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Zonal Isolation Improvement through Enhanced Cement-Shale Bonding

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Zonal Isolation Improvement through Enhanced Cement-Shale Bonding

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Dedication

To my family, for their support and encouragement.

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Abstract

Zonal Isolation Improvement through Enhanced Cement-Shale Bonding

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The incompatibility of cement and shale and the subsequent failure of primary cementing jobs is a very significant concern in the oil & gas industry. On wells ranging from hydraulically fractured shale land wells to deepwater wells, this incompatibility leads to an increased risk in failing to isolate zones, which could possibly present a well control hazard and can lead to sustained casing pressure. The cement-shale interface presents a weak link that often becomes compromised by the loads incurred either during drilling, completion/stimulation or production phases.

To formulate cements for effective zonal isolation, it is crucial to evaluate the bond strength of the cement-shale interface. Although several studies have focused on the interactions between cement and sandstone, very few studies have addressed the bonding behavior of cement with shale. The conventional push-out test protocol used to measure cement-to-sandstone shear bond strength has proven to be difficult to apply on shale due to its laminated or brittle nature that complicates sample preparation and can lead to shale or cement matrix failure instead of failure at the interface. In this paper, we present a

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novel, simple and versatile laboratory test procedure to measure the shear bond strength between cement and shale.

The new procedure was used to develop cement formulations to improve the cement-to-shale bond. Two different design approaches were investigated. One involves introducing Gilsonite into cement to maintain shale integrity. The second design involves using surfactant to improve cement interfacial sealing property. Our results indicate that bond strength of cement with shale can be enhanced significantly incorporating surfactant in cement slurries.

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CHAPTER 1 : INTRODUCTION

Gas migration remains a big challenge to the construction of wells ranging from hydraulically fractured shale wells to deepwater wells. Hydraulic fracturing, which is an important practice in recent years as a technique to stimulate production from unconventional oil and gas reservoirs, has caused public concern regarding fractures potentially contaminating ground water sources and affecting well integrity (Federal register, 2012). For offshore wells, over 8,000 wells in the Gulf of Mexico currently exhibit sustained casing pressure (Federal register, 2010). The origin of the problem in both cases, in reality, is related to the difficulties in achieving good zonal isolation across shale formations during the well construction phase, as well as maintaining it during the lifetime of the well. Poor primary cementation, or potentially compromised cementing after hydraulic fracturing, is the true source of gas migration behind casing and sustained casing pressure build up (Celia, 2004). If reservoir gas and/or fluids find a path into the formations above the top of cement and migrate upward, there is a possibility for interlayer communication to occur with the possibility to compromise the integrity of shallow aquifers.

Cement is placed in a wellbore to support casing and to provide zonal isolation of the well (Parcevaux and Sault, 1984). Historically, hydraulic and shear bond strength measurements of cement have been chosen as the means to evaluate the ability of cement to provide effective zonal isolation (Evans and Carter, 1962). Extensive work has been done to evaluate cement bonding properties to either casing or conventional formations like sandstone and limestone (Evans and Carter, 1962, Carter and Evans, 1964, Peterson, 1963, Carpenter et al. 1992). Those experimental methods developed for sandstone or pipe, however, were very difficult to apply on brittle and laminated rocks like shale

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(Ladva et al., 2004, Teodoriu et al., 2013, Opedal et al., 2014). So far, only limited information has been gathered about the strength of cement-to-shale bonding.

To overcome the difficulties in evaluating cement-to-shale bond properties under laboratory conditions, the present work provided an innovative new way to examine the shear bond and tensile bond strength between cement and formation rock. New experimental methods were developed to allow quantitative evaluation of bonding properties of various cement formulations against shale. Applying the new testing technique, two types of cement additives were evaluated for their ability to improve cement bond strength. One approach involved introducing of a dry additive, Gilsonite into cement slurries. Another approach incorporated the addition of low surface-tension surfactants to cement. An overview of the contents of this report is stated below.

Chapter 2 gives brief introduction to the concepts of cement bonding properties. The experimental methods that have been used historically to quantitatively assess individual bonding property of cement are summarized. The advantages and limitations with each method are discussed, and the need for a new suitable method for testing shale is explained.

Chapter 3 describes characteristics of cement and rock materials that were used throughout the present study. Background information on Gilsonite and surfactant types is provided, along with reasons that potentially make them great candidates for bond strength enhancement. Introduction to both shear bond strength and tensile bond strength measurement are included as well.

Chapter 4 presents the results and discussion on the investigation conducted in which both Gilsonite and surfactant are evaluated as additives for improving shear bond strength of cement with shale.

Chapter 5 gives a summary about the findings of the study and ideas for future work are recommended.

CHAPTER 2 : BACKGROUND

To appreciate the necessity to develop a superior experimental method for evaluation of cement bond strength, it is important to first understand the significance of cement bond properties for the integrity of a well and why the old testing methods are not currently meeting the needs. Thus, the first part of this chapter gives the definitions of cement bond properties, and highlights the importance of bond strength to well integrity. Next, several widely used experimental methods to characterize cement bond properties are introduced, along with their advantages and limitations. Last but not the least, a literature overview on cement interfacial bonding properties at pipe and formation surfaces is presented. It will be shown that there is a clear lack of reliable data on cement-to-shale bond strength, and that the new test methodology overcomes many of the drawbacks of established tests.

2.1   CEMENT BOND PROPERTIES

Bonding properties of cement-to-casing or cement-to-formation surfaces are important because these values directly relate to the ability of cement to prevent inter-layer communication. The bonding at these two surfaces can be characterized as:

- Shear bond

- Hydraulic bond

- Tensile bond

Shear bond is defined as the bond that mechanically supports casing in the hole (Carter and Evans, 1962). It is determined by measuring the amount of force required to initiate a sliding movement along the cement-to-casing or cement-to-formation interface. This force when divided by the contact area yields the shear bond strength. Hydraulic

bond is defined as the bond to prevent fluid communication (Carter and Evans, 1962). It is determined by pumping either fluid or gas to breach the cemented surface. The pressure necessary to initiate a leakage along the interface yields the hydraulic bond strength. Tensile bond is defined similar to the shear bond, where the stress along the interface is in tension instead of in shear (Peterson, 1963).

Cement de-bonding failure is usually a result of inadequate bond strength or cement shrinkage. External factors that could be detrimental to the cement sheath integrity and trigger cement de-bonding include near wellbore region stress variation, thermal shocks, stimulation treatments, shrinkage and expansion of casing during shut down or recommencing of production, chemical attack, and so on (Parcevaux and Sault, 1984, Carpenter et al., 1992, Teodoriu et al., 2013). Undoubtedly, it is crucial to evaluate the cement bond strengths at the design phase of the well to understand if zonal isolation could be maintained throughout the lifetime of the well.

In this chapter, an overview of the methodologies developed over the years to measure cement bond properties is provided, followed by a review of observations collected utilizing these testing techniques. More importantly, the drawbacks associated with these methods are pointed out, which clearly demonstrates the necessity to develop a better method assessing cement bond properties.

2.2   EXPERIMENTAL METHODS FOR EVALUATING CEMENT BOND PROPERTIES

2.2.1   Measurement of Shear Bond Strength

To measure the shear bond strength between cement and formation, a "push-out" shear bond test cell has been widely used in the petroleum industry for decades (Evans and Cater, 1962, Ladva, 2004, Opedal et al., 2014). As shown in Figure 2-1, a cylindrical

rock sample (sandstone or limestone or shale) is placed in the middle of a slurry mold. A plastic spacer is placed on top of the rock. Cement slurry is poured between the central rock/spacer cylinder and the mold. After certain time of curing, the mold is disassembled and the spacer is removed. Next, the cement specimen with rock inside is flipped upside down. A vertical load is applied on the rock using a load frame. The forced that is required to initiate movement between the rock and cement is the failure load. This load divided by the contact area yields the value of shear bond strength.
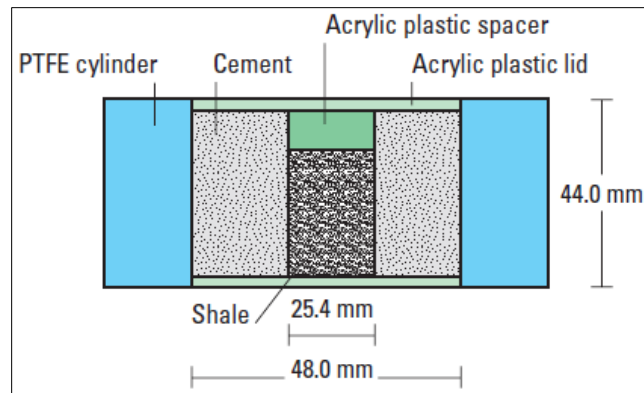
Figure 2-1: Schematic drawing of the "push-out" shear bond test cell (Nelson and Guillot, 2006).

This "push-out" method has its advantages. Besides the straightforward concept, the specimen preparation procedure is simple and repeatable. A test cell can be easily designed and used at elevated temperatures and pressures with moderate modifications. The same concept can be applied to measure the shear bond strength between cement and casing by simply replacing the core with a pipe. For these reasons, the "push-out" measurement has been the preferred choice to evaluate shear bond strength for decades. However, some major limitations associated with this measurement have been reported repeatedly in publications, which have proven this method to be ineffective in evaluating

bond strength between cement and rock, especially for brittle rocks like shale. These limitations include:

1. The failure does not always happen at the interface. Several publications have reported that failure planes are located in the rock or cement matrix rather than at the cement-rock interface. If a brittle rock such as shale is used, a thin film of shale has been found on the cement surface indicating failure in the shale (Ladva et al., 2004, Opedal et al., 2014).

2. While loading the core sample axially, the radial expansion of the internal core will exert tensile stresses on the outside cement ring, which may lead to cement tensile failure prior to shear failure (Ladva et al., 2004).

3. Some of the materials chosen to make the curing mold undergo thermal contraction during cooling. In such a situation, the specimen will be subjected to a contractive force at the end of cooling (Beirute and Cheung, 1990).

4. The reproducibility of the test is poor. A large quantity of tests needs to be performed to get a reliable average with an acceptable standard deviation.

Recently, Teodoriu et al. (2013) used a different approach to perform some cement-to-formation shear bond strength measurements. A cylindrical core sample of 2 inch diameter and 1 inch long was cemented into a composite core of 2 inch diameter and 2 inch long. As shown in Figure 2-2, the composite core was loaded in the direction parallel to the bonding surface until failure. Similar test set up has been used in the civil engineering industry to evaluate the shear bond strength between old and new concrete (Giraldo-Londoño, 2014).

This test method overall generates reproducible data. The sample preparation procedure is fairly straightforward. It is important to mention that although the stress on

the interface is primarily shear stress in the loading direction, partial tensile forces are generated from the bending tendency. Furthermore, for homogeneous materials like concrete and some homogeneous sandstone and limestone, this method could be a good choice to evaluate shear bond strength. For laminated formation materials, however, the failure would happen inbetween the lamintated layers if these are parallel to the loading direction.
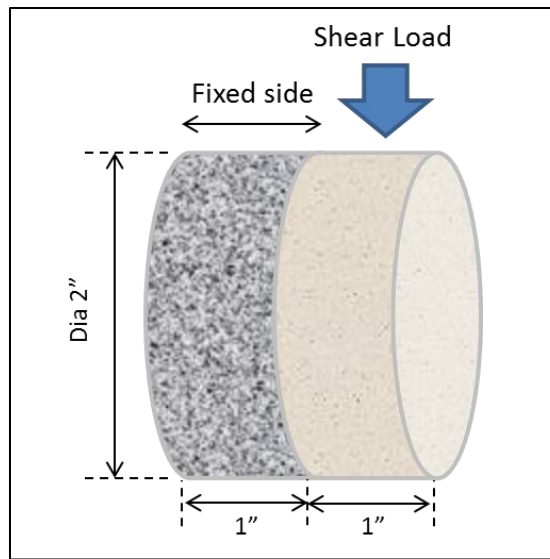
Figure 2-2: Schematic drawing of the shear bond tester using cylindrical composite cores.

