Study and Field Trials on Dissolvable Frac Plugs for Slightly Deformed Casing Horizontal Well Volume Fracturing

Peng Zhang, Chunsheng Pu,* Jingyang Pu, and Zhihui Wang

ABSTRACT: In view of the need for slightly deformed casing horizontal well fracturing, a new dissolvable frac plug has been designed using mechanical and material methods. The results of simulation analysis, laboratory research, and field test verification show that: (1) the integral half-split inlaid tooth slip and implicit-shaped single sealing element are adopted. Under the same pressure resistance index, the outer diameter and the total length of the frac plug are small, the inner diameter is big, and the pressure resistance downhole passing performance are better. (2) Using dissolvable magnesium-based powder, hydrogenated butadiene-acrylonitrile rubber, fluororubber polyglycolic acid, and the cellulose fiber and coating process, the solubility of the frac plug is observed to be more reliable. (3) Using the structural modular design, the frac plug has outstanding advantages in terms of on-site installation, preventing early seat sealing, self-separation after release, less dissolvable matter, and so on. (4) The finite element simulation and physical simulation experiments show that the frac plug can tolerate deformation in the range of 15 mm, which meets the needs for the casing usage. The temperature increases from 60 to 98 °C, and the maximum sustainable pressure differential decreases from 7.6 to 11.2 h at 70 MPa without any leakage. The frac plug can be completely dissolved within 300 h, and the final size of the residual solid is less than 0.3 mm. (5) In field trials of eight wells, through the minimum set of change inner diameter of 107 mm, the maximum wellhead fracturing pressure is 83.9 MPa in the formation, with the temperature ranging from 36 to 110 °C, and the plug can be completely self-dissolved in 16 days (the shortest time that has been found). After the fracturing operation with this dissolvable frac plug, the average daily oil production is maintained as high as 10.45 t with 22,000 m³ gas production. A new dissolvable frac plug tool solves the problem of continuous fracturing in slightly casing variable wells, restores the full bore of the wellbore after staged fracturing, and realizes the production of oil and gas wells without drilling plug and the smooth implementation of follow-up measures. It has a wider application range, higher efficiency, safety, and economy. It is of great significance for industrialized staged fracturing of unconventional oil and gas horizontal wells and platform wells.

1. INTRODUCTION

Unconventional oil and gas are important oil and gas resources. In 2009, more than 95% of wells were completed in unconventional gas horizontal wells in the United States. In 2013, the application rate of “drillable frac plug and cluster perforation” volume fracturing in North America reached 85%. Since 2010, new ideas of “volume fracturing” and “fracture-controlled reserves” transformation method have been implemented to form the “horizontal well casing completion and cluster perforation and frac plug section,” the body of the fracturing process. However, with the increase of fracturing scale and well number, there would be more and more slight casing deformations in some well sections due to various reasons. According to incomplete statistics, the average casing loss rate of shale gas horizontal wells in Changning, Wei 202, Wei 204, and Zhaotong blocks in China reached 38.46% at the end of 2019 and 30.4% at the end of 2020 in Ma#56 tight oil block in Santanghu Basin. According to the topic column of Journal of Petroleum Technology, issue 1, 2020, the casing variation rate of shale oil and gas development is 20–30%, that of Marcellus shale oil wells is 6.2%, that of Vaca Muerta formation shale gas wells in Neuca Basin of Argentina is 25%, and that of Duvernay block in Canada is 47 and 8.8%, respectively. Casing deformation caused the plug to be unable to run into the design position, resulting in a loss of reserves and failure to implement follow-up measures.
The frac plug researches can be divided into two key directions, drill frac plug at home and abroad and dissolvable bridging. After the invention of a composite drillable frac plug in the United States in 2000, the drilling and grinding time of composite drillable frac plug has been continuously improved by optimizing the structure and material design. However, the following problems are still encountered: (1) the buried depth of the reservoir, the vertical depth of the horizontal well is up to 7751.57 m (Shun Bei Ping 1H), and the horizontal section length is up to 5652 m (Purple Hayes 1H). Deep wells and long laterals present greater challenges in drilling and grinding technologies (coiled tubing length, surface horsepower, shoe grinding, etc.) for postfracturing plugs, as well as the risk of well control and downhole incidents associated with tripping strings. (2) Single well fracturing is as high as more than 50 stages, with high drilling and plug cost, and easy to cause complex wellbores. In 2014, CNPC alone spent 70–90 million RMB on drilling and plugging, and the large amount of drilling and plug residues caused by wellbore plugging has a great impact on production. In 2010, based on the research and development of dissolvable magnesium-based materials and aluminum-based materials, the research of frac plug was conducted from drillability to self-solubility. At the end of 2015, the study of dissolvable frac plug and the field test were started. Due to the limitation of structure and strength, the outer diameter of the existing dissolvable frac plug is large, which is generally 110–115 mm. However, the inner diameter of the slightly deformed casing is small and cannot be used. Therefore, it is necessary to further study the size and performance of dissolvable plugs to address the fracturing requirements of horizontal wells with slight casing variations.

In this study, first, the structure and materials of the dissolvable frac plug are designed and evaluated. The design is analyzed and studied through finite element simulation. Second, indoor experiments are carried out to evaluate the solubility and pressure-bearing capacity of the frac plug. Finally, field tests are carried out. The new type of dissolvable frac plug has a small outer diameter, large inner diameter, good high-pressure aging and dissolution performance, simple field installation process, reliable performance, and can meet the fracturing needs of 5 in. casing deformation less than 15 mm.

The research is to design a soluble bridge plug for staged fracturing of slightly casing variable horizontal wells. The bridge plug has the characteristics of small outer diameter, high pressure-bearing grade, and good solubility required by fractured wells under specific conditions. The research includes the mechanical design and material design of the bridge plug. First, the bridge plug’s structure design and material design are completed according to certain design principles (Section 2.1); second, the bridge plug performance test is completed according to the laboratory equipment (Section 2.2), and the bridge plug design is optimized. Finally, the field test of fractured wells is carried out to provide a basis for the promotion and application of the new step (Figure 1).

