DEVELOPMENT OF SMART CEMENT FOR REAL TIME MONITORING OF ULTRA DEEPWATER OIL WELL CEMENTING APPLICATIONS

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Abstract: For a cementing operations to be successful, it is important to determine the filling of cement slurry between the casing and formation, depth of the circulation losses and fluid loss, setting of cement and performance of the cement after hardening. Recent accidents on cementing failures have clearly identified some of these issues that resulted in various types of delays in the cementing operations. At present there is no technology available to monitor cementing operations in real time from the time of placement through the borehole service life. Also, there is no reliable method to determine the length of the competent cement supporting the casing.

In this study well cement was modified to have better sensing properties, smart cement, so that its behavior can be monitored at various stages of construction and during the service life of wells. A series of experiments evaluated well cement behavior with and without modifications in order to identify the most reliable sensing properties that can also be relatively easily monitored. Tests were performed on the cement from the time of mixing to hardened state behavior. During the initial setting the electrical resistivity changed with time based on the type and amount of additives used in the cement. During curing initial resistivity reduced by about 10% to reach a minimum resistance, and maximum change in resistance within the first 24 hours of curing varied from 50 to 300% depending on the additive. A new quantification concept has been developed to characterize cement curing based on electrical resistivity changes in the in the first 24 hours of curing. When cement was modified with less than 0.1% of conductive additives, the piezoresistive behavior of the hardened smart cement was substantially improved without affecting the cement rheological and setting properties. For modified smart cement the resistivity change at peak stress was about 400 times higher than the change in the strain.

1. Introduction

As deepwater exploration and production of oil and gas expands around the world, there are unique challenges in well construction beginning at the seafloor. Also preventing the loss of fluids to the formations and proper well cementing have become critical issues in well construction to ensure wellbore integrity because of varying downhole conditions (Labibzadeh et al. 2010; Eoff et al. 2009; Ravi et al. 2007; Gill et al. 2005; Fuller et al. 2002). Moreover the environmental friendliness of the cements is a critical issue that is becoming increasingly important (Durand et al. 1995; Thaemlitz et al. 1999; Dom et al. 2007). Lack of cement returns may compromise the casing support and excess cement returns cause problems with flow and control lines (Ravi et al. 2007; Gill et al. 2005; Fuller et al. 2002). Hence there is a need for monitoring the cementing operation in real time. At present there is no technology available to monitor the cementing operation real time from the time of placement through the entire service life of the borehole. Also there is no reliable method to determine the length of the competent cement supporting the casing.

1.1 Oil Well Cement

Oil well cementing is done to bond the casing to the formation so as to prevent blowout and to promote zonal isolation. The standards of API suggest the chemical requirements determined by ASTM procedures and physical requirements determined in accordance with procedures outlined in API RP 10B and ASTM. There are several types of cements that are being used for oil well cementing based on the oil well conditions. Oil-well cements (OWCs) are classified into grades based upon their $Ca_3Al_nO_p$ (Tricalcium Aluminate – C_3A) content. In general each class is applicable for a certain range of well depth, temperature, pressure, and sulphate environments. OWCs usually have lower C_3A contents and are coarsely ground with friction-reducing additives and special retarders such as starch and/or sugars in addition to or in place of gypsum.

Cements such as class G and class H, considered to be two of the popular cements, are used in oil well cementing applications. These cements are produced by pulverizing clinker consisting essentially of calcium silicates ($Ca_nSi_mO_p$) with the addition of calcium sulphate ($CaSO_4$) (John, 1992). When admixtures are added with cement, tensile and flexural properties will be modified. Also admixtures will have effect on the rheological, corrosion resistance, shrinkage, thermal conductivity, specific heat, electrical conductivity and absorbing (heat and energy) properties of oil well cement (Bao-guo, 2008). Oil well cement slurry is used several thousand feet below the ground level and hence determining cement setting time is always a challenge.

1.2 Piezoresistive Behavior

Banthia (1994) observed that strength and durability of concrete was improved by the addition of small amounts of fiber-reinforcement. Due to the fiber's high resistance to wear, heat, and corrosion, carbon fiber-reinforced concrete in particular has been shown to have excellent durability properties. Also, Chung (1996) reported that addition of carbon fiber to cement provided the strain-sensing ability and increased the tensile and flexural strengths, tensile ductility and flexural toughness, and decreased the drying shrinkage.

Chung (2001) studied the electrical resistivity of carbon fiber-reinforced cement paste and the electric polarization effect. By increasing the conductivity of the cement paste through the use of carbon fibers that were more crystalline the polarization effect diminished. It was concluded that when the four-probe method was used, voltage polarity switching effects were dominated by the polarization of the sample itself, but when the two-probe method was used, voltage polarity switching effects were dominated by the polarization at the contact sample interface. Reza (2003) proved that with the addition of a small volume of carbon fibers into a concrete mixture produced a strong and durable concrete and made the product as a smart material. It is recommended that these techniques could be used as nondestructive testing methods to assess the integrity of the composite. Vipulanandan et al. (2004 - 2013) studied the piezoresistive behavior of cementitious and polymer composites. The studies showed that the changes in resistivity with the applied stress were 30 to 50 times higher than the strain in the materials.

