INTERNATIONAL JOURNAL OF HYDROGEN ENERGY XXX (2017) 1–10

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.elsevier.com/locate/he

A comparative experiment investigate of strength parameters for Longmaxi shale at the macro- and mesoscales

Guangjian Dong [a,b,*], *Ping Chen* [a,b,**]

[a] *State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, Sichuan 610500, China*

[b] *College of Oil and Gas Engineering, Southwest Petroleum University, Chengdu 610500, China*

ARTICLE INFO

Article history:
Received 13 April 2017
Received in revised form
28 May 2017
Accepted 31 May 2017
Available online xxx

Keywords:
Hydrogen gas storage
Indentation experiment
Macroscale
Mesoscale
Shale

ABSTRACT

The strength parameters of rock is important to the storage and development of hydrogen gas and natural gas. The rock strength parameters is the basis of the establishment of storage space and wellbore, especially for the shale formation. However, the strength parameters and indentation mechanism of shale are unclear. It is necessary to research the strength parameters and dynamic indentation of shale from the multiscale perspective. A comparative experimental study of shale indentation at the macro- and mesoscales was conducted. The experimental investigate determined the four stages of shale indentation at the mesoscale. With increasing loading rate at the same load value, indentation depth decreases, and the critical fracture load of shale increases. The result shows that the indentation load can improve the rate of penetration, but the shale strength increases slightly at the same time, which suppresses the energy release of the shale fragmentation. The probability of indentation fragmentation occurs 2–3 times at the mesoscales is greater than 4 times the value at the macroscale. The macro averages of shale hardness and elastic modulus are close to the mesoscale values. The differences are 7% and 5.5%, respectively. The study results can be used in the storage and development of hydrogen gas and the optimization design of drilling tools.

Introduction

The strength parameters of rock is important to the storage and development of hydrogen gas and natural gas [1]. Mechanical parameters of rocks, especially unconfining compression strength (UCS), elastic modulus and hardness, are the basis of storage space, wellbore, rate of penetration (ROP) and rock fragmentation mechanism [2–8]. Several measurement methods are used to obtain rock mechanical parameters, which include standard rock testing [9,10], acoustic logging [11], wave velocity method [12], indentation testing [13] and reconstructed core samples [14]. However, based on the above methods, indentation testing is still a

* *Corresponding author*. State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, Sichuan 610500, China.

** *Corresponding author*. College of Oil and Gas Engineering, Southwest Petroleum University, Chengdu 610500, China.

E-mail addresses: rocdgj@hotmail.com (G. Dong), chenping@swpu.edu.cn (P. Chen).

http://dx.doi.org/10.1016/j.ijhydene.2017.05.240

0360-3199/

convenient and cheap method. Load-depth curve of indentation testing can obtain the critical fracture force, elastic modulus and hardness from the rock and drill cuttings [15,16]. Several aspects related to theoretical and important factors of indentation testing were studied. Indenter geometry is one of the most important factors that directly affect the stress distribution of rocks and broken response mechanism [17–20]. Laboratory experiments using flat, wedge, spherical and cylindrical indenters penetrating a variety of rock types were performed [21,22]. The flat-end indenter diameter is from 1 mm to 20 mm, spherical indenter diameter is 1–2 mm, and is widely used in the indentation experiment at macroscale. Penetration rate (PR) affects the rock mechanical strength, and PR is from 0.15 mm/s to 0.01 mm/s, the strength parameters of rock was proportional to the PR at the macroscale experiments [11,23]. The indentation testing may be affected by the rock sample size. Dimensions of rock specimens were variable, which is from 2 to 50 mm. There are three typical distinct zones: a zone of crushed rock (the crushed zone); a zone of intense micro- and macro-cracking that engulfs the crushed zone (inelastic, cracked zone and elastic zone), where cracks cannot be observed; and a zone that engulfs the inelastic and cracked zones. Above the results, the important research mainly forces on the macroscale. Indentation index is an important indicators to predict the UCS of rock, which were investigated by a variety of test procedures and indentation testing. Indentation index can show the relationship among rock hardness, UCS, elastic modulus and sample size [23–31].

