Characterizing Curing Cement Slurries by Electrical Conductivity

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Summary

Electrical conductivity is a parameter that can be used to monitor the entire hardening process of oilwell cement slurries. The theoretical relationship among conductivity, porosity, cement chemistry, and ion content is discussed. The theory is confirmed by experiments; the decline in the slurry conductivity is primarily a function of porosity decrease and, thus, the degree of hydration. The applied results show that the electrical conductivity of curing slurries reflects strength development and that rapid hydration will reduce the risk of gas migration.

Introduction

The main purposes of oilwell cements are to fasten the casing to the borehole wall and to seal off the rock formations. Knowledge about the entire hardening process of oilwell cement slurries is important for successful cementing operations.

Several methods exist to test cement slurries. The ubiquitous API tests[1] include procedures for finding density, free water, fluid loss, compressive strength, thickening time, rheology, and gel strength. All these tests are important for composing a successful cement recipe, but most of them consider only one or a few points of time during the setting process, or only the time period before the start of the hardening process. Thus, no continuous description of the entire setting process is obtained.

The only procedure that has won some acceptance for tracking the entire setting process is the ultrasonic cement analyzer[2] (UCA), which estimates the cement's compressive strength from the sound velocity through a cement sample. The UCA can be used throughout the entire setting process, over several days or weeks. However, it is relatively cumbersome and rather impractical for field use; therefore, there is a need for simpler test methods.

There are no reports in the literature of electrical conductivity measurements for characterizing oilwell cement, but many researchers have applied the method to concrete and other cement applications. Initially, electrical conductivity was used for finding the initial set and for tracking the rest of the curing process.[3–7] Later works include water-content assessment of fresh concrete,[8] influence of additives,[9,10] and corrosion risk of concrete reinforcements.[11,12] More recently, several publications have appeared on complex impedance,[13–16] which may be related to the cement microstructure. Work on predicting hydration and conductivity by computer modeling also has been presented.[17]

The works of the previously mentioned researchers have shown that conductivity measurements are simple, robust, and useful in monitoring the entire hardening process. Thus, the method should be well suited as a laboratory test method for optimizing the composition of field slurries, on-site quality control, and cement waiting time.

The organization of this paper is as follows. We first introduce electrical conductivity and discuss the theoretical background required to understand the method. We then present the experimental work needed to ascertain that the method can be used on oilwell slurries. Finally, practical results are discussed. Some additional results can be found in another paper.[18]

Theoretical Background

Measurement Principle. The principle of measuring conductivity, in which alternating current is transmitted through the cement slurry by two metal electrodes, is shown in **Fig. 1.** When the voltage drop over the electrodes (U) and the current through the sample (I) are known, the conductivity (σ) can be calculated as follows.

$$\sigma = \frac{\ell}{A}\frac{I}{U}, \qquad (1)$$

where ℓ=the distance between the voltage electrodes, and A=the cross-sectional area of the sample. If the geometry of the cement sample is more complex, Eq. 1 is not valid unless the geometric factor ℓ/A is replaced by an experimentally found constant, G. The simplest way to find this constant is by calibrating the measurement cell with a fluid that has a known conductivity.

Porosity. In a cement slurry, only the pore fluid contributes to the flow of electrical current. Archie[19] investigated the relationship between the conductivity and the porosity of rocks saturated with conducting water. Archie's law may also be applied as follows to cement slurries.

$$F = \frac{\sigma_f}{\sigma_c} = \frac{a}{\phi^m}, \qquad (2)$$

where F=the formation factor, σ_c=the conductivity of the cement, σ_f=the conductivity of the pore fluid, ϕ=the porosity (expressed as a fraction), and a and m=constants. The constant a is generally considered to be unity because the formation factor should be one at 100% porosity. The constant m is usually called the cementation factor and increases with increasing tortuosity. Serra[20] uses the name "tortuosity constant" for m. Archie found that the exponent m varied between 1.8 and 2 for consolidated sandstones and that it appeared to be approximately 1.3 for unconsolidated laboratory sands. For unconsolidated dispersions, it has been shown theoretically that the exponent is 1.5.[21,22] Later work[23,24] has confirmed the results of Archie, and, generally, a value of approximately 2 is used. On the other hand, the modeling work of Bryant and Pallatt[25] produced an m equal to 3.2 at porosities of less than 10%.

