Advances in Cement Research, 1997, **9**, No. 34, April, 47–54

A new technique for measuring permeability and tensile strength of a curing oil well cement

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Gas migration through cement is a problem in many wells around the world. A study of the behaviour of cement slurry during the curing period has been performed in order to relate to slurry properties such as intrusion and migration of gas in the cement. A new method for measuring the permeability and the strength of a curing cement has been developed. At specific times during the curing, water is pumped into the cement paste at a very low rate while pressure is recorded. The pumping is then stopped for a short time while the pressure drops back to normal pressure. The pump is then restarted at maximum rate causing the curing cement to fracture. The fracturing pressure appears as a sharp peak in the pressure against time curve. Using the flow rate and the pressure recording during the first low-rate pumping period, the permeability is calculated. The tensile strength of the cement is proportional to the peak pressure of the second high-rate pumping pressure. The method can be used throughout the whole time interval of interest from initial to final set, and has shown good reproducibility. The behaviour of three parameters has been found to be of importance regarding gas tightness: the permeability development, the build up of tensile strength and the hydrostatic pressure drop.

Notation

A	cross-sectional area
g	acceleration caused by gravity
k	permeability
L	distance between pressure points
p_c	capillary pressure
p_g	pressure in gas pipe
p_k	p_w during permeability recordings
p_f	p_w during fracture tests
p_{sl}	initial hydrostatic pressure of cement slurry
p_w	pressure in water pipe
q	flow rate
r	pore throat radius
Δp	$p_w - p_g$
Δt	time window between time t_1 and t_2
θ	wettibility angle (here $\theta = 0$; non-wetting)
μ	viscosity of flowing fluid
ρ	density of cement slurry
σ	surface tension between gas and solid (here $s = 100 \times 10^{-3}$ N/m)
σ_t	tensile strength

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(ACR 221) Paper received 30 January 1996; last revised 4 July 1996; accepted 24 September 1996

Introduction

When an oil well is being drilled, only predetermined sections of the well are drilled before the open hole is cased by cementing a steel tube to the well bore. The drilling is then continued with a smaller drill bit, and a new section of open hole is finished. The steel casing in this section is cemented with an overlap to the previous casing. The functioning of the steel casing is to stop the walls (the wellbore) from caving into the hole and to prevent high-pressure gas and oil escaping the porous geological formation and entering the borehole.

When a casing is cemented across a high-pressure gas zone as in Fig. 1, gas leakage behind the casing may still be a problem. If the quality of the cement is not good enough, gas may migrate from the geological formation, through the cemented annulus between the steel casing and the wellbore, and all the way to the surface.

When a casing is cemented across a gas zone, the critical time period is when the cement is changing from a liquid slurry to a weak solid material. A short time after the cement slurry has been pumped into the annulus between the casing and the wellbore, the hydrostatic pressure of the cement slurry column is higher than the gas pressure. The hydrostatic pressure

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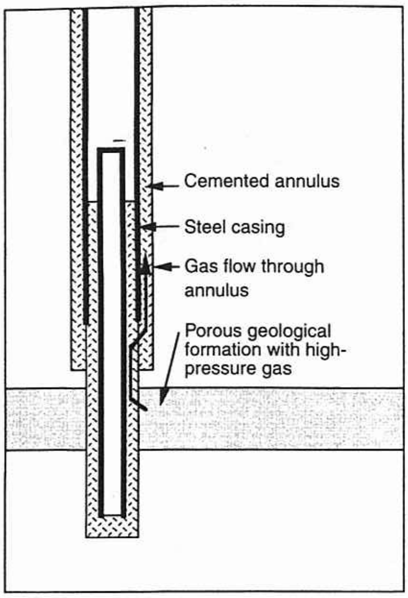

Fig. 1. Cementing operations through gas-bearing geological formations

keeps the gas in place in the formation pores and may even squeeze cement slurry filtrate into the formation pores. During the hydration process the cement slurry changes from being a liquid containing suspended solid particles of cement and solid additives to a solid material with a matrix and liquid-saturated pores.

