal chemical shrinkage is caused by formation of contraction pores which contribute to the connectivity between pores in a set cement, and hence, to permeability. The total chemical shrinkage is the sum of the external and internal shrinkage.

Chemical shrinkage of oilwell cements has been investigated by several researchers. The most extensive tests have been carried out by Chenevert and Shrestha,⁷ and by Sabins and Sutton,⁸ but other papers have also been presented.⁹⁻¹¹ The applied temperatures and pressures range from ambient conditions⁹ up to 224 °C and 121 MPa.⁷ Both total⁸⁻¹¹ and external chemical shrinkage⁷⁻⁹ were reported with a total shrinkage at 20 or 24 hours varying from 0.6 to 6 vol%, while most results were in the range of 1.5 to 3 vol%. Only Sabins and Sutton⁸ have performed measurements of external shrinkage under realistic downhole conditions. According to them, most of this shrinkage occurs when the slurry still is plastic. Their results showed an average of 0.15 vol% and from this they calculated the contraction pores to account for 97.5 to 99% of the total shrinkage. Thus, from a gas migration point of view, the formation of contraction pores is by far the largest and most important part of the chemical shrinkage.

Experimental Setup and Procedure

Permeability and Tensile Strength. The permeability and the tensile strength of a cement slurry were measured in the same cell where measurements can be done at temperatures up to 200 °C and at pressures up to 20 bars. In addition, the temperature evolution and hydrostatic pressure were recorded. **Fig. 1** shows the setup with the cell filled with cement slurry where the diameter of the cell is 5 cm and the height of the cement column is 25 cm. The water inlet is placed 5 cm from the bottom of the cell. By using four cells, measurements can be done at four different time points after mixing, and thus, generating a trend curve. To map the whole permeability and strength development 2 to 4 of these four-cell measurements had to be carried out. The top of the cell, above the cement slurry, is filled with nitrogen gas and the system is pressurized from 15 to 20 bar. A pressure transducer measures the

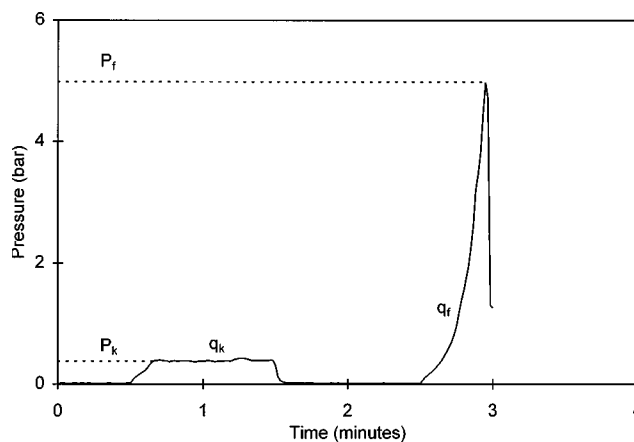


Fig. 2—Pressure behavior during a permeability and strength test.

differential pressure between the water and the gas lines which is used to find both the permeability and tensile strength. All parameters were recorded by a data acquisition card and stored in a personal computer. The slurries were prepared and mixed in accordance with API Spec. 10.¹²

Before a new slurry was tested for permeability and strength, one nondestructive test was carried out to obtain complete temperature and pressure curves for the whole setting process. This initial run was also an aid to choose the points of time when to run the permeability and strength tests.

To find the permeability, a small rate of water ($q_k=0.1$ to 0.33 mL/min) was pumped into the cement slurry until a constant pressure p_k was obtained. This is shown in **Fig. 2** where the first part of the pressure/time curve represents this part of the experiment. When the pressure reached a constant level during pumping, the flow was assumed to be Darcy flow, enabling computation of the slurry permeability.