Many macro- and microscale research results of rock indentation have been carried out. However, the heterogeneity and anisotropy of rock increase the difficulty and workload of rock mechanics experiments, especially for shale. It is difficult to design and predict the rock strength parameters under the coupled dynamic loading, and many key problems involving the rock-breaking mechanism remain unresolved. This paper focuses on the macroscopic and mesoscale indentation mechanism of anisotropic shale (Longmaxi shale) and pays attention to the influence of the multiscale. The study results can be used in the storage and development of hydrogen gas and the optimization design of drilling tools.

The experiment principles

Mesoscale experimental principles

The mesoscale indentation experiment apparatus is a surface properties tester of multifunctional materials (MFT-4000). The instrument can complete micron-grade rock performance testing. The composition of the experimental apparatus is shown in Fig. 1. The instrument have the automatic calibration module of displacement and load. The loading range is 0.5–300 N, loading accuracy is 0.5 N, loading rate is 20–100 N/min, measurement range and indentation depth are 0.5–100 μm, indenter taper angle is 120°, resolution is 0.1 μm [25,26].

The indenter acts on the sample surface. According to the loading rate, the displacement sensor and load sensor, the rock loading–depth curve and unloading–depth curve are obtained, which are used to calculate the elastic modulus and hardness of rock, as shown in Fig. 2. The key problem of mesoscale experiment is the determination of maximum load, maximum displacement and loading rate.

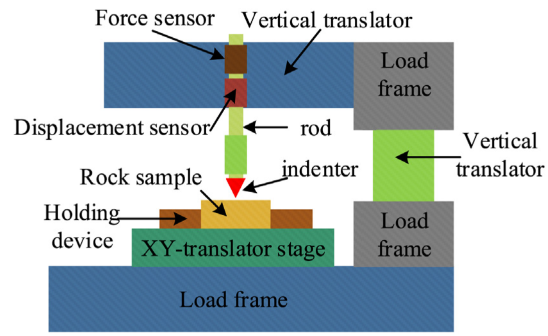

Fig. 1 – Schematic diagram of mesoscale indentation apparatus.

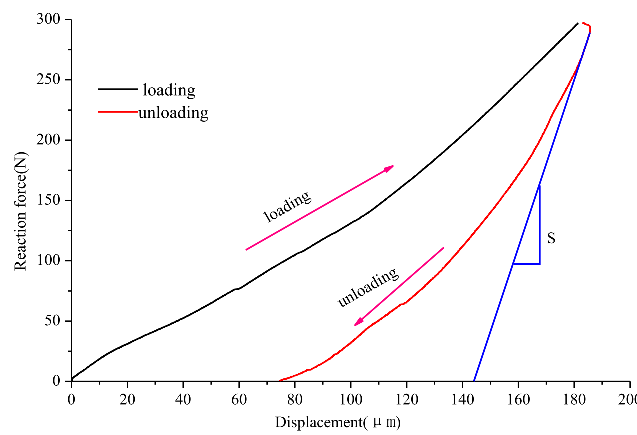

Fig. 2 – Schematic diagram of mesoscale indentation experiment.

The Oliver–Pharr method is modified and confirmed based on many experiments and has become the international standard method. Cheng modified the Oliver–Pharr method [27,28].

The calculation equation of shale elastic modulus is as follows

$$E_r \approx \frac{\pi}{20\beta^2}\frac{W_u}{W_t}\frac{S^2}{F_m} \tag{1}$$

The calculation equation of shale hardness is as follows

$$H_{IT} \approx \frac{\pi}{100\beta^2}\left(\frac{W_u}{W_t}\right)^2\frac{S^2}{F_m} \tag{2}$$

where

$$S = \left.\frac{dF}{dh}\right|_{h=h_m} = Bb\left(h_m - h_f\right)^{b-1} \tag{3}$$

where B, b, h_f are obtained by the least squares fitting coefficient; W_u is unloading work (J); W_t is total work (J); F_m is unloading maximum load (kN); β is indenter shape coefficient; E_r is elastic modulus (GPa); S is contact stiffness (N/μm); H_{IT} is indentation hardness (GPa). This paper take this method calculate the elastic modulus and hardness of shale.