Several changes in properties may be recorded during this transition: the temperature increases, shear strength builds up, and the hydrostatic pressure drops.[1] This last behaviour is due to shrinkage at the same time as the shear strength develops, and is able to hang the cement on to the wellbore and the casing. The hydrostatic pressure is then created by the water within the cement matrix structure. When the water is consumed in the curing process, the hydrostatic pressure drops even lower. When the hydrostatic pressure drops below the formation gas pressure, gas may start to flow from the geological formation pores into the cement, displacing the fluid in the cement pore structure. The gas must enter either through the pore system of the cement or break the matrix of the cement and enter through the created microfracture. Therefore, the permeability and the strength of the curing cement slurry during the transition period are important parameters.

Entry pressure and permeability

The applicability of the term 'permeability' for a curing cement slurry may be doubtful at the start of the hydration reaction. While the cement is in the state of being a liquid slurry, the solid particles are bound together by weak electrical forces. Therefore, it may be difficult to think of the solid particles as a matrix. The relative movement between the liquid and solids at this stage is caused by settling of the solids, eventually leaving clear water on the surface. This bleeding effect is usually controlled by means of additives. Suhr and Schøner[2] showed that bleeding decreases with increasing specific surface area of the cement powder. The hydration process soon creates solid reaction products of calcium and silica, producing a continuous cement matrix with an initial porosity of the order of 50–60%.

When introducing gas to the water-saturated cement pore system, the non-wetting gas phase has to overcome the entry pressure of the pore system. This is due to the interfacial tension between water and gas, as indicated in Fig. 2. The entry pressure is high when the pores are small; the permeability, on the other hand, is low. Entry pressure is, in general, inversely proportional to permeability.

Justnes *et al.*[3] have determined the pore size distribution of typical oil well cement slurries during hydration. The entry pressure may be estimated by Equation (1) (see also Fig. 2):

$$p_c = \frac{2\sigma \cos\theta}{r} \qquad (1)$$

where r is the pore size, σ is the interfacial tension between gas and water, and θ is the angle between the pore wall and the interface between the gas and the water. If $r = 10\ \mu m$, the entry pressure is of the order of 20 kPa (0.2 bar).

After having overcome the entry pressure, the differential pressure between the formation gas and the hydrostatic pressure of the cement, and the permeability, control how much gas will displace the water. There are few papers on permeability measurements in slurries. Plee *et al.*[4] have studied the permeability of bentonite–cement slurries, and found that decreasing permeability correlates linearly with increasing surface area. From an oil well cementing point of view, it is of interest that the typical permeability (50–100 mD) of a fresh cement slurry was more than three orders of magnitude larger than

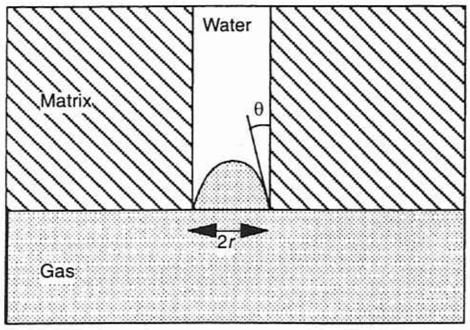

Fig. 2. Gas bubble entering a pore throat

that through a filter made of a pure bentonite suspension.

Cement strength

When the cement slurry is in the process of losing its hydraulic (liquid) properties, the strength of the cement matrix is still low. The differential pressure 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 is the highest strength parameter. Therefore, we consider that it is not very probable that gas will enter the matrix from outside and migrate through the created microfractures.

However, if gas has intruded into the cement through its pores, it is the tensile strength of the cement which has to be overcome in order to break the cement. The tensile strength is the lowest strength parameter, and we assume that this mechanism of breaking the cement matrix from inside leading to gas migration is highly probable. For the purpose of finding a characterizing parameter of the ability of the cement to resist gas intrusion, we therefore consider the hydraulic fracturing process to simulate closely what is occurring when a pressurized gas has entered the cement and fractures it from inside.

