Solid Phase Deposition Pattern Concerning Formation Oil in YD 7 Reservoir of Tarim Oilfield and Its Application: A Case Study

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ABSTRACT: Yudong (YD) 7 reservoir in the Yingmaili area of Tarim Oilfield is one of the key areas of oil and gas exploration in the Tarim Basin. However, due to the serious plugging problem caused by solid phase precipitation particles such as wax and paraffin, it is necessary to study the well flow phase behavior and solid phase precipitation law of typical high-production wells in this block to obtain the phase enveloping line and provide theoretical support for preventing solid phase precipitation of formation crude oil. In this study, the PVT tester and the self-designed microscopic solid deposition tester are used to obtain the phase enveloping line of formation crude oil, and the change law of the "gas–liquid–solid" phase behavior when the formation crude oil changes with temperature and pressure is observed. The morphological process of solid precipitation is recorded and analyzed through a microscopic visualization window. Finally, the solid phase precipitation point of formation crude oil is verified using a laser solid phase deposition tester. The experimental results show that under atmospheric pressure, the solid phase precipitation temperature point of surface crude oil is 34.05 °C, the maximum instantaneous precipitation is 0.01178%, and the maximum cumulative precipitation is 8.34%. The solid phase precipitation point of formation crude oil changes under different temperatures and pressures. Under different pressures and temperatures, it shows multiphase changes such as liquid–solid, liquid phase, gas–liquid–solid, gas–liquid, and gas phases. Limited by equipment, we can only observe the first four phase behaviors in the laboratory. In the process of solid phase precipitation, formation crude oil shows a fine needle shape at the initial stage and finally adhesions and aggregations in the form of an increasing crystal nucleus as the center, thus blocking the formation or wellbore. Combined with the analysis of production data, it can be seen that there is a solid precipitation problem in well YD 702 over 1200 m in the wellbore and the solid phase precipitation problem from the wellbore to the surface pipeline. This study provides theoretical support for preventing solid phase precipitation in the YD 7 reservoir and provides a reference for other oil fields with solid phase precipitation blocking problems.

1. INTRODUCTION

YD 7 reservoir is located in the Yingmaili area of Tarim Oilfield in western China. At present, this area is one of the key areas for oil and gas exploration in the Tarim Basin. There are oil and gas layers or oil and gas display distribution from Cambrian to Neogene, and these have great exploration potential. The structural map is shown in Figure 1. Solid precipitates such as asphaltene, paraffin, and colloid in formation crude oil plug the wellbore and reduce oil production, such as in YD 702 and YD 701H. The mechanism of solid deposition can be divided into two stages: (1) the destruction of equilibrium of the crude oil system, producing solid particles and (2) accumulation and deposition of wax and other solid particles.1,2 When the temperature is lower than the solid deposition temperature, very small coarse grain structures in the system of the asphalt and asphalt produce deposits; microcrystalline deposition includes resin, gum, oilfield water, and other particles such as silt, the coagulation decreases the strength, and the hardness of the sediments mainly depends on the composition and solid phase particle contained in the impurities. In the process of flow, if the shear stress acting on the solid deposition particle is less than the shear strength of the solid deposition, it will form an obstruction to the flowing fluid and cause blockage.3

Up to now, four electric submersible pumps in the YD 7 reservoir have been blocked by solids, no liquid has been produced, and there has been an overload shutdown of the electric pump, resulting in a sharp decrease in well production. Figure 2 shows the production curve of the electric transfer pump operation in the YD 703H well, and Figure 3 shows the
physical diagram of solid precipitation in the YD 7 reservoir. On analysis, we found that the oil and casing pressure fluctuation caused the temperature and pressure changes in the wellbore, which leads to the precipitation, deposition, and blockage of solid phase particles such as wax. At the same time, the intermittent production of the electric pump cannot ensure the continuous flow of fluid, which leads to frequent changes in temperature and pressure at the suction port of the electric pump and in the wellbore during well opening and closing, which is also one of the reasons for wellbore and pump blockage. To explore the phase behavior and the solid phase precipitation law of crude oil, this study considers the formation crude oil of the typical well YD 702 as the research object, obtains formation crude oil's phase enveloping line of YD 702, and combines it with the production system to provide effective theoretical guidance for prolonging the production life of YD 702 and predicting the precipitation law of solid under different working conditions.