The constants in Archie's law may not be valid for hydrating cement, in which the slurry initially goes from being an unconsolidated liquid suspension toward being a substance with an emerging matrix and decreasing porosity. Not much work is presented on Archie's law and cement. McCarter and Puyrigaud[8] used conductivity to estimate the water content of fresh concrete after 30 to 90 minutes, and by recalculating their data, m was found to be 1.42. The 28-day mortar data of Tumidajski *et al.*[26] gave an average m of 2.15, and their cement paste data for up to 29 years produced an exponent of 3.21. However, the data of Christensen *et al.*[14] and Coverdale *et al.*[17] do not conform to this behavior. Their results, together with our findings, are discussed later in the paper.

Ion Transport. Electrical current is transported through the slurry by ions. Thus, the conductivity is controlled by the ion concentration (c), the number of charges per ion (z), and the equivalent ionic conductivity (λ). The electrical conductivity (σ) of an aqueous solution can be calculated as follows by summing the contributions for ion j.

$$\sigma = \sum_j \left(\left| z_j \right| \lambda_j c_j \right). \qquad (3)$$

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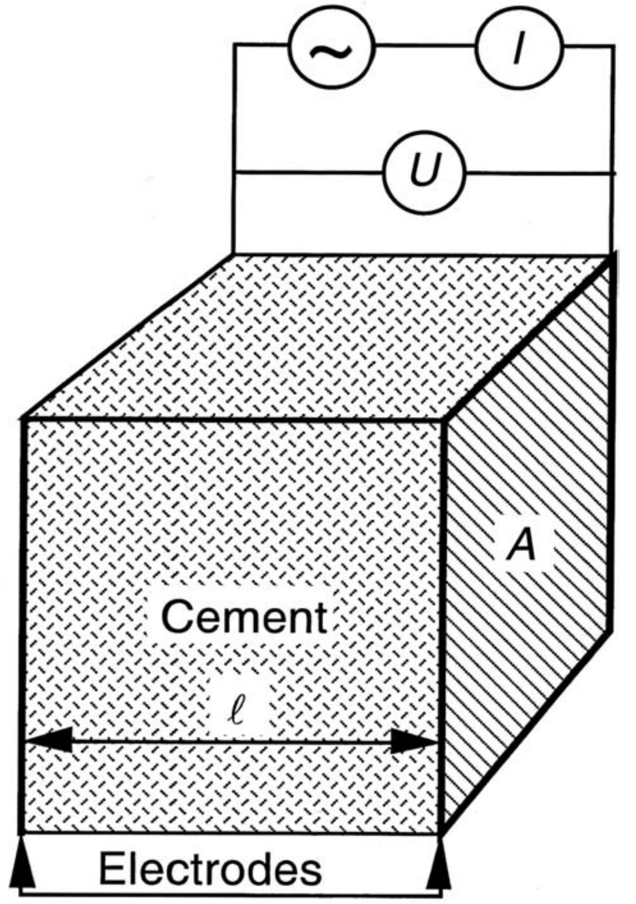

Fig. 1—Principle of conductivity measurements. The "~" symbol indicates the alternating current source.

The equivalent conductivity is a function of temperature and will increase by 1.5 to 2.5% per degree Celsius, which is mainly caused by the decrease in the water viscosity.[27]

Cement Chemistry. The ions we are likely to find in the pore fluid can be deduced from the cement chemistry. Hydration of the cement may be divided into five periods:[9,28] preinduction, induction, acceleration, deceleration, and diffusion. Their approximate periods for a neat slurry at 25°C are marked in **Fig. 2.**

The preinduction period takes place immediately after mixing and lasts for a few tens of minutes while calcium (Ca) and hydroxyl (OH) ions go into solution. In the induction, or dormant, period, little happens except a slow precipitation of semicrystalline calcium silicate hydrate gel (C-S-H) while the Ca^{2+} and OH^- concentrations continue to rise slowly. In the start of the acceleration period, the Ca^{2+} concentration reaches the saturation level, and the first hydration reactions begin, with the crystallization of solid calcium hydroxide and the deposition of C-S-H gel in pores. While the structure is building up, the porosity decreases and the availability of ions and water will be lower, eventually leading to a decelerated hydration, marking the onset of Period 4. At ambient conditions, the start of the acceleration period is approximately 3 hours, and the diffusion period begins after approximately 1 day.

