# THERMAL CRACKING AND CORRESPONDING PERMEABILITY OF FUSHUN OIL SHALE

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In this work, thermal cracking inside an oil shale sample under different temperatures was tested using a micro-CT system. The critical temperature was determined at which sharp thermal cracking in the oil shale sample occurs. It was found that the critical temperature of Chinese Fushun oil shale is 350 °C. After reaching 350 °C, all sites of the sample reveal fissures whose number, length and width increase dramatically, resulting in the formation of an enormous network of fissures. A high-temperature rock-permeability testing machine was used to test the changes in permeability at different temperatures. The results similar to those of thermal cracking were obtained. Therefore, thermal cracking can be regarded as the decisive factor to affect the changes of permeability in oil shale particle.

#### Introduction

As the demand for energy is greatly increasing throughout the world, oil shale, regarded as a potential energy source to substitute for oil and natural gas, has attracted researchers' attention for many years. There is a considerable amount of oil shale deposits in China, which rank the second in the world. The amount of oil shale in China is estimated to be 719.9 billion tons, and that of shale oil is 47.6 billion tons [1–5].

It is known that *in situ* shale oil recovery technology is an environmentally friendly method. In *in situ* process, the heating equipment is installed into an oil shale stratum, and the heat causes the organic matter to be pyrolized to form shale oil and hydrocarbon gas. Shale oil and gas are collected for the separating treatment [6–9]. The *in situ* shale oil recovery technology does not require large-scale mining and equipment for dealing with exhaust gases. It also has other advantages, such as the ability to pro-

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cess oil shale deep in the earth. With high efficiency and little pollution, however, this technology is still in the stage of pilot demonstration.

At room temperature oil shale is compact, with poor permeability. Thermal cracking occurs when oil shale undergoes high temperatures, which leads to asymmetric expansion of the mineral particles, thus changing the corresponding permeability. At the same time the fissures act as the main channels for the seepage of the heated oil and gas, at which the connectivity of the fissures directly controls the success of *in situ* shale oil recovery. Therefore, the work for optimal thermal cracking at different temperatures and the analysis of the relevant rules of permeability will be important for the development of *in situ* shale oil recovery technology.

The technological and theoretical research in the *in situ* heating process for oil shale has been carried out for many years in the Taiyuan University of Technology [10, 11]. By using the micro-CT experimental system (µm grade), the thermal cracking process is observed and analyzed when heating the Fushun oil shale from 20 °C to 600 °C. Furthermore, the formation, evolution and transfixion of fissures under different temperatures were studied. By these methods the critical temperature for thermal cracking and the corresponding changes in permeability of oil shale are determined. Test results also give us some insight into thermal cracking and the permeability change in the process of oil shale pyrolysis.

# **Experimental**

# The preparation of oil shale sample and CT test

CT testing system consists mainly of an X-ray machine, a digital plane detector, a high-precision revolving stage, a horizontal movement device, and a base seat, jig and other structural components, as shown in Fig. 1. The configuration of the interior fissures of the rock sample can be clearly observed by CT technology.

Oil shale samples were taken from Fushun deposit in China. The color of the initial sample was dark brown, its oil content 8.2%. The sample was trimmed to a cube of 7.0 mm×7.0 mm×7.0 mm, as displayed in Fig. 2. The sample was first CT-scanned at room temperature, and then put into the furnace. After the temperature of the sample reached 100 °C, the temperature was held for one hour. Thereafter the sample was cooled down to room temperature and the CT scanning was performed. Following the same fashion, with a temperature interval of 100 °C, the sample was heated at a high temperature, cooled down, and then scanned by the CT machine. The highest temperature tested in this research was 600 °C.



Fig. 1. Micro-CT experimental system.

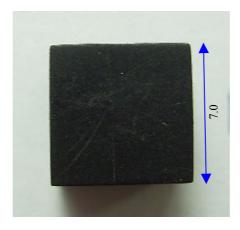


Fig. 2. Cube sample of Fushun oil shale (mm).