2. EXPERIMENTAL AND COMPUTATIONAL METHODS

2.1. Frac Plug Design Principles. China’s unconventional oil and gas horizontal wells are dominated by 5 1/2 in. casing. According to statistics, about 85% of the deformed casing wells are generally slightly deformed (casing deformation value ≤ 15 mm; the inner diameter of 5 1/2 in. casing is 121.36 mm, and the minimum inner diameter after deformation is 106.36 mm).

3. RESULTS AND DISCUSSION

3.1. Dissolvable Plug Design. 3.1.1. Structure and Parameter Design of Dissolvable Frac Plug. Based on the characteristics of the upper pressure difference in fracturing, a single-slip frac plug structure is designed, which is composed of a fracturing ball and a frac plug body. The frac plug body is divided into a sealing element seat, sealing element, antissetting scissors, lock gear ring, cone, slip, bottom cone, and a pull rod retaining ring, as shown in Figure 2. The structure, size, using
range, bearing pressure index, and the setting and dropping design parameters of the frac plug are shown in Table 1.

3.1.2. Design and Simulation of the Integral Half-Split Inlaid Tooth Slip. 3.1.2.1. Structural Design Optimization of the Slip. Slip is the important component to support the frac plug and the lock sealing element. Its performance directly determines the stability and pressure resistance level of the frac plug in casing. In 1977, Hammerlindl12 developed a permanent frac plug with an integral slip; Liu et al.13 conducted fracture tests on an integral slip and studied the relationship between the stress groove structure of the slip and fracture pressure. Deng et al.14 studied the microfracture mechanism of an integral slip. Cai et al.15 studied the structural stress of slip teeth and gave reasonable tooth profile parameters. Liu et al.16 gave reasonable spacing of slip teeth; Shahani and Sharif17 established a contact model between a slip and casing and gave the relationship between the opening radian of the slip and contact stress. Cai et al.15 also studied the influence of the parameter design of slip on the stress and slip of casing. Cai et al., Ma et al., Hao et al., and Liu et al.18−21 analyzed the distribution law of bite mark depth and equivalent stress distribution of the tooth root of slip tooth.

Based on the above findings and combined with the field experience, a kind of integral half-split inlaid tooth slip is designed. The structure and size of fracture grooves among the slip are mainly designed and optimized, which effectively guide the slip to spread evenly and anchor reliably, and ensure the uniform stress in the setting process of the blocks. The anchoring high performance of the slip is ensured by using high hardness-embedded teeth on the surfaces of the slip, as shown in Figure 3.

3.1.2.2. Working Principle of the Slip. Under the interaction of the setting adapter and tie rod, the sealing element seat of the frac plug is squeezed (the setting force comes from gunpowder or the hydraulic setting tool). After overcoming the antissetting force (antisetting shear nail), the slip tooth block slowly and evenly expands from the front end of the stress groove and goes up along the cone and continues to expand and be fixed on the inner wall of the wellbore casing through the teeth. The sealing element seat continues to press the sealing element casing downward and locks with the locking ring inside the cone to prevent it from retreating; thus, the frac plug is firmly fixed, as shown in Figure 4.

3.1.2.3. Simulation Calculation and Analysis of a Forcing State of Slip during the Setting Process. (1) Objective and methods: To optimize the size of the fracture groove, the rationality and reliability of the structural design of the slip are verified, and ANSYS finite element simulation is used to calculate and analyze the stress groove and the stress state in the process of setting and opening the slip.

(2) Research conditions and grid division: The casing material is the universal material of Project Q125; the slip fracture strain is the maximum strain before the material fracture; the fracture strain is set to 0.147; the triaxial stress is 0; the strain ratio is 0; slip teeth are G0412 hard alloy;21 and the cone and stop ring are rigid bodies. The six degrees of freedom of the stop ring and casing are fixed. The cone is subjected to a displacement load of 10−45 mm. A regular tetrahedron is selected for studying the grid property of the slip;22 the analysis step is set to display the dynamics; and the time length is 15 s.

(3) Parameter design optimization: Three schemes were used to simulate the thickness, length, and front-/back-end length of the fracture groove of the slip, and the crack-opening force value of the fracture groove was solved and extracted, as shown in Table 2.

Table 1. Design Parameters of a Dissolvable Frac Plug

| length/mm | external diameter/mm | inner diameter/mm | frac ball/mm | working pressure/MPa | casing range/mm | releasing force/kN |
|-----------|----------------------|-------------------|--------------|----------------------|-----------------|------------------|
| 430       | 103                  | 43                | 50           | 70                   | 106.36−121.36   | 140−160          |

Figure 2. Structural design of a dissolvable frac plug. (1) Sealing element seat; (2) sealing element; (3) antisetting scissors nail; (4) lock gear ring; (5) cone; (6) slip; (7) bottom cone; (8) pull rod retaining ring.

Figure 3. Structural design of an integral half-split inlaid tooth slip.

Figure 4. Schematic diagram of the working principle of a slip. (1) Sealing element seat; (2) cone; (3) casing pipe; (4) slip; (5) bottom cone; (6) pull rod retaining ring.
The following results were obtained: (1) According to the back-calculation of the crack initiation force, calculated by simulation in Table 2, the initial setting forces required by the three schemes are 15.23, 6.96, and 2.61 MPa, respectively, in 5 1/2 in. (Φ = 121.36 mm) casing, and the values of schemes A and C are much bigger or smaller than the reasonable setting pressure value (5−10 MPa) on site, so it is necessary for the setting tool to provide a greater setting force or cause of setting. (2) From Figure 5, it can be seen that the fracture groove state in the setting process of the slip can be divided into four stages: stress concentration (Figure 5a), plastic deformation (Figure 5b), local fracture (Figure 5c), and uniform fracture opening (Figure 5d). During setting, each slip tooth first opens at the front end of the slip groove where the stress is maximum, and the stress value reaches the tensile strength of the material at the middle and rear positions of the fracture groove length, and cracking begins to occur, which is in line with the design intention. As shown in Figure 6, premature or late cracking will result in uneven or difficult opening of the slip. During the whole process, the stress changes uniformly. The shape keeps well, and there is no fracture phenomenon. (3) After setting, there is no obvious stress concentration point in each slip, and the strength of the slip is high. (4) Under the ultimate bearing capacity of 70 MPa, the overall structure of the slip is complete, and there is no casing slip phenomenon, which verifies that the integral half-split slip structure and stress groove design are reasonable. Therefore, the integral half-split slip is designed with six slip lobes and six stress grooves distributed at even intervals, with the groove thickness of 2 mm, groove width of 1.0 mm, groove length of 25 mm/17 mm, and a total length of 67 mm. Teeth outside the slip lobes are hard ceramic particles, with thick teeth (Φ = 5.5 mm) and fine teeth (Φ = 2.5 mm), which are evenly distributed side by side.

