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Construction and Application of High-Strength Flexible Cement Sheath for Deep and Ultra-deep Shale Gas Cementing

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Abstract. With the gradual development of oil and gas exploration, deep and ultra-deep shale gas has become the main block for shale gas reserve growth and production addition in China. Deep and ultra-deep shale gas is characterized by deep burial depth, high pore pressure and formation fracture pressure, so the production and transformation means in the later stage are more complicated. Deep shale gas cementing puts forward higher requirements on cement slurry system, especially the mechanical properties and long-term sealing ability of cement sheath under high temperature and pressure conditions. This paper studies the cement slurry system which can meet the requirements of staged fracturing and long-term sealing. Through the physical simulation device, the mechanical property analysis and evaluation method of cement sheath under staged fracturing condition is developed. By interphase filling technology, a new type of elastic material is developed. Meanwhile, nano-emulsion filling technology is used to realize void filling and reduce porosity of cement stone and micro-ring gap. Finally, a high-strength flexible cement slurry system for deep and ultra-deep shale gas well cementing is constructed, with a temperature resistance of 200℃, elastic modulus of cement stone less than 6GPa and compressive strength ⩾25MPa/48h. It can meet the requirements of 120MPa and 25 cyclic loading staged fracturing. This cement slurry system has been widely applied in shale gas areas, and the durability and sealing ability of cement sheath have been significantly improved, which can meet the needs of large-scale staged fracturing and ensure the efficient development of deep and ultra-deep shale gas wells.

Key words: deep shale gas; high-strength flexible; cement sheath; long-term sealing

1. Introduction

With the development scale of shale gas resources gradually expanding, deep and ultra-deep shale gas resources have gradually become the main production areas for increasing oil and gas reserves in China. The deep shale gas resources are buried deep, and the pore pressure and fracture pressure of the deep shale formation are relatively high. The characteristics of high temperature and high stress put forward higher requirements on the quality of cementing and sealing ability of cement sheath, especially the mechanical properties and long-term sealing ability of cement sheath under high temperature conditions. Therefore, the research on cementing cement system of deep shale gas wells has far-reaching significance for the development of deep shale gas resources [1].

In deep shale gas high temperature, high stress formation characteristics, carried out a staged fracturing and long-term solid slurry sealing system and related technology research, using numerical simulation, in view of the staged fracturing construction was carried out by the cement ring sealing ability evaluation and analysis of the large physical simulation device, set up the deep formation, cement sheath physical simulation test evaluation method. It ensures the interlayer sealing of cement sheath in the stage fracturing process and the hollow zone pressure in the production process, and forms an improvement method to improve the sealing ability of cement sheath [2]. For deep stratum cement mechanical performance requirements, and fill the brittle transformation method and theory of porous medium filling, high flexible design method of cement slurry system is established, using the theory of cement phase crystalline phase and gel plasticizing, designed a new type of core-shell structure materials, improve the elastic material and CSH cementation, raised the temperature resistance, increased the deformation ability of cement stone. The high strength flexible cement slurry system has been formed to meet the requirements of high temperature and high stress formation. The durability and sealing ability of cement stone have been improved to meet the requirements of large-scale staged fracturing and ensure the long-term sealing requirements of deep shale gas Wells.

2. Analysis of mechanical properties of cement stone in horizontal well of deep shale gas

Compared with middle-shallow shale gas wells, the damage and the plastic deformation of cementing rock in deep formation will decrease the sealing ability of cement sheath under high stress state, leading to annular oil and gas flow. The damage analysis of cementing cement in middle-shallow shale gas Wells was carried out under the condition of stage fracturing in deep high-stress formation.

Table 1 uniaxial and triaxial mechanical test parameters of cement stone

Sample	Peak Stress (MPa)	E (GPa)	Poisson's Ratio	Loading Rate (mm/min)	Confining Pressure (MPa)
1-1	51.9	7.5	0.15	0.12	0
1-2	56.8	8.2	0.21	0.12	15

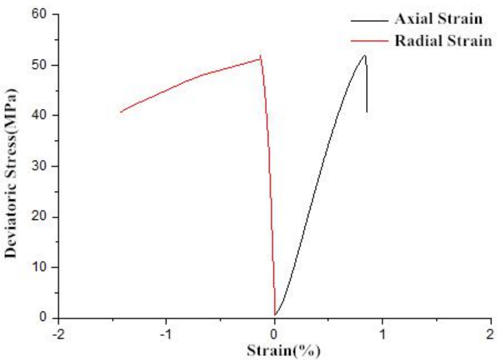

Figure 1. Stress-Strain Curve(No.1-1).