The tensile strength measurements are based on the principle of hydraulic fracturing and were usually done 1 minute after the permeability test. A high rate of water ($q_f=5$ to 20 mL/min) was pumped into the cement slurry and the pressure increased rapidly to a maximum value p_f . At this point the tensile strength of the cement slurry was exceeded, a fracture developed, and the pres-

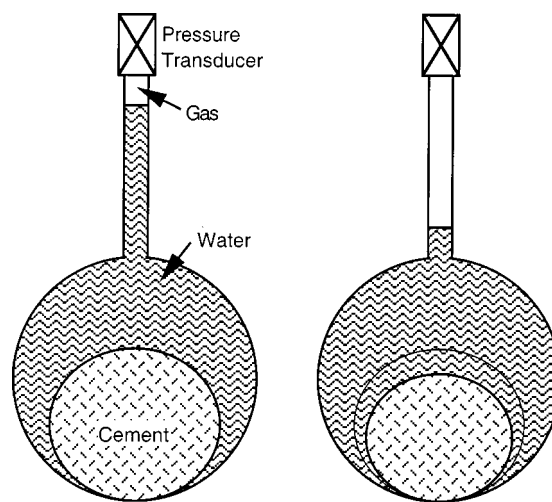


Fig. 3—Principle of the shrinkage measurements: initial conditions in the left-hand figure and to the right, conditions after some time.

TABLE 1—CEMENT SLURRY DATA, RECIPE AND RESULTS

		Slurry							
		T	B	A	C	D	E	F	G
		Slurry Data							
Test Temperature, °C		90	180	140	140	140	140	140	140
Slurry Density, g/cm ³		1.90	2.15	2.05	2.03	2.06	2.07	1.98	1.88
		Slurry Recipe							
Additive	g/cm ³								
API Class G Cement	3.22	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Fresh Water, l/hk*	1.00	43.24	54.86	45.52	39.96	44.28	44.28	44.28	44.28
Weight Material, % bwoc**	4.85		74.87	38.77	13.88	13.88	13.88	13.88	
Antistrength Retrogr., % bwoc	2.65		26.03	25.97	30.00	30.00			
Antigas Migration, l/hk	1.40		13.00	13.00	12.00				
Dispersant, l/hk	1.21		3.00	3.00	3.00	3.00	3.00	3.00	3.00
Retarder, Low Temp., l/hk	1.20	0.80							
Retarder, Medium Temp., l/hk	1.18			1.25	1.25	1.25	1.25	1.25	1.25
Retarder, High Temp., % bwoc	1.25		0.80						
Antifluid Loss, l/hk	1.04		3.00	5.00	3.00				
Calcite Flour % bwoc	2.72						30.00		
		Gas Tightness							
Gas Tightness in Test Rig		Leak	Tight	Tight	—	Tight	—	—	—
Gas Tightness Number, F_{gt}		21.20	4.9	0.6	—	3.5	3.9	4.7	8.0
		Shrinkage							
Cement, vol%		41.35	23.74	26.63	29.73	33.12	33.22	37.67	39.02
Shrinkage at 20 hours, vol%		3.92	2.61	1.79	1.98	2.58	2.49	2.84	3.11
Shrinkage at 20 hours, mL/100 g Cement		2.94	3.41	2.09	2.06	2.42	2.33	2.34	2.48
*Liters per hundred kilos of cement.									
**Percent by weight of cement.									

sure dropped instantly. In Fig. 2 the second peak of the pressure/time curve represents the fracturing pressure. After the test, a horizontal fracture at the level of the water inlet could be observed in the cement and the maximum strength that can be measured with the apparatus is 5 bar. At this strength the cement has reached final set and is quite hard (for comparison, sandstones have a tensile strength of 40 to 150 bar).

Shrinkage. For the shrinkage measurements a second cell was made. The measurements were based on the pressure/volume relationship of a gas. The principle is shown in Fig. 3, indicating initial conditions in the left-hand figure. At a later stage, shown in the right of Fig. 3, the shrinkage process leads to a lower gas pressure. When the initial temperature, pressure, and volumes are known, the shrinkage can be calculated on the basis of the continuously declining pressure during the test. The calculation is also corrected for gas temperature. Total shrinkage was measured by placing the cement in a slightly permeable paper cup containing around 50 mL of slurry. All samples were weighed before and after each test, and the volume of the hardened sample was found by applying Archimedes' principle. The applied pressure was in the range of 6 to 16 bar, which was measured with an accuracy of ±0.15%. All parameters were recorded by a data logger and stored in a personal computer.