At present, the commonly used testing methods for solid phase deposition include orthogonal polarizing microscopy, viscosity method, differential scanning calorimetry (DSC), ultrasonic method, and other testing methods (cold finger method, optical fiber method, conductivity method, rotary table method, ring path method, differential pressure method, visual method, interface tension method, fluorescence method, nuclear magnetic resonance method, etc.). Currently, the
structure of solid phase in solid phase deposition is usually studied by a variety of technical means such as small-angle neutron scattering (SANS), small-angle X-ray scattering (SAXS), mass spectrometry, and X-ray diffraction, and many molecular structural models of solid phase have been proposed.\textsuperscript{4–11} Peng photographed the microstructure of propane solid phase (908 kPa), n-pentane solid phase, and n-heptane solid phase at 120 and 1000 magnification using scanning electron microscopy. It was observed that the surface of the propane solid phase (908 kPa) was a smooth and amorphous semisolid particle, which was due to the synergistic precipitation of some semisolid colloids and propane solid phase under this test condition.\textsuperscript{12} Chen et al. believed that the solid-phase size of different measurement methods. The solid phase size measured by tunnel scanning and fluorescence depolarization technology was the smallest, about 1 nm. The size of solid phase measured by the light scattering technique is large.\textsuperscript{13} The above research cannot observe the solid phase precipitation process of formation crude oil under high-temperature and high-pressure conditions. To intuitively observe the change law of formation crude oil phase behavior and solid phase precipitation under high-temperature and high-pressure conditions, this paper uses the visual multifunctional high-temperature and high-pressure PVT tester and the high-power microscopic visual solid phase deposition tester to conduct relevant experimental research on the flow of YD 702. The equipment has the characteristics of high-temperature resistance, high-pressure resistance, and microvisualization. Finally, the solid phase precipitation point of formation crude oil is verified using a laser solid phase deposition tester.

2. EXPERIMENTAL RESULTS AND DISCUSSION

2.1. Experimental Study on PVT Phase Behavior of Formation Crude Oil. 2.1.1. Single Flash Experiment. Under constant pressure, a single flash test was carried out on formation crude oil samples. Under the conditions of formation temperature of 111.20 °C and dew point pressure above 40 MPa, the single flash test was carried out on the flow body of well YD 702, as shown in Appendix 1. According to the test results of the four components of crude oil, the asphaltene content of well Yudong 702 is 0.75 mol %, the aromatic hydrocarbon content is 16.42 mol %, the colloid content is 6.04 mol %, the saturated hydrocarbon content is 39.04 mol %, and the total yield is 62.25 mol %.

2.1.2. Constant Component Expansion (CCE) Experiment. Under constant temperature, the pressure keeps decreasing, which makes the gas in formation crude oil continuously separate out. Figure 4 shows the relationship between constant expansion pressure and volume of formation oil; test result under agitation.

expansion pressure and volume change of formation oil. At the constant temperature of 111.20 °C, the pressure gradually decreases from 40 MPa. When the pressure decreases to a certain value, the gas dissolved in the gas-containing crude oil separates out; when the pressure is 11.20 MPa, the relative volume shows a trend of rapid increase. When the pressure is 11.20 MPa, the first bubble is generated above the PVT vessel, indicating that the bubble point pressure is 11.20 MPa when the corresponding reservoir temperature is 111.20 °C.

2.1.2.1. Bubble Point Pressure Line Test. It is the dividing line of the gas–liquid two-phase region. This line represents the molar composition of the liquid phase at 100%. When the pressure drops to the bubble point pressure, the first bubbles appear in the system. Figure 5 shows the YD 702 formation crude oil bubble point diagram. In a certain temperature range, with the increase of temperature, the bubble pressure increases continuously, and the bubble point pressure is 11.20 MPa when the reservoir temperature is 111.20 °C. Figure 6 shows the formation crude oil bubble point line compared with the simulated bubble point line. The measured formation oil bubble point pressure at different temperatures is slightly higher than the calculated bubble point pressure, but there is little difference. This is mainly because the measured formation pressure of bubble point through the container pressure fluctuation corresponds to the test fluid pressure of bubble point pressure, and there is a certain lag and error caused by the equipment itself.