Overall, the current shear bond strength tests have their limitations, which prevent them in general from being used in collecting useful and meaningful data. Thus, it is essential to develop a new reliable method where the failure could always happen in shear at the interface.

2.2.2 Measurement of Hydraulic Bond Strength

Originally, hydraulic bond strength was determined utilizing a test apparatus as shown in Figure 2-3. Cement was placed in between the annulus of two pipes and the cement was allowed to harden. Hydraulic fluids were pumped through the inner pipe with the bottom of the whole specimen sealed. The pressure at which hydraulic fluid started to leak through any of the two interfaces was measured and reported as the hydraulic bond strength. Although this method does attempt to represent a downhole situation where a cement plug is placed in an open hole, this method was abandoned due to the observation that hydraulic leakage did not occur until the shear bond strength was exceeded. When the bond strength exceeded, the pipe was no longer in contact with the cement sheath thus causing the cement to be pushed out, and consequently the recorded failure pressure was higher than the actual hydraulic bond strength.
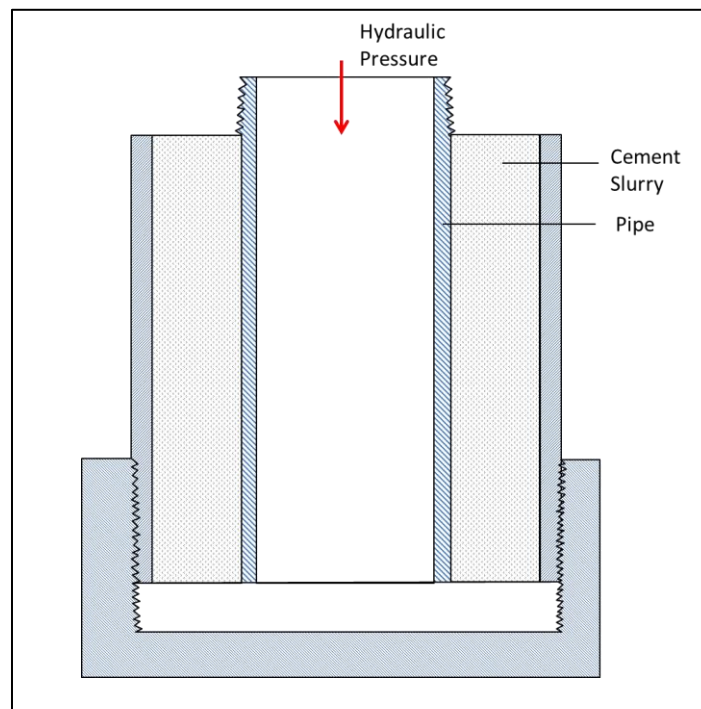
Figure 2-3: Original hydraulic bond strength test fixture (Evans and Carter, 1962).

Evans and Carter (1962) presented a modified method, which is now widely accepted as the standard method to measure hydraulic bond strength (Figure 2-4). The hydraulic pressurizing fluid could be gas (compressed air or nitrogen) or liquid (water, brine or oil). Gases are most frequently used because it causes faster bond failure progression than any of the liquids. To obtain the value of bond strength, hydraulic pressure was gradually increased until leakage occurred at the interface. The appearance of hydraulic fluid at either end of the specimen was considered as failure and the pressure at which failure occurred is considered as the hydraulic bond strength. It is important to mention that according to Evans and Carter (1962), the specimens used in this test can be of any size without significant effect on the final result. The failure of the bond with regard to dimension of the pipe is only a function of time. For instance, a longer sample would fail at the same pressure as a shorter one, however the time for gas to breach the surface would be longer.
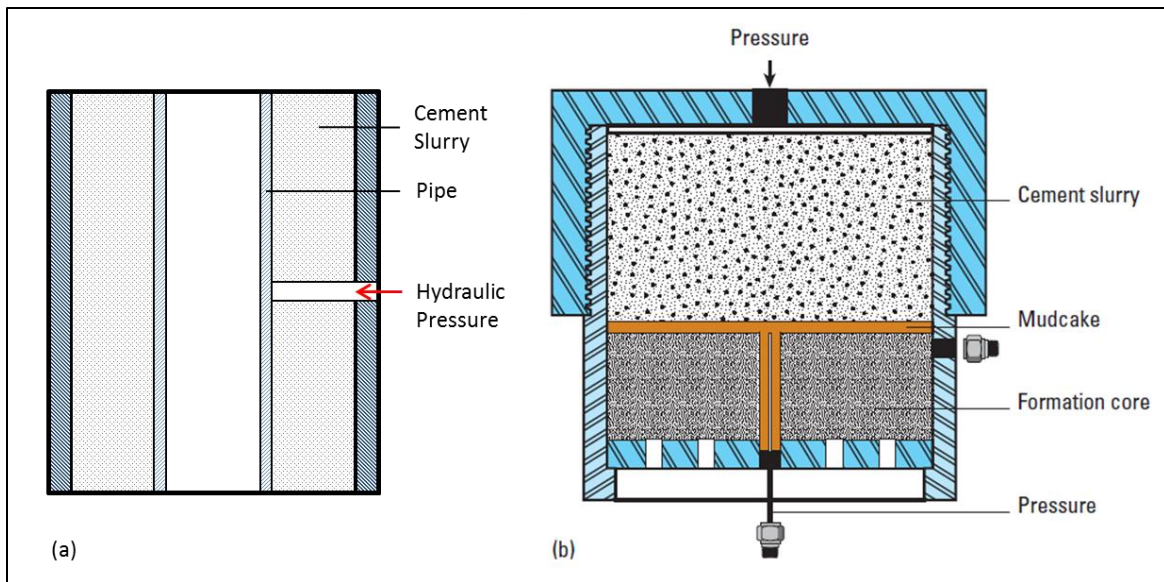
Figure 2-4: Modified and improved hydraulic bond strength test fixture for cement to (a) pipe and (b) formation (Nelson and Guillot, 2006).

2.2.3 Measurement of Tensile Bond Strength

The earliest experimental design that can be found in literature to measure the tensile bond strength was by Peterson (1963). A schematic drawing of the test setup is shown in Figure 2-5 (a). Cement was cast adjacent to the testing material (steel and sandstone in this study). A tensile load was applied perpendicular to the contact surface. The applied load was continuously increased until failure at the bond occurred. The load at failure was then equated with the tensile bond strength at the interface. Ladva et al. (2004) proposed a similar adhesion measurement and used it to determine the tensile bond strength with shale (Figure 2-5, b). The only difference with the test cell was that a pulling rod was cast inside the cement bulk to apply the tensile load where as in the previous setup the load was applied on steel or sandstone.
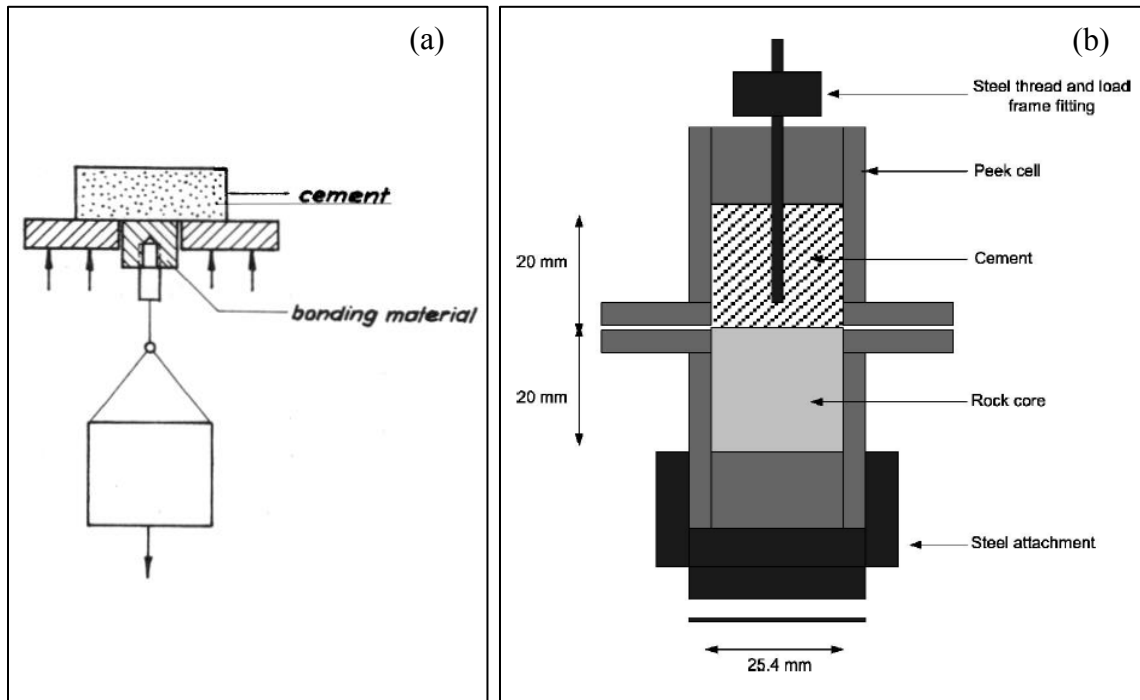
Figure 2-5: Schematic drawings of the adhesion test cell measuring tensile bond strength developed by (a) Peterson (1963), (b) Ladva (2004).

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The biggest benefit from this type of design is that because the surface to be contacted with cement is flat, the roughness and cleanliness of it can be well controlled and quantified. This is not easily achievable on the circumferential surface of cylindrical cores that are used in shear bond strength test or hydraulic bond strength test. Unfortunately, in most cases this test could not be completed successfully due to the fact that the tensile strength of most cement and rock materials is very low, such that the failure usually happens in the bulk cement or in the rock matrix rather than at the bonded interface. Very few reliable data points have therefore been collected on tensile bonding properties and behavior.

2.3   BONDING STUDIES OF CEMENT TO CASING AND FORMATION

Knowing the importance of cement bonding properties, studies have been going on for decades to find a cementation system that provides the most effective sealing in a wellbore. With the experimental methods discussed above, the bonding of casing with cement formulations was assessed under laboratory test conditions. Factors influencing the cement-to-pipe bonding properties have been identified as (1) variation between brands of cement within a given API class (Evans and Carter, 1962); (2) availability of external water for cement hydration (Carpenter et al. 1992); (3) curing pressure and curing period (Evans and Carter, 1962); (4) pipe surface finish and roughness (Evans and Carter, 1962, Carpenter et al. 1992); (5) pipe surface cleanness and presence of drilling fluids (Evans and Carter, 1962, Carter and Evans, 1964); (6) wettability of pipe surface (Peterson, 1963); (7) cement shrinkage and elasticity (Parcevaux and Sault, 1984); (8) thermal and stimulation stresses (Carter and Evans, 1964); (9) viscosity of the penetrating fluid (Carter and Evans, 1964).

