Effects of Nanoclay and Silica Flour on the Mechanical Properties of Class G Cement
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ABSTRACT: The mechanical properties of oil well cement slurry are usually measured to evaluate the durability, sustainability, and long-lasting behavior of a cement sheath under wellbore conditions. High-pressure and high-temperature (HPHT) conditions affect the mechanical properties of cement slurry such as its strength, elasticity, and curing time. In this study, an organically modified montmorillonite nanoclay (NC) and silica flour (SF) materials are used to enhance the strength of the class G cement. Four different cement slurries with the addition of different concentrations of NC (1% and 2%) and SF (20%) in a class G cement were tested under temperatures ranging between 70 and 100 °C and pressure ranging between 1000 and 3000 psia. The slurries were prepared by maintaining a water to cement ratio of 0.44. All the slurries were cured for 24 h before any test was conducted. Extensive laboratory experiments were carried out to measure the compressive and tensile strength of cement slurries cured at HPHT conditions. Compressive strength was measured using unconfined compressive strength (UCS) tests, scratch tests, and ultrasonic cement analyzer (UCA). Tensile strength was measured using breakdown pressure tests and Brazilian disc test analysis. Scanning electron microscopy (SEM), X-ray diffraction (XRD), and petrophysical analysis were also carried out to evaluate the performance of new cement additives at HPHT conditions. Results showed that the addition of organically modified NC and SF significantly increased the compressive and tensile strength of the class G cement slurry cured at HPHT conditions.

1. INTRODUCTION
The primary objective of oil well cementing is to deliver zonal isolation.1,2 Oil well cement is prepared by adding water and several additives to a cement slurry. Cement additives are used to improve the strength and curing time.3-5 A cement slurry is pumped into the annulus between the casing and formation to prevent interzonal migration of fluid inside the formation and to provide durable zonal isolation.9,10 Ensuring a strong bond between the casing and the formation is a key for any successful cementing operation.11,12 This bond can be affected by stress alteration and cement contraction due to variation of down hole temperature and pressure.13 To maintain the zonal isolation, different additives in a cement are mixed and pumped down the annulus. Cementitious materials that maintain a robust sheath throughout the operating life of the well are preferred.13 The compressive strength of cement is pivotal in determining the integrity of cement and its ability to withstand imposed stresses.

Casing cement leaks due to gas migration are caused by various factors that can take place at any time during the life of a well. The problem of gas migration can be classified into two categories.14 Category 1 is more related to slurry preparation, displacement, and hydrostatic pressure. It usually happens in early time of cementing due to reduction in the hydrostatic head of cement after it was pumped into the annulus.15 In category 2, gas migration or leakage is not just limited to cement placement; rather it is more in mechanical and thermal stresses, which compromise the cement integrity after weeks and months.16,17 Due to mechanical failure of cement sheaths, the bonds between casing and cement and formation and cement weaken, and gas or liquid get a way out. There are various physical aspects of cement that play critical roles in avoiding category 2 migration, such as cement characteristics, balanced hydrostatic head, high tensile strength, and flexibility. Usually cement does not make a bond with salt, oil sand, and shale. Bond strength (i.e., the tensile resistance of the cement–rock interface) is quite small as compared to encountered pressure, given that cycling pressure can easily debond the rock and cement. The best way to study the debonding is by a fracturing process rather than a conventional tensile pull-apart

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procedure. Due to the above-mentioned reasons, the determination of tensile strength of cement in terms of breakthrough pressure is extremely important to evaluate the oil well cement stability and integrity under the wellbore conditions. Breakdown pressure is the pressure at which a sample fails under tension. It is a direct function of tensile strength, pore pressure, and the in situ stress acting on the rock surfaces. The expression used to determine the breakdown pressure for an impermeable material is given by eq 1

\[ P_{bu} = \frac{3\sigma_h - \sigma_t + T_o - P_o}{2(1 - \gamma)} \]

where \( P_{bu} \) is the upper limit of the rock breakdown pressure, \( P_o \) is the pore pressure of the rock, \( T_o \) is the tensile strength, and \( \sigma_h \) and \( \sigma_t \) are the maximum and minimum horizontal stresses acting on the subjected rock. To include the effect of poroelasticity, Haimson and Fairhurst proposed a modified expression to determine the breakdown pressure, given by eq 2.