1.3 Theory and Concepts

It was very critical to identify the sensing properties for the cement that can be used to monitor the performance. After years of studies and based on the current study on oil well cements and drilling muds, electrical resistivity (ρ) was selected as the sensing property for cements (Vipulanandan et al. 2005, 2012). Hence two parameters (resistivity and change in resistivity) were used to quantify the sensing properties as follows:

$$R = \rho\ (L/A) = \rho\ K \qquad (1)$$

Where R = electrical resistance

L = Linear distance between the electrical resistance measuring points

A = effective cross sectional area

K = Calibration parameter is determined based on the resistance measurement method

Normalized change in resistivity with the changing conditions can be represented as follows:

$$\Delta\rho/\rho = \Delta R/R \qquad (2)$$

The modified cement materials represented in terms of resistivity (ρ) to changes (composition, curing and stress) has been quantified to evaluate the sensitivity of the selected parameter.

2. Objectives

The overall objective of the study was to develop and characterize smart cement with enhanced sensing properties that can be integrated with real-time monitoring of the operations for improving the wellbore integrity. The specific objectives are as follows:

(i) Develop smart cement and identify the sensing properties.

(ii) Characterize the rheological, curing, and electrical properties of smart oil well cement.
(iii)Characterize the piezoresistive behavior of hardened smart oil well cement.

3. Materials and Methods

3.1 Cement

In this study commercially available Class H oil well cement was used. Also a typical XRD pattern of Class H cement is shown in Fig. 1, where major constituents of the cement have been identified. Effect of carbon fiber modification to the cements with and without additives such as silica fume, fly ash, bentonite, nanoparticles, meta-kaolin and water reducing agents were investigated.

(a) Sample Preparation

The samples were prepared in accordance with API standard. For the initial mixing, a high speed propeller-type mixer was used. Varying amounts of carbon fibers (0.075% to 0.2%) were added with the cement mix as base modification. Also various other additives were added with respect to the desired result. Additives were added to the water in the mixer with the mixing intervals of 20 s at 4000 rpm. Cement, water and additive were mixed at the speed of 4000 rpm for 3 min and 35 seconds at the speed of 1200 rpm. The water/cement ratio in all formulation in this study was varied from 0.38 to 0.44. Total of four wires were placed in the mold with two wires on each side of the specimen (Fig. 3). The vertical distances between any two wires were the same. Embedment depth of the conductive wire was 1 inch. In order to have consistent result, at least three specimens were prepared for each type of mix.

For setting time monitoring and compressive loading tests, cylinders with diameter of 2 inches and a height of 4 inches were prepared. For quick results monitoring, two-probe method was chosen. During the initial stages of setting, conductivity and API resistivity meters were used to determine the curing cement resistivity and using Eqn. (1) the calibration parameter K was obtained with time.

(b) Resistivity

Different instruments were used to measure the resistivity of the cement.

(i) Conductivity Probe: Commercially available conductivity probe was used to measure the conductivity (inverse of resistivity) of the fluids. In the case of cement, this meter was used during the initial curing of the cement. The conductivity measuring range was from 0.1μS/cm to 1000 mS/cm representing a resistivity of 10,000 Ω.m to 0.1 Ω.m

(ii) Digital Resistivity Meter: This is used in the Petroleum industry to measure resistivity. It measures resistivity in the range of 0.01 Ω.m to 400 Ω.m.