The method works well but has some drawbacks. It requires precision in the initial values and it is sensitive to temperature changes. The shrinkage calculations are taken from the point of maximum pressure or when the test temperature stabilizes because we were unable to quantify the water expansion that occurs before

this point of time. This has the advantage that any shrinkage occurring before placement of the cement in a well will not be taken into account, even though some initial shrinkage may not be recorded.

Results and Discussion

Our tests were mainly performed at 140 °C, although the test program also included experiments at lower and higher temperatures as well. The recipes and data of the slurries presented in this paper are shown in Table 1. Slurry T is a very simple test mixture which

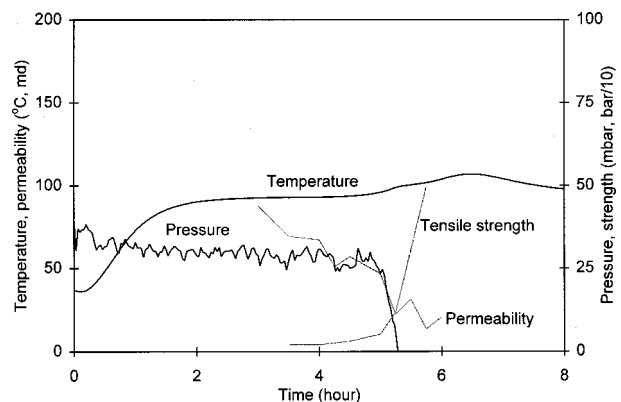


Fig. 4—Slurry T at 90 °C: temperature, pressure, strength, and permeability.

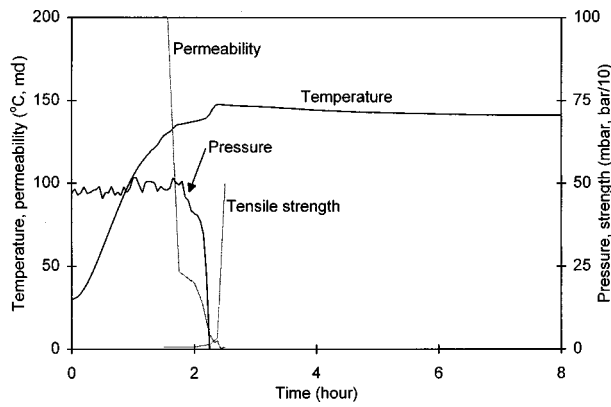


Fig. 5—Slurry A at 140 °C: temperature, pressure, strength, and permeability.

is not gas tight, while slurries A and B are commercial recipes containing all the necessary additives to avoid gas migration. Slurries C-G will be discussed below. The permeability, tensile strength, and shrinkage results of slurries T, A, and B are presented in Figs. 4 through 9, where the shrinkage is given as both vol% and mL/100 g cement. The mL/100 g values are a measure of degree of hydration.

It is difficult to compare our permeability results with some of those from the literature^{4,5} because of differing conditions. However, Appleby and Wilson's⁶ data compare well with our results where the curve shapes and the permeabilities at the temperature peak are similar. Our shrinkage results at 20 hours fall within the range presented in the literature.⁷⁻¹¹

Temperature Influence. From Figs. 4 through 9 it is evident that there is a considerable difference in behavior between the three slurries at 90, 140, and 180 °C. For slurry A at 140 °C all parameters change very rapidly when the hydration starts, whereas the other two slurries at 90 and 180 °C are slower. For all three slurries the temperature peaks coincide with the most marked changes of the other measured parameters, and the higher the cement temperature is above the set temperature, the more rapid are these changes. As an example, slurry A with the most pronounced temperature peak also shows the fastest initial shrinkage up to 1 vol% and thereafter a decaying shrinkage rate and temperature. The temperature peak of slurry B is the lowest corresponding to the slowest initial shrinkage (see Fig. 9). The same is true for its permeability and tensile strength. This correlation is to be expected as the temperature evolution is a measure of the rate of

