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Influencing factor analysis of shale micro-indentation measurement

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ABSTRACT

To address the difficulty in measuring mechanical properties within the reservoir formation during the process of drilling in shale formations, micro-indentation measurement technology is performed to determine the micro elastic modulus and the indentation hardness of a shale outcrop in the Longmaxi Formation in the Sichuan Basin (the Changning Area). Additionally, factors influencing the micro-indentation measurement are analyzed from aspects such as indenter defects, experimental parameter settings, shale surface properties, and shale composition. The results show that the elastic modulus and indentation hardness decrease with increasing indentation depth. The test is less affected by indenter passivation. The area function needs to be calibrated when the tip radius of the indenter is more than 10 μm. It is observed that the test is substantially influenced by the parameter settings, so reasonable parameters should be determined before conducting the test. The Oliver-Pharr method is applicable to shale outcrops of the Longmaxi Formation, which meet the general requirements of the method. The measured values increase with increasing packing density. Moreover, the fitting percentage of the unloading curve has an impact on the result, and the reasonable fitting percentage of the unloading curve for shale samples was determined to be from 45% to 75%.

1. Introduction

As a type of porous medium, shale is a diverse sedimentary rock that contains a variety of clay minerals, quartz, silicates and other non-clay minerals. The mechanical properties within different regions differentiate because of anisotropic effects that cause wellbore instability, and they have a strong influence on the supporting effect of the proppant used in hydraulic fracturing (Sihua et al., 2011; Yuman et al., 2012). At present, the research on the physical and mechanical properties mainly addresses the established relationship between physical properties and mechanical properties. Macro-based measurements have problems such as large sample volume, time-consuming experiments, and an unsatisfactory level of detail of the characterization of the target formation. The structure of shale results in the difficulty in obtaining unbroken shale cores in the horizontal section of a well. Conditioned logging instruments cannot be lowered to the target formation, which results in low-quality logging materials and measuring errors of mechanical parameters.

With the development of new technology, micro-mechanical measuring technology has been applied in many fields. The micro elastic modulus (E) and micro-indentation hardness (H), as important mechanical parameters, are obtained using nano-indentation tests (Oliver and Pharr, 1992). This technology can estimate the mechanical properties of materials for research on the material surface (Yang et al., 2008). As a functional index of the material characterization, indentation hardness is the overall response of material mechanics on local regions, and it attracts widespread attention. It has been demonstrated that the nano-granular mechanical behavior is related to packing density (Ulm et al., 2007). The shale model of the relationships between mechanical behavior and clay packing density were constructed (Bobko and Ulm, 2008; Ortega et al., 2010). The model provided a foundation of research on the mechanical behavior of shale on a nano scale. Based on the theoretical analysis, the dimensional functional relationships between the indentation hardness and cohesion were established (Cariou et al., 2008). According to homogenization theory, the nano-granular strength properties of shale were estimated (Bobko et al., 2011; Ortega and Ulm, 2013). Using dimensional analysis, dimensionless functions were constructed to characterize the relationship between composite and nanomechanical properties. Kumar et al. (2012) claimed that nano-indentation technology was an attractive method for implementation on the drill cuttings of shale. The shale mechanical

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behavior of nano-indentation had also been studied by other researchers (Constantinides and Ulm, 2007; Ulm et al., 2010; Deirieh et al., 2012; Mason, 2014; Eliyahu et al., 2015; Bennett et al., 2015).

However, little has been comprehensively reported on the influence factor analysis of shale micro-indentation. In contrast with nano-indentation testing, the micro-indentation tester can be directly applied in the engineering field, and the test environment of the micro-indentation is simpler. The test is determined by factors such as the instrument precision, experimental method, material properties, and mathematical analysis model. In this work, the micro-indentation of shale was performed and analyzed. The influence factors of micro-indentation of shale are estimated from aspects such as indentation load and depth, reliability of mechanical parameters, indentation range and the surface properties of shales. Ten samples from a shale outcrop are derived from the Longmaxi Formation in the Sichuan Basin (the Changning area).

2. Experiment and results

2.1. Experiment

The MFT-4000 multifunctional material performance tester is a commercial and standard set-up. The tester, based on micro-displacement measurement technology and microcomputer control technology, provides a continuous detection of the load on the indenter and the indentation depth during both loading and unloading. The tester is produced by the Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. A variety of test modules are integrated into the MFT-4000, such as indentation, scratch, reciprocating friction, and roughness detection. In this work, the indentation module parameters are as follows: the loading range is from 0.5 N to 300 N, the loading precision can reach 0.5 N, the force loading rate is from 5 N/min to 100 N/min, and the indentation displacement range is from 0.5 μm to 200 μm. Ten shale samples, originating from the Longmaxi Formation outcrop in the Changning area in China, were selected for the indentation study. The sample size was 50 mm × 25 mm × 15 mm, and the surfaces were polished by mechanical buffing at room temperature. The shales were dried at 65 °C for 12 h and stored in sealed bags.