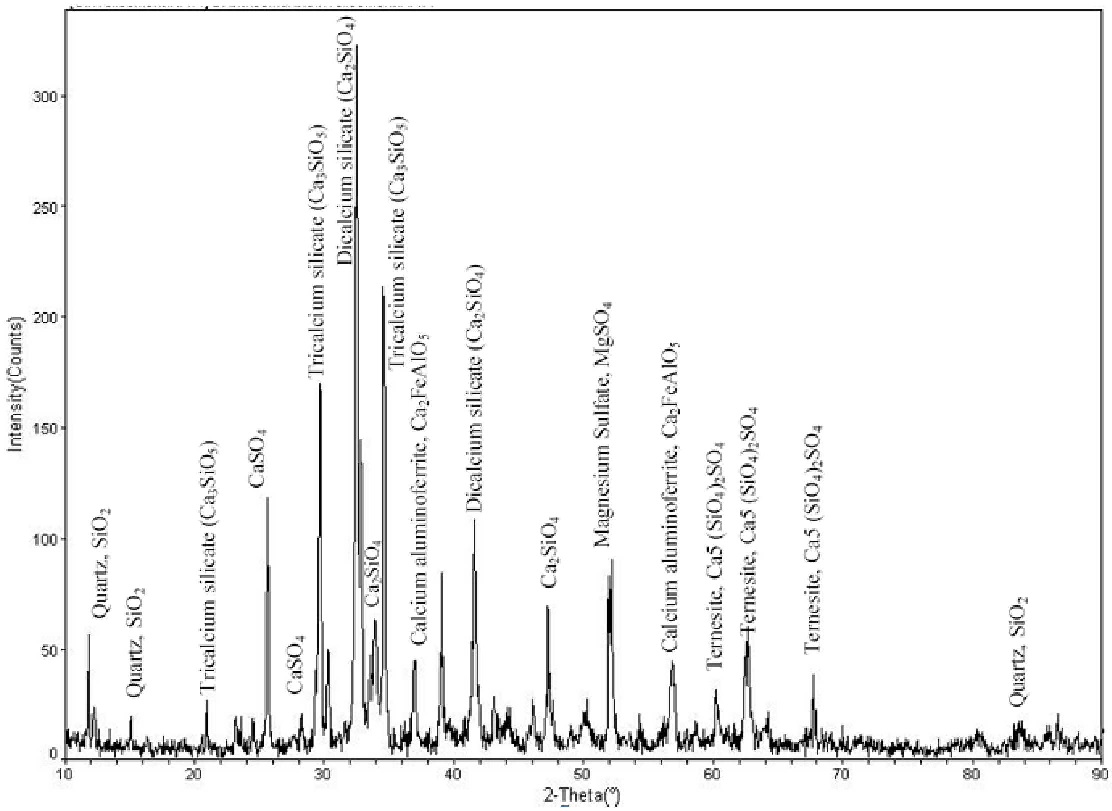

Figure 1. Typical X-ray-Diffraction (XRD) Pattern for Class H Oil Well Cement

(iii) Four and Two Probe Method: This commercially available device is used to measure the resistivity of soils. In this study it was used to measure the resistivity of curing cement and drilling mud. Measures resistivity in the range of 0.01 Ω.m to 1000 Ω.m (Fig. 2(b))

(iv) Two Wire Method: The LCR Meter was used to measure the resistance between any two wires attached to the mold using AC at relatively high frequency. This configuration is first calibrated using the same liquid (cement slurry or drilling mud) to determine the parameter K in Eqn. (1). Typical variation of parameter K with time is shown in Fig. 4.

(c) Viscosity

Standard viscometers, including Fann (high shear strain rate) and Brookfield (lower shear strain rate), were used to quantify the viscous properties (yield point, plastic viscosity) of various cement and drilling mud mixes. The viscometers were calibrated using several standard solutions.

(d) Setting Time

The Vicat setting test (ASTM C191) was used to determine the initial and final setting times for hydrating cementitious mixtures. It measures the change in the penetration depth of a plunger with a diameter of 1.13±0.05 mm under a constant applied load (300

g) as increasing structure formation acts to reduce the extent of penetration into the specimen. The test is used to determine the initial and final setting times at penetration depths of 25 mm and 0.5 mm respectively.

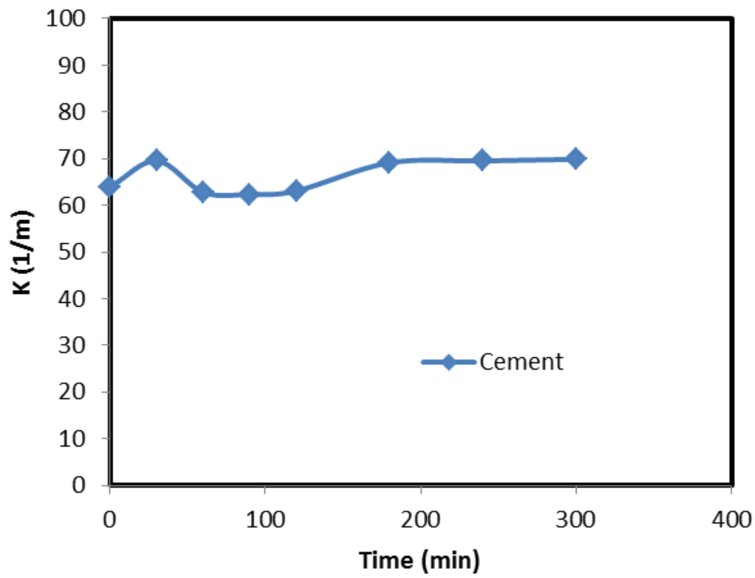

Figure 4. Typical Calibration for the parameter K in the Two Wire Method

(e) Curing Conditions

Specimens were cured at room condition (temperature of 23±2˚C) and at higher temperatures (oven cured) based on the selected testing condition.

(f) Compression Test (ASTM C39)

The cylindrical specimen was capped and tested at a predetermined controlled displacement rate. The dimension of the specimen was measured using a Vernier caliper. In order to measure the strain, a commercially available extensiometer (accuracy of 0.001% strain) was used. The change in resistance was monitored using the two probe method and the parameter in Eqn. (2) was used relate the changes in resistivity to the applied stress.