\[ P_{bh} = \frac{3\sigma_h - \sigma_t + T_o - 2\nu P_o}{2(1 - \gamma)} \]

where \( P_{bh} \) is lower limit of the rock breakdown pressure and \( \gamma \) is given by following expression

\[ \gamma = \frac{\alpha(1 - 2\nu)}{2(1 - \nu)} \]

where \( \nu \) is the Poisson’s ratio and \( \alpha \) is the Biot’s coefficient.

Nanomaterials are used in the formulation of cement to increase its strength and durability. Nanomaterials have a wide range of applications in different areas of petroleum engineering, such as exploration, drilling, and production. They have been used in other science fields such as biomedicines, electronics, polymer science, and catalysis. The application of nanotechnology in the field of oil well cementing can provide solutions to many problems related to integrity and durability of cement slurries. They can be very useful in controlling the hydration reactions of cement slurries due to their higher surface area. These materials can enhance the compressive strength and integrity of the cement sheath, as well as control and reduce fluid losses from the cement slurries. There are some applications reported in cement and concrete. For instance, Campillo et al. reported the enhancement of mechanical properties of belite cement by the addition of nanoalumina. Li et al. investigated the applications of nano-silicon dioxide (nano-SiO2) and nano-iron dioxide (nano-Fe3O4) in cement mortar used for construction material. They have found significant improvement in flexural and compressive strength due to the addition of nanomaterials. Patil and Deshpande and Senff et al. reported that nanomaterials such as nanosilica and nanoalumina reduced cement retrogression at high temperature. They also found substantial improvement in cement strength due to the addition of nanomaterials. Baig et al. and Rahman et al. used nanozeolite and carbon nanotubes in oil well cementing under HPHT conditions. They witnessed that the incorporation of nanomaterials leads to high compressive strength, less fluid loss, and quick thickening time. Hakamy et al. used calcined nanoclay (CNC) in an ordinary Portland cement (OPC) and found that the compressive, thermal, and flexural strength of the admixed cement can be improved significantly. Mahmoud et al. proved that class G cement with the addition of NC can withstand the HPHT conditions during cyclic steam stimulation. In another study, Mahmoud and Elkatatny found that the carbonation resistance of class G with the addition of NC can also be enhanced. McElroy et al. investigated the use of alumina nanofibers (ANFs) in class H cement. They have found significant improvement in the mechanical properties of class H cement by the addition of ANF.

In this study, organically modified montmorillonite nanoclay (NC) and silica flour (SF) materials are used to enhance the compressive and tensile strength of the cement slurry used for oil and gas wells operating at HPHT conditions. Organically modified NCs are a combination of organic and inorganic minerals. In the light of vast literature survey and to the best of the authors’ knowledge, little or no work has been done in the past to evaluate the breakdown pressure of the oil and gas well cements. Multistage fracturing in tight and unconventional shales is done through perforated casing, and the cement is located behind these casing strings. It is paramount to know that whether the cement behind the casing can withstand the pressure during the fracturing operations. Therefore, the breakdown pressure of the cement is indeed necessary to evaluate. The enhancement in the cement strength will maintain the wellbore integrity and protect the casing of the well in the stimulation operation of unconventional and tight reservoirs. Accurate knowledge of the breakdown pressure of the cement slurry is also very imperative to avoid gas migration or fluid leaks behind the casings. The second major objective of this work is to properly utilize the scratch test for compressive strength measurement of oil well cement slurries. The scratch test is a quick, accurate, and nondestructive technique to evaluate the material strength. For the sake of completeness, the results from all notable compressive and tensile strength measurement techniques are presented in this study. Compressive strength techniques used are UCS, UCA, and scratch test analysis. Tensile strength techniques used are breakdown pressure measurement and Brazilian disc test analysis. In addition to rock mechanical tests, petrophysical measurements such as permeability and porosity and microstructural analysis such as scanning electron microscope (SEM) on all slurries are also presented.