It became difficult when researchers tried to study the bonding between cement and formation rocks using the same experimental methods. Various types of rock, including sandstone, limestone, marble and shale, have all been used to measure their shear bond strength with cement. However, the majority of the tests showed that the failure plane was located inside the cement sheath or rock, rather than at the interface (Evans and Carter, 1962, Ladva et al., 2004, Opedal et al., 2014). Evans and Carter (1962) collected a few data points using a limestone with high compressive strength, where the shear bond strength was in the range of 800-7000 psi depending on the cement formulations. They correlated the shear bond strength to compressive strength of cement and showed a linear relation, where shear bond strength is about one tenth of the compressive strength.

When it comes to shale, shear bond tests have been performed on non-swelling shales including Catoosa and Mancos, as well as reactive shales like Oxford clay, Marcellus and Eagleford (Ladva et al., 2004, Teodoriu et al., 2013, Opedal et al., 2014). Using the push-out method (Figure 2-1), shear bond strength was only successfully characterized when the cores were pre-coated with drilling fluids, in which case the bond strength was substantially reduced to less than 100 psi or even zero. All tests at pristine condition (no presence of drilling fluid prior to cementing) concluded with a failure in the shale core itself, which leaves no baseline to compare with. Teodoriu et al. (2013) was able to perform a shear bond strength measurement on Catoosa shale using the sliding shear strength tester (Figure 2-2). He reported a bond strength value of 68 psi between Catoosa shale and Class H cement.

Ladva et al. (2004) tried to obtain tensile bond strength measurements on cement-to-steel, cement-to-marble and cement-to-Catoosa shale. Cement-to-marble interface cured for one day at 185° was reported to have a tensile bonding strength of only 25 psi,

whereas the cement-to-steel sample failed in the cement matrix at 43 psi. The same test on cement-to-Catoosa shale showed no bonding at all, where the sample fell apart on handling. It is highly doubtful if any of these measurements actually constitute valid characterizations of cement-to-rock bond strength.

2.4 CONCLUSIONS

As shown above, currently there are no standardized testing guidelines available for reliable and repeatible measurement of the shear and tensile bond strengths of cement-to-rock interfaces under laboratory conditions. The existing methods all present issues, which limit their effectiveness in evaluating interaction between cement and rocks. The present work was aimed at developing advanced testing methods that are applicable to wide variety types of rocks, which provide meaningful information to understand cement-bonding properties.

CHAPTER 3 MATERIALS AND EXPERIMENTAL METHODS

3.1 MATERIALS

3.1.1 Shale

Well-preserved shale retrieved from downhole would best represent the real interaction between shale and cement. However, well-preserved shale is very difficult to obtain in large quantities. Thus, the shale chosen for this work was Colorado oil shale outcrop, which does not contain very reactive clays and is relatively easy to handle. Mineralogy of the shale obtained from XRD analysis is listed in Table 3-1.

Table 3-1: Mineralogy of Colorado Oil Shale

Mineral	Wt%
Quartz	38
Calcite	11
Dolomite	33
Aragonite ($CaCO_3$)	16
Plagioclase Feldspar	3
Siderite	Trace
Illite	Trace

3.1.2 Experimental Fluids

The cements used in this study included API Class A, Class C and Class H Portland cements. Tests performed on neat paste slurries consisted of cement and tap water following the proportions specified in American Petroleum Institute (API) specification 10A. Table 3-2 shows particle size of cement dry powder and slurry densities after mixing. Cement slurries were mixed following API RP 10A unless otherwise specified. Additives such as surfactants and Gilsonite were added to cement to

see if they help with improving the cement bond strengths. These materials are further discussed in the following sections. All surfactants tested in this study were added after batch mixing and hand mixed with a spatula. Dry additives (e.g. Gilsonite) were blended with cement powder prior to batch mixing.

Table 3-2: Cement powder particle size distribution and slurry properties

	Cement Particle Size d_{50} (μm)	Water Content (bwoc)	Slurry Density (ppg)
Class A cement	17.2	46%	15.6
Class C cement	13.7	56%	14.8
Class H cement	16.1	38%	16.4
d_{50} = median diameter of cement particles			
bwoc = by weight of cement powder used in the formulation			
ppg = pound per gallon			

3.1.3   Surfactants

Surface active agents (surfactants) were first introduced to cement slurries and pre-flush fluids by Eric et al. (1975). Since then, various types of surfactants have been used in cement slurries for different purposes, including (1) retardation, (2) dispersion, (3) fluid loss control, (4) preparation of foamed cements, (5) preparation of pre-flush or spacer fluid, and (6) gas migration control (Hibbeler et al., 1993, Cowan and Eoff, 1993, Nelson and Guillot, 2006).

Surfactants used in pre-flushes and spacers are distinctly different in functional groups and physical characteristics than those used as dispersants (e.g. polynaphthalene

sulfonates). Surfactants contained in spacers and washes provide the essential capabilities of removing oily compounds absorbed onto the casing and formation surfaces, and even more importantly, leave a water-wet surface for cement to bond with. Examples of such surfactants include sulfates of fatty ethoxy alkylphenols (Eric et al., 1975) and amphoteric products of the primary cocoamine (Motley et al., 1974). The distinct characteristics of these surfactants are their low surface tension and low molecular weights. Cowan and Eoff (1993) were the first to evaluate these types of surfactant in cement systems with respect to their interfacial sealing properties by utilizing a customized U-Tube test apparatus. The U-Tube test set up measures the gas leakage rate through a measured volume of cement at various differential pressures. Essentially, the cement "interfacial sealing" ability to gas obtained from this U-Tube apparatus represents both cement anti-gas migration ability and cement hydraulic bonding property at the casing surface. According to their study, four surfactants investigated provided superior interfacial sealing between cement and pipe, namely sodium lauryl sulfate, coco amidopropyl betaine, ethoxylated nonyl phenol with 9-10 moles ethylene oxide, and ethoxylated C_{12-15} linear alcohol sulfate with 3 moles ethylene oxide. In addition, less volumetric shrinkage was observed during setting and hardening. The overall cost of well cementation was reduced because of the multiple functions offered by the addition of surfactants.

In the present work, we extended the research of Cowan and Eoff (1993) by looking into the bonding properties of surfactant cement between cement and shale. Surfactants listed in Table 3-3 were investigated. All surfactants were formulated at a concentration of 1% by weight of mix water and were tested at room temperature.

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Table 3-3: Description of surfactants investigated in this study

Surfactant	Description	Concentration % bwow
A	polyethylene glycol undecyl ether	1
B	coco amidopropyl betaine	1
C	ammonium C_6-C_{10} alcohol ether sulfate	1
bwow = by weight of water		

3.1.4 Gilsonite

Gilsonite, also known as uintaite, is a naturally occurring hydrocarbon bitumen found only in Northeastern Utah. Gilsonite is actually the trademarked brand name of uintaite provided by American Gilsonite Company. However, over the years the term "Gilsonite" is used interchangeably with "uintaite". Gilsonite is a relatively pure hydrocarbon with a brownish-black color. It has a very low specific gravity that is close to 1. The softening point of Gilsonite is approximately 350° F. Some of the Gilsonite products tested here have been treated with surfactant to achieve better water dispersibility.

For many years, Gilsonite has been used in drilling fluids as a lost circulation material (LCM) or a borehole stabilizer. A few early patents first proposed using Gilsonite as a filler material to treat earth formations in situations where lost circulation was encountered (H and D, 1951, Larsen, 1952, Mayhew, 1957). Through the years, Gilsonite has been proven to be effective in stabilizing problematic shale zones. Field (1968) first explained that adding asphalt-based materials could control sloughing shale by sealing or plugging the micro-fractures and minimizing shale slippage. Some

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publications confirmed this statement with field observations where cuttings obtained from wells showed Gilsonite in the micro fractures of the shale (Anderson and Edwards, 1979, Christensen et al. 1991). Davis and Tooman (1989) furthermore conducted tests on drilling fluids containing Gilsonite at downhole temperature and pressures. They concluded that Gilsonite becomes malleable at elevated temperature and intrudes into the pore spaces and micro fractures of shale, therefore bonding the clay platelets together and stopping the matrix from disintegration. Along with this theory, there is also another theory that can be found in literature. Instead of assuming that the Gilsonite is uniformly dispersed in the drilling fluid and is subsequently transported to the shale surface through filtration process, some researchers think that surface-treated Gilsonite derivatives can actually chemically bond to the surface of shale, and shield water-sensitive formations by forming a film of impermeable barrier (Lal, 1999).

In cementing applications, Gilsonite has primarily been used as a light weight additive based on its low density, chemical inertness and low water requirements. Other benefits of the addition of Gilsonite include good lost circulation control and the ability of cement to self-heal (Slagle and Carter, 1959, Leroy and Martin, 2012). No published work has been found in current published literature about the interaction between Gilsonite-modified cement and shale. Considering the affinity of Gilsonite towards shale, as mentioned above, it was considered to be very interesting to evaluate Gilsonite's ability to enhance zonal isolation across shale zones by improving the bond strength between cement and shale formations.

Three types of Gilsonite were investigated in this study. G_1 is a nonionic surfactant-coated Gilsonite, which has the best dispersibility in water. G_2 is a commercial Gilsonite product that has been widely used as a light-weight additive for

cement. G_3 is a finely ground version of G_2, with the same chemical composition. The particle size distribution of these three types of Gilsonite is listed in Table 3-4

Table 3-4: Gilsonite properties

	Particle Size Distribution, μm			Specific Gravity
	d(10)	d(50)	d(90)	
G_1	9.2	26.2	88.7	1.05
G_2	39.5	295.9	937.2	1.05
G_3	15.1	91.8	286.9	1.05

3.2   EXPERIMENTAL METHODS

3.2.1   Compressive Strength Test

Cement slurries were prepared according to the procedure stated in Section 3.1.2 and were placed in 4"×2" cylindrical plastic molds. The specimens were cured in desiccator with 70-80% humidity and maintained at 70° F. After three days curing period, specimens were de-molded and tested using a 40,000 lbf compression-testing machine at a constant loading rate of 200 lbf/s.

3.2.2   Interfacial Shear Bond Strength Test

Different from the "push-out" test protocol, a new cement-to-rock shear bond strength test method is proposed (in Figure 3-1.). A 1.5" diameter × 4" long shale core sample was cut into two at a 60 degree angle from radius direction. Cement was cast in place on top of the shale to form a bond at the inclined surface (see Figure 3-1 (a)). After

curing, the resulting cement and shale composite core plug was trimmed to reach a length (*l*) -to- diameter (d) ratio of 2.0 to 2.5. A vertical compression force was applied at constant loading rate (in this study loading rate is 0.03inch/min) until failure occurred at the contacting surface (see Figure 3-1 (b)). The maximum compressive force at which the bond breaks is denoted as F_{peak}. The shear bond strength value can be obtained by dividing the shear force applied on the interface by the interfacial area. The shear force (F_{shear}) is simply given by

$$F_{shear} = F_{peak} \times \sin 60°$$ (3.1)

The area of the elliptic interface (A) can be expressed as

$$A = \pi \times \frac{d}{2} \times \frac{1}{2}\left(\frac{d}{\cos 60°}\right) = \frac{\pi d^2}{4\cos 60°}$$ (3.1)

Where, d = diameter of the composite core sample. In this work all core samples are 1.5" in diameter.