Shear strength

Gel strength or shear stress/strength is a parameter which has been focused on in the literature as an important factor controlling gas intrusion. Sabins *et al.*[5] claimed that when the gel strength has reached 500 lb/100 ft^2 (API measurements[6]), gas will not be able to enter the cement slurry. It is not convenient to use the API gel strength parameter to characterize the strength of a curing cement because it has a limited measuring range. The gel strength in the lower range is, nevertheless, an important parameter for describing the ability of a cement to transmit hydrostatic pressure during the early stage of hydration and thus balance the gas pressure from the formation pores.

Tensile strength

Coker *et al.*[7] showed that material strength is a good indicator of gas migration problems. In a survey of eleven cement operations in a deep gas field in the North Sea, gas flow was apparent in four wells. The one common parameter that connected all four failures was the lack of compressive strength at the time of drill-out.

As previously discussed, we consider that the gas pressure will break the cement from inside after it has intruded into the pore system of the cement and migrate through microfractures. Therefore, the development of tensile strength during the critical period of time has to be measured.

Measurements of strength parameters in solid materials are well established and routinely carried out. For a given elastic material there exists a relationship between shear strength, tensile strength and compressive strength through common material-dependent constants. However, in order to measure the strength parameter in cement slurries, as they are in a state between liquid and solid, a new method had to be developed.

Hydraulic fracturing cell

Measuring principles

The permeability and tensile strength of a cement slurry were measured in the same cell. Measurements can be done for temperatures up to 200°C and at pressures up to 20 bar.

Figure 3 shows the set-up with a cell filled with cement slurry. The inner diameter of the cell was 5 cm, and the depth of liquid was 25 cm. The water inlet was placed 5 cm from the bottom of the cell. By using four cells, measurements could be done at four different time intervals after mixing, thus generating a trend curve. To map the whole strength curve development, several four-cell measurements had to be carried out. The top of the cell, above the cement slurry, was filled with nitrogen gas, and the system pressurized with nitrogen gas to 15–20 bar. A pressure transmitter measured the differential pressure, Δp, between the water line and the gas line.

In the water line the pressure, p_w, is given by Equation (2):

$$p_w = p_g + \rho g L \qquad (2)$$

where p_w is the pressure in the water line, p_g is the pressure in the gas line, ρ is the density of the

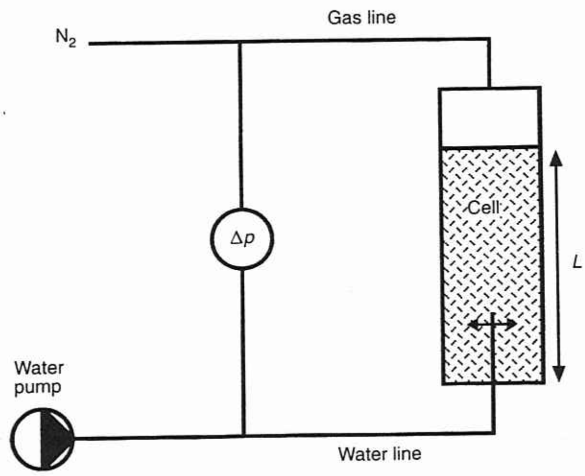

Fig. 3. Cell for testing tensile strength and permeability

cement slurry column, g is the acceleration due to gravity, and L is the height of the cement slurry column.

During the setting period, the hydrostatic pressure and the temperature of the cement slurry were continuously measured. The test was performed by pumping a small amount of water at different flow rates into the cement slurry through a steel tube in the bottom of the cell. We do not consider the small amount of water which is pumped into the cement during the permeability measurement to have any influence on the matrix strength, which is measured afterwards.