2. RESULTS AND DISCUSSION

2.1. Compressive Strength Tests. 2.1.1. Uniaxial Compressive Strength (UCS). UCS test results are reported in Figure 1. The change in temperature conditions caused the change in compressive strength. Cement mixes were subjected to two different temperature conditions. Upon increasing the temperature from 70 to 100 °C, an increase in compressive strength was observed. Due to temperature rise, class G cement strength increased 7.7%, that of class G + 1% NC BWOC increased 12.5%, that of class G + 2% NC BWOC increased 6.9%, and that of class G + 20% SF BWOC increased 20%. This is because SF provides more sand and results in strong structure by forming calcium silicate hydrate in higher percentages.

2.1.2. Ultrasonic Cement Analyzer Test. NDT was conducted on different cement slurries at two different temperatures, 70 and 100 °C. These tests were conducted on slurries for a period of 24 h using the ultrasonic cement analyzer (UCA). The evolution of compressive strength with time over a period of 24 h is shown in Figure 2.
class G cement having a value of 3015 psia. This strength was about 18% lower than the value obtained from the UCS. The addition of 2% NC increased the compressive strength to 3015 psia, the same as that of class G cement mix. The class G mix was subjected to different temperature conditions of 37, 70, 100, and 140 °C and 3000 psia pressure for a period of 24 h, and evolution of compressive strength was evaluated and compared as shown in Figure 3. It was noticed that compressive strength was impacted by both temperature and pressure conditions. Upon increase of the temperature, an increase in compressive strength was observed. At 140 °C, the development of strength was quick and reached a plateau in a short period of time. The final value of compressive strength was lower than slurries cured at 70 and 100 °C temperatures after 24 h. This data shows that class G cement alone is not feasible to pump alone in a high temperature well. The temperature impact on class G cement was evaluated and compared with the scratch test.

Figure 4 explained the effect of pressure on compressive strength. Two different slurries were exposed to same temperature but different pressure conditions, 1000 and 3000 psia, for 24 h. The slurry at high pressure started developing compressive strength earlier than the slurry subjected to 1000 psia pressure. Further, the development of compressive strength stayed higher for high pressure. At high pressure, the cement slurry compressed, and the cement particles became closer to each other, resulting in lower permeability and porosity as well. Therefore, the development rate was higher in this case. The similar case of pressure change impact was investigated in scratch testing. Figure 5 summarizes the UCA results obtained in all cases.

2.1.3. Scratch Test. The scratch test was performed on four slurry mixes for two different types of samples cured at 70 and 100 °C. For each test, a groove with 10 mm width was created.
along the whole length of the core, which generated a continuous profile of scratch strength over the entire length of the core sample.

Figure 6 shows the scratch strength along the length of the core for the four cement slurries cured at 70 °C and 1000 psia.

The four cement slurry mixes cured at 100 °C temperature and 3000 psia pressure were also tested using the scratch test. Figure 8 shows the continuous compressive strength profile of four cement slurries. Scratch test on class G cement sample resulted in a smooth cut along the length of the core sample. Class G cement + 1% NC BWOC cured at 100 °C showed a similar scratch profile to that of class G cement. Class G cement + 2% NC BWOC and class G cement + 20% SF BWOC cured at 100 °C showed higher compressive strength as compared to the class G cement and class G cement + 1% NC BWOC.

Adding SF to cement slurries make them hard, requiring more force to generate a groove on the surface of the core specimen. This causes a brittle failure, resulting in large size particles upon scratching as shown in Figure 9.

Figure 10 shows the scratch strength results for all cement slurry mixes cured under 70 °C temperature and two different pressure 1000 psia and 3000 psia. The effect of curing pressure on scratch strength was not significantly observed except for 2% NC slurry, which showed 27.396% increment.