3.1.3. Design and Performance Experiment of Implicit Groove Sealing Element. High contact stress is the key to achieve high sealing performance of the sealing element, and the structure and material design of the sealing element determine whether a high contact stress can be obtained.23 A concave barrel-shaped single sealing element structure is designed, as shown in Figure 7. A smooth recessed circular surface is designed in the middle of the inner circular side of the integral barrel-shaped single sealing element, and when the sealing element is extruded, it is guided to bulge from the middle along the circular arc line at the first time (the initial seal of the sealing element is uniformly spread from the middle along the circumferential direction to form a high contact stress, which is also the reason why the hardness value of the central sealing element of the three split sealing elements is lower than that of the sealing element on both sides), thus avoiding the surface of the barrel-shaped sealing element of equal thickness from being wrinkled and being sheared and torn during extrusion. Material design of sealing element: (1) A water-dissolvable sealing element is used when the reservoir temperature is lower than 100 °C. HNBR rubber is used as the main material, and an accelerator is added to adjust its hardness and hydrophilicity, with a tensile strength of ≥28 MPa and shore hardness of ≥85. It is muddy after dissolution. Laboratory experiments show that the hardness of the sealing element remains unchanged for 2−3 days in a water bath at 95 °C, which meets the requirements of pressure resistance and aging in fracturing operations, as shown in Figure 8. (2) When the reservoir temperature is higher than 100 °C, a degradable sealing element is used. The main material is fluororubber, and a certain amount of polyglycolic acid, dissolvable metal powder, and fiber are added. When these materials are dissolved, water molecules enter the fluororubber structure

Table 2. Simulation Calculation Scheme and Crack Initiation List of a Slip

| project | front/back length/mm | groove length/mm | groove width/mm | groove thickness/mm | split number | teeth number | Crack-opening force/kN |
|---------|----------------------|------------------|----------------|-------------------|-------------|-------------|------------------------|
| A       | 15/2                 | 50               | 1.0            | 3.5               | 6           | 96          | 176                    |
| B       | 25/17                | 25               | 1.0            | 2.0               | 6           | 96          | 82                     |
| C       | 30/27                | 10               | 1.0            | 2.0               | 6           | 96          | 31                     |

Figure 5. Fracture simulation diagram of an integral half-split slip in the setting process.

Figure 6. Mises’ stress distribution diagram of the stress block (without front and back ends) of slip.

Figure 7. Schematic diagram of an implicit-shaped single sealing element.
through the fiber tissue, and the polyglycolic acid and dissolvable metal powder react chemically to form scale substances. Thus, the rubber is decomposed into fluororubber particles from the fiber. The test parameters are shown in Table 3.

3.1.4. Properties and Dissolution Principle of Dissolvable Materials. Because of the requirements of self-dissolution, high mechanical strength, lightweight, and easy to flow back with the fracturing fluid, the frac plug is made of magnesium alloy.

3.1.4.1. Study and Analysis of the Dissolution Principle and Influencing Factors. (1) The standard electrode potential of magnesium is $-2.37 \, V$, which is an extremely active metal and easily forms oxide film structures in air. The outermost layer is a small plate structure with thickness of 2 $\mu m$; the middle layer is a dense layer with a thickness of 20–40 nm, and the inner layer is a honeycomb layer of 0.4–0.6 $\mu m$ thickness, and all three layers are porous, as shown in Figure 9. The porous characteristic of the oxide film on the surface of magnesium alloy is a necessary condition for dissolution.

(2) The negative differential effect of magnesium-based alloys in Cl$^-$ medium is the key to realize dissolution. The NDE-negative differential effect refers to the phenomenon that the “self-dissolution current density” of magnesium-based alloys does not decrease but increase with the increase of anodic polarization current or potential under the condition of applying anodic current or potential. Ying, Robinson, and King and others believe that the dissolution reaction of magnesium-based alloys occurs at the porous defect of the oxide film on its surface, and the negative differential effect makes the pore diameter of the oxide film increase and the self-dissolution rate increase.

(3) Cl$^-$ has the characteristics of small volume, low hydration degree, and fast movement speed, which reliably guarantee its dissolution through the protective film of magnesium-based alloys.

In sum, dissolving process can be achieved by pitting dissolution both inside and outside the erosion hole to form a micro thermocouple (outside the hole and the hole for the anode of the cathode), ongoing acidification autocatalytic, or generate oxide-oxide decomposition-show lively matrix-again accelerated dissolution. The chemical reaction of hydrogen dissolution is given as below which is given priority to accelerate the decomposition of general dissolution to pulverization process.

$$
\text{Mg} \rightarrow \text{Mg}^+ + \text{Mg}^{2+} + \text{H}_2 \uparrow
$$

(anodic reaction, oxide film formation)

$$
2\text{H}_2\text{O} + 2\text{e}^- \rightarrow 2\text{OH}^- + 2\text{H}_2 \uparrow
$$

(cathodic reaction, electrochemical hydrogen evolution dissolution)

$$
\text{Mg}^{2+} + 2\text{OH}^- \rightarrow \text{Mg(OH)}_2
$$

(dissolution products)