5. Results and Discussion

The main focus of this study was to enhance the sensing capabilities of cement slurry from the time of mixing to solidified state. Also Class H and Class G oil well cements were used to demonstrate the potential of making the material more piezoresistive and sensing without significantly affecting the rheological properties. Although several modified cement slurries were studied only the performance of selected materials are presented. The test results form the unmodified cement was used as the baseline for comparison. Also various types of drilling muds were modified and tested to quantify the sensitivity of the changes.

5.1 Smart Cement

(a) Density

The average density of the cement mix was measured immediately after mixing. For the cement mix with the water-to- cement (w/c) ratio was 0.35 the density was 2019 kg/m^3. The density with the addition of 0.075% carbon fibers was 2026 kg/m^3, hence the addition of 0.075% of carbon fibers had minimal effect on the density.

(b) Rheological Properties

The effect of carbon fiber additive on plastic viscosity and yield point of API class H cement slurries was studied. All additives were weighed by weight of cement. Standard API high speed mixer was used at 4000 and 1200 rpm to mix the slurries. Slurries were mixed without and with 0.1% carbon fiber based on weight of cement (BOWC). FANN standard viscometer (shear strain rate of 512 and 1024 sec^{-1}) was used to determine the shear stress – strain rate relationships for the various cement mixes and the responses are shown in Fig. 5.

Table 5. Effect of Carbon Fiber Addition on the Rheological Properties of Cement

Composition	Carbon Fiber (BOWC)	Plastic Viscosity (PV) (cPs)	Yield Point (YP) (lb/100 tf^2)	Y_P/PV ratio	Remarks
Water-to-cement ratio of 38%	0% CF	71	24	0.34	Minor changes in the plastic viscosity. Reduced the Y_P.
	0.1% CF	65	22	0.34	
	0.2% CF	73	20	0.27	

(i) Fibers

***Carbon Fiber**:* The Bingham plastic relationships for cement with and without carbon fibers are compared in Fig. 5. Addition of 0.2% carbon fiber to the cement slurry with a w/c ratio of 0.38 increased the plastic viscosity by 2 cps and reduced the yield point by 4.5 lb/100 ft^2 (Table 5). Also the ratio of YP/PV reduced from 0.34 to 0.27. With 0.1% carbon fiber the effects on the rheological properties were less and the ratio of YP/PV remained unchanged.

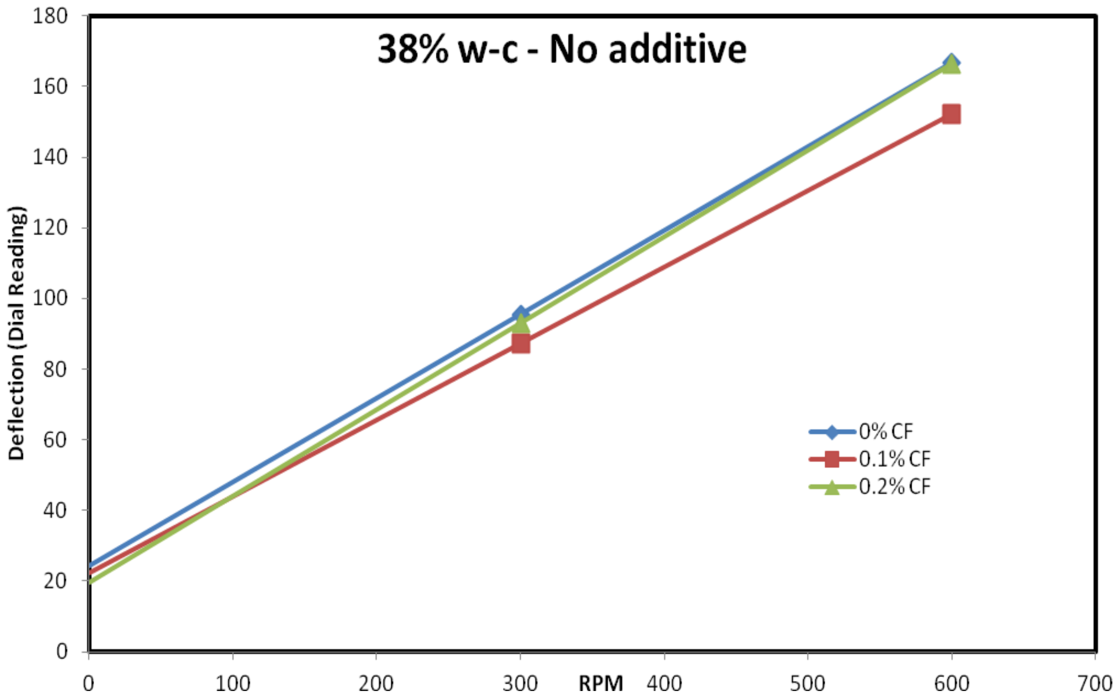

Figure 5. Variation of Shear Stress- Strain Rate Bingham Relationships for Cement with and without Carbon Fibers