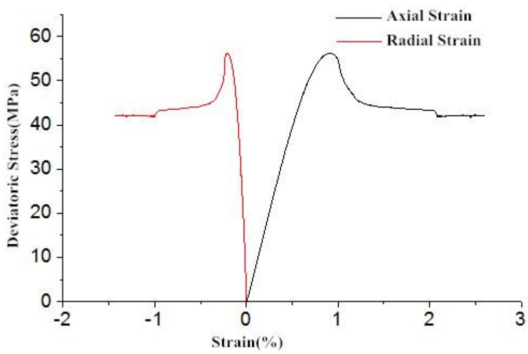

Figure 2. Stress-Strain Curve(No.1-2).

The peak strength, elastic modulus and Poisson's ratio measured by triaxial measurement are all higher than those measured by uniaxial measurement. The confining pressure has a great influence on the basic mechanical parameters, and the triaxial test results are more real and closer to the field. Under triaxial condition, the cement stone has obvious post-peak stage and higher residual strength. It is in sharp contrast with the brittleness of uniaxial test.

At present, the cement slurry system used in the cementing of middle-shallow shale gas is ductile cement slurry, the basic formula is: Cement(G) + 35% Silicon powder+3% SFP (elastic material) + 3% DZJ-Y (water loss agent) + 3% DZP (expansion agent) + 4%SCLS (liquid silicon) + 44% field water. The density of cement slurry is 1.88 g/cm3, the elastic modulus of cement is 6.6-7.2 GPa, and the API water loss is less than 40 mL. In order to explore the failure mechanism of cement sheath, a large-size cement sheath seal ability evaluation device was used to simulate the condition of shale gas well staged fracturing and evaluate the sealing ability of cement sheath. The principle of the device is

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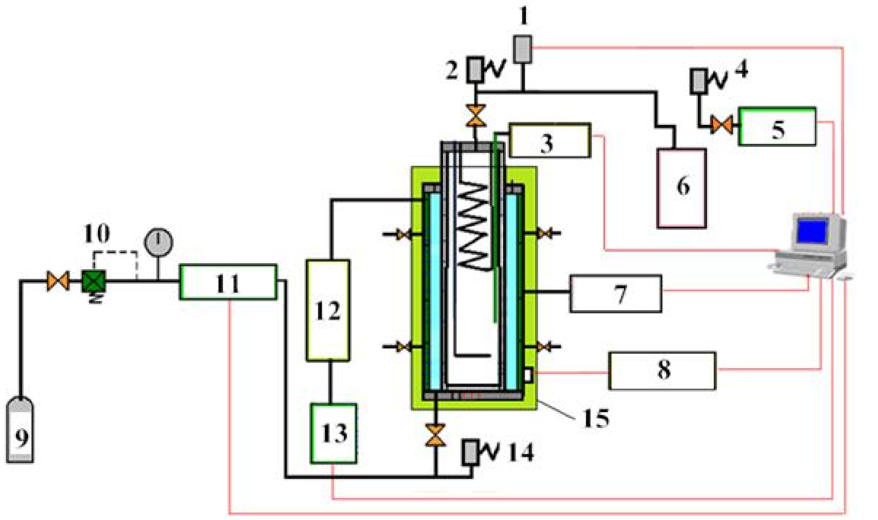
shown in Figure 3. The principle and test method of the device: the plane strain theory is adopted to simulate the deformation of the cement ring under uniform stress in the fracturing process, which is consistent with that of the stratum, so as to realize the same mechanical constraint of the stratum on the cement sheath. The cement slurry was poured, the curing time was 72h, and the loading was carried out according to the fracturing construction load.



1. Pressure sensor; 2. Safety valve; 3. Temperature controller; 4. Safety valve; 5. Automatic pressure control pump; 6. High pressure buffer tank; 7. Strain detector; 8. Acoustic detection system; 9. Nitrogen tank;10. Relief valve; 11. High pressure flowmeter; 12. Gas flowmeter; 13. Bubble detection unit; 14. Safety valve; 15. Lagging

Figure 3. Sealing evaluation device for cement sheath.