Permeability measurement

Initially, water at a low flow rate ($q_1 = 6{-}20$ ml/h) was pumped into the cement slurry until a constant pressure, p_k, was obtained. This is shown in Fig. 4, where the first part of the pressure–time curve represents this part of the experiment. When the pressure reached a constant level during pumping (60 s), the flow was assumed to be a Darcy flow, and the permeability, k, of the cement slurry can be computed:

$$k = \frac{q\mu L}{\Delta p\, A} \tag{3}$$

The permeability measurements can be done while the cement slurry hydrates until the final set is reached and the cement is hard. The permeability figure which was calculated in this way is not the true permeability because the geometrical factor in Equation (3), L/A, is not correct. The formula applies to flow in a tube of equal diameter with length L and cross-section A, whereas we are using water flowing from a point source. However, for comparing permeabilities in cement slurries measured with the same cell geometry and procedure, the apparent permeability figures calculated by Equation (3) may be applied.

During permeability measurements, Plee *et al.*[4] observed a sudden increase in hydraulic water loss when differential pressure was gradually increased. The observed phenomenon could be an indication of hydraulic fracturing. This behaviour was only seen after the induction period (2–3 h), and was interpreted to be associated with a stress threshold representing the rupture of the interparticular bonds generated by the hydration of the cement.

Tensile strength measurement

The method used for the tensile strength measurements is based on hydraulic fracturing. The strength measurement was usually done 1 min after the permeability test. Water at a high flow rate ($q_2 = 600{-}1200$ ml/h) was pumped into the cement slurry. The pressure increased rapidly to a maximum value, p_f ('f' denotes fracturing). At this point the tensile strength of the cement slurry is exceeded, a fracture develops and the pressure drops steeply. In Fig. 4 the second peak of the pressure–time curve represents the fracturing pressure. Afterwards, when cleaning the cell, a horizontal fracture at the level of the water inlet could be observed in the cement core.

The recorded tensile strength of the cement slurry is defined by Equation (4):

$$\sigma_t \propto p_f - p_w \tag{4}$$

The maximum strength that can be measured with the apparatus is approximately 5 bar. At this strength the cement has reached final set and is quite hard (for comparison, sandstones have a tensile strength of 40–150 bar).

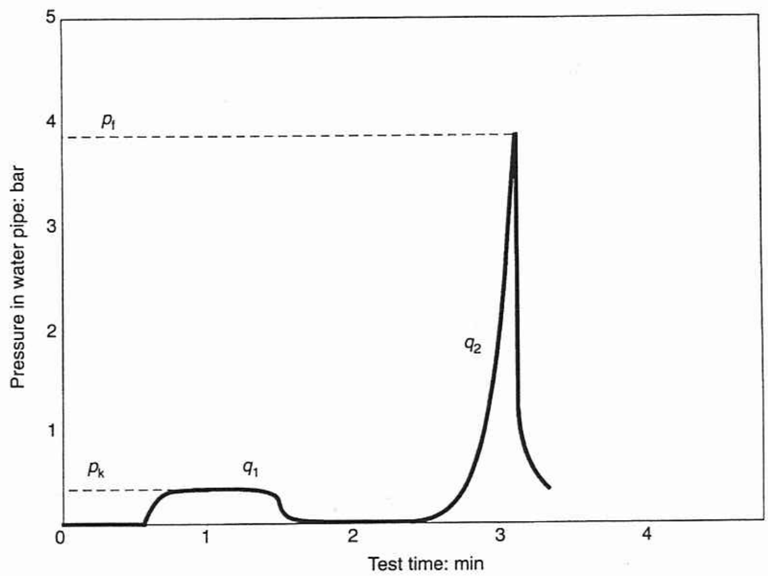

Fig. 4. Typical pressure variation during a flow test

Procedure

The slurries were mixed in a Waring Blender in accordance with a procedure suggested in API RP 10B.[6]

Each slurry was tested with three to six different batches. The cement and the air temperature in the heating chamber, the hydrostatic pressure of the slurry and the system pressure (5–20 bar) were measured during the test. The hydrostatic pressure and the cement temperature indicated when the slurry started to set. Therefore, an experiment with pressure–time recording of each slurry always preceded the permeability and tensile strength measurements.