3.1.4.2. Analysis of Factors Affecting the Dissolution of Magnesium-Based Alloys. Indoor dissolution experiments were carried out in different solution temperature environments with $\Phi = 3.5 \times 5 \, cm$ magnesium-based alloy cylinders for 60 min. Drying–weighing method was adopted (drying at 105 $^\circ\text{C}$ for 60 min, $v = (m_2 - m_1)/60, v$: dissolution rate, g/min; $m_1$: original weight, g; $m_2$: weight after dissolution, g) to calculate the dissolution rate. The following experimental results were obtained: (1) The dissolution rate of magnesium-based alloy continues to accelerate with the increase of the solution medium temperature, and the dissolution rate is divided into three regions: zone I (<50 $^\circ\text{C}$) has a small dissolution rate and slow dissolution, and the influence of this stage on the bearing capacity of the frac plug is negligible; in zone III (50–80 $^\circ\text{C}$), the dissolution rate is slowly accelerated, and obvious bubbles appear and gradually increase. In zone III (>80 $^\circ\text{C}$), the dissolution rate begins to accelerate and bubbles become obvious. (2) The dissolution rate of the magnesium-based alloy in Cl$^-$ solution is higher than that in saline solution as a whole, which is in accordance with the dissolution mechanism of negative differential effect of magnesium-based alloys.

Table 3. Performance Parameters of Degradable Rubber Cylinder

| temperature resistance/$^\circ\text{C}$ | hardness/A | breaking strength/MPa | elongation/% | adapt temperature/$^\circ\text{C}$ | dissolution time/h |
|--------------------------------------|------------|-----------------------|-------------|-----------------------------------|-------------------|
| 120                                  | 90–93      | 20–25                 | 250–300     | 110–137                           | 50–120            |
| 93                                   | 78–82      | 20–25                 | 350–450     | 82–115                            | 50–120            |
|                                      | 90–93      | 20–30                 | 250–350     |                                   |                   |
|                                      | 78–82      | 20–30                 | 380–480     |                                   |                   |

Figure 8. Immersion hardness curve of water-dissolvable sealing element.

Figure 9. Schematic of the natural oxide film on the magnesium alloy surface.
alloys in $\text{Cl}^-$ solution, and the dissolution rate also accelerates with the increase of $\text{Cl}^-$ concentration or salinity, as shown in Figure 10.

3.1.4.3. Dissolution Rate Control and Analysis of Magnesium-Based Alloy. A cylinder sample with a diameter of $\Phi = 12.7 \times 25.4$ mm is made from magnesium-based alloy powder (matrix) and then the surface is coated with micro-nano metal/ceramic materials by the forging and extrusion process, with a total thickness of 30–2000 nm; the samples were immersed in 3% KCl solution at 93°C to simulate the downhole environment, and the dissolution rate (ROD) was evaluated by the weight loss method (24 h). The mechanical strength was evaluated by the uniaxial compressive strength test (ASTM E9 standard).

The following results were obtained: (1) The microstructure of the sample coating is observed by a scanning electron microscope. It is found that the higher the strength, the denser the surface is, as shown in Figure 11. (2) The mechanical strength of samples increases with the increase of the substrate surface coating thickness, and the dissolution rate increases first and then decreases with the increase of the substrate surface coating thickness, indicating that the combination of the coating formula has a great influence on the dissolution rate, as shown in Figure 12. (3) The ceramic coating can significantly reduce the dissolution rate compared with the metal coating, as shown in Table 4. (4) The maximum dissolution rate generally decreases with the increase of strength. When the dissolution rate of sample A is 100 mg/cm²/h, the strength is between 37 and 68 ksi, which is higher than the requirements of fracturing greater than 30 ksi, as shown in Figure 13. (5) When the compressive strength is basically the same, the dissolution rate of the forging process is 5–10 times that of the extrusion process, as shown in Figure 14.

Therefore, the substrate surface coating thickness and material composition can effectively regulate and control the dissolution rate and mechanical strength of the material, which plays a key role in high-pressure aging and uniform decomposition in a specified time, and is the core of the frac plug material design.

3.1.4.4. Preparation Method of Magnesium-Based Alloy Matrix. It can be divided into the sintering method and high-
temperature (250−550 °C) drawing method. The test results of mechanical properties are howed in Table 5. The tensile strength is not less than 320 Mpa, and the compressive strength is not less than 78 Mpa, meeting the technical requirements of pressure performance of the 70 Mpa volume fracturing frac plug.

Therefore, magnesium alloy has good solubility, high specific strength, high specific stiffness, and good machining performance, which can meet the mechanical strength requirements of volume fracturing with small section size and is easy to manufacture. Its density is 1.78 g/cm³, only 1/4 of steel, and the residue is easy to discharge from the wellbore, meeting the fracturing design requirements for different well conditions and reservoirs.

### 3.2. Experimental Study on the Performance Test of Dissolvable Frac Plug

#### 3.2.1. Setting and Releasing Test

Dissolvable frac plug was connected to the setting—hydraulic type frac plug setting tool—matching the tubing nipple in turn and loaded into the casing of the test wellbore. The tight tubing nipple was fixed, and the hydraulic plunger pump was connected, slowly pressed into the tubing, and the change of the pump pressure curve was recorded, as shown in Figure 15.

Laboratory experiments show that the plug starts to set at 7.07 MPa and is successfully set and released at 15.37 MPa. Compared with the design value (70−80 kN converted to 6.1−7.0 MPa; 140−160 kN was converted to 12.2−14.0 MPa), the slightly larger experimental value was related to the small displacement piston pump used in laboratory experiment, which proved that the design of the frac plug and setting matching was reasonable, the matching was good, and the releasing mode and releasing value were relatively stable.31−33

#### 3.2.2. Sealing Pressure Aging Test

##### 3.2.2.1. Experimental Preparation

The tubing sub and setting tool were pulled out. Before the experiment, the test wellbore was filled with 1% NaCl solution, and a 50 mm dissolvable frac ball was put in and then the test wellbore was heated to 60 °C.