2.2. Surface Crude Oil Solid Deposition Experiment. In the solid deposition experiment of surface crude oil in YD 702, a differential thermal scanning test of degassed crude oil was carried out using DSC.\textsuperscript{15} When solid phase particles, such as wax, precipitate from crude oil, the heat release phenomenon occurs. The differential thermal scanner can accurately monitor the change value of heat flow during solid phase heat evolution. When the temperature decreases from the high temperature above the deposition point to the vicinity of the precipitation point, the value of the inflection point when the heat flow rises is the initial deposition temperature of the solid phase. As the system temperature continues to decrease, the components in the crude oil gradually precipitate into solid phase crystals from heavy components to light components, and the cumulative deposition of solid phase in the crude oil gradually increases.\textsuperscript{16–18} Since the precipitation process of solid phase in crude oil is similar to the wax precipitation process, the same method can be used to quantitatively analyze the precipitation process and the amount of wax in crude oil in this study. The calculation steps are as
follows. (1) Determining the cooling rate \( K \), which is set by the experimental instrument, generally \( 5 \, ^\circ C/min \), \( k = 5/60 = 1/12 \, ^\circ C/s \). (2) Selection of the base value: first, we determine the wax precipitation point from the wax precipitation characteristic curve, then take 5–10 points above the wax precipitation point, and calculate the average value of heat flow. This value is the base value. (3) Select the analysis range. Generally, \(-20 \, ^\circ C \sim \) wax precipitation points are selected. First, subtract the average value from the heat flow value of each point to obtain column I, and then the dimension of column I is \( W/g \), which becomes \( J/g \) after multiplying by \( \Delta t \). (4) The \( \Delta t \) calculation method: we observe the temperature on the abscissa and select \( \Delta t \). (5) We multiply column I by \( \Delta t \) to obtain column J, sum up column J to obtain the total heat release, then divide the total heat release by the enthalpy of wax precipitation (generally \( 200\,\sim\,230 \, J/g \)), and then the wax content can be obtained.

Through the experimental test and calculation, the curve of change of instantaneous precipitation of solid phase with decreasing temperature and cumulative deposition of surface crude oil in well YD 702 were obtained, as shown in Figures 7–9. According to the results, the surface oil deposition temperature point is \( 34.05 \, ^\circ C \), the maximum instantaneous precipitation is 0.01178%, and the maximum cumulative precipitation is 8.34%.

2.3. Experimental Study on the Wax Precipitation Point. 2.3.1. Solid Precipitation Point Test: Laser Method. Solid precipitation points are tested using a laser solid phase deposition tester. Through the uniform fluid, the laser almost does not produce fluctuations or light signals. When solid particles are generated in the fluid to block the laser signal from passing through, the optical power received by the laser signal receiver decreases sharply, and the corresponding inflection point is the solid deposition point. Figure 10 shows the solid precipitation curve of formation crude oil at different temper-
formation crude oil. In the process of exploration, the reservoir has a microscopic pore-roar structure, and solid wax is easily adsorbed on the rock surface or stuck at the end of the pore throat. If the wax mud has good fluidity, the rock surface or the pore throat end can fall out under the driving force. With the further decrease of the temperature, the heavy components in the solid phase are further increased, the consistency of wax mud increases, the fluidity becomes worse, and the wax mud gradually accumulates and stays on the surface and the end of the pore throat, reducing the pore permeability of formation and plugging the flow of oil and gas in the wellbore. At the same time, wax precipitates to increase oil consistency, reduce oil fluidity, resulting in oil retention and accumulation, and further reduce formation pore permeability. The wax in the pipeline also narrows the oil and gas seepage channel, which causes pipeline blockage and stops injection and production. Therefore, it is very important to study the solid precipitation point of formation crude oil. Figures 11–16 show the black solid phase spots precipitated in the visualization window when the first batch of solid phase particle precipitates in the crude oil at different temperatures and pressures. The solid phase precipitation temperature points measured in the experiment under the corresponding pressure are consistent with the laser method test results, with only a small deviation.