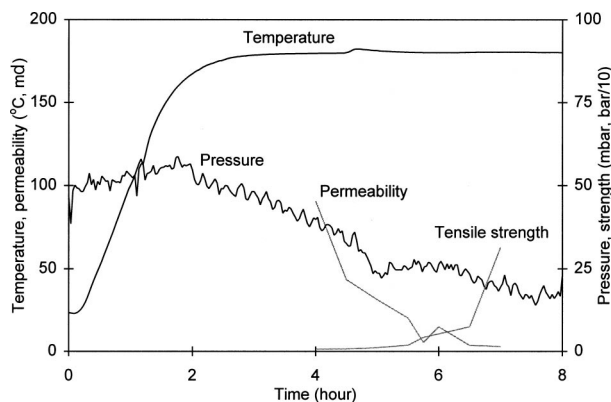


Fig. 6—Slurry B at 180 °C: temperature, pressure, strength, and permeability.

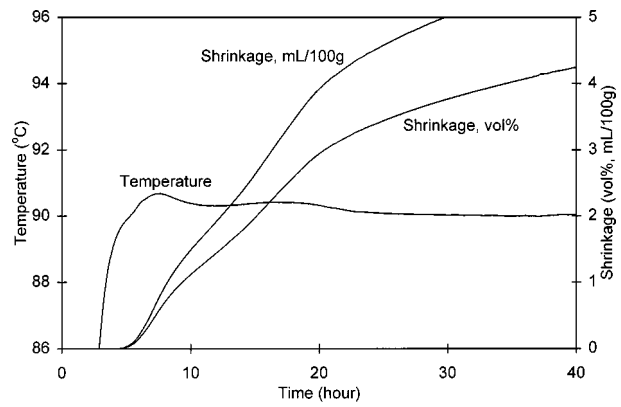


Fig. 7—Slurry T at 90 °C: temperature and shrinkage.

hydration, and hence, the parameter behavior. Later in the curing process, after around 15 hours, the shrinkage measured in mL/100 g is higher for slurries T and B than for slurry A. More interesting from a hydration point of view, though, are the two temperature peaks of slurries T and B, the first being at 5 to 7 hours and the second around 15 to 20 hours, seen in Figs. 7 and 9.

We also tested several other slurries at the temperatures 90, 140 and 180 °C and they exhibited the same behavior, demonstrating that this is a general trend. A closer look at the literature also confirm this behavior. The freshwater-based slurries of Chenevert and Shrestha⁷ at 93, 121, and 177 °C are not strictly comparable as they measured the external and not total shrinkage. Nevertheless, their medium temperature slurry does show the fastest initial shrinkage. Sabins *et al.*¹³ carried out a substantial number of compressive strength tests and their 24 hour results are plotted in Fig. 10. Apart from two slurries at 143 °C with a strength above 20 MPa, the strength of the cements above 135 °C is lower than those below this temperature. Sabins and Sutton¹⁴ extended this work and the results follow the same trend although not so clearly.

However, one question remains, how can this temperature influence be explained? The recipes of the slurries A and B at 140 and 180 °C, respectively, are very similar, but their behavior is quite different, implying that this difference may be a temperature effect on the cement hydration chemistry. We leave this question open.

Gas Tightness. With respect to gas migration, a short transition period (as with slurry A) will limit the period in which gas can enter the cement, thus reducing the hazard of gas migration. All three slurries were also tested in the gas migration rig of Jamth *et al.*,¹⁵ where the T slurry was leaking and the other two were

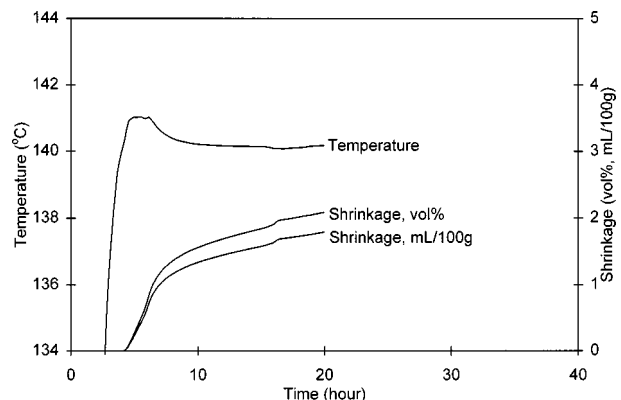


Fig. 8—Slurry A at 140 °C: temperature and shrinkage.

