INTRODUCTION

The mechanical properties of rock at high temperature and high pressure are of importance in many underground high-temperature engineering works such as geothermal energy development, deep underground geological disposal of nuclear waste, underground coal gasification and in situ pyrolysis of oil shale, servo-controlled rock testing machine was used in a study of the variation in elastic modulus of Jimsar mudstone (the roof stratum of Jimsar oil shale) when subjected to triaxial compression at high temperatures. The elastic modulus exhibited nonmonotonal variation when heated to 400°C while subjected to triaxial compression. This occurred in three stages. An empirical model involving piecewise linear function was used to quantitatively describe the relationship between elastic modulus and temperature. Elasticity increased between room temperature and 265°C, then weakened between 265 and 341°C, then increased at 341-400°C, in accordance with the calculated result from the empirical model. Thermal expansion, thermal cracking, water loss, and structure reconstruction had a combined effect on the elastic modulus of the mudstone. The results will be helpful for understanding the mechanism of strata movement in some underground high-temperature engineering operations.

KEYWORDS
elastic modulus, high temperature and pressure, mechanical properties, mudstone, thermal cracking, thermal expansion

Abstract
The elastic modulus is a key parameter in the constitutive equation of rock material, which changes with exterior conditions, including temperature. To study the evolution of the elastic modulus of the surrounding rock in some high-temperature underground engineering works (e.g., underground coal gasification, in situ pyrolysis of oil shale), the servo-controlled rock testing machine was used in a study of the variation in elastic modulus of Jimsar mudstone (the roof stratum of Jimsar oil shale) when subjected to triaxial compression at high temperatures. The elastic modulus exhibited nonmonotonal variation when heated to 400°C while subjected to triaxial compression. This occurred in three stages. An empirical model involving piecewise linear function was used to quantitatively describe the relationship between elastic modulus and temperature. Elasticity increased between room temperature and 265°C, then weakened between 265 and 341°C, then increased at 341-400°C, in accordance with the calculated result from the empirical model. Thermal expansion, thermal cracking, water loss, and structure reconstruction had a combined effect on the elastic modulus of the mudstone. The results will be helpful for understanding the mechanism of strata movement in some underground high-temperature engineering operations.

KEYWORDS
elastic modulus, high temperature and pressure, mechanical properties, mudstone, thermal cracking, thermal expansion

1 INTRODUCTION

The mechanical properties of rock at high temperature and high pressure are of importance in many underground high-temperature engineering works such as geothermal energy development, deep underground geological disposal of nuclear waste, underground coal gasification and in situ pyrolysis of oil shale/low metamorphic coal. In these operations, the temperature is a key factor for accurately evaluating the physico-mechanical behavior of rocks. The physico-mechanical parameters obtained at room temperature cannot be used in the computation or analysis and subsequent guidance of these activities. Hence, it is imperative that the physico-mechanical parameters of rocks be measured under high-temperature and pressure conditions.

Extensive studies of the physico-mechanical behavior of igneous rock such as granite at high temperature have been done in the context of geothermal development and nuclear geological disposal. Commonly, the results have indicated that the peak strength of granite decreased and peak strain increased with rising temperature during uniaxial compression tests. Temperature and confining pressure were two key factors in the failure of granite. With increasing temperature, granite experienced brittle to...
plastic deformation, and its elastic modulus decreased with the increase of temperature. In addition to experimental studies, thermal damage model coupled by thermo-mechanical fields has been also presented to simulate the response of rock to coupling effect.

Limestone, sandstone, and mudstone are three main types of sedimentary rock closely located above and below of coal beds, oil and gas reservoirs, and oil shale. Hence, the mechanical behavior of these kinds of rocks at high temperature has also been studied in order to guide some underground high-temperature engineering works such as underground coal gasification. Changes in peak strength, peak strain, failure mode, elastic modulus, and P-wave velocity of sandstone and limestone are found to be consistent with granite after high-temperature treatment. For example, Zhang et al reported that the peak strength decreased slowly, the elastic modulus decreased at a relatively rapid rate, and the peak strain increased slowly when sandstone was subjected to high-temperature treatment. Mudstone is a clay-mineral-rich rock, and the variation of its mechanical properties at high temperature are more complicated. Liu et al reported that the ductility of mudstone increased at high temperatures, and the elastic modulus increased from 25 to 400°C, then decreased at 400-800°C. A study of the mechanical properties of two Australian mudstones found that the tensile strength and low-strain shear stiffness were both significantly increased at 100-450°C and decreased at 450-900°C, and further concluded that the behavior variability was attributable to mineral change and thermal fracturing at high temperatures.