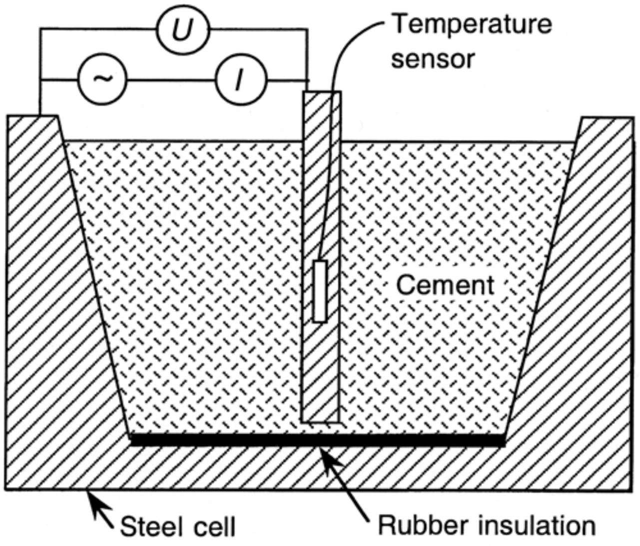

Fig. 3—Principle drawing of the conductivity cell. The "~" symbol indicates the alternating current source.

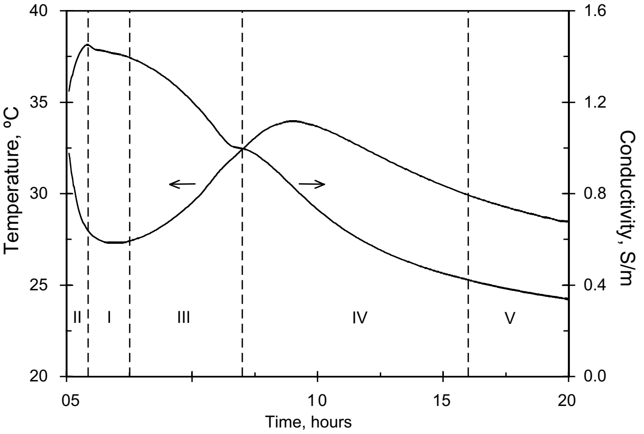

Fig. 2—Temperature and conductivity behavior of a neat slurry at 25°C. The five hydration periods (1 through 5) are shown.

From this discussion, it follows that the Ca^{2+} and OH^- ions are the most important from an electrical conductivity point of view. In addition, significant concentrations of Na^+, K^+, and SO_4^{2-} ions can be found in the cement pore fluid. Data in the literature[14,29–31] show that the Ca^{2+} and SO_4^{2-} concentrations decline slowly when hydration starts and that the Na^+, K^+, and OH^- concentrations increase slowly. Thus, one cannot expect the conductivity of the cement pore water to remain constant. This is borne out in the data of Christensen *et al.,*[14] in which the pore water conductivity increases significantly toward 1 week of hydration.

It should be noted that the previous results are based on cements cured at ambient conditions.

Experiment

Experimental Setup. The principle of the conductivity measurements is shown in Fig. 1, as previously described. Alternating current was used to avoid electrolysis of the cement slurry. The applied voltage was 1 to 6 V, and the current was in the milliampere range. All data, including cement temperature, were recorded and stored in a personal computer.

Several different cell geometries were used; the one used in this paper is shown in **Fig. 3,** with one center electrode and the circumference of the cell acting as the second electrode. The bottom is electrically insulated with rubber, forcing the current to flow radially between the electrodes. It had an internal diameter of 85 mm and a height of 50 mm, and it was designed for pressures of up to 20 bar and temperatures of up to 200°C. Heating was provided by an electric hot plate with surrounding insulation and a proportional/integral/derivative (PID) controller to regulate temperature. Samples were prepared according to API specifications.[1]

Suitability of Conductivity Measurements on Cement. A couple of tests were carried out to see if flow of electrical current through the slurry altered the hardening process of the cement. The setting process is exothermic and exhibits a characteristic temperature curve; if the temperature evolution is identical for two parallel tests, one with and one without current through the sample, it can be concluded that conductivity measurements do not influence the setting. Tests at room conditions and at 90°C confirmed that passing current through the slurry has no adverse effect.

TABLE 1—CEMENT SLURRY RECIPES

		Slurry Test Temperature (°C)			
Additive	g/cm^3	25	90	140	180
API class G cement	3.22	100.00	100.00	100.00	100.00
Freshwater, L/hk*	1.00	50.00	43.24	45.52	54.86
Weight material, %bwoc**	4.85			38.77	74.87
Antistrength retrograde, %bwoc	2.65			25.97	26.03
Antigas migration, lhk	1.40			13.00	13.00
Dispersant, L/hk	1.21			3.00	3.00
Antifluid loss, L/hk	1.04			5.00	3.00
Retarder, low temperature, L/hk	1.20		0.8		
Retarder, medium temperature, L/hk	1.18			1.25	
Retarder, high temperature, %bwoc	1.25				0.80

*L/hk = Liters per hundred kilos of cement
**%bwoc = percent by weight of cement

Pore Fluid. Pore filtrate was pressed out of three slurries at 25, 90, and 140°C (recipes in **Table 1**) at several points in time during the setting process and tested for ion content and conductivity. A standard API filter press was used, except for the 90°C slurry, in which a larger, similar cell was used to produce enough filtrate. Tests were carried out until the yield volumes became too low.