Through the evaluation of the sealing ability of cement stone, it can be found that under 90MPa fracturing load, after 11 fracturing loads were loaded, gas channeling occurred in the annulus(Figure 4). Cement was tested by triaxial pressure testing machine, in 15MPa confining pressure, 40MPa axial stress condition of rocks stress-strain hysteresis curve(Figure 5), found 0.74% of cement with residual strain, resulting in cement sheath is greater than 0.1 mm ring gap caused by gas channeling. Combined with the porosity distribution test after loading cement stone, it can be found (Figure 6) that due to the collapse of the macropores of the cement stone, the decrease of the macropores of the cement stone and the increase of the pores lead to the irreversible deformation of the cement stone, which leads to the formation of micro-annulus gap of the ductile cement stone (Figure 6) and the occurrence of gas channeling.

The test shows that while further optimizing the mechanical properties of cement and reducing the elastic modulus of cement, it is necessary to effectively reduce the porosity of cement, so as to effectively control the residual strain of cement and improve the sealing annulus ability of cementing cement sheath.

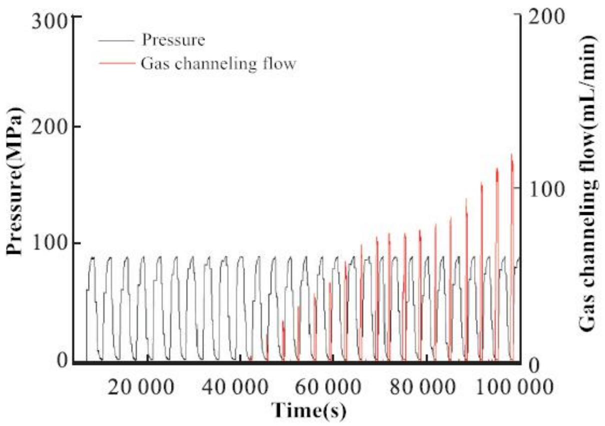


Figure 4. Analysis of loading and unloading of ductile cement.

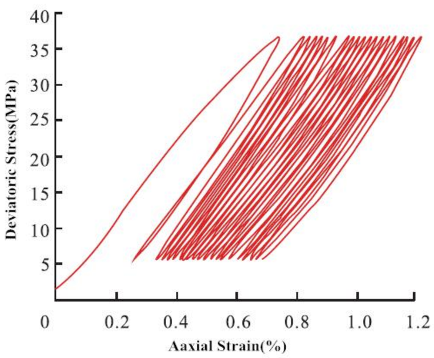


Figure 5. Residual strain test of ductile cement under cyclic loading.

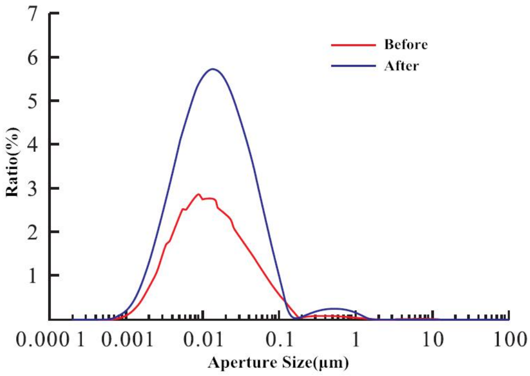


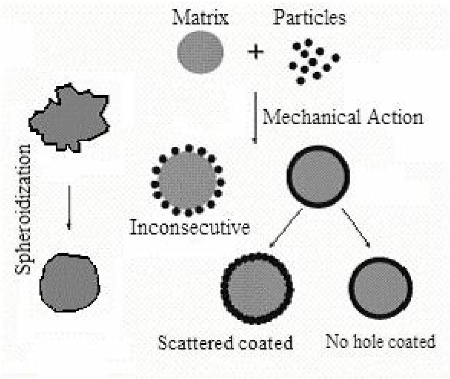
Figure 6. Porosity test of cement after loading.