# The high-temperature permeability test of oil shale

By using the high-temperature rock-permeability testing machine designed at the Taiyuan University of Technology, the permeability coefficients of oil shale at different temperatures can be measured. This machine (displayed in Fig. 3) consists mainly of systems of axial and lateral pressure, heating, seal-

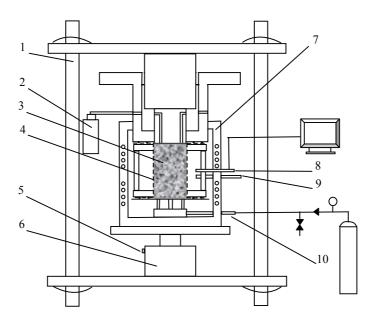


Fig. 3. Block diagram of high-temperature rock-permeability testing system. 1 – framework; 2 – gas-collection instrument; 3 – oil shale sample; 4 – high-temperature alloy seal jacket; 5 – oil entrance; 6 – axial actuator; 7 – electric furnace; 8 – inlet of confining fluid; 9 – thermocouple; 10 – inlet of pore fluid.

ing and gas input, respectively. The seepage gas is N<sub>2</sub>. By Darcy's law, the permeability coefficient is determined as

$$K = \frac{QL\rho g}{A(p_1 - p_2)},$$

where K is permeability coefficient, Q – the discharge volume of gas, L – the length of the sample,  $\rho$  – gas density, g – acceleration of gravity, A – the cross-sectional area of the sample, and  $p_1$  and  $p_2$  – gas pressure at the inlet and outlet, respectively.

The Fushun oil shale sample was trimmed into a cylinder with a diameter of 5 cm and a height of 10 cm. The test procedures include three steps. Firstly, the sample is wrapped with alloys resistant to high temperatures. Secondly, it is loaded in a cell with triaxial pressures at room temperature. Thirdly, by setting surrounding and axial pressures to measure the pore pressures, the permeability coefficient can be obtained. The temperature is increased to 50 °C and maintained for one hour. The permeability test is performed and the permeability coefficient is calculated. Taking 50 °C as the temperature interval, and following the same procedure, the permeability coefficient is obtained for each temperature until the temperature reaches 500 °C. Finally, the sample is cooled down to room temperature and removed from the cell. Figure 4 shows the comparison of the samples before

and after testing. It can be seen that the original oil shale sample is highly compact, with no fissures on the surfaces, whereas at a high temperature fissures are subparallel along the direction of deposition after undergoing heating.



Fig. 4. Comparison of an oil shale sample before and after the permeability experiment.

## Results and discussion

#### Thermal cracking of oil shale

Figure 5 shows the images of the thermal cracking evolution in the oil shale sample under different temperatures from 20 °C to 600 °C. At a magnification of 37 times, the fissures whose width is larger than 5  $\mu m$  can be clearly seen. As shown in Fig. 5, the process of thermal cracking can be divided into two stages: 20 °C - 300 °C and 300 °C - 600 °C. The two stages are described as follows:

The first stage:  $20 \, ^{\circ}\text{C} - 300 \, ^{\circ}\text{C}$ 

No obvious fissures in the oil shale are found at room temperature. At 100 °C, a large fissure along the depositional bedding direction appears on the left side of the sample, whereas no obvious fissures are observed on the other sides. Apparently the original clayey cementation between bedding planes is very weak and easy to be broken with small changes in thermal stress. The rupture extends further along the bedding planes to form a large-scale fissure. When the temperature increases from 200 °C to 300 °C, a few micro-fissures are initiated from the edges of hard mineral particles (pointed by arrows in Fig. 5). It is clear that the new fissures of different sizes occur parallel to the bedding planes. As thermal characteristics of hard mineral particles and ambient rock particles differ, thermal stress focuses on the