The OH^- concentration was found by measuring the pH of the pore filtrate, while the amount of Ca^{2+} and Mg^{2+} was measured by titration according to an API procedure.[32] Because a cement slurry contains little or no magnesium, the measured API value represented calcium only. The theoretical conductivity was calculated from the measured ion concentrations with Eq. 3.

The concentrations and conductivities for the neat slurry at 25°C are shown in **Fig. 4.** The increase in OH^- concentration and decrease of Ca^{2+} has also been observed by other authors.[14,29–31] The difference between the calculated and measured conductivities is most likely caused by other ions in the pore fluid. The conductivity of the pore filtrate is approximately 4 S/m and rises toward 5 S/m as the final set approaches. The values for the 90°C slurry remained relatively constant at approximately 4 S/m. For the 140°C slurry, pore filtrate was extracted only once after 30 minutes, and the conductivity was found to be somewhat lower at 3.3 S/m. Values in the literature[8,14,26] vary between 0.6 and 15 S/m, with most of the data in the range of 3 to 5 S/m. One interesting observation by Christensen *et al.*[14] is a pore fluid conductivity of 10 to 15 S/m after 1 week of curing.

Porosity. The porosity of a cement slurry may be estimated by Archie's law (Eq. 2) but, as previously mentioned, conflicting data exist on the value of the constants in the law.

Our conductivity data were compared to the porosity data of Justnes *et al.*,[33,34] one at an ambient condition and four at 150°C in the time interval of 100 to 270 minutes. In addition, the initial porosity of all the 33 tested slurries (25 to 195°C) was calculated from the cement recipes and compared to the conductivity at the onset of the induction period of hydration. In these calculations, it was assumed that the conductivity of the pore fluid was constant-equal to 4 S/m. The results are shown in **Fig. 5,** where our data follow the same trend as those by Coverdale *et al.*[17] and Christensen *et al.*[14] A least squares fit to all these data leads to the following version of Archie's law.

$$F = \frac{0.126}{\phi^{5.77}} \quad \ldots\ldots (4)$$

Christensen *et al.*[14] note that the special values of constants *a* and *m* may be attributed to the more tortuous pore structure of cement slurries compared to that of rocks. Bryant and Pallatt[25] mention a similar explanation for low-porosity rocks.

Temperature Influence on Conductivity. Because conductivity increases with temperature, the cement conductivity has to be corrected to compare conductivity curves of slurries at different temperatures. Usually, the conductivity is corrected to a temperature of 25°C.

The same problem arises when the open, noncased borehole is logged. Arps[35] introduced the following formula for this purpose.

$$\sigma_{25} = \frac{46.5\sigma_T}{21.5+T}, \quad \ldots\ldots (5)$$

where T=the temperature in °C, σ_T=the conductivity at that tem-

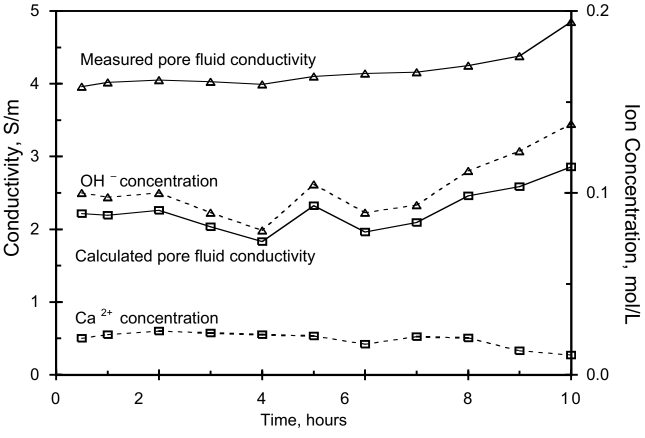

Fig. 4—Ion concentrations and conductivities of the pore fluid for the neat slurry at 25°C.