The above experimental studies were mainly performed under uniaxial stress and posthigh-temperature treatment. For igneous rock, monotonal variation of mechanical behavior was observed. The mechanical properties of sedimentary rocks have no uniform tendency due to the structural changes in the clay minerals. Whereas our previous studies had indicated that igneous rock such as granite experienced nonmonotonal variation of elastic modulus, permeability, and thermal expansion when they were subjected to high temperatures up to 600°C and high triaxial stresses. Hence, the mechanical behavior of mudstone is more complicated at high temperatures while under triaxial stress than after high-temperature treatment alone.

In this study, a servo-controlled high-temperature test machine for rock mechanics, developed by the authors, was employed to study in detail the variation of the elastic modulus of mudstone at temperature up to 400°C while being subjected to triaxial compression. The mechanism of the nonmonotonal change in the elastic modulus is also further discussed.

2 | EXPERIMENTAL SETUP AND METHOD

2.1 | Samples

Mudstone blocks were collected from the roof strata of an oil shale deposit in an open pit mine in Jimsar, Xinjiang, China. All weathered material was removed to ensure that fresh mudstone blocks were used. Standard-size samples with 50 mm in diameter and 100 mm long, shown in Figure 1, were processed in the laboratory. The constituent minerals were obtained by X-ray diffraction were quartz (57%), plagioclase (10%), siderite (4%), and clay minerals (29%). The clay minerals were mainly illite/smectite (82%), illite (9%), kaolinite (8%), and chlorite (1%).

2.2 | Experimental arrangement

An IMT-HTP-100F servo-controlled high-temperature test machine, developed by the authors, was used to measure the elastic modulus of the mudstone. Shown in Figure 2A, this was capable of measuring the real-time physical and mechanical properties of rock subjected to triaxial compression and high temperatures up to 600°C.

The sample was placed in a vessel (Figure 2B) and inert gas or heat-transfer oil was used to confine the sample at pressures up to 70 MPa, was applied by a pump. An axial pressure up to 500 MPa was applied to the sample by a piston. The sample was heated up to 600°C by electrically heated rods. The heating was controlled at rates from 0.04 to 10°C/min. Two grating rulers (resolution 1 μm) measured the axial displacement. The displacement of the axial piston on the sample was from 10^{-2} to 10^4 μm/s. The maximum duration of the constant applied high temperature exceeded 720 h.
2.3 | Experimental method

The elastic modulus of rock describes the deformation of rock material. Generally, it is determined from a stress-strain curve, which may be divided conceptually into five stages for rock in uniaxial compression: (a) natural crack closure stage; (b) elastic deformation stage; (c) stable crack propagation stage; (d) unstable crack growth stage; and (e) failure stage. The average elastic modulus is the average slope of the approximately linear portion of the axial stress-strain curve.

**FIGURE 2** A. Servo-controlled high-temperature test machine for rock mechanics, model IMT-HTP-100F. B. Sample assembly in high-temperature test machine shown in Figure 2A.
In an experiment for rock mechanics, a single sample obtains only one elastic modulus if an experiment on the full stress-strain curve, including failure deformation, is performed at a preset temperature. A number of samples are therefore needed to observe the evolution of the elastic modulus with temperature change. However, this cannot eliminate the effect of rock heterogeneity on the elastic modulus, since rock is a heterogeneous, anisotropic material. Hence, it is appropriate to measure the elastic modulus of one sample at different temperatures. Taking into consideration that the elastic modulus is obtained from a linear fit of the data recorded within the elastic deformation stage, the sample will not be destroyed if it experiences only elastic deformation at the different temperatures. Hence, the stress-strain tests at different temperatures were performed only within the elastic range to obtain the evolution of the elastic modulus with temperature. The procedures are described below.