Therefore the shear bond strength (σ_s) can be calculated as

$$\sigma_s = \frac{F_{peak} \times \sin 60°}{\dfrac{\pi d^2}{4\cos 60°}} = \frac{4F_{peak}\sin 60° \cos 60°}{\pi d^2}$$ (3.3)
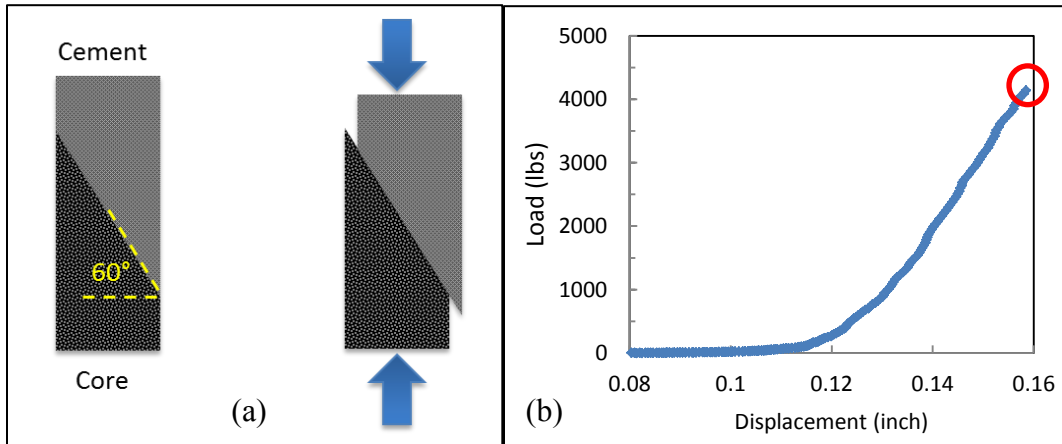
Figure 3-1: Cement-to-rock shear bond strength testing method. (a) Illustration of the testing method. (b) Example of Force vs Displacement data obtained from the test.

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The reason core samples are cut diagonally at 60 degrees is based upon Mohr-Coulomb failure analysis. The basis of Mohr-Coulomb envelope is given in Figure 3-2. The effective normal stress (σ_n) and shear stress (τ) on the failure plane can be graphically analyzed with respect to the effective principle stresses. σ_1 and σ_3 are effective maximum and minimum principle stresses respectively. In an unconfined compressive strength (UCS) test, where an intact rock sample is compressed axially until it fails, the angle at which the fault develops can be described by β (Figure 3-2). β is the angle between the maximum principle stress (σ_1) and the fault normal. According to Byerlee's law for earthy materials, this angle is approximately 60° regardless of rock type (Zoback, 2007).
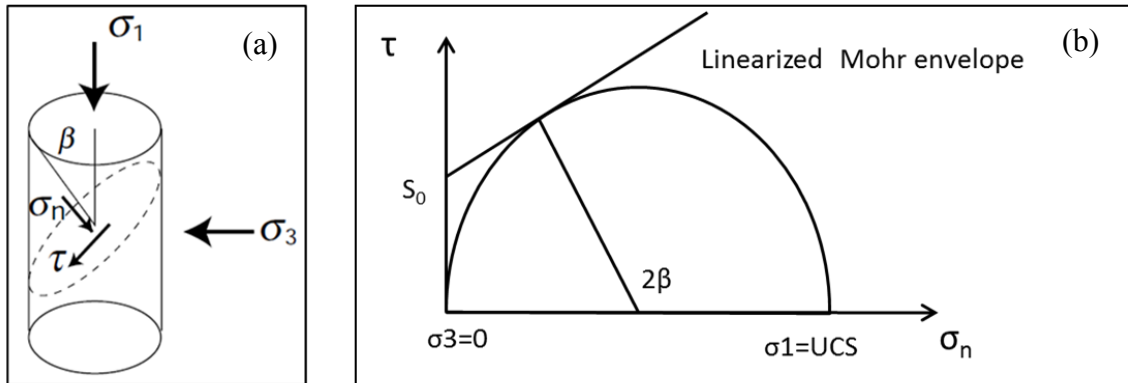
Figure 3-2: Illustration of unconfined compressive strength test (where $\sigma_3 = 0$) (a) samples typically fail in compression when a through-going fault develops. The angle is described by β. (b) The normal stress σ_n and shear stress τ on the fault plane correspond to the point where the Mohr circle hits the linearized Mohr failure envelope (Zoback, 2007).

In the case of making a sample composed of cement and rock with a contacting angle at 60°, if a cement-to-rock bonding is strong enough such that the bonding is stronger than the material, a failure in cement or rock bulk phase (whichever has a lower UCS) should be expected. On the other hand, if the cement-to-rock bonding is weaker

than each of the individual material strengths, then the failure will happen along the interface so that a bond strength value can be calculated using Equation 3.3. Therefore the bonding properties of cement-to-formation can be measured by loading a composite sample as proposed.

It is important to mention that for this testing method, there are a few critical details that need special attention. First and foremost during sample preparation it is vital to ensure that additional stresses are not imparted on the sample during removal of the mold, prior to testing. Depending on the formation type and curing condition, there are a few options in choosing the material to make the cement cast mold (see Appendix A). Secondly, coring high-quality rock samples requires extensive practice and a significant amount of core. High precision coring and trimming equipment is essential for the success of getting an intact sample with desired dimensions. Last but not least, a repeatable surface finishing method needs to be deployed on all specimens to obtain comparable bonding values. Upon collaboration with experienced personnel, both in universities and in industrial laboratories, some recommended best practices on core handling and sample preparation have been obtained and are summarized in Appendix A.

One of the biggest benefits from the current experimental design is that the influences of core surface roughness can be quantified and controlled. Figure 3-3 shows the effect of three different shale core surface finishes on shear bond strength measurement. Method 1 was using an industrial tile saw with a diamond blade to cut the inclined surface without checking the surface roughness. The average was obtained from six samples from two mixes. The error bar was based on standard deviation from six samples, which reflects a very poor reproducibility. In method 2, core samples were cut the same way as in method 1, whereas a surface roughness measurement was performed

on every sample at multiple locations. Only those cores with less than 2 μm roughness on the inclined surface were chosen for the shear bond test. Clearly, it can be seen that the error bar was greatly reduced to less than 15%. In the third method, core samples that were cut the same way as in method 1 and were next polished with 60 grit emery cloth. In comparison with method 2, the standard deviation for method 3 was about the same however the average shear bond strength reduced by nearly 50%, which leaves narrow window for comparing different cement formulations. Overall, method 2 offers the best repeatability and simplest sample preparation procedure and was therefore applied on all shear bond test samples throughout this study.
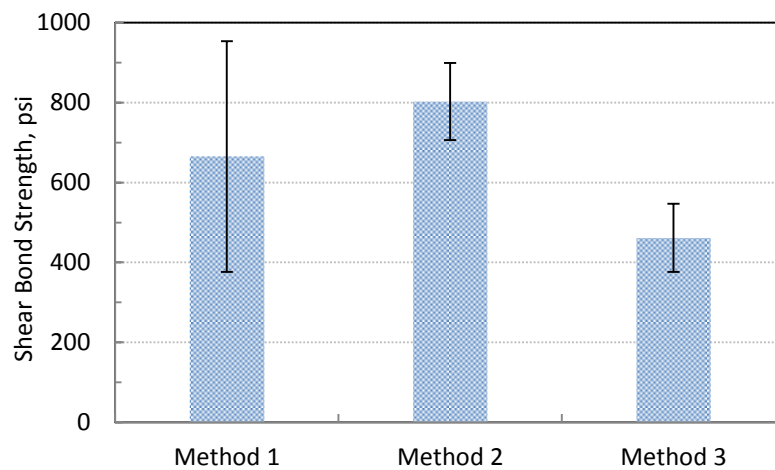
Figure 3-3: Shear bond strength test results at the cement-to-shale interface applying three different core surface finishing techniques.

Based on all the data collected in this study, the overall repeatability of the shear bond strength tests was good. Table 3-5 shows typical examples of sample test results. Among the tests that have been performed in this study, majority of the tests had a standard deviation within 15%. Additional work is ongoing to perfect the current design to incorporate more realistic (downhole) curing and loading conditions.

Table 3-5: Shear bond strength test results

Shear Bond Strength (psi)	70°F (21.1°C)		150°F (65.6°C)	
	Slurry A	Slurry B	Slurry A	Slurry B
Sample 1	848	966	853	1352
Sample 2	691	1165	1111	1397
Sample 3	858	1167	1065	1173
Average	803	1100	1010	1307
Standard Deviation	97	115	138	118

To summarize, compared with the "push-out" method, the current shear bond strength measurement has some major advantages. First of all, the roughness of core surface can be quantified by performing surface roughness measurements. The finish of the surface can be easily controlled with certain cutting or polishing techniques to achieve the same level of surface roughness among all samples. Secondly, unlike the push-out method where the cement ring will be under tension while loading, both cement and core will be experiencing compression load during the entire measurement. This eliminates the possibility of tensile failure in the cement ring prior to the shear bond failure. Lastly, as long as the UCS values of the individual materials is not exceeded, failure will always happen at the interface instead of inside the cement or rock matrix.

3.2.3   Interfacial Tensile Bond Strength Test

A test method was designed to measure the tensile bond strength at the cement-to-rock interface based on the Brazilian splitting test. The Brazilian splitting test was first introduced by Carneiro and Barcellos from the Brazilian Union of Testing and Research

Laboratories in 1953. This test measures the compressive force required to create a maximum shear stress at the top and bottom of a cylindrical rod to split the sample into two halves, from which the tensile strength of the material can be calculated. Applying a similar concept as the Brazilian splitting test, a cylindrical sample composed of half rock and half cement was designed to obtain the tensile bond strength between the two materials (Figure 3-5). Appendix B gives the detailed description of how the cement/formation rock composite sample was prepared.