##### 3.2.2.2. Experimental Process

The hydraulic plunger pump was used to pressurize the test well (top of the frac plug) to simulate the volume fracturing process. The pressure was kept at 50 MPa for 5 h and then the pressure was increased to 70 MPa (the pressure change was closer to the pressure fluctuation in the field fracturing process). Pressure drop and leakage were observed and recorded. After that, the plug was replaced, and the same process was replicated at 80 and 98 °C, respectively.

The results show that the dissolvable plug can withstand the upper pressure difference of 70 MPa and the pressure stability of 15 min when soaked for 11.2 h at 60 °C, and the leakage is less than 1%. After soaking at 80 °C for 8.8 h, the plug can withstand the upper pressure difference of 70 MPa and the pressure stability of 15 min, and the leakage is less than 1%. Soaked at 98 °C for 7.5 h, the frac plug can still withstand the upper pressure difference of 70 MPa, keep the pressure stability for 15 min, and the leakage is less than 1%, as shown in Figures 16 and 17.

Therefore, the dissolvable frac plug seal compression performance under different temperature conditions meets the requirements of temporary plugging of separate wellbores; considering the biggest flow rate fracturing operation, frictional resistance, well depth, and the next to layer pressure, the fracture load under the frac plug net pressure difference value is less than 70 MPa.

#### 3.2.3. Dissolution Performance Test of Sealing Element Seat and Frac Ball

The sealing element seat and frac ball can effectively create the fracturing interval. The separation efficiency of the sealing element seat and frac ball is evaluated at different temperatures and concentrations of NaCl solution. The dissolution trial has been conducted with a certain set of sealing element seat numbers with the constant temperature water bath and certain mesh barrier at the end of the sink. The decomposition products are completely dissolved after passing through the filter screen, and the dissolution time is recorded.

![Figure 14. Performance parameters of two kinds of technology.](image1)

![Figure 15. Laboratory-tested curve of the frac plug setting and release.](image2)

| number  | tensile strength/MPa | compressive strength/MPa | unproportional elongation strength/(N mm⁻²) | elongation after fracture/% |
|---------|----------------------|--------------------------|--------------------------------------------|-----------------------------|
| GWKR-E1 | 342                  | 78                       | 272                                        | 1.5                         |
| GWKR-E2 | 320                  | 80                       | 276                                        | 1.5                         |
| GWKR-E3 | 322                  | 84                       | 252                                        | 1.5                         |

Table 5. Mechanical Property Test of Three Dissolvable Metals

https://doi.org/10.1021/acsomega.1c06928

ACS Omega 2022, 7, 10292−10303

ACS Omega 2022, 7, 10292−10303

10298
The following results were obtained: (1) The total dissolution time of the sealing element seat is negatively correlated with temperature and the salt solution mass fraction, as shown in Figures 18 and 19. (2) The dissolution rate of frac ball is faster than that of the sealing element seat in the same dissolution environment, which is in line with the useful characteristics of frac ball dissolving first, leaving a larger channel for fluid flowback and accelerating the dissolution of the frac plug body, as shown in Figure 20. (3) The duration of the stable zone (7.0–8.0 h) at 1% NaCl 80–90 °C is consistent with the results of the pressure aging experiment (7.6–8.9 h), indicating that the stable zone has a good pressure aging. (4) In the decomposition process, the geometric size and dissolution uniformity of the sealing element seat and frac ball are maintained well, which meet the requirements of fracturing for long-pressure aging, as shown in Figures 21 and 22. (5) The dissolved residue is fine sand which turns into powder after drying, as shown in Figure 23. The particle size measured by the laser particle size analyzer is less than 0.3 mm, which meets the requirements of bringing oil and gas wells back to the ground during the production stage.

3.3. Comparison of Characteristics of New Dissolvable Frac Plug. 3.3.1. Advantages. According to the published literature, compared with similar 70 MPa plug products in 5 1/2 in. casing, the structural design of the new dissolvable plug has obvious advantages. Under the same pressure resistance index, the outer diameter and total length of the frac plug are small, the inner diameter is large, and the pressure aging, downhole passing, and dissolution performance...
are better, and it is suitable for long horizontal section wells and slightly casing variable wells, as shown in Table 6. With the modular design of the dissolvable plug structure, field installation takes only three steps, and the convenience, reliability, and safety of the frac plug are more prominent.

3.3.2. Limitations. Because the dissolvable material is greatly affected by temperature, when the reservoir temperature exceeds 100 °C, the sealing pressure aging time is short and the requirements for on-site frac organization are high. If the fracturing cannot be completed within 7 h, the frac plug needs to be pulled again to ensure the sealing.

3.4. Field Trial Evaluation. The new dissolvable frac plug and its supporting tools were tested in eight wells in the Ma#56, Lu1, Niudong, and Shengbei blocks of Tuha Oilfield, fracturing 39 stages in total. The plug passed through the change point position one time, setting and releasing reliably, and the fracturing curve was stable during fracturing, achieving the expected results. As shown in Table 7, one program well was transferred by tubing and seven program wells were transferred by cable in the test well. The inner diameter of the maximum casing change point position was φ = 107 mm, the maximum reservoir temperature was 110 °C, the minimum reservoir temperature was 36 °C, the maximum fracturing wellhead pressure was 83.9 MPa, the maximum displacement was 14 m³/min, and the maximum single-layer fluid volume was 1575.9 m³. With the tubing probe plug, the shortest 16 day frac plug has been dissolved, in accordance with the higher design index.

In the Ma#56-183H well field trial, the following results were obtained: (1) The well proved that the casing deformation point was 2700 m, the minimum inner diameter of the deformation point was φ = 107 mm, and the frac plug design position was 2738 m. The actual setting position of the frac plug was 2738.1 m by using the tubing transfer program, which was set at 16 MPa and released smoothly, and the exploratory plug had no displacement of 4 tons, proving that the frac plug setting performance was well. (2) The well adopts a water injection energy increase and volume fracturing scheme, that is, water injection of 5000 m³ at 2 m³/min was used to increase the energy, and then large-volume fracturing was carried out. The highest fracturing pressure was 65.97 MPa, the maximum fracturing displacement was 13.09 m³/min, the total fluid volume was 587.74 m³, the sand volume was 57.1 m³, and the highest sand ratio was 40.11%. The fracture curve is shown in Figure 24.