2.3.2.1. Morphology Change Process of Solid Precipitates. Solid deposition occurs in formation crude oil below the bubble point pressure. At the same pressure, with a decrease of temperature, the morphology of solid phase changes obviously. Taking the pressure of 11.70 MPa as an example, the gas separation and solid phase precipitation can be clearly observed by the visual system as the temperature gradually decreases, as shown in Figures 17–22. When the pressure remains constant and the temperature is 20.2 °C, the precipitate of solid phase in the crude oil can be clearly observed, and black solid phase particle can be seen in the yellow crude oil. When the temperature decreases to \( T = 16.2 \) °C, the black solid phase gradually aggregates and precipitates and increases gradually. When the temperature decreases to \( T = 8.4 \) °C, most of the solid phase in the crude oil precipitates and aggregates, and finally when the temperature decreases to \( T = 5.2 \) °C, the three-phase coexistence of dissolving gas, precipitated solid phase particle, and crude oil can be observed.

2.3.2.2. Phase Envelope Line of Formation Crude Oil. It can be seen from the above study that under different
pressures, the type of solid phase particle precipitated from crude oil is different. When the pressure is higher than the bubble point, solid phase particle precipitates only at low temperatures. When the pressure drops below the bubble point pressure, the light components become less soluble in the liquid phase and the gas is released from the oil, while the solid phase slowly precipitates as the temperature decreases. The bubble point line and the solid precipitation point of crude oil in the above experiment are plotted in the same curve, and the phase enveloping line in well YD 702 is obtained, as shown in Figure 23. The experimental results and simulated bubble point lines were plotted in the same curve, as shown in Figure 24.

### 2.4. Field Application

#### 2.4.1. Wellbore T and P and Phase Behavior of Well YD 702

The sampled depth of PVT phase behavior of YD 702 was 4946.00–4986.00 m. The temperature and pressure gradient data on October 21, 2020, showed that the average temperature gradient and pressure gradient of YD 702 were 1.8567 °C/100 m and 0.6398 MPa/
100 m, respectively. Figures 25 and 26 show the curves of temperature, pressure, and phase behavior for the wellbore depth of 1000 and 1200 m in YD 702. According to the curve, well YD 702 is in the “liquid−solid”, “gas−liquid−solid”, “liquid”, and “gas−liquid” multiphase mixing zone with a well depth of more than 1200 m; therefore, crude oil above 1200 m produces gas separation and solid phase precipitation problems. In the wellbore depth below 1200 m, crude oil is in a single liquid zone; therefore, gas separation and solid precipitation problems do not occur.

2.4.2. Oil Pressure, Back Pressure, Casing Pressure, and Phase Behavior Curve of YD 702. The oil pressure, back pressure, casing pressure, and phase curve of YD 702 were drawn according to the production data of well YD 702 from September 2017 to March 2021, as shown in Figure 27. The oil pressure, casing pressure, and back pressure of YD 702 are mostly located in the gas−liquid−solid zone and the gas− liquid zone; therefore, the solid precipitation of formation crude oil occurs in the wellhead.

3. CONCLUSIONS AND SUGGESTION

In this paper, the formation crude oil of YD 702 was studied using the visualized PVT tester, the visual high-power microscopy tester, and a laser solid phase deposition tester. The experiment is related to the PVT phase behavior and solid phase precipitation law. Based on the production data of well YD 702, we analyzed the solid precipitation from the formation to the wellbore and from the wellbore to the surface. The main conclusions and suggestions are given below.

(1) The bubble point pressure of formation crude oil is 11.2 MPa when the reservoir temperature of YD 702 is 111.20 °C.

(2) The solid precipitation temperature of surface crude oil in YD 702 is 34.05 °C. When the temperature is lower than 34.05°C, at the initial stage of temperature decrease, the instantaneous precipitation of solid phase increases greatly. When the system temperature reaches 16.2 °C, the instantaneous precipitation of solid phase reaches the maximum value of 0.01178%, and then the instantaneous precipitation gradually decreases. At the temperature of −35 °C, 8.34% of solid phase particle was accumulated.

