UNIAXIAL COMPRESSIVE STRENGTH AND FAILURE CHARACTERISTICS OF ARKOSIC SANDSTONE AFTER THERMAL TREATMENT

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Experiments were conducted to study the mechanical characteristics of arkosic sandstones sampled from Pingyi, China. Rock samples were all thermally treated under the temperature ranging from room temperature to 800 ^{o}C. Results show that as the treatment temperature rises, the arkosic mineral composition does not change obviously, but the mechanical behaviors change regularly. Variation trend changes dramatically at 200 ^{o}C, 400 ^{o}C and 500 ^{o}C. With thermal expansion of mineral particles being the dominant factor, mechanical behaviors barely change below 200 ^{o}C. When temperature ranges from 200 ^{o}C to 400 ^{o}C, it has an important effect on the mechanical properties because of the thermal fracture. From 400 ^{o}C to 500 ^{o}C, mechanical properties change dramatically as a result of the mutual influence of thermal fracture, fusion and recrystallization, but the thermal fracture is the leading factor. Because of the fusion and recrystallization, fractures are partly filled, which results in partial recovering of the mechanical strength. With the combined action of thermal fracture, fusion and recrystallization after 600 ^{o}C, mechanical performance of arkosic sandstones degrades rapidly. Generally, the porosity and peak strain of arkosic sandstones increase with the temperature rising. However, the peak stress, elastic modulus and deformation modulus decrease simultaneously. Influenced by mineral particles' thermal expansion, thermal fracture, fusion, and recrystallization and so on, the variation trend and amplitude are not the same at different temperature ranges, and the damage mechanism of sandstones also makes a difference.

Key words: *rock mechanics; arkosic sandstone; thermal treatment; mechanical property*

1 Introduction

Currently, human beings have managed to exploit solid mineral resources at about 4 km underneath and mechanical tools can also reach strata of more than 10000 m underground [1]. The research on high geothermal properties of rock mass is a must for deep mining because of the common high temperature problem. Rock mass engineering including underground coal gasification and pyrolysis, geothermal exploration, rock mass support of tunnels and roadways in coal mine after fire, underground nuclear waste disposal and so on all involves relatively higher temperature. The study of high temperature mechanical property of rock mass thus has broad engineering significance.

Sandstone, a typical sedimentary rock widespread in the crust, is also the most common rock in coal measure strata. Due to the complicated diagenetic environment, the mineral compositions, particle morphology and cement types of sandstones vary greatly, leading to obvious distinctness of rock strength. The mechanical property is also prone to be affected by temperature. At present, researches on high temperature mechanical properties of sandstone are focused on the post-high-temperature experiment, with test approaches such as uniaxial compression experiment, acoustic emission, scanning electron microscope, XRD composition analysis and ultrasonic inspection and measurement etc. Zhao Hongbao et al [2] studied the ultrasonic transmission of roof sandstone

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from Guhanshan coal mine after high temperature treatment, and found that the sandstone could be badly damaged as the temperature increased. The research of Wu Gang et al[3] on the mechanical property of sandstone from Jiaozuo coal mine after thermal treatment within 1200 ^oC showed that, the temperature below 400 ^oC had little influence on the mechanical property of sandstone; the peak strength at 400 ^oC was the highest, and began to decrease when the temperature kept increasing. Chen Lunjian et al [4] adopted SEM, polarizing microscope and thermal analyzer to study the sandstone of Hebi coal mine and concluded that the sandstone began to crack at 400 ^oC, and the particles began to burst at 600 ^oC. Zhang Yuan et al [5] considered that as the temperature increased, the permeability of sandstone would increase in general. The uniaxial compression test on sandstone after thermal treatment [6] indicated that 600 ^oC is the threshold temperature of sandstone fragility-ductility damage; within this range, the uniaxial compressive strength and elastic modulus of sandstone presents a falling trend on the whole. Other experiments also demonstrated that from room temperature to 500 ^oC, the compressive strength of sandstone after thermal treatment first took on an upward trend, and then fell dramatically after 500 ^oC [7]. Wu G et al [8] found that the uniaxial compressive strength of sandstone increased slightly from room temperature to 400 ^oC, but declined sharply afterwards, while it's elastic modulus showed a decreasing trend. The experiment of Zhang L Y et al [9] manifested that 600 ^oC was the turning point temperature of sandstone uniaxial compressive strength and elastic modulus variation. The mechanical property of sandstone after this point decreased significantly. Tian H et al [10] summarized and analyzed the existing data of compressive strength and elastic modulus of sandstone, and thought that there was a threshold temperature for compressive strength and elastic modulus. Before the point, there were three possible trends of sandstone mechanical parameters: increase slightly, remain unchanged, and decrease slightly. After the point, there would be a sharp decrease. The general threshold temperature was 500 ^oC. On the whole, previous studies have shown that temperature has obvious effect on mechanical properties of sandstone; the influence before the temperature range of 400 ^oC ~600 ^oC is different, but relatively definite after 600 ^oC; it is widely believed that the thermal expansion and thermal fracture of sandstone mineral particles are dominant factors for the effect of temperature on sandstone mechanical properties.

In order to explore the mechanical properties of sandstones in the deep high geothermal engineering, sandstones were sampled and thermally treated. Then, methods including X-ray diffraction(XRD), scanning electron microscope(SEM), mercury intrusion porosimetry(MIP) and uniaxial compressive system(UCS) were comprehensively adopted to study the mechanical behavior and the damage mechanism of sandstones thermally treated from room temperature to 800 ^oC, from perspectives of macro mechanical property, micro mineral composition and pore structure.