Figure 11 compares the average scratch strength obtained for four different cement slurries at two different temperatures of 70 and 100 °C at constant pressure of 1000 psia. At 100 °C, the average scratch strength for class G cement increased by 33.83% as compared to the mix cured at 70 °C. The effect of incorporating NC at 1% and 2% BWOC at higher temperature is also evident. Addition of 1% NC results in slightly lower compressive strength compared to the class G cement. This was also observed in the tests conducted by UCA at 70 °C.
The scratch test also captures this effect. But increasing the temperature increased the scratch strength by 75.59% in 1% NC slurry. Similarly increased in strength was found in 2% NC cement slurry with the temperature. The addition of 20% SF enhances the compressive strength by only 6.34% as compared to the strength at 70 °C.

Further, the effect of depth of cut on compressive strength was studied. For this purpose, only class G cement sample prepared at 70 °C and 3000 psia conditions was taken. Three different cuts of 0.5, 0.6, and 0.7 mm depth were tested with same cutter width along the length of the whole core. Mean scratch strength values are shown in Figure 12. It can be observed that the increase in depth of cut did not result in change of compressive strength. The variation of compressive strength for all cuts was very tiny. It showed uniformity and a good mix of cement slurry. It means the cuts were in the same range, and the variation in strength was negligible for those depths of cuts.

2.2. Tensile Strength Tests. Tensile strength of the different cement samples was measured by breakdown pressure test and Brazilian disc tests.

2.2.1. Breakdown Pressure. Breakdown pressure tests were carried out at an ambient temperature of 27 °C. Deionized water was used as fracturing fluid. There was no confining pressure applied on the cement sample. The fracturing fluid was continuously injected at a flow rate of 5 cm³/min through a central bore hole, as a result of which a gradual increase of injection pressure was observed. Injection pressures were recorded until a specimen breakdown happened. Figures 13 and 14 show the breakdown pressure curves for four cement slurries, class G cement, class G + 1% NC BWOC, class G + 2% NC BWOC, and class G + 20% SF BWOC prepared at two different curing pressures of 1000 psia and 3000 psia at constant temperature of 70 °C.
2% NC BWOC, and class G + 20% SF BWOC, prepared at temperature 70 °C and pressure 1000 psia. Addition of 2% NC and 20% SF increases the tensile strength of the cement sample. Further, enhancement in the strength was observed when the curing temperature increased from 70 to 100 °C.

Figure 15 summarizes the breakdown pressure results obtained for four different samples cured at constant pressure of 1000 psia and at two different temperatures of 70 and 100 °C. Similar to compressive strength results, increase in temperature resulted in increase in breakdown pressure. The most significant increment was observed in SF, which is 117.59%. This shows that the SF plays an important role in preventing strength retrogression at higher temperatures.

2.2.2. Brazilian Disc Test. The indirect tensile strength of the prepared cement samples was evaluated using the Brazilian disc test. Figure 16 summarizes the indirect tensile strength results obtained for four different samples cured at constant pressure of 1000 psia and at two different temperatures of 70 and 100 °C. For the case of indirect tensile strength, a similar trend to that for breakdown pressure was observed.

2.3. Petrophysical Analysis. Figure 17 summarizes the permeability and porosity measurements for four different samples cured at constant pressure of 1000 psia and at two different temperatures of 70 and 100 °C. In all cement mixes, the values of permeability and porosity decrease with the increase of temperature. The nonuniform distribution of small particles of NC and SF filled the small pore spaces in the
simple class G cement, which resulted in reduction of porosity and permeability. This decrease in permeability and porosity is also attributed to the increase in the strength, sealing capability, and durability of the cement samples.