3. Construction of high strength flexible cement slurry system

3.1. Selection and modification of high temperature resistant elastic materials

Conventional cement stone has the inherent properties of high compressive strength, poor deformation ability and brittle failure. To improve the hardness and brittleness of cement stone and increase the deformation ability of cement stone, it is mainly through the crystallization phase plasticizing, gel phase plasticizing and the pore increasing of cement stone matrix. The methods of adding pore to cement matrix mainly include increasing the porosity of cement or filling cement with low modulus material. At present, the method of filling cement with organic elastic admixture has a great influence on the strength of cement. The main factor is the hydrophobic organic elastic material, which leads to the interface with low bonding strength between the particle interface and cement hydration products, thus affecting the mechanical properties of cement. In view of the technical problems existing in the current material, adopts nonporous coating technology, the existing modification on elastic material, cover a layer of organic material in the elastic material, form the new sexual elasticity materials to after the cement hydration and cement hydration products realize the seamless bonding, at the same time of lower young's modulus of cement stone, reducing the loss of strength of cement stone.

The rubber particles and nano-silica fume were added into the Particle Composites System in a certain proportion, and the experimental process parameters were adjusted to make the nano-silica fume fully contact and mix with the rubber particles, forming a core-shell structure of silica fume coated rubber particles [3].



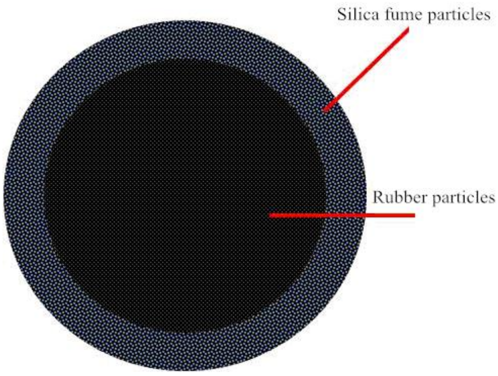


Figure 7. Composites of micro and nano particles. **Figure 8.** Composites of core-shell structure.

Elastic particles can obviously reduce the compressive strength of cementitious materials. In order to ensure that the compressive property of the material remains above 14MPa, silica fume is needed to strengthen the cement. In particular, the surface of the elastic particles is coated with silica fume, so that these elastic particles which have no affinity with cement have good compatibility with cement. Especially when the elastic particles are irregular in shape, the good bonding with cement stone means that the rigid particles around the elastic particles can allow a certain non-destructive slip when subjected to external forces, and achieve the effect of reducing the elastic modulus comprehensively.

Table 2 Influence of different elastic materials on mechanical properties of cement

Material	Elasticity Modulus and Strength			
	+2%	+4%	+6%	+8%
Conventional elastic particle	8.5/32.3	7.4/26.2	7.1/18.9	6.6/14.2
Modified elastic particle	8.6/34.2	6.7/30.8	6.2/28.6	5.6/24.8

3.2. Optimization and evaluation of high temperature resistant toughening agent

Conventional cement-based materials are typical brittle materials with low tension/compression ratio and low ultimate elongation. When organic fibers are added to cement-based materials, the fibers bond the cracks, transferring stress to the upper and lower surfaces of the cracks. With the increase of fiber content, this effect will become more obvious, so the stress concentration at the crack tip will tend to ease and may disappear. The fracture toughness and tensile strength of the composites will be improved due to the cracking resistance of the fibers.

In view of the environmental temperature of the design and the feasibility of the field construction of the high temperature resistant cement slurry in the deep formation, the new mineral fiber is selected for research and evaluation, so as to improve the deformation ability of the cement slurry and the effect of toughening and cracking prevention [4].