To measure the tensile bond strength, the composite core sample was removed from the mold and placed in a 1,000 lb-f load frame with the interface perpendicular to the loading platform (as shown in Figure 3-4). The sample was placed between two pieces of semi-circular stainless steel plates to ensure that the sample was loaded along the bond. A compression force was loaded at a constant rate of 0.003 inch/min. A typical tensile bond test profile is presented in Figure 3-5. The tensile bond strength can be calculated as follows:

$$\sigma_t = \frac{2F_{max}}{\pi LD} \tag{3.4}$$

Where:

σ_t = splitting tensile bond strength, MPa (or psi),

F_{max} = maximum applied load indicated by the load frame, N (or lbf),

L = thickness of the specimen, mm (or in.), and

D = diameter of the specimen, mm (or in.).
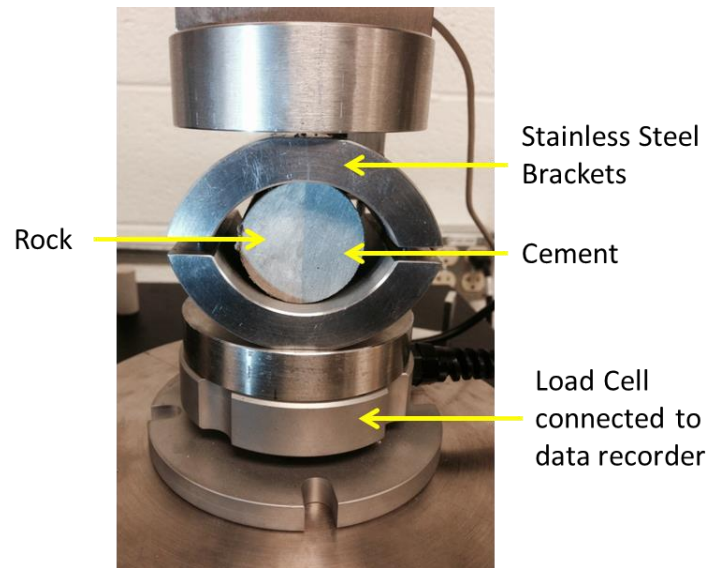
Figure 3-4: Photograph of splitting tensile bond strength test
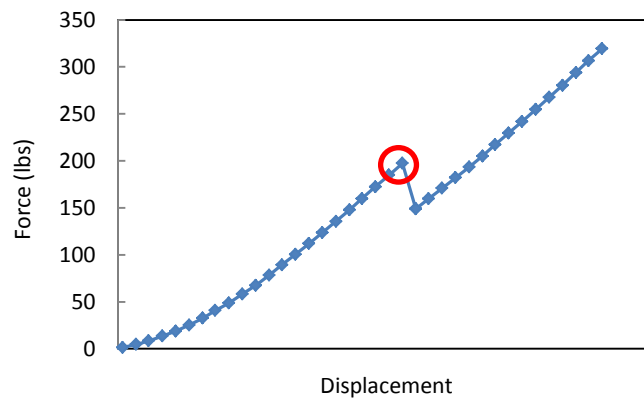
Figure 3-5: Tensile bond strength test profile

Compared with the adhesion tester described in Chapter 2, the tensile bond strength measurement proposed as above overcomes the difficulty that both cement and rock materials have very low intrinsic tensile strength. The current measurement avoids applying tension on either side of the composite core. The actual load is in compression, which is transferred to the interface as a tensile load. Several proof of concept tests have been performed. The peak load, which represents tensile bond failure, can be captured as

shown in Figure 3-5. Ongoing work is focused on assessing the repeatability of the tensile bond strength measurement and ways to improve sample preparation method.

3.2.4 Visual examination of the cement-to-shale interface

FEI Quanta 650 Scanning Electron Microscope (SEM) was used to observe the cement-to-shale interface as a way to characterize cement behavior against shale. A sample similar to that of a shear bond strength sample was prepared where the bottom half consisted of a 60 degree angled rock along with cement on the top half. Cement was allowed to harden for 3 days. From this sample a 1.5 inch diameter and half inch thick composite disc was obtained. This disc was immersed in ethanol for 3 days to fully displace residual water and stop cement hydration following which the sample was embedded in epoxy. The epoxy was cured for 24 hours at room temperature. To get a smooth and flat surface, the core disc that was embedded in epoxy was polished following four stages of dry grinding with silicon carbide paper (#60, #180, #600, #1200) and then four stages of polishing with diamond pastes (6μm, 3μm, 1μm, ¼ μm). The sample was cleaned between each stage with ethanol. After each stage of polishing, a light microscope was used to assess the smoothness of the surface. Since both cement and shale are not conductive, the specimen surface was coated with Palladium to ensure that electrons could be dissipated from the sample surface to acquire a good image.

CHAPTER 4 : RESULTS AND DISCUSSIONS

Based on the shear bond strength testing method proposed in the previous chapter, the bonding strength of Portland cement against Colorado oil shale was determined. Various types of surfactants and Gilsonites were evaluated of their effectiveness as a bond strength enhancer. Notice that all slurries were cured for 72 hours at 70° F inside a desiccator with humidity maintained at 70~80%.

For all compressive strength, shear bond strength and tensile bond strength tests three samples with the same formulation from one batch mix were tested, and the average and standard deviation values are reported. If the standard deviation was more than 15% of the average value, a repeat test was performed.

4.1   SHEAR BOND STRENGTH RESULTS - NEAT PORTLAND CEMENT SLURRY

The strengths of neat cement pastes were first determined to understand the bonding character of cement with no additives. Figure 4-1 (a) shows the 72 hours cement-to-shale shear bond strength and Figure 4-1 (b) shows the compressive strength for API Class A, Class C and Class H cements. Also plotted in Figure 4-2 is the percentage of shear bond strength relative to compressive strength. From Figure 4-1 (a) it can be seen that the Class C cement slurry provides the highest shear bond strength among the three. The shear bond strength of Class C cement slurry was higher than that of Class A by 40% and higher than that of Class H by 64%.
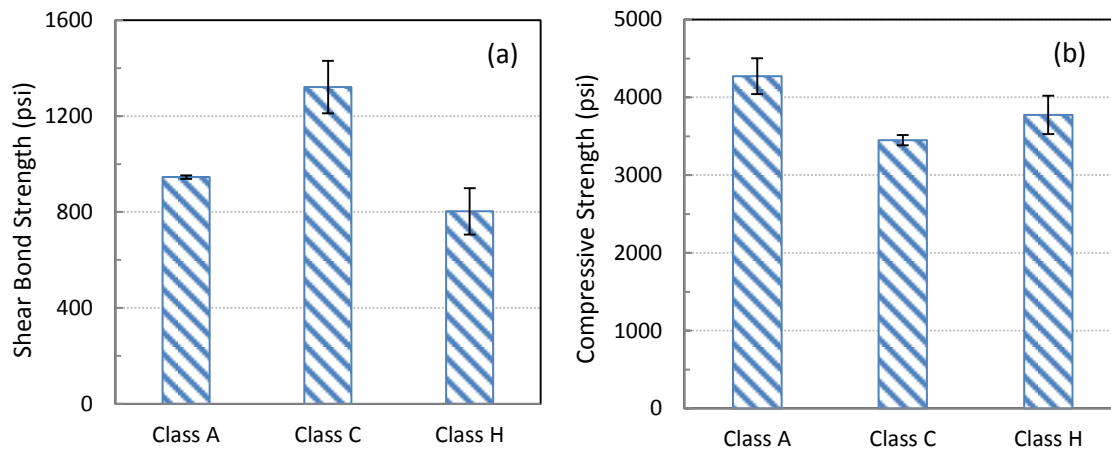
Figure 4-1: 72 hours (a) cement-to-shale shear bond strength and (b) compressive strength for neat paste cement slurries at 70° F
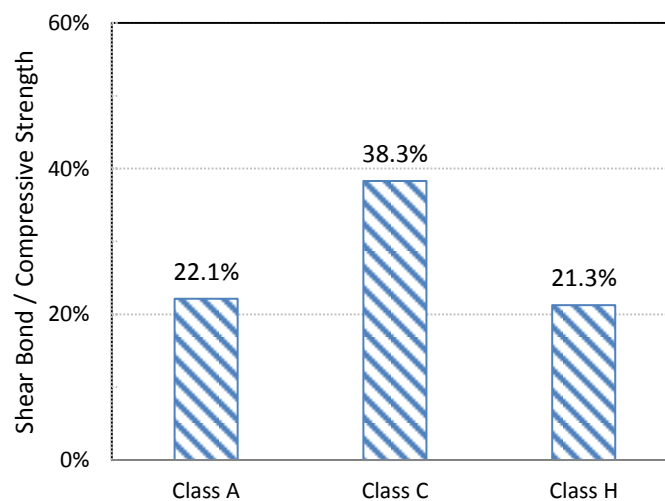
Figure 4-2: 72 hours cement-to-shale shear bond strength relative to compressive strength for neat paste cement slurries

4.2 EVALUATION OF MULTIPLE APPROACHES TO ENHANCE SHEAR BOND STRENGTH

4.2.1 Gilsonite

As pointed out in chapter 3, Gilsonite, when used as a light weight agent, is usually formulated as a solid additive. Additional water needs to be added to maintain the slurry's pumpability. However, to purely investigate the performance of Gilsonite as a bonding enhancement agent, Gilsonite was first introduced by addition based on the weight percentage of cement, in which case the water to cement ratio was maintained constant at 38% without adding any additional water.

In order to evaluate Gilsonite's potential to improve shear bond strength at the cement-to-shale interface, a range of dosages was investigated on three types of Gilsonites to determine whether any of these Gilsonite worked better than others at the specified concentration. The bond strength values were compared with that of neat class H cement. Based on the test results collected at dosages of 2%, 5%, and 10% bwoc, no improvement on shear bond strength was observed for all three types of Gilsonites. Figure 4-3 shows an example of all three Gilsonite samples at a dosage of 2% bwoc. And Figure 4-4 shows shear bond strength results for G_1 Gilsonite at all three dosages. Overall, it can be seen that as the dosage of Gilsonite was increased up to 10% bwoc, the shear bond strength values decreased by 50% compared to that of the neat slurry. SEM imaging was performed on a cement-shale composite core sample where 5% G_1 Gilsonite bwoc was added into the slurry (Figure 4-5). The SEM image showed no accumulation of Gilsonite at the interface. Higher concentrations of Gilsonite were not tested because cement slurries became too viscous to mix without the addition of dispersants or additional water.
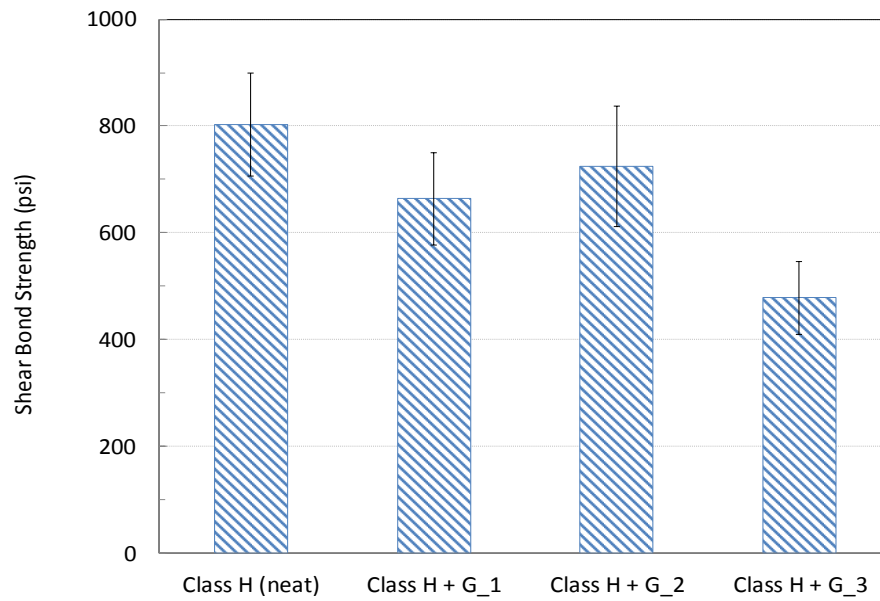
Figure 4-3: 72 hours cement-to-shale shear bond strength for three types of Gilsonite modified cement slurries at 70°F
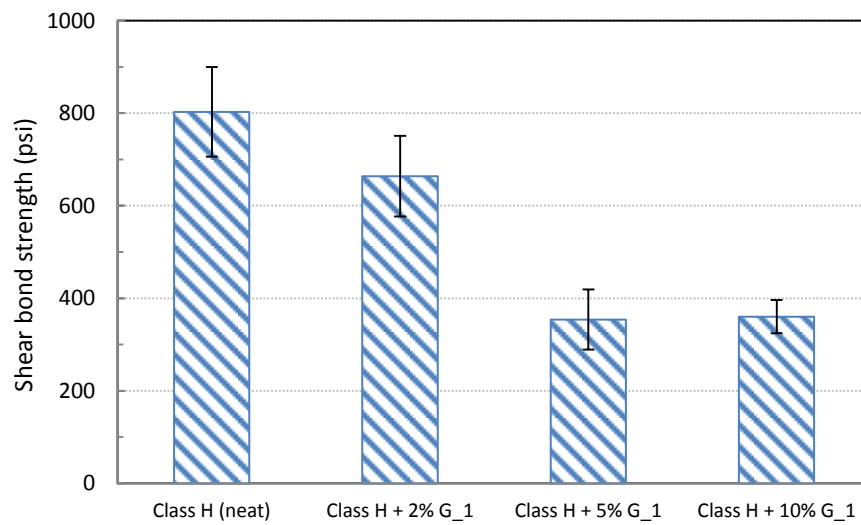
Figure 4-4: 72 hours cement-to-shale shear bond strength for G_1 Gilsonite at different dosages at 70°F
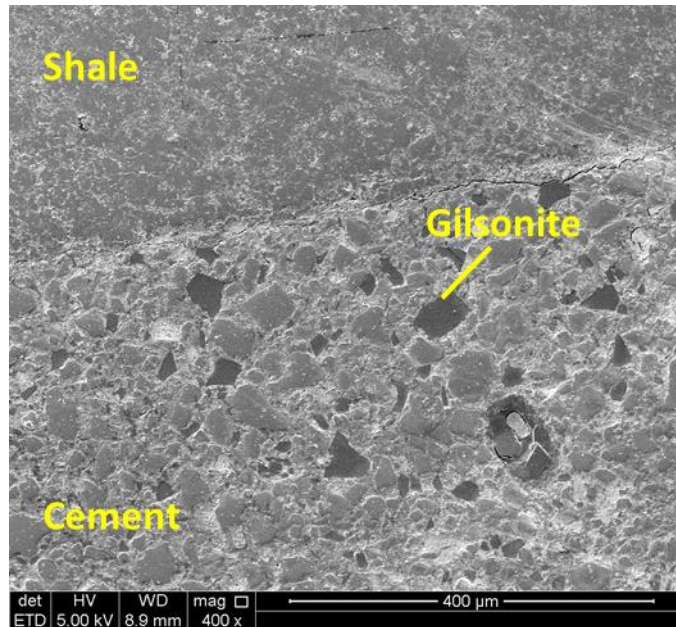
Figure 4-5: SEM image showing Gilsonite evenly distributed in cement and at the cement-to-shale interface.

