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Low elastic modulus and expansive well cement system: The application of gypsum microsphere

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HIGHLIGHTS

- We invent the method for preparing gypsum microsphere.
- Gypsum microsphere is used to achieve low elastic modulus and expansive well cement.
- Cement sheath with gypsum microsphere has brilliant resistance to stress and strain.
- Massive crystals formed by gypsum powder decrease interfacial bonding strength.

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ABSTRACT

The research objectives in this paper are designed to use gypsum microsphere to achieve low elastic modulus and expansive well cement system. Results show that gypsum microsphere has the ability to compensate for volume shrinkage of hardened cement. Expansive crystals produced by the reaction of tricalcium aluminate and dihydrate gypsum exist around gypsum microsphere. Though compressive strength of hardened cement is improved by gypsum powder, existence of massive crystals increases brittleness of cement sheath. Compared with gypsum powder, due to porous structure, gypsum microsphere decreases elastic modulus of hardened cement, which shows brilliant resistance to stress and strain.

1. Introduction

Cement slurry is pumped into the annular space between the casing and formation via the inner of casing to form cement sheath [1]. Due to no repeatability, cementing engineering is a crucial step for the whole construction process of well [2]. If there is a problem with cementing quality, it not only needs a large number of costs to repair wells, more likely induces wells scrapped [3]. With the development of offshore, unconventional oil and gas resources and new sources of energy, the importance of cementing engineering is more obvious [4–6]. Portland cement has been used as the primary oil well cement [7]. One of the major requirements for oil well cement is to create a very low permeability barrier for fluids [8]. However, autogenous and drying shrinkage may increase the risk of cracking the cement materials and induce the interface decementation. Moreover, Portland cement is quite brittle and can be broken easily due to very small compressive deformation, which negatively increase the permeability. Therefore, volume shrinkages and intrinsic brittleness of cement are the main reasons of sealing failure [9].

Expansive additives have been widely used to compensate the shrinkage of cement-based materials to avoid cracking. The expansion can be realized by the hydration of expansive agents mixed in cement such as sulfo-aluminate, free calcium oxide and uncombined magnesia [10]; however these additives might increase brittleness of cement [11]. In order to satisfy the requirements of sealing, the flexible and expanding cement materials have been proposed [12–15]. Some lab and field experiments show that gas bubbles could reduce the elastic modulus and brittleness of the cement [16–18]; therefore, swelling gas bubbles are beneficial to compensate volume shrinkage and improve cement flexibility. Cement expands through entrapping swelling gas bubbles artificially by adding aluminum powder during mixing. However, the formation and distribution of gas bubble are very difficult to

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control [9]. Some researchers propose adding rubber and synthetic elastic particles in cement matrix to reduce the elastic modulus [19,20]. However, the application of rubber particles in cement is not so technically successful [21]; furthermore, rubber and synthetic elastic particles have no ability to compensate the shrinkage of cement.

In this paper, gypsum microspheres are prepared and used in cement to achieve low elastic modulus and swelling cement system. The microstructure of gypsum microsphere was observed by SEM. The volume swelling performance of hardened cement was evaluated, and the mechanism was analyzed by combining orthogonal polarization microscopic and SEM. The elastic modulus, compressive strength, and interfacial bonding strength of cement with gypsum powder or gypsum microsphere were tested. Furthermore, considering complicated downhole pressure conditions which might be caused in perforating, pressure testing and stimulation operations, interfacial bonding strength was also tested under alternating pressure condition. Finally, the micro structure of interface was analyzed by SEM.

2. Experimental

2.1. Materials

Dihydrate gypsum (chemically pure), anhydrous gypsum (chemically pure) and α-semi hydrated gypsum (chemically pure) were provided by Sinopharm Chemical Reagent Co., Ltd. Class G oil well cement was used in the experiments. The typical mineral composition and physical properties of class G oil well cement are given in Table 1. Typical chemical composition is given in Table 2. Corn oil used in this study was a commercial product.

Two particle size ranges of gypsum microsphere including 50–100 and 100–200 μm were prepared. Two dosages of dihydrate gypsum powder including 1.5% and 3.5% by weight of cement (BWOC) were used. In order to ensure the volume expansion of hardened cement is basically similar to cement with gypsum powder, two dosages of gypsum microsphere including 25% and 30% by weight of cement (BWOC) were studied. Furthermore, in order to reveal the influence of gypsum microsphere on the cement slurry, two dosages of dihydrate gypsum powder including 25% and 30% by weight of cement (BWOC) were also prepared. Micro silicon was used in mixture to prevent the sedimentation of gypsum microsphere. Nine cement samples in which S1 is a plain slurry were prepared as shown in Table 3. Due to the high dosage of gypsum microsphere, water to solid ratio was used to ensure the flowability of mixture.

2.2. Method

2.2.1. The preparation of gypsum microsphere

Cone oil (200 g) was placed in a 500 ml three-necked flask equipped with a stirrer at the speed of 500 r/min. Anhydrous gypsum (6 g) and α-semi hydrated gypsum (14 g) were mixed in tap water (16 g) to form water phase, and then the mixture was added to flask at room temperature. After stirring for 2 h, gypsum microspheres with a particle size of 100–200 μm were obtained. Gypsum microspheres with a particle size of 50–100 μm were prepared by using 250 g cone oil and a stirrer at the speed of 700 r/min. Gypsum microspheres were dried at 40 °C and atmospheric pressure.