2.4. Microstructural Analysis. The microstructural analysis of the prepared cement slurries was performed by carrying out scanning electron microscopy (SEM). SEM images help in determining the pore structure, composition, and topography. A scanning electron microscope from JEOL (JSM-6610LV) was utilized to identify the structure of prepared cement slurries. Figure 18 shows the SEM images of the four cement slurries. Class G cement upon hydration produced portlandite and calcium silicate hydrate. The structure was dense but with many voids. The number of capillary pores present in the class G cured at 70 °C was very high compared to the cement slurries prepared with 1% NC, 2% NC, and 20% SF. Capillary pores are the empty cavities that are not filled by the cement gel during the hydration process. Water tends to reside in these pores. Upon heating the water present in the pores evaporates and leaves behind the tiny holes. These capillary pores cause high porosity in the cement slurry that ultimately leads to low strength. In cement slurries with NC, highly dense calcium silicate hydrate gel, CSH (II) \([\text{Ca}_2\text{SiO}_4\cdot 3\text{H}_2\text{O}]\) and C2SH2 \([\text{Ca}_2\text{SiO}_4\cdot 2\text{H}_2\text{O}]\), was produced due to high availability of silica, which was crystalline, providing high compressive strength. The small size of NC filled the capillaries and resulted dense structure as compared to class G, so the set cement retained a high compressive strength.

In cement slurry with 20% BWOC SF, the higher availability of silica produced C2S6H5 that had good crystals of needle shape. These needle shaped C2S6H5 products could interlace and knot with each other to build an ideal and well-proportioned network structure in the hardened paste (see Figure 19). This makes the set cement retain a high compressive strength.

2.5. Effect of Nanoclay and Silica Flour on Bulk Density. The NC and SF admixed cement slurries were subjected to density measurements. The densities of four cement slurry systems having 1% and 2% nanoclay contents, 20% SF content, and class G cement slurry were measured in the laboratory (see Table 1).

| slurry type       | density, lb/gal |
|-------------------|-----------------|
| class G           | 15.80           |
| 1% NC BWOC        | 15.7            |
| 2% NC BWOC        | 15.5            |
| 20% SF BWOC       | 16.5            |

3. CONCLUSIONS

In this study, extensive laboratory experiments were carried out to measure the compressive and tensile strengths of oil well cements with the addition of organically modified NC (1% and 2%) or SF (20%) as additives. Based on the results obtained and discussion mentioned in the paper, the following conclusions can be drawn:

1. NC as an additive at 2% BWOC and 20% SF BWOC in class G cement increases the compressive and tensile strength significantly.
2. An increase of about 40−50% in compressive strength and 20−40% in tensile strength was observed when temperature increased from 70 to 100 °C in all mixes except SF, which showed significantly higher strength due to the temperature increment.
3. Samples cured at higher pressure also showed higher compressive and tensile strength values.
4. The compressive strengths for all mixes obtained from macrolevel crushing strength were significantly higher than the ultrasonic measurements of compressive strength evolving with time.
5. Scratch test to determine the strength of the cement was presented and compared with the other conventional techniques in practice including the crushing compressive strength at macroscale and evolution of compressive strength using ultrasonic tests.
6. The scratch test method is the best way of describing the continuous scratch strength profile along the entire scratched length of the sample. The test results showed that the compressive strength of the cement predicted...
from the scratch test is reasonably close to those obtained by UCS.  
7. Breakdown pressure tests are very important to evaluate cement strength against any leak and gas migrations.  
8. From petrophysical analysis, it was observed that the addition of NC and SF reduces the porosity and permeability in the cement samples because of the nonuniform distribution of the particles. The 2% NC and 20% SF BWOC provide very low permeability. Also, permeability and porosity of the samples decreases with the increase of temperature, which is attributed to the increase in cement sample strengths.  
9. The microstructural analysis depicted that addition of NC and SF leads to dense structure with minimum cavities.

4. MATERIALS AND METHODS

4.1. Materials. In this study, all test specimens were prepared using class G cement complying with American Petroleum Institute (API) specifications. The class G cement has a density of 3.15 g/cm³. The composition of class G cement was characterized by XRD and is given in Figure 20. The phase composition of class G cement is listed in Table 2.