After every test the hard cement was pressed out of the cells using a manual hydraulic press.

Test results

General

Both gas-tight and gas-untight slurries were tested, characterized by their control data in Table 1, designed by four different cement service companies operating in the North Sea. The five chosen gas-untight slurries (T30–T180) were simple, basic slurries with no gas-tight additives, in contrast to the eight chosen gas-tight pastes (A140–H180). In Table 2, three examples of composition are shown.

Permeability and tensile strength

The results from the basic slurry T90 and the commercial gas-tight slurry A140 are shown in Figs 5 and 6, respectively. A characteristic feature for all

Table 1. Control parameters for all slurries. Slurries denoted T are basic slurries, those denoted A through H are gas-tight slurries, designed by different service companies

Slurry	BHST: °C	SG	t_{set} (h:min) to reach			FW: ml	FL: ml	PV: cP	YP: lbs/100 ft^2
			30 Bc	40 Bc	70 Bc				
T25	25	1·89	2:30	3:12	2:40	3·5	> 50	52	110
T60	60	1·87	2:56	3:15	3:30	9·5	> 50	32	125
T90	90	1·86	3:00	3:17	3:25	8·5	> 50	62	96
T140	140	2·04				2·0	> 50	20	28
T180	180	2·13				1·0	> 50	15	20
C140	140	2·05	5:53	5:58	6:01	0	40		3·5
E140	140	2·05	2:29	2:30	2:33	0	35	90·0	15·0
A140	140	2·05	3:58	4:08	4:09	0	–		3·0
G140	140	2·05	2:40	3:12	3:18	> 0·1	24	31·6	7·5
D180	180	2·15	4:49	4:50	4:51		28		3·0
F180	180	2·15	3:02	3:17	3:18	0	14	58·5	11·5
B180	180	2·15	3:23	3:24	3:25	0	20		2·3
H180	180	2·15	5:25	5:26	5:26	< 0·1	30	43·2	10·9

BHST, bottom hole static temperature; SG, specific gravity (cylinder filled with 250 ml of slurry); FL, fluid loss (ml/30 min); t_{set}, time to reach a consistency of specified value; FW, free water on top of glass (ml/250 ml after 2 h); PV, plastic viscosity; YP, yield point; Bc, Beardon unit of consistency.

Table 2. Slurry data

		Slurry			Unit
		T90	A140	B180	
Test temperature		90	140	180	°C
Slurry density		1·90	2·05	2·15	g/cm^3
Slurry recipe	Density: g/cm^3				
API G class cement	3·22				
Water, fresh	1·00	43·24	45·52	54·86	1/100 kg*
Material density	4·85	–	38·77	74·87	%BWOC†
Silica flour	2·65	–	25·97	26·03	%BWOC
Dispersant	1·21	–	3·00	3·00	1/100 kg
Anti-fluid loss	1·04		5·00	3·00	1/100 kg
Anti-gas migration	1·40	–	13·00	13·00	1/100 kg
Retarder, low temperature	1·20	0·80	–	–	1/100 kg
Retarder, medium temperature	1·18	–	1·00	–	1/100 kg
Retarder, high temperature	1·25	–	–	0·80	%BWOC

* Litres per 100 kg of cement.
† Percentage by weight of cement.