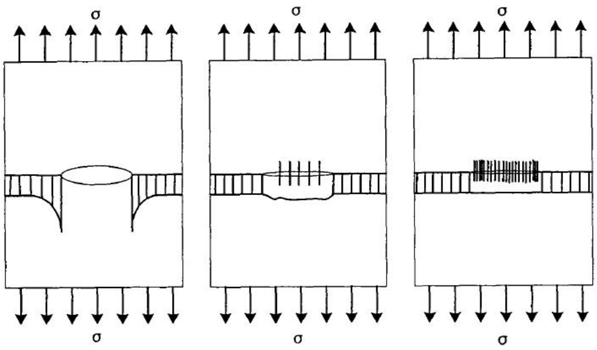
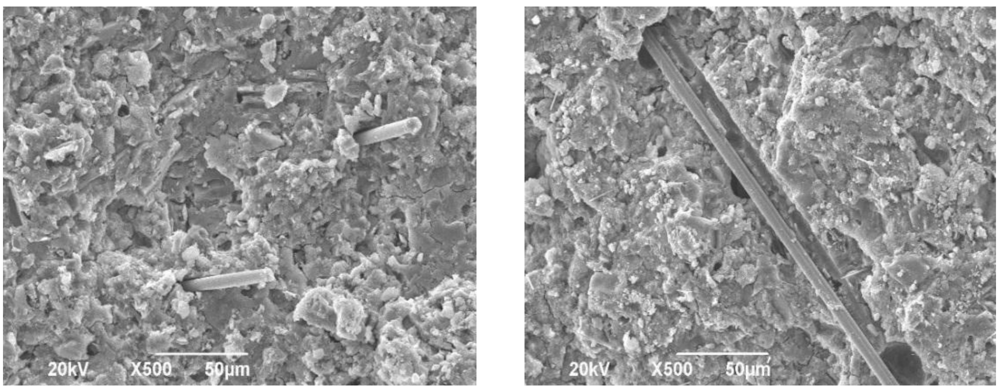


Figure 9. Fiber crack resistance

Before loading and initial cracking, the fiber reduces the number and size of crack sources by inhibiting the shrinkage of matrix. When the strain reaches the strain limit of the ordinary matrix, the composite material does not break immediately due to the improvement of the crack influence factors, and the strain will continue to rise until the matrix cracks, thus the overall strength of the composite material is improved. After cracking, the composite material can continue to bear the load due to the existence of the fiber. If the bearing capacity of the fiber is enough, the strength of the composite material will continue to improve [5].

Figure 10 shows the situation of the organic fiber being pulled out (a) and peeled off (b) on the fracture surface of the organic fiber cement stone specimen. From the morphology of the cement specimen after destruction, some fibers on the damaged interface of the specimen were pulled out or broken, and some were peeled off. When cracks appear in the organic fiber cement specimen, the cement matrix no longer bears tensile force, and the tensile load is borne by the fibers at the crack interface. The fibers transfer the tensile stress to the matrix through the bonding force and mechanical biting force, adjusting the stress distribution in the cement specimen and preventing the expansion of local cracks in the cement specimen. However, when the development of cracks is inevitable, the reinforced fibers at the cracks will be stretched, pulled out or broken, which can consume a lot of energy, thus greatly improving the fracture toughness of cement materials.



(a) (b)

Figure 10. Micromorphology of failure section of high temperature resistant organic fiber cement matrix

3.3. Optimization of filling materials for pore improvement

By revealing the failure mechanism of cement seal, improving the porosity structure of cement is an effective method to reduce residual strain of cement. At present, the method to improve the pore structure of cement stone mainly adopts particle grading technology and nano-material filling. Because the powder material used in the current system is mainly cement, it is difficult to achieve by adjusting the particle grading of the powder material, so nano-material filling is an effective method to improve the pore structure.

In order to solve the problem that the high shrinkage characteristics easily lead to the deterioration of cement sheath bonding, a set of preparation technology of "physical dispersion + chemical polymerization inhibition" nano silicon emulsion was formed, and an enhanced silicon based emulsion was developed [6-7]. The spherical nano-silica at 50nm-300nm was selected as the raw material. The agglomeration of nano-dispersed particles was dispersed by impact and shear action of high-energy physical ball milling. The agglomeration of nano-dispersed particles was prevented and the stability of the dispersion system was improved based on chemical methods such as the spatial hindrance effect, Zeta potential and thermal motion resistance of the lifting particles.

The enhanced silicon-based emulsion can inhibit the hydration and shrinkage of cement, and the relative volume expansion rate reaches 37%. The cement bonding strength is increased to 2.55MPa, which is 30% higher than that of traditional cement slurry. At the same time, it also has a strong filling and plugging effect, which can reduce the permeability of cement to less than 0.02mD and effectively prevent gas/liquid channeling between the interface and the body [8-9].