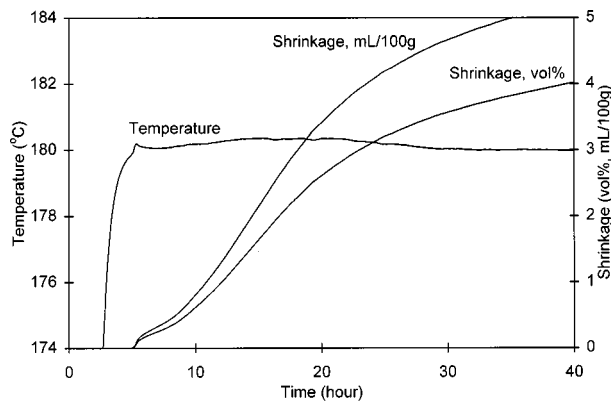


Fig. 9—Slurry B at 180 °C: temperature and shrinkage.

tight. This may be due to the simple composition of this slurry.

In order to relate the hydrostatic pressure, tensile strength, and permeability to gas tightness, a systematic interpretation of the results for several slurries were performed. For the untight cements, the hydrostatic pressure dropped almost immediately down to the water column pressure (30 mbar in Fig. 4), stayed constant for some time, and then fell abruptly. The gas tight pastes in Figs. 5 and 6 were better at maintaining the hydrostatic cement pressure until the onset of hydration. Buildup of tensile strength was generally also faster for the tight cements, indicating the importance of a short transition period.

Based on the tensile strength buildup we have defined a time window, $\Delta t = t_2 - t_1$. The two points of time t_1 and t_2 represent a tensile strength of 0.3 and 5 bars, respectively. At time t_2 the cement is hard. The strength buildup should be rapid, thus Δt should be small. For the basic slurry, we found in Fig. 4 that Δt is 2 hours, while in Fig. 5 Δt is 15 minutes for the gas tight slurry A. Slurry B at 180 °C exhibited a time window of 1.6 hours. At the same points of time, the permeabilities k_1 and k_2 are recorded. In order to avoid gas intrusion into the pores, the permeability should be low within the time window. The theoretical initial hydrostatic pressure of the fresh cement slurry is denoted p_i . The drop of the hydrostatic pressure at the time when the strength starts building up, i.e., p_1 at t_1 , should be small. Therefore, the value of $(p_i - p_1)$ should be small.

The three parameters, the time window, the permeabilities, and the hydrostatic pressures may be combined in various ways to find

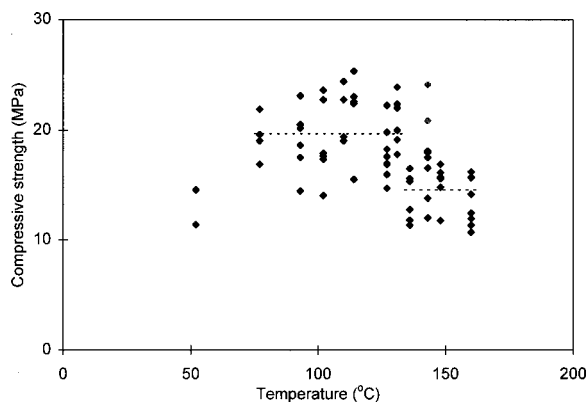


Fig. 10—Compressive strength at 24 hours. The dotted lines represent the average within the two temperature ranges (excluding the two gray points). Data from Sabins *et al.* (Ref. 13).