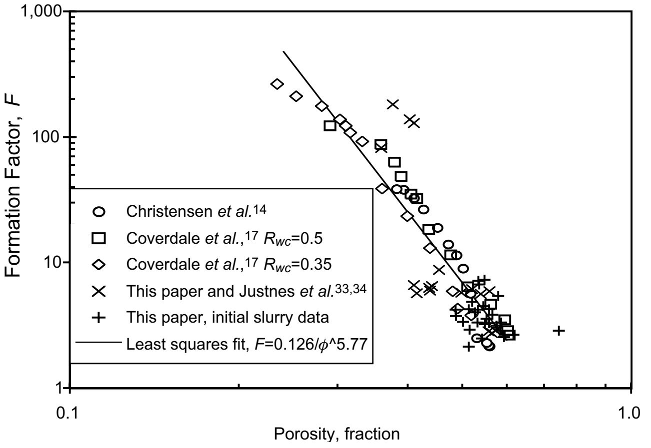

Fig. 5—Porosity vs. formation factor. Data from Christensen *et al.*,[14] Coverdale *et al.*,[17] Justnes *et al.*,[33,34] and this work.

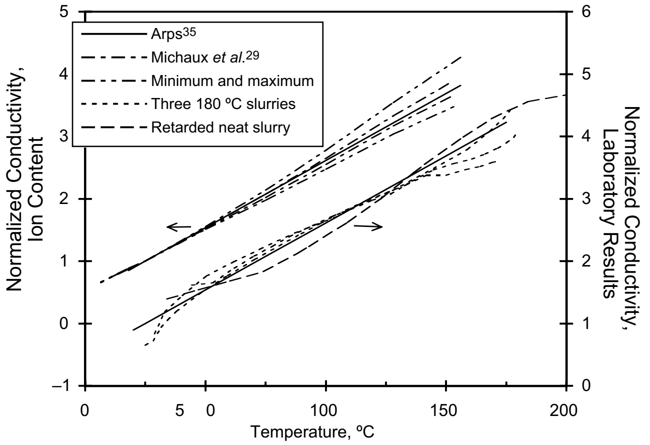

Fig. 6—Conductivity increase as a function of temperature. The left-hand scale represents theoretical behavior based on ion concentrations and tabulated equivalent conductivities, whereas the right-hand scale shows results from laboratory tests. Data from Arps,[35] Michaux *et al.,*[29] and this work.

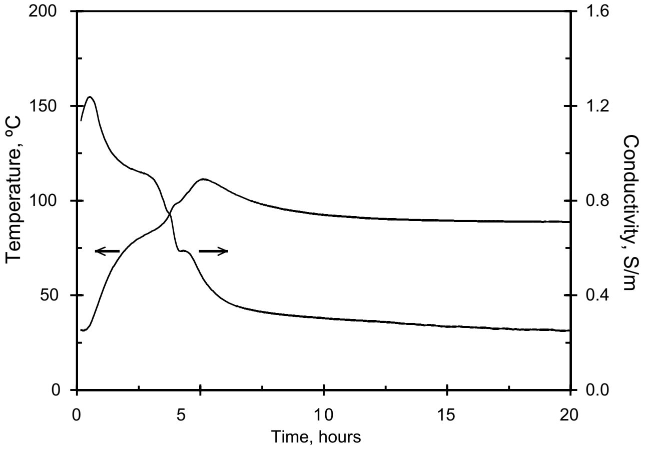

Fig. 7—Conductivity and temperature behavior of the 90°C slurry.

perature, and σ_{25}=the corrected conductivity at 25°C. This correction is based on sodium chloride solutions at temperatures of between 0 and 156°C and is reasonably accurate. Because cement slurries contain other ions, Eq. 5 may not be valid for cement.

Two approaches were employed to find an equivalent correction for cement—a theoretical and a practical one. The first approach is based on tabulated equivalent conductivities[36] of up to 156°C and the ion composition of cement pore fluids. Michaux *et al.*[29] and Vidick *et al.*[30] investigated the ion concentration for API G-class cement for up to 6 hours, whereas Christensen *et al.*[14] and Taylor's[31] results are for ordinary Portland cement up to and past the final set. These results are for cement pastes at ambient conditions. With Eq. 3, the theoretical conductivity of the pore fluid was calculated for each set of ion-concentration data found in the literature. The results are presented in **Fig. 6,** where the maximum derivation from Arps are shown, as well as the curves for the two API G-class cements used by Michaux *et al.*[29] The latter two are close to the Arps curve (Eq. 5), which suggests that the Arps correction can be used on cement without gross errors.