The NC material used in this study was organically modified and prepared by modifying the natural montmorillonite with a quaternary ammonium salt. It is composed of the smallest particles, comprising three main constituents, silica, alumina, and water. The montmorillonite is a layered magnesium aluminum silica, which was organically modified by cation exchange reaction by using quaternary ammonium salt to transform it to a hydrophobic nanoclay. The montmorillonite-based nanoclay was modified with methyl, Tallow (65% C18, 30% C16, 5% C14), bis 2-hydroxyethyl quaternary ammonium chloride. Table 3 provides the characteristics of the nanoclay used in this study. NC comprises octahedral sheets of magnesia or alumina sandwiched between two tetrahedral sheets of silica. A high concentration of oxides of silica and alumina existed in the tested NC as shown in Figure 21. Further, morphological characterization of NC was performed using SEM as shown in Figure 22. In the SEM image, the highlighted white surface indicates the presence of organic material in the NC.

![Figure 20. Chemical composition of class G cement.](image)

![Figure 21. Elemental composition of NC.](image)

![Figure 22. Scanning electron microscopy (SEM) of NC.](image)

| Chemical Composition | Concentration (%) | Chemical Composition | Concentration (%) |
|----------------------|-------------------|----------------------|-------------------|
| Tricalcium aluminate  | C₃A               | 3CaO·Al₂O₃           | 21.6              |
| Tricalcium silicate  | C₃S               | 3CaO·SiO₂            | 3.3               |
| Dicalcium silicate   | C₂S               | 2CaO·SiO₂            | 4.9               |
| Tetracalcium aluminoferrite + tricalcium aluminate | C₄AF + 2C₃A | 4CaO·Al₂O₅ + Fe₂O₃ + 3CaO·Al₂O₃ | 1.1 |
|                      |                   |                      | 2.2               |
|                      |                   |                      | 0.6               |
|                      |                   |                      | 0.3               |
|                      |                   |                      | 0.41              |

Table 2. Class G Cement Phase Composition

| Material          | Color       | Density (g/cm³) | d-spacing (nm) | Aspect Ratio | Surface Area (m²/g) | Mean Particle Size (µm) |
|-------------------|-------------|-----------------|----------------|--------------|---------------------|-------------------------|
| Nanoclay          | Off-white   | 1.98            | 1.85           | 200–1000     | 750                 | 6                       |

SF was added into the cement to reduce permeability and enhance compressive strength under HPHT conditions. It is recommended for use in cementing wells where static temperature exceeds 230 °F (110 °C). Above this temperature, most cement compositions lose strength. SF prevents the cement strength retrogression problem by chemically reacting with the cement at high temperature. The formulations were subjected to similar conditions of pressure and temperature for
uniformity. The water to cement ratio (WCR) of 44% BWOC was kept constant in all formulations.

4.2. Particle Size Distribution of Cement Additives. The particle sizes of class G, NC, and SF powders were measured using a HELOS Particle Size Analyzer. This instrument measures the particle sizes using laser diffraction method. Particle size distribution is useful in understanding the physical and chemical properties of the samples. The median diameter or median values of particle size distribution ($D_{50}$) of class G cement, NC, and SF are given in Table 4. The $D_{50}$ value is the value at 50% in the cumulative distribution, which means that 50% of the particles are smaller than this value and 50% of particles are greater than this value. This value is useful in characterizing particle sizes. The $D_{50}$ of cement particle size distribution is 20.17 μm, that of NC is 12.30 μm, and that of SF is 14.94 μm. Figure 23 shows the graphs of particle size distributions for class G cement, NC, and SF.

Table 4. Particle Size Distribution of Cement and NC

| sample          | $D_{10}$ (μm) | $D_{50}$ (μm) | $D_{90}$ (μm) |
|-----------------|--------------|--------------|--------------|
| cement          | 3.28         | 20.17        | 57.33        |
| nanoclay        | 2.43         | 12.30        | 21.88        |
| silica flour    | 1.77         | 14.94        | 45.00        |

Figure 23. Particle size distribution for class G, NC, and SF.