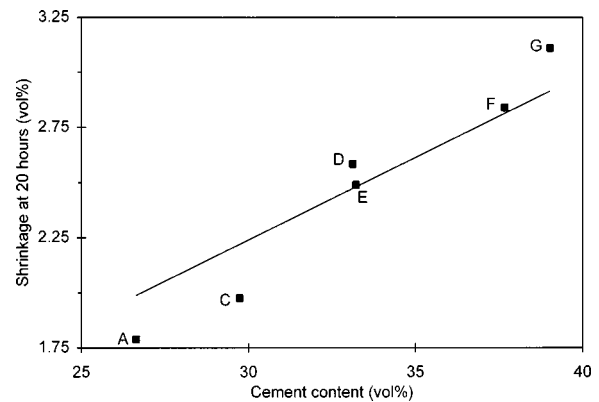


Fig. 11—Shrinkage at 20 hours vs. cement content.

a factor that can characterize a cement's ability to resist gas migration. We have tested the following combination of the parameters, called the gas tightness factor:

$$F_{gt} = \sqrt{\Delta t} \times \frac{k_1 - k_2}{k_1} \times k_2 \times \left(\frac{p_i - p_1}{p_i} + 1 \right). \quad (1)$$

From the discussion above, we conclude that the factor should be as small as possible to have a gas-tight cement.

In Table 1 the leaking slurry T has a F_{gt} value above 20 whereas the three tight slurries exhibit values below 5. Thus, based on these slurries, the threshold is somewhere between 5 and 20. To further test the F_{gt} factor, slurry A was used as a starting point for simplifying the cement composition to study the influence of additives on gas migration. The recipes and F_{gt} factors of slurries D–G are shown in Table 1 and the results show that simplifying the recipe will increase the F_{gt} factor. These slurries were not tested in the gas rig, but slurry G with a value of 8 would probably leak due to its simple recipe. Later results, however, show some overlap between tight and leaking slurries; but the general trend is still clear, i.e., a short transition period is essential for a gas-tight cement.

Shrinkage vs. Cement Content. With one addition, the 140 °C slurries tested for the F_{gt} factor were also tested for shrinkage. The recipes and data of slurries C–G are shown in Table 1. The shrinkage at 20 hours was found to be correlated to the cement content, as shown in Fig. 11, where the curve is forced through zero, as zero-cement content should yield no shrinkage. This correlation is self-evident and has been reported previously,^{7,8} without being linked to the gas migration problem, however. As long as the strength development of the cement slurry is satisfactory, more use of inert extender will reduce the shrinkage and the risk of gas migration. The data in Table 1 support this conclusion, where the only leaking recipe, slurry T, shows the highest absolute shrinkage at 20 hours.

Conclusions

1. Two cells were successfully developed to study hydrostatic pressure, temperature evolution, tensile strength, permeability, and total shrinkage during cement hydration.
2. A new mechanism for gas migration is proposed. First, the gas has to overcome the entry pressure of the cement pores, and once inside the cement (caused by the continuously changing pressure conditions) the gas may fracture the cement structure.
3. There is a marked contrast in behavior of slurries at 90, 140 and 180 °C where the 90 and 180 °C slurries exhibit two temperature peaks. This difference is most likely due to a temperature effect on the cement hydration chemistry.

4. To predict migration problems, a gas-tightness factor based on the collected data was defined and compared to results in a gas migration test rig. A short transition period is important.

5. There is a positive correlation between total chemical shrinkage and cement content. A low shrinkage will reduce the risk of gas migration.

Nomenclature

- F_{gt} = gas tightness factor as defined in Eq. (1)
 k = permeability, L^2 , md
 p = pressure, m/Lt², bar, mbar, MPa
 q = flow rate, L³/t, mL/min
 t = time, t , hour, minute
 Δt = time difference, t , hour, minute

Subscripts

- 1,2 = point of time
 f = fracture
 i = initial
 k = permeability

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SI Metric Conversion Factors

bar × 1.0*	E-01 = MPa
°F (°F-32)/1.8	= °C
ft × 3.048*	E-01 = m
g/cm ³ × 1.0*	E+03 = kg/m ³
in. × 2.54*	E+00 = cm
lb/100 ft ² × 4.788 026	E-01 = Pa
lbm × 4.535 924	E-01 = kg
md × 9.869 233	E-04 = μm ²
mL × 1.0*	E+00 = cm ³
psi × 6.894 757	E-03 = MPa
gal × 3.785 412	E-03 = m ³

*Conversion factors are exact.

SPEDC

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