(b) Setting Behavior

(i) Resistivity

Based on the current study and past experience of the researchers, the change in resistivity with time can be represented as shown in Fig. 6. Hence several parameters can be used in monitoring the curing (hardening process) of the cement. The parameters are initial resistivity ($\rho_{initial}$), minimum resistivity ($\rho_{minimum}$), time to reach the minimum resistivity (t_{min}), resistivity after 24 hours of curing (ρ_{24}) and percentage of maximum change in resistivity $[(\rho_{24}-\rho_{min})\ /\rho_{min}]100\%$. The test results from various cement compositions are summarized in Table 6. The initial resistivity of cement with w/c ratio of 0.38 and 0.44 were 0.98 Ω.m and 1.33 Ω.m, a 35% increase in the resistivity. Also the time to reach the minimum resistance was reduced by 30 minutes when the w/c ratio increased from 0.38 to 0.44. The maximum change in resistivity for cement with w/c ratio of 0.38 and 0.44 were 67% and 306% respectively. These observed trends clearly indicate the sensitivity of resistivity to the changes occurring in the curing of cement (Table 6).

(i) Fibers

Carbon Fiber*:* Addition of 0.2% of carbon fiber (CF) to cement with w/c ratio of 0.38 reduced the resistivity to 0.94 Ω.m, a 4% reduction from the neat cement with w/c ratio of 0.38. The maximum change in resistivity in the first 24 hours with 0.2% CF was 52% and the time to reach the minimum resistivity was 70 minutes. Addition of CF to the cement with a w/c ratio of 0.44 had minimal effect on the initial resistivity but reduced the maximum change in resistivity after 24 hours (Table 6).

(ii) Filers

Silica fume: With 10% silica fume and w/c ratio of 0.38, the initial resistivity was 1.11 Ω.m, a 13% increase in resistivity compared to the cement only mix. The time to research minimum resistivity was 50 minutes and was not affected by the addition of up to 0.2% CF. The maximum resistivity change was 87%. With and without CF, the observed trends were similar, hence addition of carbon fibers had minimal effect on the curing of the cement with 10% silica fume.

Bentonite: With 2.5% bentonite and w/c ratio of 0.38, the initial resistivity was 1.26 Ω.m, a 28% increase in resistivity compared to the cement only mix. Addition of 0.1% CF didn’t affect the initial resistivity. The time to research minimum resistivity was 190 minutes and adding 0.1% CF reduced it to 90 minutes. The maximum resistivity change was 120%. With 0.1% CF, the maximum resistivity change was 214%. Hence adding carbon fibers influenced the resistivity of the curing cement with 2.5% bentonite.

***Barite**:* With 4% barite and w/c ratio of 0.38, the initial resistivity was 1.97 Ω.m, a 100% increase in resistivity compared to the cement only mix. Addition of 0.1% CF reduced the initial resistivity to 1.32 Ω.m. The time to research minimum resistivity was 40 minutes and was not affected by the addition of 0.1% CF. The maximum resistivity change was 111% and with carbon fiber addition it was 124%. Addition of carbon fiber influenced the resistivity changes in the curing cement with 4% barite.

Fly ash: With 5% fly ash and w/c ratio of 0.38, the initial resistivity was 0.83 Ω.m, a 15% reduction in resistivity compared to the cement only mix. Addition of 0.1% and 0.2% CF increased the initial resistivity. The time to research minimum resistivity was 30 minutes and adding CF didn’t affect the time to research the minimum resistivity. The maximum resistivity change was 255%. With 0.1% and 0.2% of CF, the maximum resistivity change was reduced to 135% and 128% respectively.

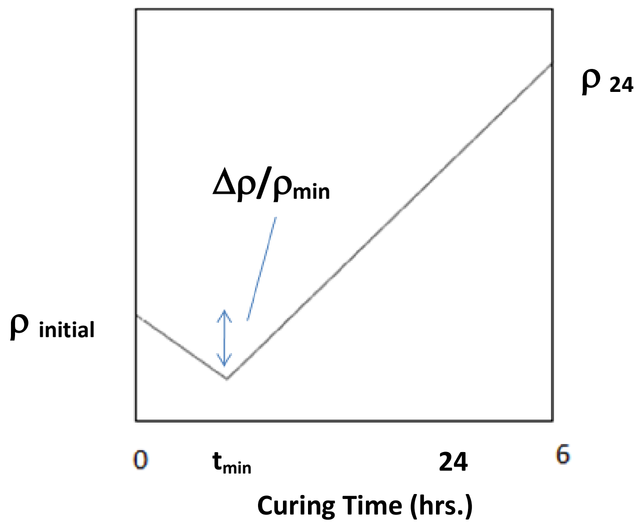

Figure 6. Typical resistivity–time relationship during the curing process

Table 6. Resistivity Change with Curing Time for Oil Well Cement (class H) with different additives and Carbon Fibers