(3) When the pressure is higher than 11.70 MPa, the solid precipitation temperature of formation crude oil gradually decreases with the decrease of pressure. When the pressure is equal to about 11.70 MPa, it produces a turning point. When the pressure is further reduced to 11.70 MPa, the dissolved gas in the formation oil gradually decreases, and the light components in the crude oil decrease, while the proportion of heavy components increases. Therefore, the lower the pressure, the higher the temperature point of solid phase precipitates, and the easier for the solid phase particle to precipitate.

(4) According to the microscopic test of the solid precipitation envelope of formation crude oil, black solid spots can be clearly seen in the visualization window, and the solid precipitation temperature point under the corresponding pressure is consistent with the laser test result. According to the results of solid phase precipitation, the precipitated solids are fine and needlelike in the initial stage, and the solid phase particle is always aggregated in the form of a crystal nucleus and gradually increases, which eventually blocks the oil and gas flow. Therefore, the solid phase particle precipitation should be avoided as far as possible in the actual production process.

(5) The phase behavior test results show that, under the temperature/pressure changes, four phases can be observed, liquid−solid, “liquid phase”, gas−liquid−solid, and gas−liquid, which shows that the formation crude oil presents multiphase changes in the process of temperature/pressure change and not only two liquid or gas−liquid equal forms. This change characteristic also makes it more difficult for us to prevent and control solid phase precipitation.

(6) Combined with the production parameters and phase testing results of YD 702, it can be seen that the solid phase precipitation points of YD 702 are mainly located above 1200 m in the wellbore and in the Christmas tree, and there is basically no solid precipitation in the bottom hole and reservoir. To prevent solid precipitation of crude oil, it is necessary to adjust the wellhead pressure/temperature or manual and mechanical intervention is
required to prevent solid deposition produce, such as wax, paraffin, and colloid, which are not generated when the oil and gas are transported from formation to the wellhead.

4. EXPERIMENT

4.1. Experimental Material. The experimental formation crude oil was obtained from the well depth of 4946.00–49860.00 m, reservoir temperature was 111.20 °C, reservoir pressure was 38.10 MPa, gas–oil ratio was 95 m³/m³, and the well number was YD 702.

4.2. Experimental Equipment. The entire experimental process was completed in a high-temperature and high-pressure visualization system. The experimental devices consist of an injection system, a PVT tester manufactured by France, a differential scanning calorimeter, a flash evaporator, an Agilent 7890A oil/gas chromatograph, a self-developed solid phase deposition instrument, and a laser solid phase deposition instrument manufactured by France, as shown in Figure 28. The experiment consists of three parts: formation crude oil phase experiment, solid deposition experiment of surface crude oil, and solid precipitate point test of formation crude oil. The
Table 1. Formation Crude Oil Composition Analysis Results of Well YD 702\textsuperscript{a,b}