In the practical approach, the conductivity curve of the 180°C slurry in Table 1 was plotted in Fig. 6 with two other 180°C slurries. These slurries were chosen because they exhibit no significant hydration before the set temperature is reached. The fit with the Arps equation is good; the low temperature discrepancy is caused by the preinduction period of the cement hydration, and the deviations at the higher temperature may result from an initial slow hydration. Further testing included slow heating of an overretarded neat slurry up to 200°C, which was subsequently cooled to investigate any possible hysteresis effect. No hysteresis effect was found, and the results plotted in Fig. 6 again show reasonable agreement with Eq. 5.

Because both the theoretical and practical approaches confirm that Arps' correction has sufficient accuracy to be employed on cement, it was consequently used for all our experiments.

Results and Discussion

Effect of Test Temperature. Conductivity tests were carried out on 25 commercial and eight simple, basic cement slurries, in which the applied bottomhole static temperatures ranged from ambient to 195°C. These data revealed that there is a considerable change in behavior over the temperature range. To exemplify, curves for three slurries, with test temperatures of 90, 140, and 180°C, are shown in **Figs. 7 through 9.** Their recipes are listed in Table 1.

The conductivity curve of the 140°C slurry declines rapidly when the hydration starts (at approximately 3 hours), whereas the other two slurries at 90 and 180°C are slower, with this observation being particularly true for the 180°C slurry. Several other slurries at the same three temperatures were tested and exhibited the same behavior, demonstrating that this may be a general hydration trend. This trend is also confirmed by previous results with identical slurry recipes that measured permeability, tensile strength, and shrinkage.[37]

Conductivity vs. Cement Strength. A relationship between slurry conductivity and porosity has been established previously. However, for oilwell use, compressive strength is more important than porosity because a certain minimum cement strength is required before drilling operations can resume. Conductivity is a candidate for estimating strength because the reverse side of the porosity decrease is growth of crystals and, thus, strength buildup.

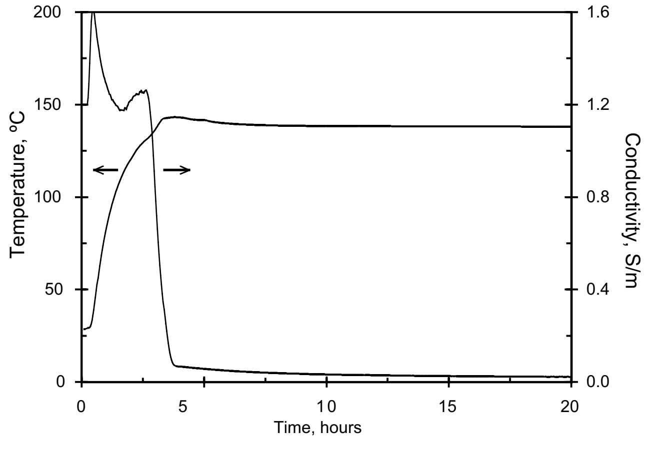

Fig. 8—Conductivity and temperature behavior of the 140°C slurry.

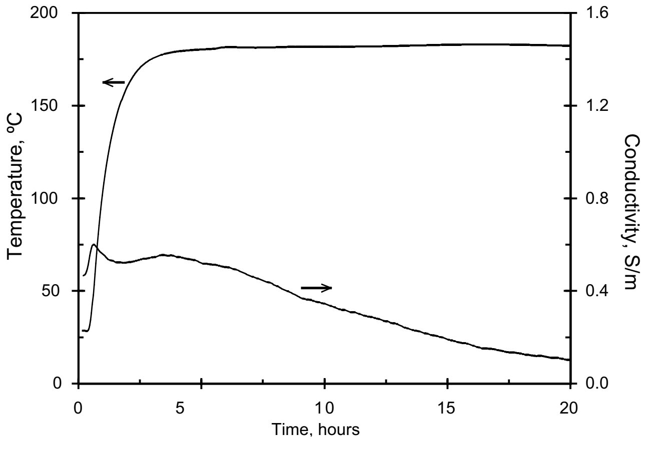

Fig. 9—Conductivity and temperature behavior of the 180°C slurry.

To find a correlation with strength, our conductivity data were compared to the strength data obtained from the UCA. A literature search was also carried out to find correlations between strength and porosity, which, again, are related to conductivity through Archie's law (Eq. 2).[19]

Several models exist for porosity vs. strength. Most are for mortar and concrete, and they are almost exclusively focused on the later stages of hydration (i.e., from 1 day to up to several weeks). Nevertheless, extrapolation of these correlations into the early setting period may still shed light on the early time strength behavior. We will discuss the following three models.