The overall results can be summarized as follows: (1) the new dissolvable frac plug takes 45 h to complete fracturing. In the fracturing stage, the construction displacement was stable, and the wellhead oil pressure did not drop significantly, which proves that the frac plug maintains good pressure aging. (2) Continuous high-displacement water injection for 42 h to increase the energy before volume fracturing verified that the dissolvable frac plug has good adaptability to the high-volume water injection and volume-fracturing process. (3) After 82 days of self-production, the plug probe confirmed that all of the frac plugs dissolved during the pump production operation, realizing the wellbore recovery without intervention. (4) The frac plug showed a long time of pressure aging, which is related to the cooling effect of large-displacement water injection in the well on the wellbore and near the well zone. The continuous low temperature has a certain inhibitory effect on the frac plug dissolution.

4. CONCLUSIONS

In this study, a dissolvable frac plug was designed and evaluated through finite element simulations, indoor experiments, and field tests. From this work, the following conclusions can be drawn:

(1) The dissolvable frac plug is designed with an integral semisplit teeth-inserted slip and a recessed barrel-shaped single sealant cylinder. Under the same pressure resistance index, the outer diameter and total length of the frac plug are small; the inner diameter is large; and the pressure aging, downhole passing, and dissolution performance are better; and it is suitable for long horizontal section wells and slightly casing variable wells.

(2) With the modular design of dissolvable plug structure, field installation involves only three steps: pull rod stop and bottom cone limit the self-release design, when pumping in the wellbore, and slip and sealing elements are not affected by the compression force, to prevent early setting; the design of automatic separation between the bottom cone and the body reduces the total length.
of the frac plug by 8.1% after setting. The design of the dissolving ring reduces the dissolvable content of the frac plug by 0.83%. The convenience, reliability, solubility, and safety of the frac plug are more prominent.

(3) The tests show that the new dissolvable plug can meet the design and application requirements in terms of pressure resistance, temperature resistance, solubility, and permeability and can meet the fracturing operation of 5 1/2 in. casing deformation no more than 15 mm. It has important engineering significance to solve the reconstruction problem of slightly deformed wells, which accounts for 85% of the horizontal wells with variable casing at present.

(4) Through systematic analysis and research of dissolvable frac plugs and design perfection of construction technology, the frac plug has been improved to adapt to oil reservoirs, well conditions, on-site installation, selection of running procedures, and so forth. The study of pressure resistance and aging in different environments provides more direct reference and guidance values for engineers in fracturing design and engineering organization.

Table 6. Comparison of Characteristics of Similar Dissolvable Frac Plugs at Home and Abroad (According to the Literature Published at This Stage)*

| Company          | Schlumberger | Haliburton10 | Baker Hughes31 | Vitai Oil and Gas Energy Group | MAGNUM | Petrochina Exploration Institute31 | Petro-king oil | New dissolvable frac plug |
|------------------|--------------|--------------|----------------|--------------------------------|--------|-----------------------------------|----------------|--------------------------|
| Product          | Infinity     | Illusion     | SPECTRE        | WIZARD/CHAMELEON™             | MVP    | SIIARK                            | Thunder        | Y257R-103.70              |
| Materials        | Aluminum-based metal | Aluminum base + dissolvable rubber cartridge | Electrolytic nano metal + polyurethane rubber cylinder | Magnesium aluminum alloy + dissolvable rubber or plastic rubber cylinder | Biodegradable material + ethylene glycol polymer/polyster cartridge | Magnesium base metal+dissolvable rubber | Magnesium base alloy + composite coating + water dissolvable/degreaded rubber cylinder |
| Mechanical structure | Single slip (tee + ball) | Bidirectional slip + partitioned inlaid ceramic grain slip | Unidirectional slip + integral non-valves nickel - base alloy-coated tile | Single/bi-directional slip + split inlaid cast iron slip | Bidirectional slip + partitioned inlaid alloy grain slip | Bidirectional slip + partitioned slip | Unidirectional slip + integral half petal inserts ceramic grain slip |
| Seal element structure | Tee/ball metal seal | Barrel type single rubber drum seal | Barrel type three rubber drum seal | Barrel type single rubber drum seal | Recessed single rubber drum seal |
| Prod. dim. ratios | φ112.7xφ82.3 | φ111.1xφ44x4 | φ111.1xφ38x550 | CHAMELEON™ φ111xφ35x660 | φ104.8xφ2437 | φ110xφ28x680 | φ110xφ35x600 | φ103xφ43x430 |

*Note: the dimensions (OD × ID × total length, unit mm) are compared with similar products of 5 1/2 in. casing, and the working pressure is 70 MPa.

Table 7. Field Application Statistics of Dissolvable Frac Plug and Supporting Tools*

| Well number | Transport type | Fracturing section number | Maximum pressure (MPa) | Maximum pump discharge (m³/min) | Maximum liquid content of monolayer (m³) | Maximum sand in single layer (m³) | Full completion fluid volume (m³) | Inner diameter of casing change point (mm) | Pass the casing deformation point | Days of plug (dissolved) |
|-------------|----------------|--------------------------|------------------------|----------------------------------|----------------------------------------|---------------------------------|------------------------------------|--------------------------------------|----------------------------|----------------------|
| *Ma 56-183H | Pipeline       | 1                        | 65.1                   | 13                               | 573                                    | 56.5                            | 573                                | 107                                  | yes                       | 82                   |
| *Lu 1-182   | Cable          | 4                        | 63.4                   | 14                               | 1049.8                                 | 89                              | 3594                               | 108                                  | yes                       | 16                   |
| *Lu 1-16H   | Cable          | 3                        | 62.8                   | 13.2                             | 1249.5                                 | 113.2                           | 3388.9                             | 110                                  | yes                       | 72                   |
| Niudongce   | Cable          | 2                        | 40                     | 10                               | 826.9                                  | 30.3                            | 1562.4                             | 108.62                               | yes                       | 50                   |
| Lingping    | Cable          | 3                        | 66.5                   | 9                                | 764                                    | 56                              | 2084                               | 108.62                               | yes                       | 42                   |
| Shengbei    | Cable          | 12                       | 83.9                   | 12                               | 1575.9                                 | 87.7                            | 16190.8                            | 114.3                                | yes                       | 97                   |
| *Huo 805H   | Cable          | 7                        | 81                     | 7.7                              | 1158.6                                 | 80                             | 7272.8                             | 108                                  | yes                       | --                   |
| *Ma L1-20H  | Cable          | 7                        | 62                     | 14                               | 1499                                   | 135.64                          | 9667.5                             | 121.36                               | yes                       | --                   |