Mix proportions		Initial resistivity (Ω-m)	Minimum resistivity ρ_{min}(Ω-m)	t_{min} (min)	1Hour resistivity (Ω-m)	24Hour resistivity ρ_{24}(Ω-m)	$[(\rho_{24}-\rho_{min})/\rho_{min}]100\%$
W/C=0.38	Cement (C)	0.98	0.86	70	0.87	1.43	67%
	C+0.2%CF	0.94	0.85	70	0.88	1.30	52%
	C+10%SF	1.11	0.94	50	1.01	1.75	87%
	C+10%SF+0.1CF	1.08	0.96	50	0.96	1.84	91%
	C+10%SF+0.2CF	1.08	0.98	50	1.00	1.79	82%
W/C=0.44	Cement (C)	1.33	1.06	40	1.13	4.30	306%
	C+0.1%CF	1.30	1.20	40	1.26	2.66	121%
	C+0.2%CF	1.32	1.04	70	1.23	2.26	118%
W/C=0.38	C+2.5%Bent	1.26	1.13	190	1.16	2.48	120%
	C+2.5%Bent+ 0.1%CF	1.29	1.00	90	1.02	3.14	214%
	C+4%Barite	1.97	1.91	40	1.95	4.03	111%
	C+4%Barite+ 0.1%CF	1.32	1.24	40	1.28	2.77	124%
	C+4%Barite+ 0.2%CF	1.37	1.28	50	1.30	2.70	112%
	C+5%FA	0.83	0.83	30	1.07	2.96	255%
	C+5%FA+0.1%CF	0.89	0.82	30	0.83	1.93	135%
	C+5%FA+0.2%CF	1.02	0.95	30	0.99	2.17	128%

(ii) Hydration of Cement

A high-performance semi-adiabatic calorimeter (P-CAL 1000, Calmetrix Inc.) was used to record the temperature change during the hydration (7 days) of Class H oil well cement with different additives. The volume of the specimen tested was 1.64 L. As shown in Fig 7 and summarize in Table 7, 0.075% carbon fiber with and without 0.075% Fe/Ni nanoparticles reduced the peak temperature of the cement. Further addition of 1% biosurfactant reduced the peak temperature by 21.5 ºF, and it also delayed the time to reach the peak temperature by 0.7 h.

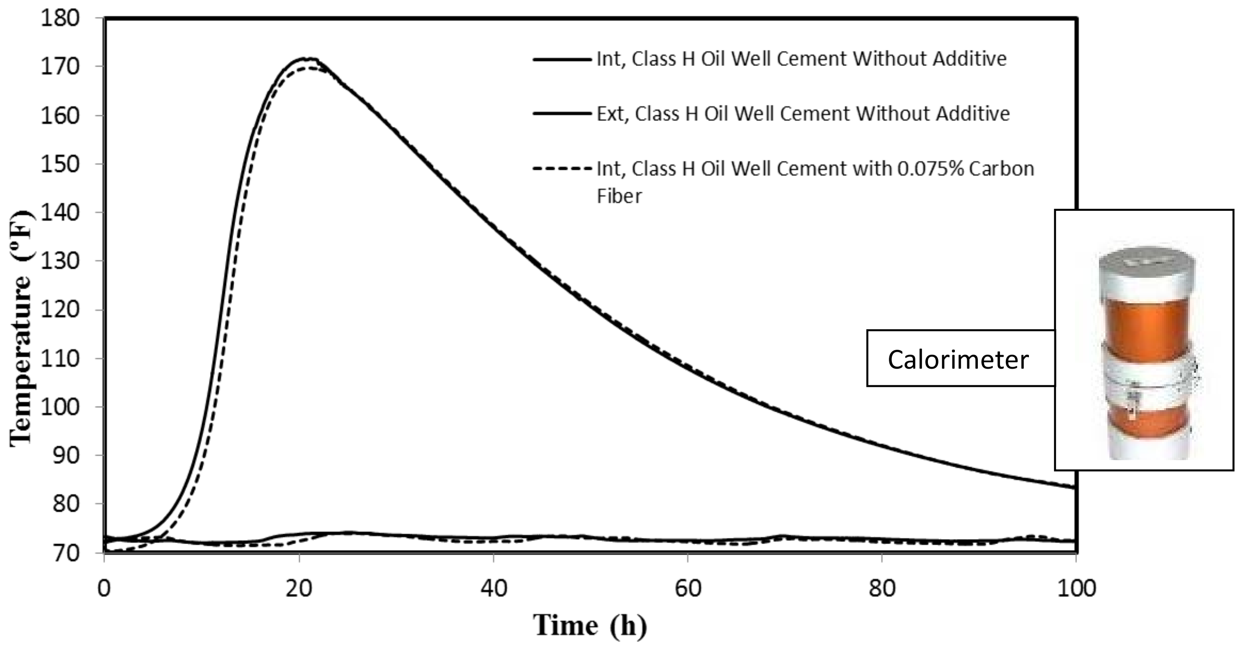

Figure 7. Hydration Temperature with Time for a Class H Oil Well Cement (Int: Internal temperature or hydration temperature; Ext: External room temperature).