Hooke’s law gives the stress-strain relationships as

\[ \varepsilon_1 = \frac{1}{E} (\sigma_1 - 2\mu \sigma_2), \quad (1) \]

\[ \varepsilon_2 = \frac{1}{E} [\sigma_2 - \mu (\sigma_1 + \sigma_2)], \quad (2) \]

where, for a cylindrical sample \( \varepsilon_1 \) is the axial strain; \( \varepsilon_2 \) is the lateral strain; \( \sigma_1 \) is the axial stress; \( \sigma_2 \) is the confining stress; \( E \) is the elastic modulus; and \( \mu \) is Poisson’s ratio. If the sample is first loaded from 0 to hydrostatic stresses \( \sigma_1 = \sigma_2 = \sigma_3 = \sigma_0 \), Equation (1) can be rewritten as

\[ \varepsilon_a = \frac{\sigma_a}{E} (1 - 2\mu), \quad (3) \]

where \( \varepsilon_a \) is the axial strain corresponding to \( \sigma_a \). Then the sample is compressed by increasing the axial stress while confining stress is maintained as \( \sigma_a \). The axial strain increment is then given by

\[ \varepsilon_1 - \varepsilon_a = \frac{1}{E} (\sigma_1 - 2\mu \sigma_a) - \frac{\sigma_a}{E} (1 - 2\mu). \quad (4) \]

The elastic modulus is then derived from Equation (4) to give

\[ E = \frac{\sigma_1 - \sigma_a}{\varepsilon_1 - \varepsilon_a} \quad (5) \]

Equation (5) illustrates that the elastic modulus of a triaxially stressed sample can also be obtained by a linear fit of the data during elastic deformation stage.

As described above, the following steps were taken to measure the elastic modulus of mudstone at elevated temperatures. First, the axial and confining stresses were increased to 6 MPa at room temperature. The axial stress was then increased at the rate of 0.1 MPa/s to about 32 MPa, which was approximately 50% of the peak strength of the sample at room temperature when a confining stress of 6 MPa was maintained; or axial stress was no longer increased when the total axial strain reached 0.003.

The increasing stress and strain were recorded automatically by the test machine. The elastic modulus of the sample at room temperature was calculated from Equation (5). The axial stress was then reduced to 5 MPa and confining stress was maintained at 6 MPa. This stress state was considered to be equivalent to the in situ rock stress at a depth of 200 m (taking the lateral coefficient as 1.2).

Second, the sample was heated to 100°C at the low heating rate of 10°C/h, as shown in Figure 3. The low heating rate eliminated any thermal shock effect in the sample during heating. This temperature was maintained for two hours to ensure uniform temperature distribution in the sample after heating to 100°C. 41

Finally, following the first step, the elastic modulus of the mudstone at 100°C was obtained. The temperature increase was then continued to 200, 250, 300, and 400°C, and the corresponding elastic modulus was determined by repeating the first two steps.

3 | RESULTS

3.1 | Effect of temperature on the elastic modulus of mudstone

Figure 4 illustrates the stress-strain curve at different temperatures. From the method described above, the origin of the ordinate of the stress-strain curve would be expected to be 6 MPa. However, the actual data showed that the strain was zero over a large stress range. For example, the strain at 100°C remained zero as the axial stress increased from 6 MPa to about 22 MPa. The linear strain began to increase at 100°C, when the axial stress exceeded 22 MPa. Here, the stress value at which the axial strain began to increase was defined as the initial stress. The difference value between the starting stress and the confining stress was defined as the starting stress difference (\( \sigma_{\text{std}} \)). Both mudstones exhibited a starting stress difference. Since \( \sigma_{\text{std}} \) at room temperature was observed at the lowest temperature, it is conjectured that the reason is that the samples became stiffer due to thermal expansion at higher temperatures. We have no more reasonable explanation for that at present.

The linear portion of the plotted data on the stress-strain curve was fitted to obtain the elastic modulus at each temperature. Figure 5 shows the variation of the elastic modulus of the two mudstones with temperature increase. The elastic
modulus of mudstone experienced nonmonotonal variation with temperatures up to 400°C. The variation was divided into three stages, as follows.

Stage I (room temperature to 200°C): The elastic modulus of mudstone during this stage increased almost linearly with rising temperature. The elastic modulus of sample 1# was approximately 3 GPa at room temperature, increasing to about 4.5 GPa at 200°C. The elastic modulus of sample 2# at 200°C was about 1.6 times the at room temperature value. Thermal expansion of the samples resulted in the increment at this stage.

Stage II (200-300°C): The elastic modulus of the two samples decreased as the temperature increased. For example, sample 1# showed a drop of 1.7 GPa from 200-250°C and continued to decrease at 250-300°C. The elastic modulus of sample 1# at 300°C was about 0.8 times the value at 200°C. The same trend was also observed in sample 2#. In particular, the elastic modulus of the two samples showed a smaller value than at room temperature. This was attributed to severe thermal cracking in the samples at 300°C.