Gilsonite was next formulated with additional water to achieve mixable and pumpable slurry for testing. The industrial accepted water requirement for Gilsonite was applied, where 0.04 gallon water was added with each pound of Gilsonite (Nelson and Guillot, 2006). As shown in Figure 4-6 (a-b), both shear bond strength and cement compressive strength values reduced upon addition of the extra water. Compared to strengths at 2%, the reduction in shear bond strength and compressive strength of Gilsonite modified cement was exacerbated at 5% due to the increased water to cement ratio. A further reduction is anticipated at higher dosages but more tests needs to be performed to confirm this trend.

Overall, within the limited range of dosages covered in the present test matrix, Gilsonite did not improve the shear bond strength between cement and shale. On the contrary, the increased amount of Gilsonite leaded to a decrease in bonding strength. The additional water added for rheological purpose further worsens the results. Additional

work is ongoing to look at the influences of Gilsonite at higher dosages (with additional water) and at elevated temperatures.
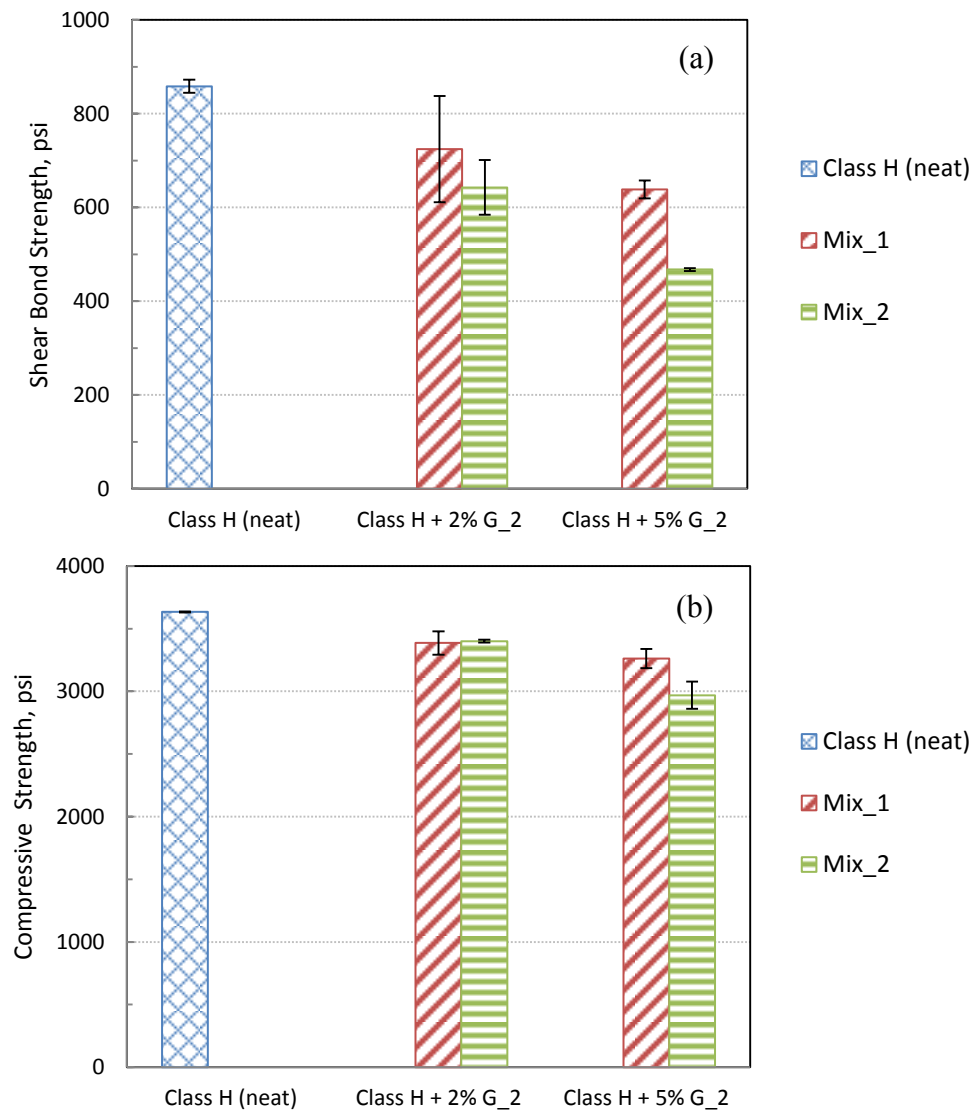
Figure 4-6: 72 hours (a) cement-to-shale shear bond strength and (b) compressive strength for Gilsonite modified cement with or without additional water at 70°F. Mix_1 represents slurries mixed without additional water. Mix_2 represents slurries mixed with additional 0.04 gallon water per pound of Gilsonite

4.2.2   Surfactants

As discussed in Chapter 3, surfactant has been shown to improve interfacial sealing between cement and casing. In this section, same types of surfactant were further tested on their ability to improve cement bonding properties against shale.

Figure 4-7 (a-b) shows the influences of surfactant on cement-to-shale shear bond strength and cement slurry compressive strength. It can be seen that with the use of both coco amidopropyl betaine (B) and polyethylene glycol undecyl ether (A) the shear bond strength increased up to 50% when compared to the shear bond strength of neat class H at 70° F. Figure 4-8 shows the ratio of shear bond strength relative to compressive strength, which indicates that the increase in shear bond strength is no purely because of the higher compressive strength of the cement. This improvement in shear bond strength can partially be attributed to a better spreading of cement on the rock surface. The surface of Colorado Oil Shale is naturally oil-wet. The surfactant might emulsify the oil present on the core leaving a less oil-wet surface, which results in the formation of a better bond between cement and rock. Another benefit from surfactant addition is less volumetric shrinkage of cement slurries. Powers (1935) stated that in cement as hydration products grow, water is consumed and is absorbed onto solid phases. The recession of water from pore surfaces exerts capillary tension inside the pore structures and consequently internal and external volumetric shrinkage happens during cement setting and hardening. If this is true, the reduced solution capillary tension due to the presence of surfactant prevents the tendency for shrinkage. This effect combined with a better spreading of cement on rock surface promotes the cement sealing property. Additional studies are ongoing to understand interaction between the function groups and shale.
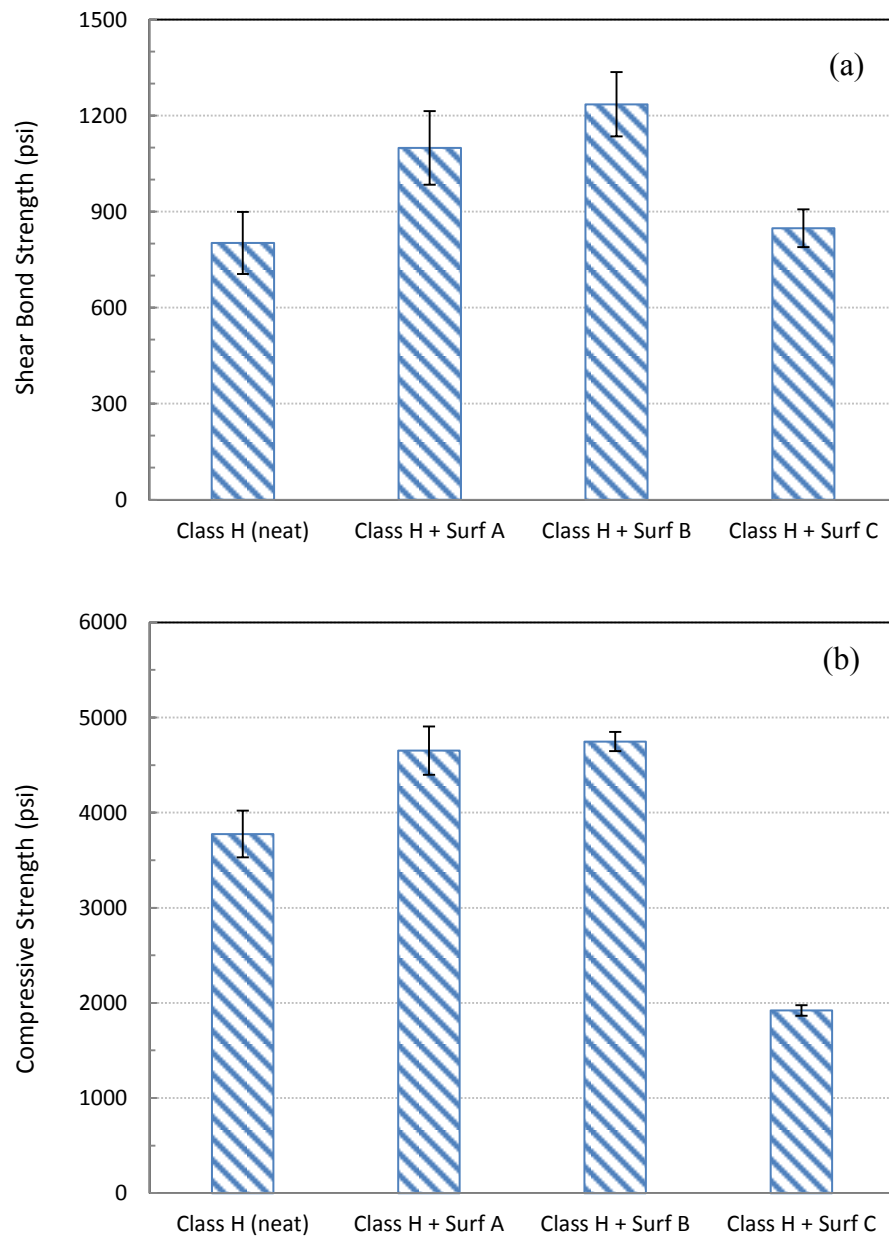
Figure 4-7: 72 hours (a) cement-to-shale shear bond strength and (b) compressive strength for three types of surfactant modified cement slurries at 70°.
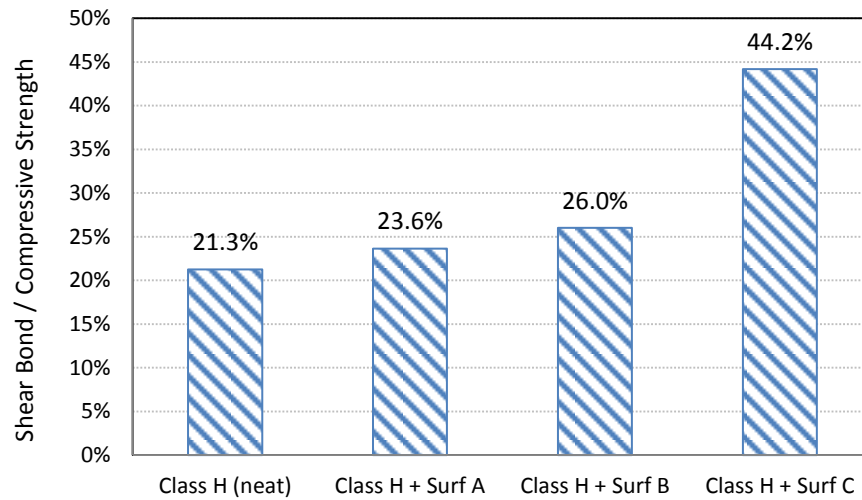
Figure 4-8: 72 hours cement-to-shale shear bond strength relative to compressive strength for different types of surfactant modified cement slurries