Table 7. **Peak Temperature and Corresponding Time for a Class H Oil Well Cement Hydration with Different Additives Recorded by P-CAL 100 Calorimeter**

Additives	Peak Temperature (ºF)	Peak Time (h)
With no Additive	171.7	20.5
With 0.075% Carbon Fiber	169.8	20.7

Note: Water to Cement Ratio = 0.4. The weight percentage of carbon fiber, Fe/Ni nanoparticles and biosurfactant was determined according to the weight of cement powder.

Based on Vicat needle test at room condition the initial setting time was determined to be after 6 hours and the final setting time was about 8 hours. As shown in Fig. 12 the cement continues to hydrate beyond the experimentally determined setting time based on the Vicat needle test (determined by hardness).

(i) Fibers

Carbon Fibers: Addition of 0.075% of carbon fibers to the cement with a w/c ratio of 0.4 reduced the peak temperature by about 2^oF degrees and increased the time to peak reach the peak temperature by 0.2 hr. Hence adding 0.075% carbon fiber (amount used in the piezo-resistive studies) had minimal effect on the hydration process of the cement. This was also reflected in the resistivity and changes in resistivity during the initial 24 hours of curing.

(ii) Surfactant

Biosurfactant: Addition of 1% of biosurfactant to the cement with 0.075% of Fe/Ni Nanoparticles and 0.075% carbon fibers reduced the hydration temperature by 21.5^oF degrees and increased the time to reach the peak temperature by 0.7 hours. Hence adding the biosurfactant affected the hydration of the cement.

(c) Solid Behavior

Piezo-Resistive Behavior

Carbon Fiber: After 7 days of air curing, the specimen (w/c = 0.4) with 0.075% carbon content (total weight of cement mix) was tested under compression loading. Typical change in resistivity with compressive stress is shown in Fig. 15. The change in resistivity in the bulk material was much higher than the strain response. For example, the change in bulk resistivity was 45% as compared to a strain of 0.05%, at a compressive stress of 5 MPa. This shows the magnification of the resistivity response of the modified cement with 0.075% of carbon fibers. The axial compressive strain was about 0.2% at failure for the fiber modified cement and the resistivity change was 80%. The resistivity change was about 400 times higher than the change in strain.

For 0.125% carbon fiber content specimen, the resistivity change response was different where the resistivity initially reduced with applied compressive stress (Fig. 16). Once the crack formed, may be due to cracking of the matrix between carbon fibers the change in resistivity increased considerably. The specimen was tested after 7 days of specimen made. The percentage change in resistivity at failure was around 150%, nearly double the amount observed with 0.075% fibers.