| components | flash oil composition | well fluid composition |
|------------|----------------------|------------------------|
|            | mol % | wt % | mol % | wt % |
| H\textsubscript{2}S | 0.00 | 0.00 | 0.00 | 0.00 |
| N\textsubscript{2} | 0.00 | 0.00 | 2.74 | 1.16 |
| CO\textsubscript{2} | 0.00 | 0.00 | 0.31 | 0.13 |
| C\textsubscript{2} | 0.00 | 0.00 | 67.05 | 28.25 |
| C\textsubscript{3} | 0.00 | 0.00 | 11.73 | 4.94 |
| C\textsubscript{4} | 0.00 | 0.00 | 8.10 | 3.41 |
| C\textsubscript{5} | 0.00 | 0.00 | 3.32 | 1.40 |
| nC\textsubscript{6} | 0.00 | 0.00 | 4.22 | 1.78 |
| iC\textsubscript{6} | 8.39 | 3.89 | 1.45 | 5.46 |
| nC\textsubscript{7} | 4.24 | 1.96 | 0.95 | 2.85 |
| C\textsubscript{7} | 13.73 | 7.41 | 0.12 | 8.00 |
| C\textsubscript{8} | 13.36 | 8.24 | 0.01 | 7.74 |
| C\textsubscript{9} | 9.00 | 6.19 | 0.00 | 5.21 |
| C\textsubscript{10} | 6.62 | 5.14 | 0.00 | 3.83 |
| C\textsubscript{11} | 5.49 | 4.73 | 0.00 | 3.18 |
| C\textsubscript{12} | 4.62 | 4.36 | 0.00 | 2.67 |
| C\textsubscript{13} | 4.28 | 4.43 | 0.00 | 2.48 |
| C\textsubscript{14} | 3.88 | 4.36 | 0.00 | 2.25 |
| C\textsubscript{15} | 3.43 | 4.18 | 0.00 | 1.98 |
| C\textsubscript{16} | 2.73 | 3.62 | 0.00 | 1.58 |
| C\textsubscript{17} | 2.58 | 3.68 | 0.00 | 1.49 |
| C\textsubscript{18} | 2.11 | 3.22 | 0.00 | 1.22 |
| C\textsubscript{19} | 1.85 | 2.99 | 0.00 | 1.00 |
| C\textsubscript{20} | 1.70 | 2.87 | 0.00 | 0.98 |
| C\textsubscript{21} | 1.51 | 2.67 | 0.00 | 0.88 |
| C\textsubscript{22} | 1.35 | 2.52 | 0.00 | 0.78 |
| C\textsubscript{23} | 1.15 | 2.25 | 0.00 | 0.66 |
| C\textsubscript{24} | 1.02 | 2.09 | 0.00 | 0.59 |
| C\textsubscript{25} | 0.85 | 1.81 | 0.00 | 0.49 |
| C\textsubscript{26} | 0.81 | 1.79 | 0.00 | 0.47 |
| C\textsubscript{27} | 0.70 | 1.62 | 0.00 | 0.41 |
| C\textsubscript{28} | 0.65 | 1.56 | 0.00 | 0.38 |
| C\textsubscript{29} | 0.54 | 1.34 | 0.00 | 0.31 |
| C\textsubscript{30} | 0.48 | 1.25 | 0.00 | 0.28 |
| C\textsubscript{31} | 0.41 | 1.09 | 0.00 | 0.24 |
| C\textsubscript{32} | 0.36 | 0.99 | 0.00 | 0.21 |
| C\textsubscript{33} | 0.31 | 0.87 | 0.00 | 0.18 |
| C\textsubscript{34} | 0.27 | 0.80 | 0.00 | 0.16 |
| C\textsubscript{35} | 0.23 | 0.71 | 0.00 | 0.14 |
| C\textsubscript{36} | 0.21 | 0.67 | 0.00 | 0.12 |
| C\textsubscript{37} | 1.13 | 4.69 | 0.00 | 0.66 |
| C\textsubscript{38} | 100.00 | 100.00 | 100.00 | 100.00 |

\textsuperscript{a}C\textsubscript{36}+ molecular weight, 645. \textsuperscript{b}C\textsubscript{36}+ density, 0.9375 g/cm\textsuperscript{3}.

Core devices are high-temperature and high-pressure PVT tester, solid phase deposition tester, and a laser solid phase deposition tester.

4.2.1. High-Pressure Fluid PVT Instrument. Phase behavior was studied using the visual multifunctional high-temperature and high-pressure PVT tester, manufactured by France. It can be used to observe the bubble point and the dew point pressure of formation fluid, as well as expansion test, flash test, differential degassing test, and constant volume depletion test.\textsuperscript{14} The device is mainly composed of a visualization window, monitoring system, temperature control system, and a compression pressure-resistant kettle body, as shown in Figures 29 and 30. The maximum working pressure is 100 MPa, the maximum working temperature is 200 °C, PVT kettle volume is 240 mL, and visualization; the PVT reactor has a sapphire window with a diameter of 60 mm, the test accuracy pressure of ±0.01 MPa, volume ±0.0005 mL, and temperature of ±0.1 °C.