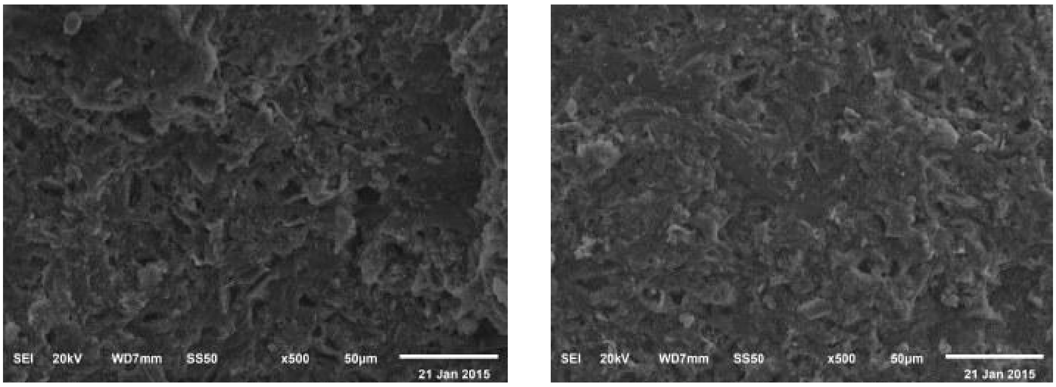


Figure 11. Microstructure of silicon-based emulsion reinforced cement stone
(Blank sample on the left and 8% silicon-based emulsion addition on the right)

3.4. Evaluation of high strength flexible cement slurry system

Based on the conventional ductile cement slurry system used in middle and shallow shale gas Wells, a new elastic material and silicon-based emulsion were used to modify the cement slurry system. The orthogonal design method was adopted to optimize the cement slurry system with different material dosage, and the conventional and mechanical properties of the cement slurry were tested. The five optimal formulas were as follows:

Table 3 Optimized formulation of high strength flexible cement slurry system

Serial Number	Cement(G)	Silicon Powder	SFP-H	DZJ-Y	DZP	SCLS	Field Water
1#	100%	35%	3%	3%	3%	4%	44%
2#	100%	35%	6%	3.5%	3%	6%	44%
3#	100%	35%	8%	3.5%	3%	8%	44%
4#	100%	35%	8%	3.5%	3%	6%	44%
5#	100%	35%	6%	3.5%	3%	8%	44%

Note: SFP-H is modified elastic particle. DZJ-Y is water loss agent. DZP is expansion agent. SCLS is silicon emulsion

Through evaluation of cement slurry for slurry rheology, filtration, sedimentation stability, strength, elastic modulus, permeability and other performance (Table 4), under the condition of ensuring good liquidity, control the slurry sedimentation stability within the $0.03g/cm^3$, safeguard cement modulus below 6.5 GPa conditions water mudstone intensity is high, as far as possible. At the same time, it can meet the low permeability, so No.5 is selected as the cementing field formula for deep shale gas horizontal Wells.

Under the condition that 5# cement slurry meets the construction requirements, the sealing ability and residual strain of cement stone are tested and evaluated. The large-size physical simulation device is used to test the sealing ability of cement ring under the condition of 120 MPa, and 30-stage staged fracturing is simulated without gas conduction (Figure 12). The residual strain of cement stone is tested. The residual strain of cement stone was reduced by 42.89 %(Figure 13), and the laboratory test shows that it can meet the needs of horizontal well cementing in deep and ultra-deep shale gas wells

Table 4 Basic properties of cement slurry

Serial Number	Rheological Property	API Water Loss (mL/30min)	Sedimentation Stability (g/cm^3)	Strength (MPa/48h)	Elasticity Modulus (GPa)	Permeability ($10^{-3}\mu m^2$)
1#	221/143/100/82/4/2	49	0.04	32	7.1	0.12
2#	281/172/110/97/8/7	37	0.01	29.1	6.3	0.08
3#	300^+/285/200/129/21/17	33	0	25.6	5.8	0.08
4#	300^+/271/182/116/18/14	33	0	24.2	5.5	0.10
5#	285/177/115/100/12/10	34	0	28.6	5.7	0.05