Powers,[38–40]

$$S = S_0 x^3 , \tag{6a}$$

$$x = \frac{1-\phi}{1+0.47(1/\alpha-1)\phi} , \tag{6b}$$

$$\alpha = \frac{R_{wc} - \phi(R_{wc}+0.32)}{0.18} ; \tag{6c}$$

Balshin,[39,41]

$$S = S_0(1-\phi)^y , \tag{7a}$$

$$y = \frac{2.35}{(\alpha-0.19)^{0.45}} ; \tag{7b}$$

and Schiller[41]

$$S = b\ln\frac{\phi_i}{\phi} , \tag{8}$$

where S_0=the end compressive strength, α=the degree of hydration, ϕ=the porosity, ϕ_i=the initial porosity, R_{wc}=the water:cement ratio, and b=a constant. To compare these models to conductivity, a few assumptions have to be made: S_0 was chosen to be 30 MPa, which is a reasonable oilwell cement strength, and the constant b in Schiller's model was chosen such that the end strength is 30 MPa. Finally, Nielsen[39] states that the cement exhibits no coherence or strength before the degree of hydration is more than the water:cement ratio divided by two (e.g., for a water:cement ratio of 0.5, the degree of hydration should be more than 0.25). In our case, with Eqs. 4 and 6c, Nielsen's[39] coherence criterion leads to the statement that the slurry exhibits no strength before the normalized conductivity reaches 0.6 (i.e., when the conductivity is 60% of the conductivity level during the induction period of the hydration). Normalization is especially important when comparing fresh- and saltwater-based slurries because the absolute conductivity of the latter is two to three times higher than for the former.

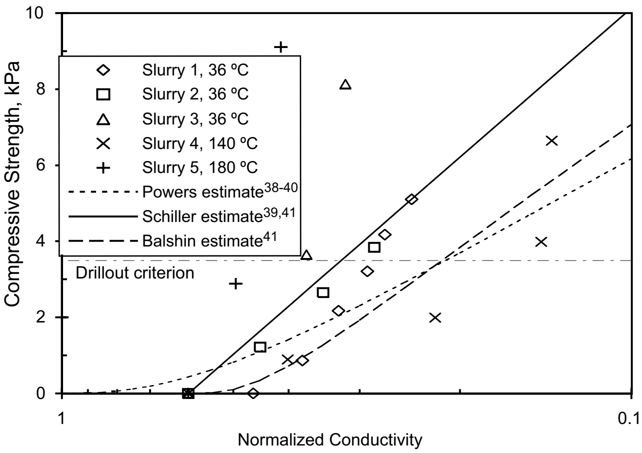

Fig. 10—Normalized conductivity correlated to UCA strength (markers) and conductivity/porosity/strength relationship (lines).

The strength estimates from the three models are shown in **Fig. 10** with the UCA data correlation. The correlation point on the time scale is when the UCA strength starts to build up and the normalized conductivity reaches a value of 0.6. All three strength estimates are in the same range as the correlation with the UCA. Rae[42] states that a compressive strength of 3.5 MPa is sufficient to continue drillout. A conservative estimate based on Fig. 10 shows that this strength is reached when the normalized conductivity approaches 0.2.

These results indicate that there is a correlation between conductivity and strength. Because of the empirical nature and extrapolations, future work should include a direct comparison between conductivity and compressive strength.

Conductivity vs. Gas Migration. Several slurries have been tested in a gas-migration test rig,[43,44] and tightness properties were compared to conductivity. This rig consists of a 2-m vertical steel pipe with an inner diameter of 0.102 m, in which a differential pressure is applied between the bottom and top of the cement column. Flow into and out of the column is recorded by mass flowmeters.

Pressure loss in the cement column during hydration is inevitable and is the main driving force behind migration; eventually, the cement pressure will decline to less than the pore pressure, which may allow gas flow. Many remedies have been suggested,