Stage III (300-400°C): The elastic modulus of the two samples again increased with increasing temperature at this
stage. Sample 1# was about 4.5 GPa at 400°C, which was about twice the value at 300°C, even higher than at 200°C. Sample 2# also experienced a significant increment in elastic modulus at this stage. However, it was smaller than at 200°C.

3.2 | Empirical model of elastic modulus evolution with temperature

Based on elastic modulus change with temperature, we defined a parameter (elastic modulus factor, $D$) to describe the influencing extent of temperature to modulus. The factor is the ratio of the elastic modulus at a given temperature ($E_T$) to that at room temperature ($E_0$), shown as

$$D = \frac{E_T}{E_0}. \quad (6)$$

The factor represented thermal damage in the mudstone at different temperatures. The relationship between $D$ and temperature is plotted in Figure 6. The factor also exhibited three stages with temperature.
rising. Every stage is expressed as a linear function, seen in Equation (7):

$$D = aT + b. \quad (7)$$

Hence, the three stages corresponding to elastic modulus change are expressed by three linear functions.

An average elastic modulus was constructed for both samples to calculate the elastic modulus factor. The average factor was shown as the pink five-star line in Figure 6. Three linear functions were used to fit the relationship between the average factor and temperature are.

$$D = \left\{ \begin{array}{ll}
3.05 \times 10^{-3}T + 0.94 & (T = 20 - 200^\circ C) \\
-7.95 \times 10^{-3}T + 3.11 & (T = 200 - 300^\circ C) \\
5.87 \times 10^{-3}T - 1.00 & (T = 300 - 400^\circ C) 
\end{array} \right. \quad (8)$$

As seen in Figure 6 and Equation (8), the elastic modulus factor increased continuously at RT–200°C and $D > 1$, indicating that the elasticity of the mudstone was strengthened. At 200-300°C, the factor decreased with rising temperature, showing that the rock exhibited a drop of elasticity. In particular, the elasticity was weakened within a certain range, and the factor was less than 1. The factor increased again above 300°C and was greater than 1, which indicated that the elasticity was strengthened again at 400°C. Using Equation (8), the temperature at the inflection point may be calculated in detail, showing the elasticity change from strengthening ($D > 1$) to weakening ($D < 1$), then strengthening again. The elasticity was strengthened at room temperature (RT)–265°C and 341–400°C, and weakened at 265–341°C.

4 | MECHANISM OF EVOLUTION OF ELASTIC MODULUS OF MUDSTONE WITH TEMPERATURE

As mentioned above, the mudstone exhibited enhancement and weakening of mechanical properties at RT–400°C. These changes were closely related to variation in the constituent minerals at high temperatures. The samples used in the test were mined from the roof of oil shale strata in an open pit mine located in Jimsar, Xinjiang. The minerals were mainly quartz (57%), plagioclase (10%), siderite (4%), and clay minerals (29%). The clay minerals mainly consisted of illite/smectites (82%), illite (9%), kaolinite (8%), and chlorite (1%). Different minerals exhibited different properties when heated. Quartz, plagioclase, and siderite have a stable structure and which is not altered at elevated temperature up to 400°C, but they do experience thermal fracturing, which results in many microcracks that weaken the mudstone. Conversely, clay minerals generally have a lamellar structure and the interlaminar bonding force is weak. In addition, there was a great deal of free water between layers, which is easily removed when the rock is heated. Bound water in the structure of minerals is also removed at high temperatures and such water loss further damages the lamellar structure, causing structural change to the clay minerals. These changes have a significant effect on the mechanical properties of mudstone. In all tests, mudstone was under the condition of drainage during the whole heating process. Hence, vapor phase of water in the mudstone had little effect on elasticity change even if at high temperatures.

The variation of the elastic modulus of the mudstone at high temperatures is attributable to the overall effect of thermal expansion and change of mineral structure. Each such effect has both advantages and disadvantages in weakening the mudstone’s properties. As the temperature increases, uncoordinated deformation occurred among the different mineral particles due to the differences in their thermal expansion coefficients, resulting in many microcracks, termed “thermal cracking”. Cracking that takes place at the boundary of two adjacent mineral particles is termed “intergranular thermal cracking”. Cracking that takes place within crystalline minerals is “transgranular thermal cracking”. Both intergranular and transgranular thermal cracking result in a weakening of the mudstone properties because of the thermally induced microcracks occurring throughout the whole heating process.