Cowan and Eoff (1993) showed a decrease in compressive strength of cement when incorporating the same surfactants at temperatures up to 200° F. In the current study, however, an increase in compressive strength was observed by the addition of coco amidopropyl betaine and polyethylene glycol undecyl ether. This difference in trend could be because in the current study, the samples were cured in a dessicator at 70% humidity whereas in Cowan and Eoff's work (1993), the samples were completely submerged in water during curing. So the current tests were repeated in a water bath and as shown in Figure 4-9, the compressive strength data decreased in comparison to those cured in the desiccator. The cause of this phenomenon is not fully understood yet. One hypothesis to explain this observation is that the cement slurries with surfactant forms more porous hydration products due to the low surface tension, consequently allowing the water to escape when the curing environment is not fully saturated. This leads to a reduction in the water-to-cement ratio, which in turn yields a higher compressive strength for samples cured in desiccator.
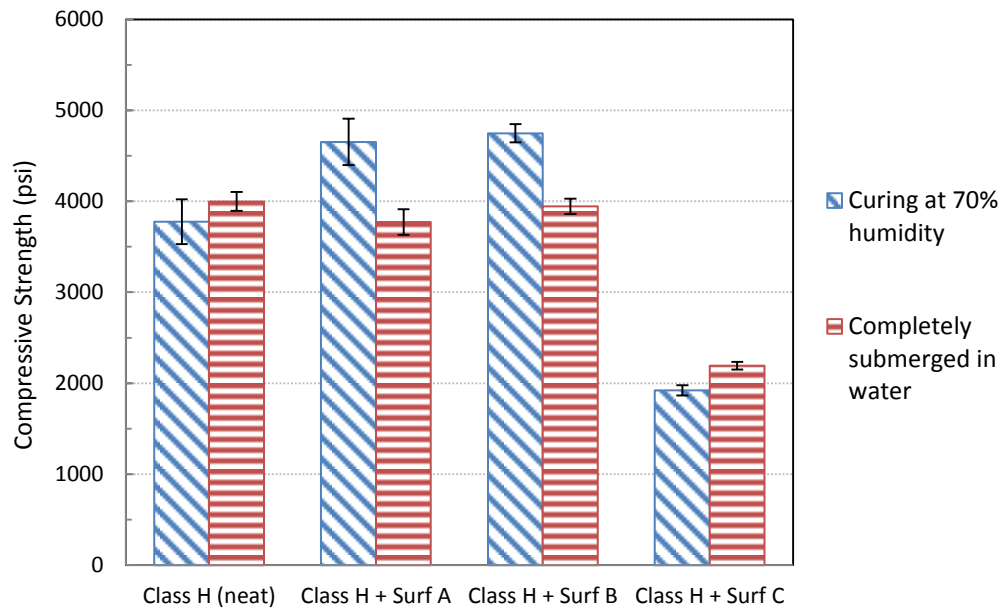
Figure 4-9: 72 hours compressive strength for surfactant modified cements cured under two conditions

Overall, by incorporating the surfactants studied in this work, bonding strength of cement was improved by up to 50% at room condition. As described above, this could be a result of reduced volumetric shrinkage in cement, or because of the effect of surfactant on the oil-wet shale surface or a combination of both. Further research is ongoing to find out the influences of surfactant on cement hydration as well as on tensile bonding characteristics. Four types of surfactants including cationic, anionic, nonionic and amphoteric surfactants are under investigation. The key deliverable of this project is to provide a surfactant package that significantly improves cement bonding capability to ensure an effective zonal isolation.

CHAPTER 5 : CONCLUSIONS AND FUTURE WORKS

5.1 CONCLUSIONS

The main goal of the present work was to develop new and improved experimental methods to evaluate cement bonding properties against formation rocks. In this work, a new shear bond strength test method is proposed based on the intrinsic rock properties, and a tensile bond strength test is designed based on the Brazilian splitting test.

The new shear bond strength testing method shows great potential as an alternative method for quantitative evaluation of cement bonding performance. It was successfully applied on brittle rocks, more specifically shales, which were never achieved utilizing the old push-out testing techniques. Based on the tests conducted so far, all failures occurred at the cement-to-shale interface. The overall repeatability of the newly developed method was good. More than 70% of the test results had a standard deviation within 15%. Recommendations were included in this report about sample preparation procedures and set up assembling.

Utilizing the shear bond strength testing method, two types of additives were studied individually of their potential as bond strength enhancer at atmosphere condition, and some preliminary results were presented. Firstly, three different grades of Gilsonite were evaluated at up to 10% bwoc dosages. No improvement on shear bond strength was observed on all Gilsonite type and concentration combinations. While formulating Gilsonite, the addition of water into slurries for achieving a pumpable rheology further worsened bond strength. Secondly, surfactants of three categories were evaluated at 1% by weight of mix water. Two surfactants showed great performance in improving shear bond strength. They are coco amidopropyl betaine and polyethylene glycol undecyl ether, and the former increased shear bond strength by 50%.

5.2   FUTURE WORK

Chapter 3 detailed the newly proposed shear bond testing method. Currently, the experimental set up has only been used in room condition or been submerged in water bath for high temperature tests (up to 150° F). Modifications to the sample mold and sample preparation procedures are required to allow experiments at elevated pressure and elevated temperature.

The tensile bond strength test method has been successfully applied and tested on a few cement-shale composite cores. Additional tests need to be conducted to (1) prove the concept; (2) justify the test reproducibility; and (3) finalize the sample preparation method.

With the scope of the present report, Gilsonite did not show any positive influences on cement bonding performance. However, as discussed in Chapter 3, Gilsonite is more effective in stabilizing shales at temperatures that are closer to its softening / melting point. Therefore additional tests will be performed at higher temperatures to further explore the Gilsonite's effect on cement bonding properties.

The current study has covered three types of surfactant, which include nonionic, anionic and amphoteric surfactants. Ongoing research is testing one more cationic surfactant to complete a comparison among four categories of surfactants. Surfactants are generally more effective at elevated temperatures (exceptions exists where surfactant becomes unstable at high temperature), thus the effects of temperature will be investigated. In the meanwhile, screening tests will be continued to find the optimum surfactant package. Material characterization methods including SEM imaging and Computerized Tomography (CT scan) will be utilized to visually understand the impact of each of the additives on the cement bonding properties.

Appendix A: Sample Preparation for Shear Bond Strength Tests

In chapter 3 a new method to measure the interfacial shear bond strength between cement and shale was introduced. Using this new method, the biggest challenge in producing reproducible results involves high degree of precision during coring the rock samples as well as during the preparation of the composite cement/rock specimen. In this appendix, some recommended best practices on core handling and sample preparation developed in collaboration with experienced personnel and industrial laboratories are provided.

Plugging core samples, especially from shale outcrop, can be difficult due to the innate fragility of shale. High precision coring and trimming equipment is essential for successfully obtaining an intact sample with precise dimensions. The best method to core shale is to use a vertical knee mill along with a core sampling bit and lubricant swivel attached to the mill head. The mill should also be connected to a lubricant circulation and sedimentation system for cooling the bit and for debris removal.