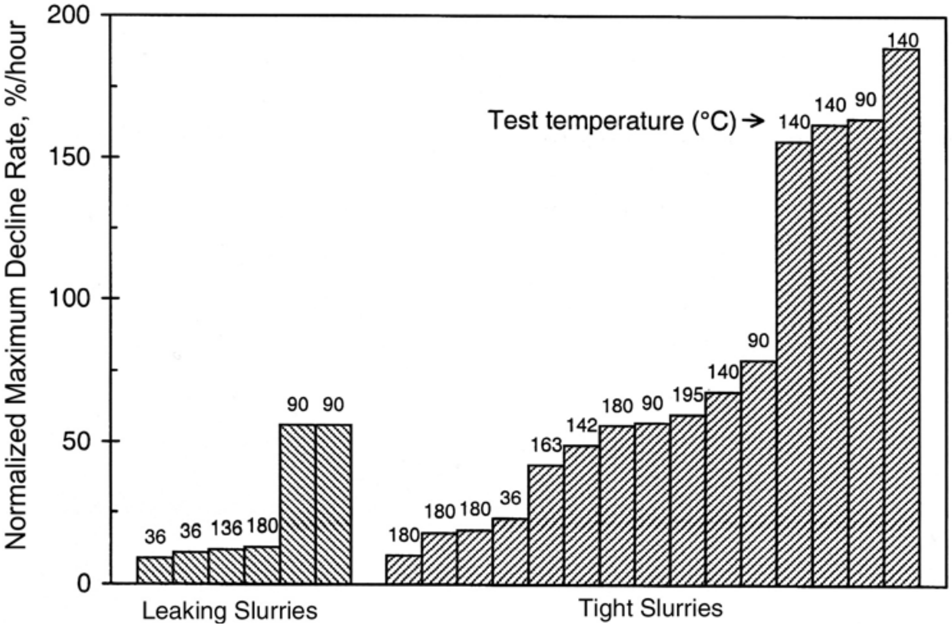

Fig. 11—Normalized maximum conductivity decline rates vs. gas tight and leaking slurries.

including that a rapidly setting slurry should reduce the problem;[45] if no significant pressure loss is experienced before the initial set, the following rapid matrix buildup and pore size reduction will hinder gas intrusion and flow.

The maximum normalized conductivity decline rate is a measure of the hydration rate. The decline rates are shown in **Fig. 11,** where the tight slurries generally exhibit higher hydration rates than leaking. There is some overlap, indicating that rapid setting alone is not sufficient to avoid gas flow; downhole conditions and other cement properties, such as fluid loss and gelling, have to be taken into account when designing a slurry. However, conductivity is an excellent method to pick out rapid setting, which, together with other design criteria, will reduce the risk of gas migration.

Conclusions

Electrical conductivity of oilwell cement slurries has been related to cement chemistry and physical properties. On the basis of data from literature and experiments, a relationship between electrical conductivity and porosity has been found. The measurements were found to have no adverse effect on the hydration.

A relationship between conductivity and compressive strength has been established, and a conservative drillout strength criterion based on conductivity is presented. A comparison of results from a gas migration test rig indicates that the risk of gas flow through the cement is reduced with a rapidly setting slurry.

Our laboratory work on electrical conductivity has proven the method to be sensitive for monitoring the hydration of oilwell cement slurries. The simplicity and robustness of the measurements should make them well suited as a laboratory test method for optimizing field slurries, on-site quality control, and cement waiting time when run parallel with the cementing operation.

Nomenclature

a = constant in Archie's law, dimensionless
A = cross-sectional area, L^2, m^2
b = constant in Eq. 8, m/Lt^2, MPa
c = ion concentration, n/L^3, mol/m^3
F = formation factor, dimensionless
G = geometric constant$=\ell$/A, l/L, m/m^2
I = electric current, q/t, A
ℓ = length, L, m
m = exponent in Archie's law (Eq. 2), dimensionless
R_{wc} = water to cement ratio, dimensionless, kg/kg
S = compressive strength, m/Lt^2, MPa
T = temperature, T, °C
U = electric potential difference, mL^2/qt^2, V
x = intermediate variable in Eq. 6, dimensionless
y = intermediate variable in Eq. 7, dimensionless
z = charge number, n
α = degree of hydration, dimensionless, fraction
ϕ = porosity, dimensionless, fraction
λ = ionic equivalent conductivity, tq^2/nm, Sm^2/mol
σ = electrical conductivity, tq^2/mL^3, S/m

Subscripts

c = cement
f = pore fluid
i = initial
j = ion j
T = conductivity at temperature T
0 = end compressive strength
25 = conductivity at 25°C

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SI Metric Conversion Factors

°F (°F − 32)/1.8 = °C
in. × 2.54* E − 02 = m
in.^{2} × 6.4516* E − 04 = m^2
in.^{3} × 1.638 706 E − 05 = m^3
lbm × 4.535 924 E − 01 = kg
lbm/ft^3 × 1.601 846 E − 02 = g/cm^3
liter × 1.0* E − 03 = m^3
mho × 1.0* E + 00 = S
psi × 6.894 757 E − 03 = MPa

*Conversion factor is exact.

SPEDC

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