4.2.2. Solid Phase Deposition Instrument. 4.2.2.1. Microscopic Solid Deposition Tester. A high-magnification microscopic visualized solid phase deposition tester was used to determine the solid phase precipitation in the process of the gas-containing crude oil changing with pressure and temperature. Solid phase precipitation temperature, solid phase formation temperature, and solid phase formation shape and size of formation crude oil can be observed; therefore, the experimental results can be directly observed. The device mainly consists of a visualization window, a monitoring system, a temperature control system, and a compression pressure-resistant kettle body, as shown in Figure 31.
The working pressure range of the visual solid deposition tester is 0−150 MPa, the working temperature range is −30−200 °C, the volume of the kettle is 300 mL, and the solid phase size can be observed by the visual sapphire window of 1−4 μm. The main steps of the wax precipitation point test for formation crude oil are as follows. (1) The sample of formation crude oil is transferred into the reactor, and the sample is stirred evenly under the formation pressure and balanced at the formation temperature for 4 h. (2) After the system is stabilized, the experimental pressure is kept constant and the system temperature is slowly reduced at a rate of 0.5−1.0 °C/min. During the experiment, the samples are always fully agitated to ensure that the system is in a state of equilibrium. As the pressure continues to drop during the cooling process of the system, it is necessary to set the pump to a constant pressure mode and supplement the pressure at any time. The system pressure was kept constant, the wax crystals generated in the system were observed through a microscopic visualization window, and the corresponding temperature and pressure of the first solid deposition in the system were observed and recorded, namely, the temperature of the solid deposition point under the test pressure. (3) After testing the temperature of the solid deposition point at one pressure, the system increases the pressure again and stirs into a single-phase behavior, and then the temperature of the solid deposition point at the next pressure point is tested. (4) The above steps to measure the temperature of solid deposition points corresponding to different pressure points are repeated until the end of the experiment, and the relationship curve of solid deposition points (P−T) is drawn. The test of pressure and temperature of solid deposition points should be within the limit range of the equipment (Table 1).

4.2.2.2. Laser Solid Phase Deposition Tester. To further verify whether the precipitation points of solid phase deposition measured by the microscopic method are accurate, the wax deposition points in the process of formation crude oil changing with pressure and temperature were measured by the laser method. Figure 32 shows the French ST company manufacturing laser method solid phase deposition tester (Photograph courtesy of Jie Wang. Copyright 2021).

Figure 32. French ST company manufacturing laser method solid phase deposition tester (Photograph courtesy of Jie Wang. Copyright 2021).

Figure 33. P = 0.1 MPa and deposition temperature point = 21.3 °C.

Figure 34. P = 2.0 MPa and deposition temperature point = 20.6 °C.

Figure 35. P = 4.0 MPa and deposition temperature point = 20.2 °C.

Figure 36. P = 6.0 MPa and deposition temperature point = 19.5 °C.
laser solid phase deposition test system manufactured by France. The core components of the device are a high-pressure vessel with visualization windows on both sides and a laser test device. The laser tester is mainly composed of a laser light source, optical fiber, and a laser signal detector. The laser light source and laser intensity detector are, respectively, arranged on both sides of the high-pressure vessel. The laser light emitted by the laser source can pass through the high-pressure vessel window through the system to be tested, as shown in Figure 32. The performance index of the solid phase deposition tester is the highest working pressure of 70 MPa and the pressure resolution of 0.01 MPa; the maximum operating temperature is 200 °C, and the temperature resolution is 0.1 °C. The maximum sample volume of the PVT cylinder is 130 mL, and the volume resolution is 0.01 mL (Figures 33−42).

APPENDIX 1. FORMATION CRUDE OIL COMPOSITION ANALYSIS RESULTS OF WELL YD 702

APPENDIX 2. LASER TEST CURVE OF EACH SOLID PHASE DEPOSITION POINT

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Figure 37. P = 8.0 MPa and deposition temperature point = 18.9 °C.

Figure 38. P = 11.7 MPa and deposition temperature point = 18.4 °C.

Figure 39. P = 16.0 MPa and deposition temperature point = 19.0 °C.

Figure 40. P = 34.1 MPa and deposition temperature point = 21.2 °C.

Figure 41. P = 36.4 MPa and deposition temperature point = 21.9 °C.