Once a core (1.5" diameter in this study) is plugged, it is important to ensure that the core sample is long enough for a shear bond strength test. As shown in Figure A-1 (a) each core is cut at a 60-degree angle, which means that the cores have to be at least 4" in length to obtain two good rock samples for shear bond strength test. Obtaining shale cores that are at least 4" long for this study requires extensive practice. The amount of force that is applied during coring varies by the strength of different layers. Care should be taken to plug the cores manually (and not through automatic feed) to avoid failure along the rock laminations. While cutting the 60 degree sample, it is essential to cut the sample slowly to avoid chipping off the edge of the sample.
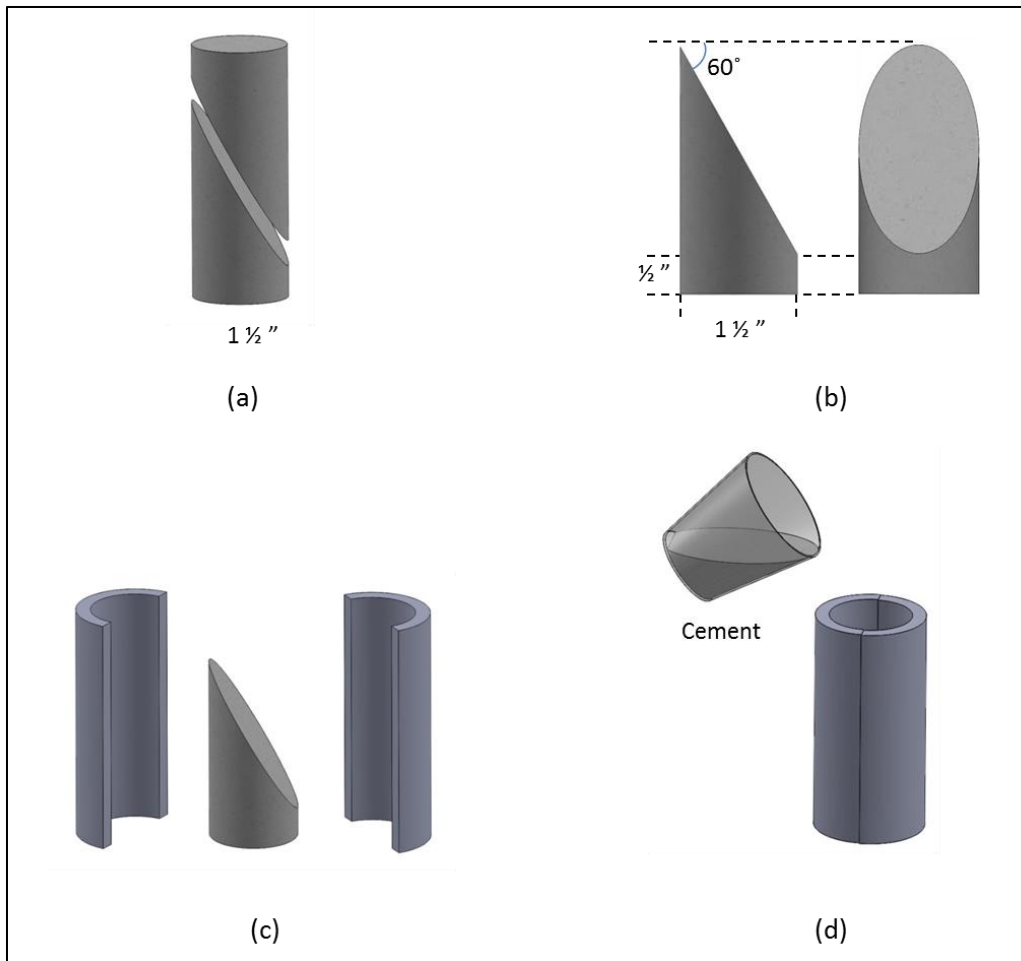
Figure A-1: Sample preparation procedure. (a) Plug a core sample of 1.5 inch diameter and 4 inches long. Cut diagonally at 60˚. (b) Dimensions of the final core sample. (c) Place the core inside the stainless steel clamshell brackets along with a plastic sheet if required for a tight fit. (d) Secure the mold with Velcro straps and then pour the cement slurry.

Another factor that affects the cement bonding property is the surface roughness of the rock. The surface of the 60-degree rock sample should neither be too rough nor too smooth, and the surface pore matrix must be kept as natural as possible to best simulate downhole cementing conditions against rock formations. A repeatable surface roughness

is essential to obtain comparable bonding values. As shown in Figure 3-3, the bond strength values vary with varying degrees of surface roughness. As discussed in Chapter 3, an industrial tile saw was used to perform this task, after which all samples were checked with a surface roughness tester. Only samples with a surface roughness less than 2 μm were used in this study.

Given below are step-by-step instructions for preparation of samples for shear bond strength test:

1. Plug a 1.5-inch diameter core sample that is at least 4 inches in length from an outcrop block. Trim the ends to obtain flat surfaces.

2. Cut the core sample at a 60 degree angle from radius direction (Figure A-1 (a) and (b)).

3. Measure surface roughness of the 60-degree-angled core surface to ensure that all cores have a surface roughness value of less than 2 μm. Roughness of the core-bonding surface should be measured at minimum 4 locations along two perpendicular directions. If surface roughness is found to be slightly higher than 2 μm, an emory cloth can be used to finish the surface. But care should be taken to avoid fine polishing of the surface to prevent a smooth finish.

4. The core should be placed in a high humidity environment to saturate the pores for at least 48 hours prior to testing.

5. Two stainless steel clamshell brackets are joined together with velcro to form a cylindrical mold with inner diameter of 1.5 inch and length of 4 inches (see Figure A-1 (c)). This bracket will form the mold that holds both the core sample and the cement slurry for curing prior to performing the shear bond stress test.

6.   Place one 60 degree core plug into the clamshell mold. Generally, the core drilled with a 1.5 inch coring bit has a diameter slightly smaller than 1.5 inch. In these cases, a thin film of plastic sheet can be inserted between the brackets and the core to allow the core to fit snugly in the clamshell. This prevents the cement from leaking into the micro-annulus between clamshell and the core, and secures the core from slippage during the curing process. This plastic sheet also ensures that the cement slurry does not stick to the steel clamshells and will help with the de-molding process.

7.   Pour cement slurry on top of the mold (Figure A-1 (d)). Tap the mold repeatedly to remove gas bubbles as much as possible.

8.   At room temperature, the samples should be cured either in a dessicator maintained at a high humidity or submerged in water to prevent loss of water from the cement slurry. For samples cured at elevated temperatures, the samples can be placed in a curing chamber or water bath filled with water.

9.   When performing a test at high temperatures, slowly cool down the curing chamber prior to removing samples to avoid thermal shocking.

10.   Care should be taken to ensure that the sample is removed from the curing assembly and returned to ambient conditions prior to testing.

11.   Remove the composite samples from the clamshell mold and trim the cement side of the sample down so that the final sample length to diameter ratio is in the order of 2-2.5.

12.   Test the shear bond strength in a 10,000 pound-force load frame, cushioned by retaining cups on top and bottom to ensure a level surface contact. The specimens should to be tested in the same direction as they were cured (cement on top and

core on the bottom). The loading rate must be constant for all tests. (0.03 inch/min for this study). Record peak load and calculate shear bond strength.

Appendix B: Sample Preparation for Tensile Bond Strength Tests

This section summarizes the sample preparation procedure for tensile bond strength test. Figure 3-4 shows an example of tensile bond test sample composed of half rock and half cement. The most critical factor in the sample preparation procedure is to ensure that the cement-to-rock interface overlaps with the composite core diameter so that the vertical load will be transferred along the interface without any deviation. Figure B-1 (a) is an example of a perfect sample and Figure B-1 (b-c) show examples of not so perfect scenarios that should be avoided. To achieve this, it is crucial to cut a core to a perfect half cylinder. Throughout this study, all samples are 1.5 inch diameter. The cutting and curing procedure is summarized below.

1. The 1.5" diameter by 1" long core is cut longitudinally into two halves (one half is shown in Figure B-2 (a)). Before cutting with a saw, the core needs to be moved towards one side by half of the blade thickness, or kerf, so that one half of the cut sample has a thickness of exactly 0.75". This will ensure that the scenario shown in Figure B-1(c) is avoided.

2. The half core sample with 0.75" thickness is placed upright in a plastic 1.5 inch inside diameter mold. A 1.5 inch diameter number 400 mesh screen is placed on one end of the core along with a 1.5 inch plastic disc with holes (Figure B-2 (a)). The mesh screen was used to allow moisture supply to the cement and prevent loss of water from the cement to the environment.

3. Pour cement slurry to fill the other half of the mold (Figure B-2 (b)). Place another #400 mesh screen along with a plastic disc on top and squeeze the sample gently by hand to allow any excess gas to escape from the mold.

4. Place the sample horizontally with the core at the bottom in a humidity and temperature-controlled environment (Figure B-2 (c)).

5. After three days, remove the sample from the mold and placed in a 1,000 lb-f load frame with the interface perpendicular to the loading platform (as shown in Figure 3-4). The sample needs to be placed between two pieces of semi-circular stainless steel plates to ensure that the sample was loaded along the bond. Load the sample at a constant loading rate (0.003 in/min) until de-bonding is observed.
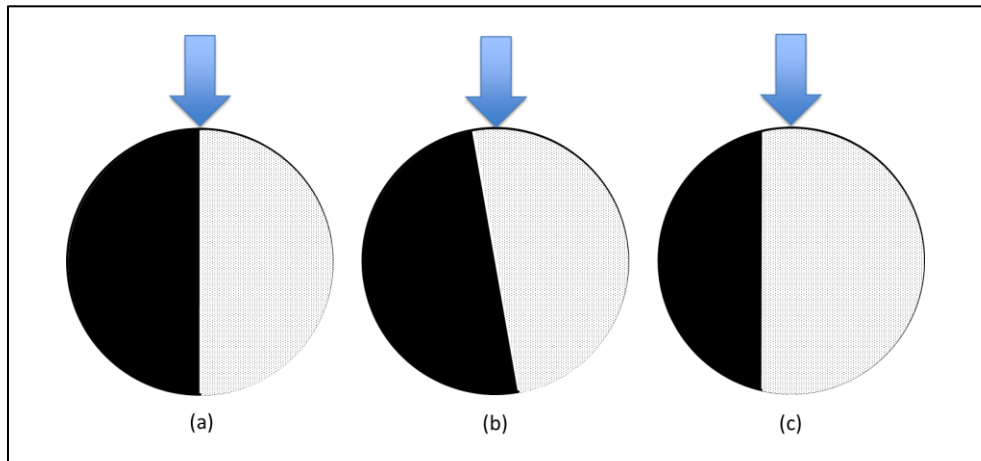
Figure B-1: Importance of having a perfect half cement and half rock sample for tensile strength test. (a) The interface overlaps with sample diameter thus the compressive load acts along the interface. (b) The sample is not placed properly such that the vertical load is transformed to a compressive stress and a shear stress on the interface instead of tensile stress. (c) A sample that is not composed of two half cylinders. The vertical load completely acts on one side of the sample.

Figure B-2: Sample preparation procedure for splitting tensile bond strength measurement. (a) Glue the rock to the plastic cylindrical tube with a #400 mesh screen on the bottom. (b) Pour cement to fill the other half of the cylinder mold and place 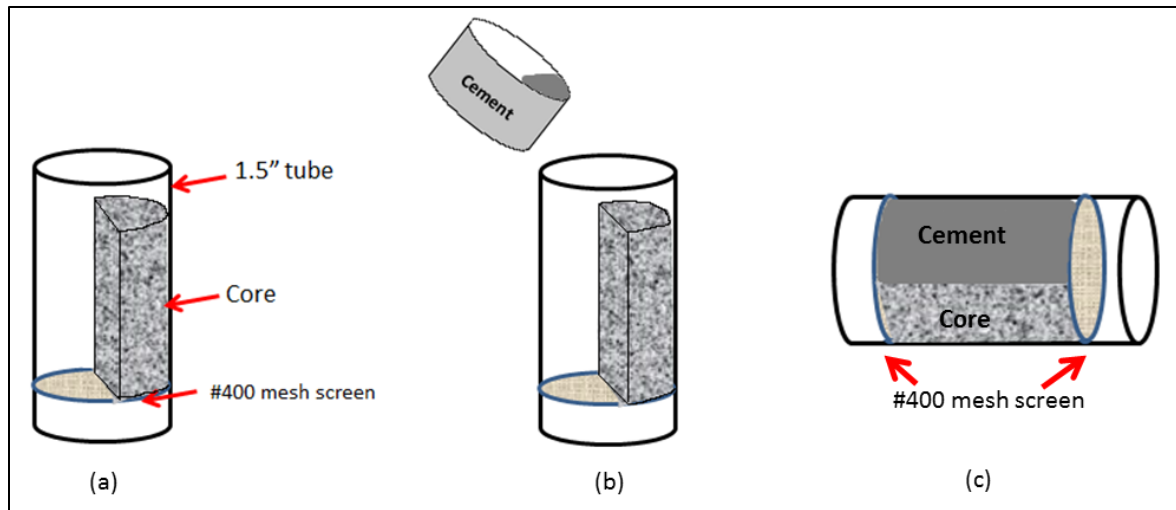another #400 mesh screen on top. (c) Place the mold horizontally in a humidity controlled environment at constant temperature.

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