4.3. Sample Preparation. In this study, four different types of cement slurries were synthesized and tested. These cement slurries were class G cement, class G + 1% NC BWOC, class G + 2% NC BWOC, and class G + 20% SF BWOC. All cement slurries were prepared using a high-speed cement blender according to API specifications and cured for 24 h. The dry mixing procedure was used in which additives and cements were uniformly blended before adding water. First, the NC was dry blended with cement. The dry-blended mixture of cement and NC was added to distilled water within 15 s. Then the high-speed mixer was run at a speed of 12000 rpm for 35 s to obtain a homogeneous cement slurry. The cement slurry was conditioned in an atmospheric consistometer for a period of 30 min. All samples were cured for a period of 24 h. WCR was kept constant at 0.44 in all samples. The cement slurry was kept in water before performing the experiments.

4.4. Experimental Program. Figure 24 shows the complete experimental program. All cement slurries for compressive and tensile strength tests were prepared at two different temperatures, 70 and 100 °C, and two different pressures, 1000 psia and 3000 psia. Only for UCA test, cement slurries were prepared at other temperature conditions, such as 37, 70, 100, and 140 °C.

4.5. Methods. 4.5.1. Compressive Strength Test. UCS test was performed according to the methods of American Society for Testing and Materials (ASTM) and International Society for Rock Mechanics (ISRM). UCS tests were performed on cement cubes of different formulations, such as class G cement, class G + 2% NC BWOC, and 20% BWOC SF. All the cement mixes were cured at 70 °C temperature and 3000 psia pressure except class G cement mix, which was prepared at 70 and 100 °C. For the UCS test, the samples were cast in the shape of cubes. The length and width of the cubes were 2 in. After curing, the samples were end faced, to avoid experimental artifacts in compression testing. UCS was calculated using eq 4:

$$\sigma = \frac{F}{A}$$

where $\sigma$ is the UCS, $F$ is the maximum axial force applied on the sample, and $A$ is the cross-sectional area of the sample.

4.5.2. Ultrasonic Cement Analyzer. The UCA method indirectly measures the compressive strength of the sample over time by recording ultrasonic wave velocities. UCA is a nondestructive test (NDT). In this test, sonic waves pass through the cement to determine the evolution of compressive strength. The oil well cement slurry is placed inside the cell, which is loaded in the UCA with simulated down hole conditions applied. A sonic signal is transmitted through the cement sample. As the compressive strength develops with time, the ultrasonic signal passes through the sample at a higher rate, providing a measure of the development of compressive strength with time. Sonic signals can give the compressional (P) wave and shear (S) wave travel time of the sample. From this test, the dynamic Young’s modulus, $E_{\text{dyn}}$, and dynamic Poisson’s ratio, $\nu_{\text{dyn}}$, can also be measured. $E_{\text{dyn}}$ and $\nu_{\text{dyn}}$ can be estimated by eqs 5 and 6.
where \( V_P \) and \( V_S \) are the compressional and shear wave velocities in km/s and \( \rho \) is the bulk density in g/cm³.

4.5.3. Scratch Test. The scratch test is used to provide the continuous compressive strength of the sample over the entire scratched length.\(^5\) Scratch test measurement has certain significant advantages over the conventional UCS test. First, it needs minimum sample preparation, second it is a NDT, and third it has a high repeatability.\(^5,5\) The scratch test proceeded with the estimation of normal force, \( F_N \), and tangential force, \( F_T \), along the axis of the sample. The forces recorded were used to calculate the intrinsic specific energy (\( \varepsilon \)), \( \varepsilon \) is the amount of energy needed to scratch a unit volume of the sample and is linearly correlated with the UCS.\(^5\) Schei et al.\(^5\) carried out scratch test analysis on different sedimentary rocks such as carbonates and sandstones and found a linear correlation between \( \varepsilon \) and UCS.

In this study, a PDC cutter was used to create the groove on the surface of the sample. During the test the cutter velocity, cutter width, and rake angle were kept constant while the depth of cut (DOC) was varied between 0.5 and 0.7 mm. The width of the cutter was 10 mm, cutter velocity was set to 10 mm/s, and rake angle was 15°. Scratch tests were performed on different cement samples cured at different temperature and pressure conditions.