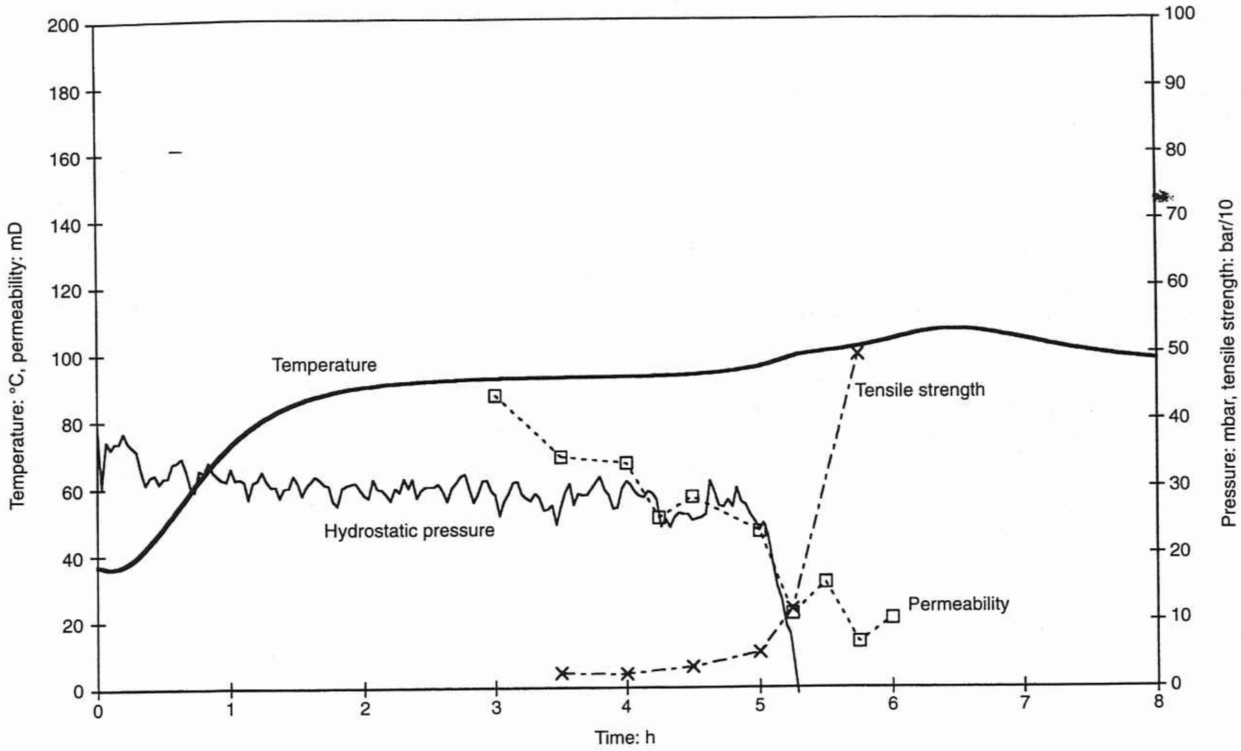

Fig. 5. Tensile strength and permeability of T90 cement slurry

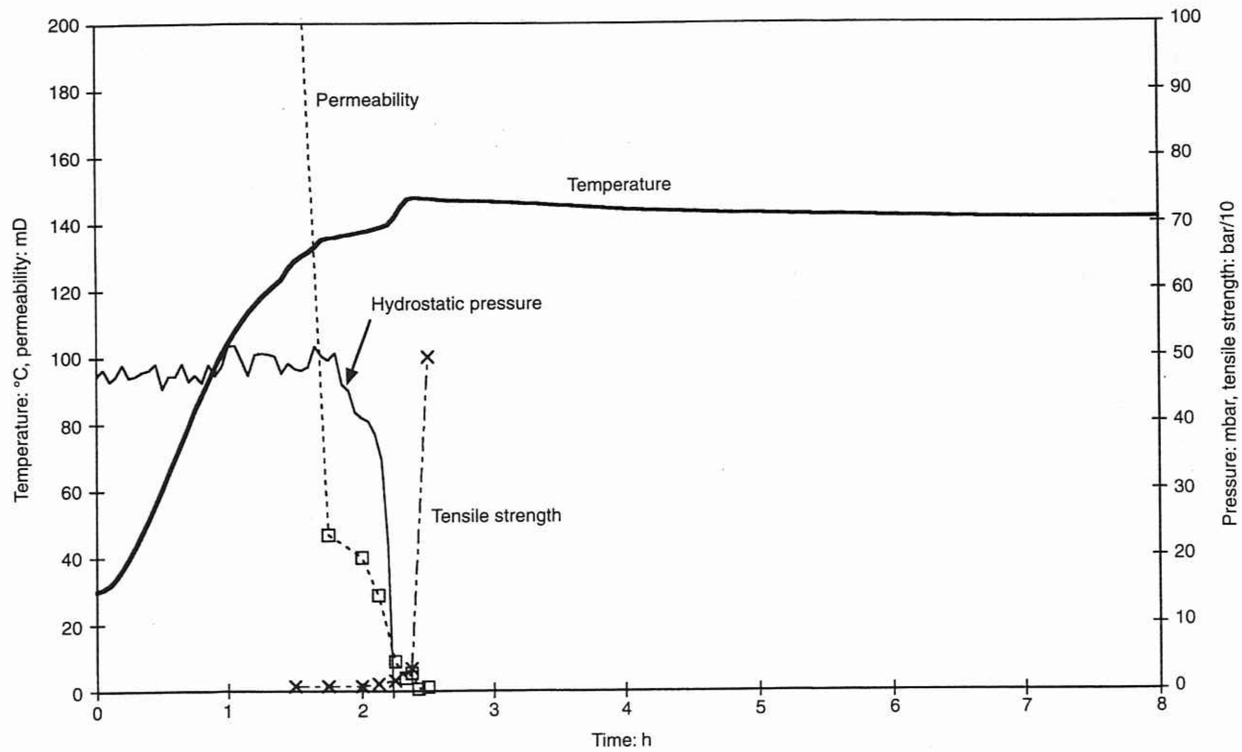

Fig. 6. Tensile strength and permeability of A140 cement slurry

slurries is the permeability decrease when the cement sets and the cement strength increases. A remarkable difference between the basic slurry, T90, and the commercial slurry, A140, is that the tensile strength increases and the permeability decreases faster for the commercial slurry. The normal strength range for a slurry which has not yet set is 0·05–0·20 bar, while the permeability is in the range of 50–100 mD. At final set the strength is above 5 bar and the permeability is in the range of 1–5 mD.

Discussion

A systematic interpretation of the relationship between all measured parameters, for all basic slurries and all the gas-tight slurries, revealed that the key difference between the two types of cement may be seen in three parameters: the hydrostatic pressure, the permeability development and the tensile strength behaviour. For the gas-untight cements, the hydrostatic pressure dropped almost immediately down to the water column pressure (30 mbar in Fig. 5), stayed constant for some time and then fell abruptly. The gas-tight pastes maintained the hydrostatic cement pressure for a time until a very sharp decline occurred (50 mbar in Fig. 6). The build-up of tensile strength was generally also much faster for the gas-tight than for the gas-untight cements; hence, the critical period for gas intrusion was much shorter.