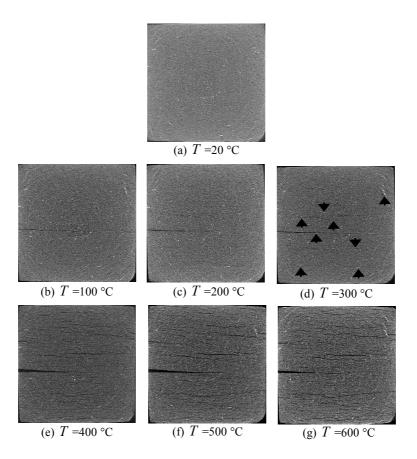


Fig. 5. CT sections showing thermal cracking of oil shale under different temperatures.

edges of hard particles, resulting in thermal cracking. Thus these micro-fissures can extend along the bedding direction with little resistance.

The second stage: 300 °C -600 °C

As the temperature increases from 300 °C to 600 °C, thermal cracking in the interior of oil shale occurs, especially when the temperature increases from 300 °C to 400 °C. Fissure number, length and width increase dramatically in the sample. Most of them are subparallel and relatively straight. This is due to the organic mater pyrolyzed at higher temperatures, resulting in production of a large amount of oil and gas from oil shale in the form of CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>, CO<sub>2</sub>, H<sub>2</sub>, CO and H<sub>2</sub>S. In addition, after being heated to higher temperatures, shale oil and gas can spew out at a high speed. The corresponding volume will expand synchronously, leading to the formation of localized high-pressure zones in oil shale. When the pressure exceeds the tensile strength of oil shale, new fissures with increased lengths and widths will form. Therefore, the factor controlling thermal cracking in oil shale is the pyrolysis

reaction at temperatures of 300 °C – 600 °C, which causes impermeable oil shale to become relatively permeable, containing a large number of fissures.

By analyzing the number of the fissures with lengths greater than 0.2 mm in CT images at different temperatures (as shown in Fig. 6), it is clear that a sudden increase in the fissures occurs when the temperature is about 300 °C. If the temperature is less than 300 °C, the fissures increase slowly, and the curve is relatively flat. Thus, for the sample in this work, the critical temperature for thermal cracking is estimated to be 300 °C. On the other hand, the number of fissures reaches a plateau when the temperature is at 500 °C. A decrease is observed within the temperature range from 500 °C to 600 °C. This decrease in fissure number can be regarded as the stage at which a connected fissure is forming, as shown in Fig. 7.

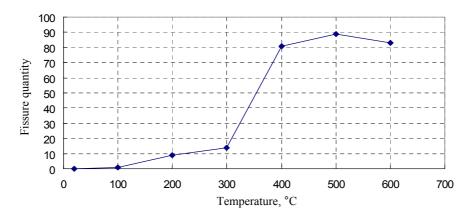


Fig. 6. Relation between temperature and fissure quantity.

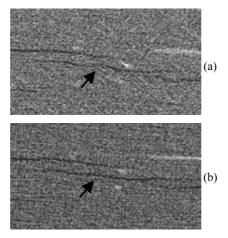


Fig. 7. Connection of fissures at high temperatures. (a)  $T = 400 \,^{\circ}\text{C}$  (b)  $T = 500 \,^{\circ}\text{C}$ 

# Permeability of oil shale sample

Figure 8 shows the change of the permeability coefficient of oil shale with temperature. It is obvious that with the increase of temperature the permeability coefficient increases very slowly before 350 °C, while the permeability coefficient increases sharply after reaching 350 °C. Therefore, 350 °C can be considered the critical temperature for the change in permeability of the Fushun oil shale. The maximum permeability coefficient was obtained at the temperature of 500 °C. Compared to the initial value of  $0.008 \times 10^{-3}$  cm/s, the coefficient increases to  $2.040 \times 10^{-3}$  cm/s, which turns the oil shale from a low-permeable medium to a much more permeable medium. The latter provides a smooth pathway for the evolution of shale oil and gas during *in situ* shale oil recovery.