However, thermal expansion also enhanced mudstone properties by opposing deformation due to the applied compression. Mineral structure changes also have a significant impact on the elastic modulus due to the loss of water within the structure and its consequent reconstruction at high temperatures. Hence, the observed nonmonotonal changes of elastic modulus were the combined effect of thermal cracking, thermal expansion, water loss, and reconstruction of clay minerals.

In stage I at RT–200°C, all minerals in the mudstone, especially the hard minerals such as quartz, plagioclase, and siderite thermally expand to strengthen the elasticity. In rock mechanics, the convention is that compressive deformation is positive and expansive (tensile) deformation is negative. Figure 7 shows that sample 1# thermally expanded at elevated temperatures up to 400°C. Thermal cracking of the mudstone was represented by cumulative counts of acoustic emission in Figure 7. The AE counts began to occur at about 85°C and increased to 1200 at 180°C. This was followed by a rapid increase at 180–200°C, and then continued to increase to 3100. Hence, in stage I, thermal cracking was relatively slight and had little weakening effect on the elastic modulus.

Figure 8 shows the comparative thermal analysis curves for smectite, illite, and kaolinite. Smectite and illite show an endothermic peak at around 130°C, indicating that a large amount of interlaminar water and adsorbed water were removed at this stage. The large width of the endothermic
peak indicates that most of the water removal occurred below 200°C. However, the space occupied by the water was reduced by thermal expansion. The structure had not yet reconstructed at this stage due to the relatively low temperature. Hence, thermal expansion of minerals dominated the variation of elastic modulus, and the elasticity of the mudstone exhibited enhancement at this stage.

In stage II at 200-300°C, the hard minerals continued to expand. Thermally induced microcracks continued to increase. The total acoustic emission (AE) counts reached 3100, indicating that thermal cracking was stronger at this stage than in stage I. Bound water (mainly hydroxyls, -OH) in the molecular structure of the clay minerals was removed at these temperatures, destroying the crystal lattice and forming a further amorphous phase. Therefore, both thermal fracturing and bound water removal resulted in a reduction of elastic modulus, which showed a reduction at 200-300°C.

In stage III at 300-400°C, the thermal expansion of hard minerals continued to increase. Thermal cracking was intense and promoted the weakening of elasticity. Nevertheless, the amorphous phase recrystallized to form new crystal structures which strengthened the elasticity of the mudstone. For example, plasticity of kaolinite was eliminated as heated to 400°C, indicating that elasticity was strengthened. The mixture of illite/smectites changed into illite and then transformation of tetrahedral sheet took place. The semi-quantitative XRD...
analysis showed that the content of illite/smectites would decrease from 20% to 5% with temperature increasing from room temperature to 750°C. It indicated that significant structure variation of clay minerals appeared during heating. This is a very common phenomenon, and is the main reason that clay minerals are used to make bricks and ceramics. Hence, mudstone exhibited a rise in the elastic modulus at 300-400°C, despite the increasingly severe thermal cracking.

5 | CONCLUSIONS

The elastic modulus of Jimsar mudstone was studied in detail using standard sized samples 50 mm diameter and 100 mm long at temperatures up to 400°C and triaxial stresses. The effect of thermal expansion, thermal cracking, water loss and structure reconstruction on the elastic modulus is discussed. Notable conclusions were drawn, as follows.

1. The elastic modulus of Jimsar mudstone experienced nonmonotonal variation in the process of heating up to 400°C under a triaxial stress of 17 MPa. The variation in elastic modulus was divided into three stages: increasing with temperature ranging from room temperature to 200°C, decreasing at 200-300°C, and rebound at 300-400°C.

2. An empirical model involving piecewise linear function was used to quantitatively describe the relationship between the elastic modulus and temperature. Elasticity enhancement occurred at RT–265°C and 341-400°C, and weakened at 265-341°C according to the calculated result of empirical model.

3. Thermal expansion, thermal cracking, water loss and structure reconstruction had a combined effect on the elastic modulus of the mudstone. Thermal expansion and structure reconstruction were the predominant factors inducing elasticity enhancement at stages I (room temperature to 200°C) and III (300-400°C). Thermal cracking and water loss dominated elasticity weakening at stage II (200-300°C).

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CONFLICT OF INTEREST

The authors have no conflicts of interest.

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