We defined a time window, $\Delta t = t_2 - t_1$, based on the tensile strength. The two points of time, t_1 and t_2, correspond to values on the tensile strength curve of 0·3 and 5 bars, respectively. At time t_2 the cement is hard. The strength build-up should be rapid, thus Δt should be small. For the basic slurry, we found that $\Delta t = 2$ h (Fig. 5), while $\Delta t = 15$ min for the gas-tight slurry (Fig. 6).

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 hydrostatic pressure of the fresh cement slurry is denoted p_{sl}. In Fig. 6, $p_{sl} = 50$ mbar. The drop in 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 $(p_{sl} - p_1)$ should be small.

The three parameters – tensile strength measured within the time window Δt, permeability, and hydrostatic pressure – may be combined in various ways to find a 'factor' that can characterize the ability of a cement to resist gas migration.

We have tested the following combination of parameters, called the 'gas tightness factor' (GT factor):

$$GT = \sqrt{\Delta t}\,\frac{k_1 - k_2}{k_1}\,k_2\left(\frac{p_{sl} - p_1}{p_{sl}} + 1\right)$$

From the previous discussion, we conclude that the GT factor should be as small as possible to have a gas-tight cement. In Fig. 7 the GT factors for the tested cements are presented. Tests so far, compared to results from a gas migration rig,[8] have indicated that cement slurries with GT factors exceeding 5 are not gas tight. However, we should add that this GT factor definition presented here is only preliminary.