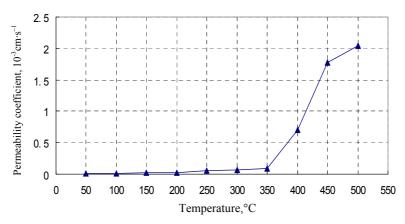


Fig. 8. Relation between temperature and permeability coefficient.

## The correlation between thermal cracking and permeability

By combining the data shown in Figs. 6 and 8, Fig. 9 shows the dependence of both thermal cracking and permeability at different temperatures. It is clear that two curves have a similar tendency. From 20 °C to 350 °C, thermal cracking of oil shale is not obvious, the number of fissures is low, accompanied by narrowly dispersive characteristics in fissure orientation and little connected networks.

As a result, thermal cracking makes tiny contribution toward increasing the permeability of oil shale; and the permeability coefficient increases very slowly with increasing temperature. Starting from 350 °C, however, thermal cracking of oil shale becomes very active, resulting in the instant formation of a large number of new fissures with serried distribution. As shown in Fig. 10, the length and width of the original fissures are dramatically increased and the fissures cross each other, providing connected networks. This implies that thermal cracking can greatly enhance the permeability of

oil shale, leading to a rapid increase in the permeability coefficient of oil shale at a higher temperature. According to these results, thermal cracking of oil shale can be considered the decisive factor in controlling corresponding changes in permeability.

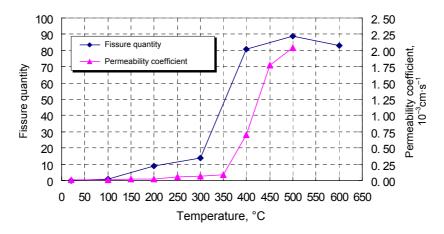


Fig. 9. Comparison of the changes between the amount of fissures and the permeability coefficient with temperature.

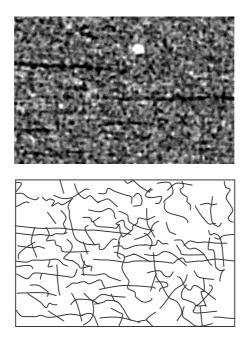


Fig. 10. Network structure and trace designs of fissures ( $T = 500 \, ^{\circ}\text{C}$ ).

#### **Conclusions**

In this work the micro-CT experimental system (µm scale) was used for investigation of thermal cracking of the Fushun oil shale. The oil shale sample was heated to different temperatures from 20 °C to 600 °C. Furthermore, the permeability coefficients were determined at different temperatures by the high-temperature rock-permeability testing system. From the results obtained, the following conclusions can be obtained:

- 1. In the range of 20 °C–300 °C, a few small and narrow micro-fissures were formed in the Fushun oil shale sample, which originated mostly from the edges of hard mineral particles. In contrast, when the temperature increased from 300 °C to 600 °C a drastic thermal cracking occurred, resulting in an obvious increase in the amount, length and width of subparallel fissures.
- 2. In the range of 20 °C–350 °C, the permeability coefficient of the Fushun oil shale was generally low. When the temperature exceeded 350 °C, the permeability coefficient of the oil shale sharply increased. Therefore, 350 °C is regarded as the critical temperature for permeability mutation in the Fushun oil shale. The permeability coefficient changed from  $0.008 \times 10^{-3}$  cm/s to  $2.040 \times 10^{-3}$  cm/s.
- 3. In the range of 20 °C–350 °C, thermal cracking made a small contribution to the increase of the permeability of the Fushun oil shale, mainly due to the unlikeliness of engendering connected networks. When the temperature exceeded 350 °C, a large number of new fissures were instantly generated, linking with each other to form extensive networks and leading to a rapid increase in the permeability coefficient. Therefore, thermal cracking can be regarded as the decisive factor in controlling permeability changes in the Fushun oil shale.

## Acknowledgments

The authors are grateful for financial support from the Ph.D. programs of the Special Scientific Research Foundation of the Ministry of Education of China (20101402120011), and for Nature Science Foundation of Shanxi Province (2010011014-2).

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Presented by S. Li Received November 25, 2010