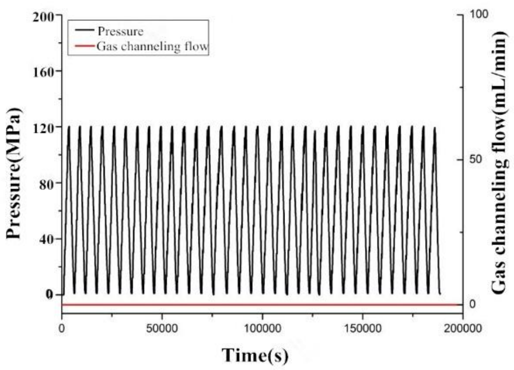


Figure 12. Analysis of loading and unloading strength of high strength flexible cement stone

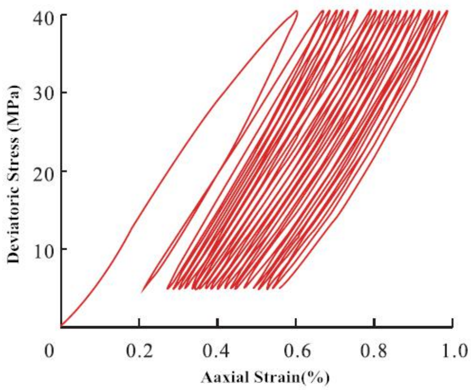


Figure 13. Residual strain test of high flexible cement stone under cyclic loading

4. Application of high strength flexible cement slurry system

At present, deep shale gas target layer vertical depth is more than 3500m, using three open system well structure, a open surface casing sealing Maokou group and other shallow gas reservoirs; Double drive ϕ 244.5mm casing into the Longmaxi group 50m; The triple-opening ϕ139.7mm production casing runs to the design depth and seals the target zone.

Field tests using the high-strength flexible cement slurry system were conducted to effectively seal the target zone and prevent post-fracture annular pressure. The test well adopts a two-stage cement slurry structure, in which the lead slurry adopts a low density elastic ductile cement slurry system, and the tail slurry adopts the high strength flexible cement slurry system designed in this paper. The lead slurry is returned to the well depth of 1000-1500m, and the tail slurry is returned to the technical casing shoe of 200-300m.

The cementing quality has reached high quality. No casing annular pressure has occurred in subsequent tests and oil and gas production, and the application effect is shown in the table below. The results show that the high strength flexible cement slurry system and reasonable slurry column structure designed in this paper, together with the supporting cementing technology, can effectively improve the long-term sealing ability of cement sheath, reduce the annulus pressure ratio of production casing, and ensure the safe and efficient production of shale gas wells after fracturing.

Table 5 Field implementation and application

Serial Number	Fracturing Segments	Max Fracturing Pressure (MPa)	Cementing Quality	Addition of SFP-H (%)	Elasticity Modulus (GPa)	Annulus pressure (Yes/No)
JYA-3HF	22	115.6	Perfection	6	5.7	No
JYA-4HF	22	125.0	Perfection	6	5.5	No
JYA-6HF	20	120.0	Perfection	6	5.8	No
JYB-2HF	24	109.8	Perfection	6	5.5	No
JYB-3HF	21	113.5	Perfection	6	5.7	No
JYC-2HF	25	115.7	Perfection	6	5.5	No
SYA-1HF	20	121.5	Perfection	6	5.7	No

The reduced annular pressure ratio of the production casing reduces the corrosion of the outer surface of the production casing and the inner wall of the casing due to the contact with the formation fluid to a certain extent, which effectively prolongs the production life of the shale gas well, and saves the extra cost of annular pressure management and well abandonment and plugging in the later stage.

5. Conclusions

(1) The physical simulation test shows that the demand of elastic toughness of cement sheath in deep shale gas horizontal well is higher than that of conventional shale gas horizontal well.

(2) The hydration products of cement were filled with new elastic particles to further improve the hardness and brittleness of cement, and the elastic modulus of cement was further reduced. Nano materials were used to reduce the porosity of cement, reduce the residual strain of cement, and improve the sealing ability of cement sheath.

(3) The field application shows that through the design of reasonable slurry column structure and the use of high strength flexible cement slurry system with excellent performance, the phenomenon of casing annulus pressure in the production of horizontal Wells in deep shale gas can be effectively alleviated, and the high strength flexible cement slurry system has great popularization value in the horizontal Wells in deep shale gas staged fracturing.

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