*Note: * indicates that the ID of the casing in the test well was φ = 121.36 mm; -- indicates that no plug testing operation was conducted.
(5) With the development of unconventional reservoirs becoming increasingly complex, higher requirements and challenges are put forward for fracturing technology and supporting tools. The precise controllability and technical specifications of dissolvable plugs need to be continuously improved to adapt to more complex reservoirs, wellbores, and fracturing conditions. In particular, development of dissolvable frac plugs suitable for ultra-deep and high-temperature reservoirs can meet the higher requirements of low cost and efficient development of unconventional reservoirs.

**AUTHOR INFORMATION**

**Corresponding Author**
Chunsheng Pu — School of Petroleum Engineering, China University of Petroleum (East China), Qingdao 266580, China; Key Laboratory of Unconventional Oil & Gas Development (China University of Petroleum (East China)), Ministry of Education, Qingdao 266580, P. R. China; orcid.org/0000-0002-0724-5591; Email: chshpu_tq@126.com

**Authors**
Peng Zhang — School of Petroleum Engineering, China University of Petroleum (East China), Qingdao 266580, China; Key Laboratory of Unconventional Oil & Gas Development (China University of Petroleum (East China)), Ministry of Education, Qingdao 266580, P. R. China; TUHA Petroleum Research & Development Center, Hami, Xinjiang 839009, China

Jingyang Pu — School of Petroleum Engineering, China University of Petroleum (East China), Qingdao 266580, China; Key Laboratory of Unconventional Oil & Gas Development (China University of Petroleum (East China)), Ministry of Education, Qingdao 266580, P. R. China

Zhihui Wang — Gas Lift Innovation Center, Laboratory of Multiphase Pipe Flow, Yangtze University, CNPC, Wuhan 430100, China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.1c06928

**Notes**
The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

Funding for this study was provided by National Natural Science Foundation of China (grant number 52104057 and 62173049), Natural Science Foundation of Shandong Province (grant number ZR202103010885), and China Postdoctoral Science Foundation (grant number 2021M693506). The authors would like to thank Yan’an Zhongshida Oil and Gas Engineering Technology Service Co., Ltd. for the scale-up production and the participation in the field trial. This paper only reflects the views of the authors and does not necessarily reflect the views of the company.

**REFERENCES**

(1) Dejam, M.; Hassanzadeh, H.; Chen, Z. Semi-analytical solution for pressure transient analysis of a hydraulically fractured vertical well in a bounded dual-porosity reservoir. *J. Hydrol.* 2018, 565, 289–301.

(2) Bai, M.; Zhang, Z.; Chen, Q.; Weifeng, S.; Du, S. Research on the Enhanced Oil Recovery Technique of Horizontal Well Volume Fracturing and CO2 Huff-n-Puff in Tight Oil Reservoirs. *ACS Omega* 2021, 6, 28485–28495.

(3) Lei, Q.; Yang, L.; Duan, Y.; Weng, D.; Wang, X.; Guan, B.; Wang, Z.; Guo, Y. The “fracture-controlled reserves” based stimulation technology for unconventional oil and gas reservoirs. *Pet. Explor. Dev.* 2018, 45, 770–778.

(4) Durey, D. A.; Degner, D. L. Bridge-Plug Millout With Coiled Tubing—Case Histories. In SPE/ICoTA Coiled Tubing Roundtable, 2000; p SPE-60725-MS.

(5) Xu, Z.; Agrawal, G.; Salinas, B. J. Smart Nanostructured Materials Deliver High Reliability Completion Tools for Gas Shale Fracturing. In SPE Annual Technical Conference and Exhibition, 2011; p SPE-146586-MS.

(6) Yartys, V. A.; Lototskyy, M. V.; Akiba, E.; Albert, R.; Antonov, V. E.; Ares, J. R.; Baricco, M.; Bourgeois, N.; Buckley, C. E.; Bellosta von Colbe, J. M.; Crivello, J.-C.; Cuevas, F.; Denys, R. V.; Dornheim, M.; Felderhoff, M.; Grant, D. M.; Hauback, B. C.; Humphries, T. D.; Jacob, I.; Jensen, T. R.; de Jongh, P. E.; Joubert, J.-M.; Kuzovnikov, M. A.; Latroche, M.; Paskevicius, M.; Pasquin, L.; Poplevsky, L.; Skripnyuk, V. M.; Ribkin, E.; Sofianos, M. V.; Stuart, A.; Walker, G.; Wang, H.; Webb, C. J.; Zhu, M. Magnesium based materials for hydrogen based energy storage: Past, present and future. *Int. J. Hydrogen Energy* 2019, 44, 7809–7859.

(7) Zhang, Z.; Xu, Z.; Salinas, B. High Strength Nanostructured Materials and Their Oil Field Applications. In *SPE International Oilfield Nanotechnology Conference and Exhibition*, 2012; p SPE-157092-MS.

(8) Liu, X.; Zhong, M.; Chen, X.; Zhao, Z. Separating lithium and magnesium in brine by aluminum-based materials. *Hydrometallurgy* 2018, 176, 73–77.

(9) Li, M.; Chen, L.; Wei, R.; Liao, C.; Fu, T. The Application of Fully Dissolvable Frac Plug Technique in Weiyuan Gas Field. In SPE Kingdom of Saudi Arabia Annual Technical Symposium and Exhibition, 2018; p SPE-192422-MS.