Conclusions

A new apparatus and method for studying the permeability and tensile strength of curing cement

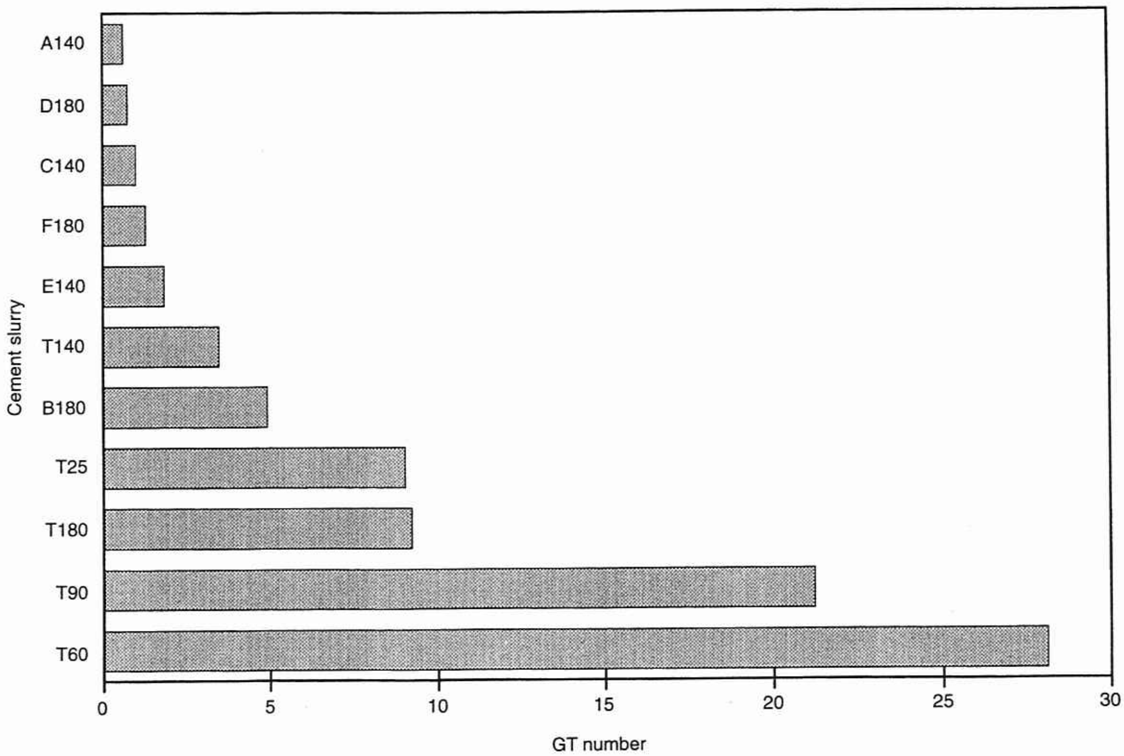

Fig. 7. GT factor for different gas-tight slurries (A through H) and basic, gas-untight slurries (T)

slurries have been developed and tested. The permeability is measured by pumping water through the cement slurry at a very low rate while recording the pressure, and the tensile strength is measured by hydraulic fracturing of the cement. The methods show reproducible results, and are promising for classifying cement slurries relative to their ability to withstand gas migration through the GT factor.

Acknowledgements

The authors would like to extend their thanks to the sponsors of this work: The Research Council of Norway, Norsk Hydro a.s., Saga Petroleum a.s. and Statoil a.s., and their respective representatives, Svein Joakimsen, Øystein Kalvenes, Jan Erik Olvin and Nils Egil Nødland, for their personal engagement and contributions.

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Discussion contributions on this paper should reach the editor by 26 September 1997