Figure 42. P = 38.5 MPa and deposition temperature point = 22.4 °C.
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Notes
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REFERENCES

(1) Pelet, R.; Behar, F.; Monin, J. C. Resins and asphaltenes in the generation and migration of petroleum. Org. Geochem. 1986, 10, 481–498.
(2) Chu, Y. P.; Hu, Y. F.; Ming, Y. F.; Guo, T. M. Experimental study on asphalten precipitation mechanism and law during high pressure gas injection. J. Univ. Pet. 2003, 4, 74–77.
(3) Colin, J. M.; Boulet, R.; Escalier, J. C. Characterization of Heavy Crude Oils and Petroleum Residues; Fuel Science and Technology International: USA, 1989; Vol. 7.
(4) Kirtley, S. M.; Mullins, O. C.; van Elp, J.; et al. Nitrogen chemical structure in petroleum asphaltene and coal by X-ray absorption spectroscopy. Fuel 1993, 72, 133–135.
(5) Schwager, I. Characterization of the microstructure and macrostructure of coal-derived by nuclear magnetic resonance spectrometry and X-ray diffraction. Anal. Chem. 1983, 55, 42–45.
(6) Zaki, N. S.; Barbootti, M. M.; Baha-Uddin, S. S.; et al. Determination of trace metals and their distribution in heavy crude oil distillates (350°C) by atomic absorption spectrophotometry. Appl. Spectrosc. 1989, 43, 1257–1259.
(7) Benkhedda, Z.; Landais, P.; Kister, J.; et al. Spectroscopic analysis of aromatic hydrocarbons extracted from naturally and artificially matured coals. Energy Fuels 1992, 6, 166–172.
(8) McKay, J. F.; Latham, D. R. Fluorescence spectrometry in the characterisation of high-boiling petroleum distillates. Anal. Chem. 1972, 44, 2132–2137.
(9) Poveda, J. C.; Molina, D. R. Average molecular parameters of heavy crude oils and their fractions using NMR spectroscopy. J. Pet. Sci. Eng. 2012, 84–85, 1–7.
(10) Yen, T. F.; Erdman, J. G.; Pollack, S. S. Investigation of the structure of petroleum asphaltenes by X-ray diffraction. Anal. Chem. 1961, 33, 1587–1594.
(11) Kelemen, S. R.; George, G. N.; Gorbaty, M. L. Direct determination and quantification of sulphur forms in heavy petroleum and coals: 1. The X-ray photoelectron spectroscopy (XPS) approach. Fuel 1990, 69, 939–944.
(12) Peng, P.; Fu, J.; Sheng, G.; et al. Ruthenium-iron-catalyzed oxidation of an immature asphaltene: Structure features and biomarker distribution. Energy Fuels 1999, 13, 266–277.
(13) Chen, C. Observation of the type of hydrogen bonds in coal by FTIR. Energy Fuels 1998, 12, 446–449.
(14) John, R. F. Petroleum Engineering Handbook, Volume I: General Engineering; Society of Petroleum Engineers, 2006.
(15) Coto, B.; Martos, C.; Espada, J. J.; et al. Analysis of paraffin precipitation from petroleum mixtures by means of DSC: Iterative procedure considering solid–liquid equilibrium equations. Fuel 2010, 89, 1087–1094.
(16) Alan, S. S.; Singh, K.; Mohamed, A. S. et al. In Organic Deposition: From Detection and Laboratory Analysis to Treatment and Removal, SPE Asia Pacific Oil and Gas Conference and Exhibition; OnePetro, 2013.
(17) Uetani, T. In Wettability Alteration by Asphaltene Deposition: A Field Example, Abu Dhabi International Petroleum Exhibition and Conference; OnePetro, 2014.
(18) Wang, S.; Civan, F. In Productivity Decline of Vertical and Horizontal Wells by Asphaltene Deposition in Petroleum Reservoirs, SPE International Symposium on Oilfield Chemistry; Society of Petroleum Engineers, 2001.
(19) Burke, N. E.; Hobbs, R. E.; Kashou, S. F. Measurement and modeling of asphaltene precipitation. J. Pet. Technol. 1990, 42, 1440–1446.
(20) Leontaritis, K. J. In Asphaltene Deposition: A Comprehensive Description of Problem Manifestations and Modeling Approaches, SPE Production Operations Symposium; OnePetro, 1989.
(21) Yen, T. F.; Wu, W. H.; Chilingar, G. V. A study of the structure of petroleum asphaltenes and related substances by infrared spectroscopy. Energy Sources 1984, 7, 203–235.
(22) Zhou, X.; Thomas, F. B.; Moore, R. G. Modelling of solid precipitation from reservoir fluid. J. Can. Pet. Technol. 1996, 35, No. 3.