2.2.2. The testing of basic properties of oil well cement

2.2.2.1. Manufacturing process. Cement slurry was mixed based on API Spec. 10B-3-2004. After being prepared, the basic properties were tested as follows:

2.2.2.2. Determination of slurry density. The method for measuring the density of cement slurry is performed by using the fluid density balance. The density was tested at room temperature and atmospheric pressure.

2.2.2.3. Cement slurry stability. The purpose of this test is to determine the static stability of the cement slurry, and to determine if the cement slurry experiences particle sedimentation. The slurry is poured into a sedimentation tube. The sedimentation tube should have an inner diameter of 25 ± 5 mm. The most common tube length is approximately 200 mm. The inside of the tube, and all joints, should be lightly greased to become leak-tight and ensure that the set cement can be removed without damage. After curing the slurry at 75 °C for 24 h, the tube is cooled down to room temperature and the set cement is removed. The cement sample is immersed and kept in water as much as possible to prevent it from drying out. The length of the set cement specimen should be measured. The specimen is marked approximately 20 mm from bottom and top sides of the sample. The middle section, between the marks, is divided into roughly equal pieces with 5 segments. The sections must be kept in the same order. The sections are immersed and kept in water until each is weighed. The volume of every section is obtained by testing the weight of the sample suspended in water and the density of every section is calculated by applying the principle of Archimedes. The stability is evaluated by the percentage of density difference between the top and the bottom segments.

Table 1
Phase composition and physical properties of class G oil well cement.

C_3S (wt%)	C_2S (wt%)	C_3AC (wt%)	C_3AF (wt%)	Specific density (kg/L)	Specific surface area(m^2/kg)
53.7	30.46	2.8	8.0	3.17	332

Table 2
The main chemical composition of class G oil well cement.

SiO_2 (wt%)	Al_2O_3 (wt%)	Fe_2O_3 (wt%)	CaO (wt%)	SO_3 (wt%)	MgO (wt%)	K_2O (wt%)	Loss on ignition (wt%)
22.70	3.39	4.81	65.60	1.21	0.90	0.37	0.49

Table 3
Mix proportions of cement slurry.

Sample [a]	Gypsum		Si/C (%)	D/C (%)	De/C (%)	Water–solid ratio
	Type	G/C (%)				
S1	–	0	8	0.5	0.5	0.5
S2	Dihydrate gypsum powder	1.5	8	0.5	0.5	0.5
S3	Dihydrate gypsum powder	3.5	8	0.5	0.5	0.5
S4	Gypsum microsphere (50–100 μm)	25	8	0.5	0.5	0.5
S5	Gypsum microsphere (50–100 μm)	30	8	0.5	0.5	0.5
S6	Gypsum microsphere (100–200 μm)	25	8	0.5	0.5	0.5
S7	Gypsum microsphere (100–200 μm)	30	8	0.5	0.5	0.5
S8	Dihydrate gypsum powder	25	8	0.5	0.5	0.5
S9	Dihydrate gypsum powder	30	8	0.5	0.5	0.5

[a] C, G, Si, D and De are, respectively, the weight of cement, gypsum, micro silicon, dispersant (sulfonated ketone/aldehyde polycondensates), defoamer.

2.2.3. The testing of porosity

The porosity of gypsum microsphere was measured in the State Key Laboratory of Heavy Oil Research using the specific surface area and pore physical sorption analyzer whose type was ASAP 2020 (USA Micromeritics Company).

2.2.4. The testing of mechanical properties

2.2.4.1. Interfacial bonding strength. Interfacial bonding strength testing device which consists mainly of steel sleeve, cap, and casing, etc. is shown in Fig. 1. The inner pressure of casing can be stressed by piston pump. The cement slurry was placed into the annual space between casing and steel sleeve, and then was cured at 75 °C and atmospheric pressure for 96 h. To consider an alternating pressure condition, the cement slurry was cured at 75 °C and atmospheric pressure for 72 h and subsequently curing temperature was kept constant and the inner pressure of casing changed periodically as follows: 5 MPa (6 h) → 15 MPa (6 h) → 5 MPa (6 h) → 15 MPa (6 h) → atmospheric pressure.

The testing procedures to determine interfacial bonding strength were as follows: Opening the cap, installing lock block and cushion block as shown in Fig. 2; increasing the pressure on the lock block and recording the maximum pressure (P1) when cement and casing sliding occurs. P1, which was divided by the contact area of cement-sheath and casing, was bonding strength.

2.2.4.2. Compressive strength. Cement slurry was placed into compressive-strength molds (50 mm × 50 mm × 50 mm). Considering the temperature circumstance in downhole, the device and molds were put into high temperature curing chamber, and cement slurries were cured at 75 °C and atmospheric pressure, which is chosen to simulate the downhole temperature. After curing, the set cement cubes were