Indentation tests were performed on the surfaces of the shale samples. The micromechanical response of a random indentation on the surface is not necessarily representative of the overall response. Grid indentation technology is a tool that provides a means of obtaining a statistical average. Additionally, all of the grid indentation points were normal to the bedding plane in the experiment. The test temperature remained at 24 °C ± 2 °C. A Berkovich indenter was selected for the experiment.

The test procedure is composed of three phases: the loading phase, the holding phase and the unloading phase. First, the indenter tip comes into contact with the shale surface without interaction force. Once the indentation test begins, the force is loaded at a constant rate. When the maximum load is reached, this load is held for 15 s before the load decreases at a constant rate. Then, the indenter is retracted and moved to the next test position. In this paper, the number of grid indentation points is 20 on each shale surface.

2.2. Results

The micromechanical behaviors of shale are evaluated. First, the valid data of load–displacement are obtained by the indentation-measuring instrument. Then, the microscopic elastic modulus and the indentation hardness are evaluated by the methodology of Oliver-Pharr. Fig. 1 shows the box chart of the micro elastic modulus of ten shales. The overall change is from 2.4 GPa to 31.6 GPa. The distribution deviation from 25% to 75% is less than 8 GPa. Fig. 2 shows the box chart of the micro-indentation hardness of the 10 shale samples. The overall change is from 0.07 GPa to 3.6 GPa. The distribution deviation from 25% to 75% is less than 1.2 GPa. It is clear that the modulus distribution is slightly more scattered than the hardness distribution.

3. Influence factors

3.1. Indenter tip radius

As a key component of the instrument, the indenter is one of the important factors influencing the micro/nano-indentation test. In the calculation, the indenter tip is usually regarded as an ideal sharp indenter. In fact, the indenter tip has a radius that is more blunt than that of an ideal indenter (Shih et al., 1991). The geometry of the indenter tip determines the functional relationship between the contact area and the indentation depth. For the Berkovich indenter, the actual contact area is larger than the ideal contact area. This phenomenon results in a higher indentation hardness in the shallow indentation test (Chen et al., 2007; Torres-Torres et al., 2010). The qualitative analysis of ideal/non-ideal cone indenters shows that when the indentation depth is 10 nm, the actual contact area is 10 times as large as the ideal contact area; when the indentation depth reaches 100 nm, the actual contact area is 1.2 times as large as the ideal contact area (Shih et al., 1991). To reduce the deviation, the contact area should be calibrated.

An iterative method is therefore proposed to calibrate the contact area by Oliver and Pharr, and this method is quite complicated and time consuming (Pharr et al., 1992). Liu Dongxu proposed a simple method for finite element analysis (Liu and Zhang, 2004). The combination of a sphere and a cone, as the harmonic average, is a good substitute for the profile of the indenter tip. When the indentation depth is less than the height of the spherical cap, the contact area function can be written as:

$$A = -\pi h_c^2 + 2\pi h_c R \tag{1}$$

where A is the contact area (μm^2), h_c is the contact depth (μm), and R is the radius of the spherical cap (μm).

When the indentation depth is greater than the height of the spherical cap, the contact area function can be rewritten as:

$$A_c = \pi a^2 = \pi\left[\frac{h_c}{\cot\theta} + R\left(\frac{1-\sin\theta}{\cos\theta}\right)\right]^2 \tag{2}$$

where a is indentation radius (μm), and θ is the equivalent semi-conical angle (degrees).

The contact indentation depth is typically written as:

$$h_c = h_m - \varepsilon\frac{F_m}{S} \tag{3}$$

where ε is the indenter constant (dimensionless), F_m is the maximum force (N), h_m is the maximum depth (μm), and S is the contact stiffness (N/μm).

Using the calibrated method from Equations (1)–(3), shale micro-indentation tests of different indenter radii are evaluated. Fig. 3 shows the relationship between the elastic modulus ratio (E/E_0) and the contact depth (h_c), where E is the test modulus with an indenter tip radius and E_0 is the ideal modulus without a tip radius. It is noted that the test results can be affected by the indenter radius to a certain extent. When the indenter tip radius (R) is 0.1 μm, the elastic modulus ratio ($E_{R=0.1\ \mu m}/E_0$) is close to 1 and has almost no