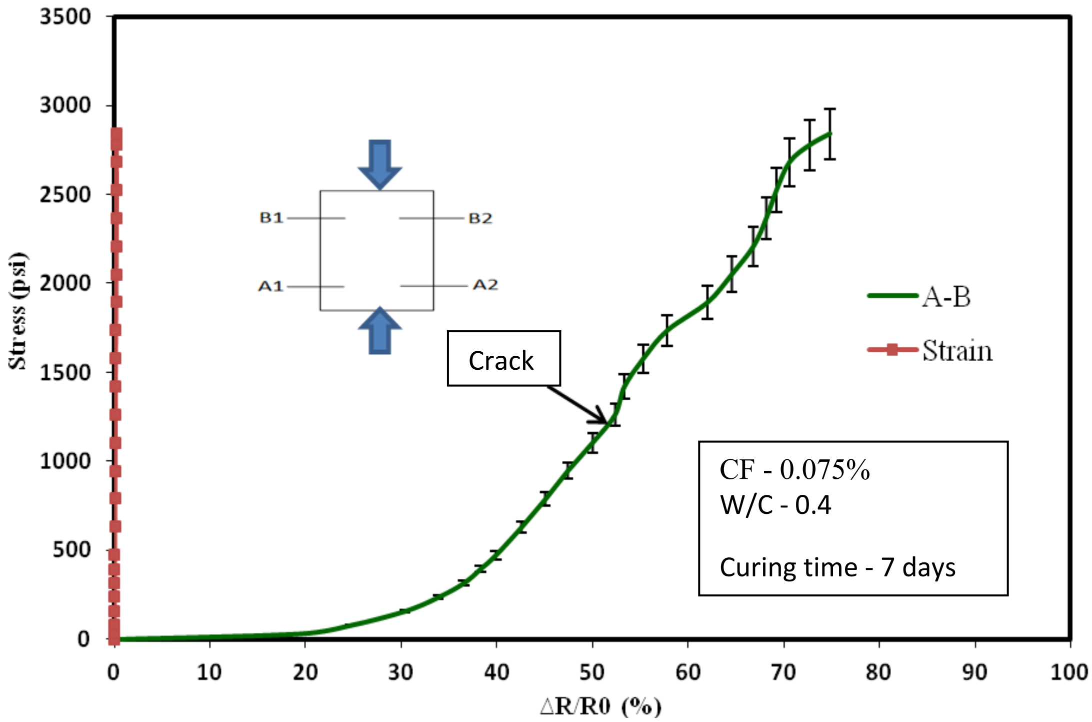

Figure 8. Piezo-resistivity Behavior of Oil Well Cement with 0.075% CF

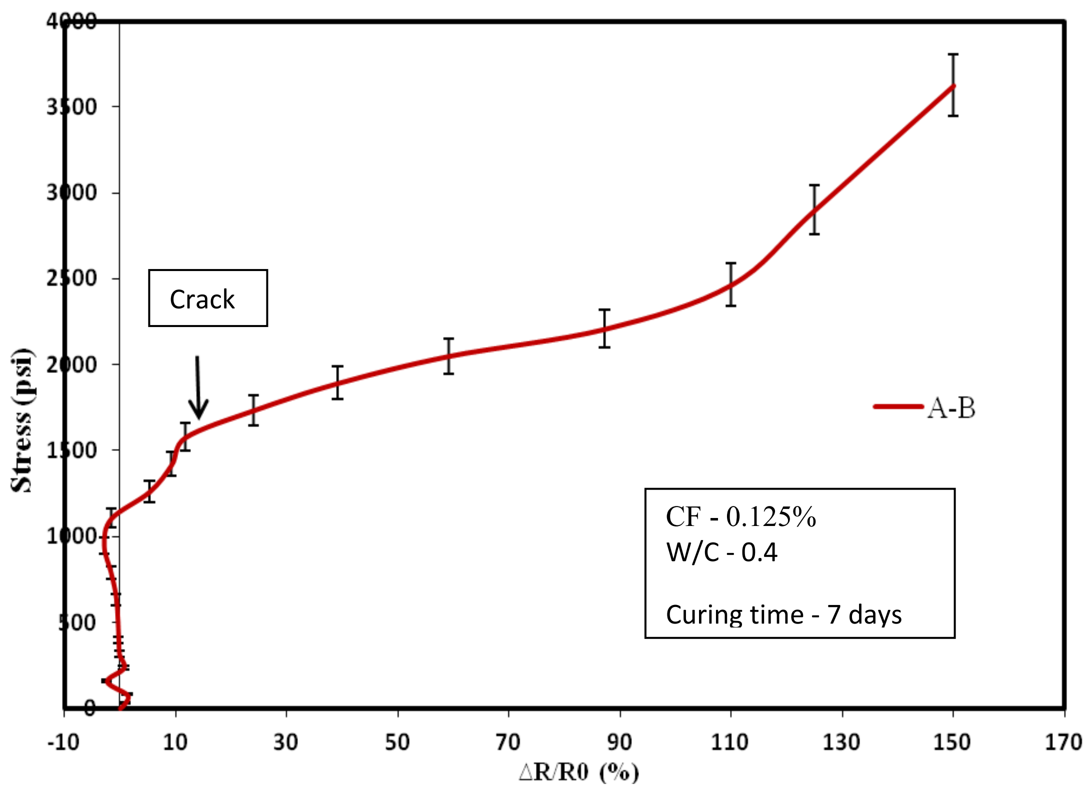

Figure 9. Piezo-resistivity Behavior of Oil Well Cement with 0.125% CF

6. Conclusions

Based on the experimental study in characterizing the modified smart cement and drilling mud/fluid following conclusions are advanced.

(1) Material resistivity will be the sensing property selected to represent both cement slurry and drilling mud/fluids. This selection will unify the real time monitoring, using the same instruments, for both oil well cement and drilling mud.

(2) Resistivity was sensitive to the types and amount of additives used in the oil well cement. Contamination of the cement by bentonite can be detected based on the change in resistivity.

(3) Several resistivity parameters have been identify to characterize the curing of the oil well cement. Changing the water-to-cement ratio from 0.38 to 0.44 increased the resistivity by 30% and reduced the time to reach the minimum resistivity by 30 minutes. During the curing of the cement, initial resistivity reduced by about 10% to reach a minimum resistance and maximum change in resistance within the first 24 hours of curing varied from 50 to 300%.

(4) Addition of 0.1 % carbon fiber to the cement reduced the initial resistivity and made the material piezoresistive. The resistivity change due to applied stresses was 30 to 80 times higher than the change in the strain in the material, making the cement material smart and sensing. Also the addition of 0.1% carbon fiber did not affect the rheological properties of the cement. For the carbon fiber modified smart cement, the resistivity change at peak stress was about 400 times higher than the change in the strain.

(5) Addition of 0.1% of carbon fiber made the cement with other additives such as nanoparticle and silica fume more piezoresistive.

7. Acknowledgement

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