4.5.4. Breakdown Pressure Measurement. The fracture breakdown pressure measurement experimental setup consists of an ISCO pump, a vacuum pump, two accumulators, a core holder, HPHT valves, pressure transducers, and fittings. A core holder can accommodate a 1.5 in. diameter sample with length up to 12 in. The length of the samples used in this study was 2 in. A schematic diagram of the breakdown pressure experimental setup is given in Figure 25.

To apply injection pressure, a bore hole was drilled at the center of the sample. The diameter of the bore hole drilled was 3.5 mm. The depth of the drilled hole was 19 mm. Stainless-steel tubing of outer diameter 3 mm was inserted inside the bore hole to a depth of 13 mm. An open hole section of 6 mm was left for fracturing fluid to initiate the fracture. Stainless-steel tubing was fixed to the cement sample using HPHT epoxy. The tensile strength of the epoxy confirmed by the manufacturer was 4200 psia. Once the epoxy was attached to the sample, the sample was placed inside the fracturing cell. Fracturing fluid was continuously injected, and the injection pressure was logged. The fracturing fluid was injected at the flow rate of 5 cm³/min until the specimen failed.

Figure 26a shows the view of the three cement samples with 3 mm stainless-steel tubing inserted and attached with HPHT epoxy. Figure 26b shows the view of fractured sample. All the experiments were carried out at zero confining pressure conditions.

4.5.5. Brazilian Disc Test. The indirect tensile strength of the prepared cement samples was evaluated using the Brazilian disc test. Cylindrical samples were prepared for the Brazilian disc test. The samples were 1.5 in. diameter and 0.75 in. length. The indirect tensile strength was determined by measuring the maximum load the sample can withstand before failing under tension. Equation 7 is used to compute the tensile strength.

\[
\sigma_t = \frac{2P}{\pi dl}
\]

4.5.6. Petrophysical Properties Measurement. Petrophysical properties of the studied cement samples were evaluated using permeability and porosity. Permeability measures the ability of fluid to flow through porous media, whereas porosity defines the void spaces between the grains where fluid can be stored. These properties can help in determining the long-lasting behavior of a cement sheath. As discussed earlier, the purpose of an oil well cement sheath is to provide complete zonal isolation. Therefore, lower permeability and porosity is required. Low permeability not only reduces gas migration but also reduces communication between different layers and corrosion of the casings. In this study, permeability and porosity were measured using an automated porosimeter/permeameter (AP-608). The equipment is capable of measuring porosity and permeability under a confining pressure. The AP-608 can take samples with diameter of either 1.5 in. or 1.0 in. inside a Hassler type core holder. The
permeability measurements were made using an un-steady-state pulse decay technique.

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Notes
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■ NOMENCLATURE

ANF = alumina nanofiber
API = American Petroleum Institute
ASTM = American Society for Testing and Materials
BWOC = by weight of cement
CT = computer tomography
CNC = calcined nanoclay
E = Young’s Modulus, GPa
Edyn = dynamic Young’s modulus, GPa
Fe = maximum axial force, N
HPHT = high-temperature, high-pressure
ISRM = International Society of Rock Mechanics
NDT = nondestructive testing
NMR = nuclear magnetic resonance
NC = nanoclay
P-wave = compressional wave
PDC = poly diamond crystalline
PFD = process flow diagram
PR = Poisson’s ratio
S-wave = shear wave
SEM = scanning electron microscopy
SF = silica flour
SRV = stimulated reservoir volume
UCA = ultrasonic cement analyzer
UCS = uniaxial compressive strength
Vp = compressional wave velocity, km/s
Vs = shear wave velocity, km/s
XRD = X-ray diffraction

WCR = water to cement ratio

Greek Symbols

α = Biot’s coefficient
ρ = bulk density, g/cm³
υ = static Poisson’s ratio
υdyn = dynamic Poisson’s ratio

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