(10) Nichols, M.; Eis, A. Self-Removing Fracturing Plugs: A Study of Initial Adoption in the Williston Basin. In SPE Oklahoma City Oil and Gas Symposium, 2017; p D021S001R001.

(11) Duan, P.; Sadana, A.; Xu, Y.; Deng, G.; Pratt, B. Degradable Packing Element for Low-Temperature Fracturing Applications. In *Offshore Technology Conference*, 2018; p D011S005R001.

(12) Hammerlindl, D. J. Movement, Forces, and Stresses Associated With Combination Tubing Strings Sealed in Packers. *J. Pet. Technol.* 1977, 29, 195–208.

(13) Liu, W.; Xu, S.; Li, Q. Experimental study on fracture performance of ultra-high toughness cementitious composites with J-integral. *Eng. Fract. Mech.* 2012, 96, 656–666.

(14) Santecchia, E.; Hamouda, A. M. S.; Musharavati, F.; Zalnezhad, E.; Cabibbo, M.; El Mehtedi, M.; Spigarelli, S. A Review on Fatigue...
Life Prediction Methods for Metals. Adv. Mater. Sci. Eng. 2016, 2016, 9573524.

(15) Cai, B.; Wang, J.; Sun, L.; Zhang, N.; Yan, C. Experimental study and numerical optimization on a vane-type separator for bubble separation in TMSR. Prog. Nucl. Energy 2014, 74, 1–13.

(16) Liu, Z.; Zhang, L.; Wang, F.; Li, S.; Wang, P.; Cai, M.; Han, L.; Ma, Y.; Ma, Z.; Yan, B. Study on Optimization Design of Permanent Packer Slip Structure. J. Failure Anal. Prev. 2021, 21, 50–60.

(17) Shahani, A. R.; Sharifi, S. M. H. Contact stress analysis and calculation of stress concentration factors at the tool joint of a drill pipe. Mater. Des. 2009, 30, 3615–3621.

(18) Li, Y.; She, C.; Liu, N.; Zhang, H.; Zhang, L.; Zhu, D. Completion difficulties of HTHP and high-flowrate sour gas wells in the Longwangmiao Fn gas reservoir, Sichuan Basin, and corresponding countermeasures. Nat. Gas Ind. 2016, 3, 269–273.

(19) Liu, Z.; Zhang, L.; Wang, F.; Li, S.; Wang, P.; Cai, M.; Han, L.; Ma, Y.; Ma, Z.; Yan, B. Study on Optimization Design of Permanent Packer Slip Structure. J. Failure Anal. Prev. 2021, 21, 50–60.

(20) Wang, Q.-H.; Wu, Z.-H.; Xu, Z.-J.; Fang, X.-L.; Zhao, H.; Wang, Y.-J.; Deng, D.-X. Optimization of the coupling groove parameters of composite porous vapor chamber. Appl. Therm. Eng. 2022, 205, 118007.

(21) Zou, Y.; Chai, Y.; Wang, D.; Li, Y. Measurement of elastic modulus of laser cladding coatings by laser ultrasonic method. Opt. Laser Technol. 2022, 146, 107567.

(22) Zhang, H.; Yang, S.; Liu, D.; Li, Y.; Luo, W.; Li, J. Wellbore cleaning technologies for shale-gas horizontal wells: Difficulties and countermeasures. Nat. Gas Ind. 2020, 7, 190–195.

(23) Lan, W.-J.; Wang, H.-X.; Zhang, X.; Chen, S.-S. Sealing properties and structure optimization of packer rubber under high pressure and high temperature. Pet. Sci. 2019, 16, 632–644.

(24) Liang, T.; Zhou, F.; Shi, Y.; Liu, X.; Wang, R.; Li, B.; Li, X. Evaluation and optimization of degradable-fiber-assisted slurry for fracturing thick and tight formation with high stress. J. Pet. Sci. Eng. 2018, 165, 81–89.

(25) Baril, G.; Pèbère, N. The corrosion of pure magnesium in aerated and deaerated sodium sulphate solutions. Corros. Sci. 2001, 43, 471–484.

(26) Lin, S.; Niu, L.; Pan, X.; Liang, Z.; Zhang, T. Study on preparation and recovery of cobalt hydride and cobalt carbonate. Hydrometallurgy 2021, 203, 105518.

(27) Holze, R. Encyclopedia of electrochemistry (A. J. Bard and M. Stratmann eds), vol 3: instrumentation and electroanalytical chemistry (P. R. Unwin ed). J. Solid State Electrochem. 2007, 11, 134–135.

(28) Zhang, Z.; Xu, Z.; Salinas, B. J. In-situ Disintegrating Completion Tools by Means of Controlled Microgalvanic Cells. In SPE Annual Technical Conference and Exhibition, 2013; p D0215025SR007.

(29) Li, S.; Li, S.; Guo, R.; Zhou, X.; Wang, Y.; Chen, J.; Zhang, J.; Hao, L.; Ma, X.; Qiu, J. Occurrence State of Soluble Organic Matter in Shale Oil Reservoirs from the Upper Triassic Yanchang Formation in the Ordos Basin, China: Insights from Multipolarity Sequential Extraction. Nat. Resour. Res. 2021, 30, 4379–4402.

(30) Song, G.-L. I-Corrosion behavior and prevention strategies for magnesium (Mg) alloys. In Corrosion Prevention of Magnesium Alloys; Song, G.-L., Ed.; Woodhead Publishing, 2013; pp 3–37.

(31) Yerokhin, A. L.; Nie, X.; Leyland, A.; Matthews, A.; Dowey, S. J. Plasma electrolysis for surface engineering. Surf. Coat. Technol. 1999, 122, 73–93.

(32) Liang, F.; Sayed, M.; Al-Muntasheri, G. A.; Chang, F. F.; Li, L. A comprehensive review on proppant technologies. Petroleum 2016, 2, 26–39.

(33) Jin, N.; Zeng, Q. Dissolvable Tools in Multistage Stimulation. In SPE/IATMI Asia Pacific Oil & Gas Conference and Exhibition, 2017; p D0125056R067.