2 Methodologies

2.1 Sandstone samples

The experiment samples are sandstone taken from Pingyi, Shandong province of China. They are grey green with major mineral composition such as quartz, potassium feldspar, plagioclase feldspar, calcite, dolomite and some clay minerals, among which the content of feldspar reaches 69%. The samples are cylinder, with diameter and height of 50 mm and 100 mm, respectively. The unevenness of the surface is less than 0.05 mm. The deviation of top and bottom diameter is no more than 0.3 mm, and the axial deviation is less than 0.25°. Drying treatment was conducted on all the samples. Each test involves no less than three specimens, and more would be added if a large discrete result was measured.

2.2 Thermal treatment

SX_2-4-10A Chamber Electric Furnace and K-serial Temperature Controller were adopted to heat the samples. The maximum heating temperature is 1000 ^oC, and the temperature control accuracy is ±1 ^oC. The experiment temperature is set as 200 ^oC, 400 ^oC, 500 ^oC, 600 ^oC, 700 ^oC and 800 ^oC, respectively, with heating rate of 2 ^oC /min. After reaching the test temperature, samples were kept for heat preservation of 2 h, and then left for nature cooling to room temperature.

2.3 Experiment procedure

(1) Put the samples into the drying cabinet of 105 ^oC for 24h, leave them naturally cooled in the cabinet to ensure that the samples are all completely dried.

(2) Select samples randomly and divide them into groups, mark them by temperature of room temperature, 200 ^oC, 400 ^oC, 600 ^oC and 800 ^oC, and ensure that there are at least three samples in each group.

(3) Heat the grouped samples in the high temperature oven, set the temperature as 200 ^oC, 400 ^oC, 500 ^oC, 600 ^oC, 700 ^oC and 800 ^oC, respectively, with heating rate of 2 ^oC /min. After reaching the preset temperature, keep the samples for heat preservation of 2h, and then leave them for nature cooling to room temperature.

(4) Conduct uniaxial compression experiments at each test temperature on MTS C64 hydraulic universal testing machine in the State Key Laboratory of Coal Resources and Safe Mining. The stress control model was adopted. The loading rate is 0.5MPa/s.

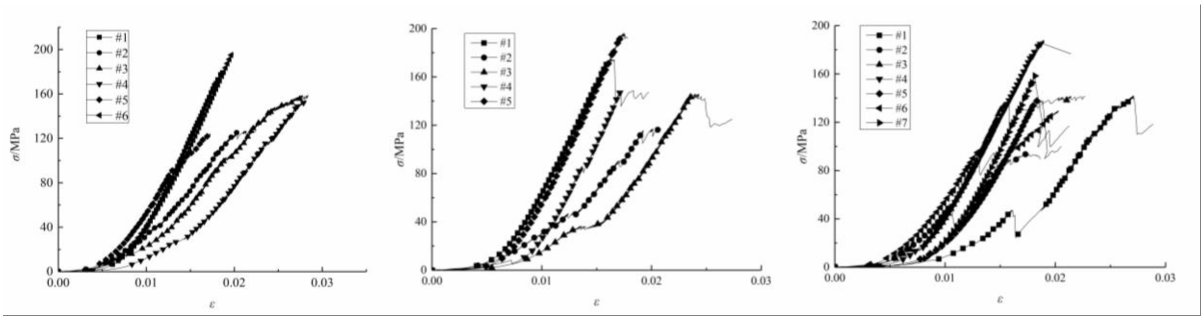
(5) Take samples from the crushed specimens after experiment. Use the German D8 ADVANCE X-ray diffractometer to analyze the mineral composition of samples after heating treatment. Apply the American QuantaTM 250 SEM to observe the microstructure of samples, and adopt American PoreMaster-60 GT Mercury Injection Apparatus to test the pore structures.

3 Experimental results

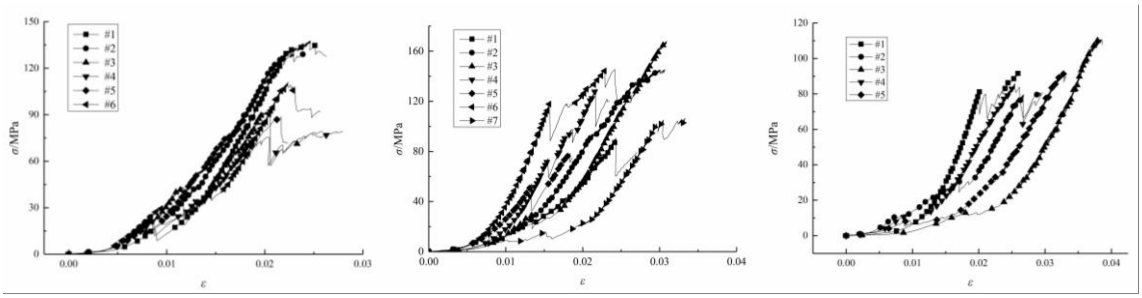
3.1 Mechanical performance

1) Stress-strain characteristic

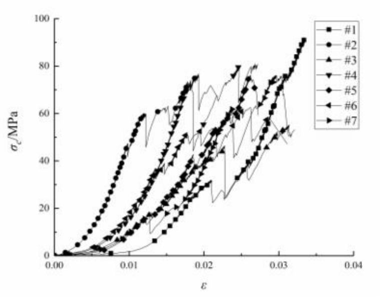
Uniaxial compression experiment results of sandstone samples treated with each temperature are illustrated in Fig.1.



(a) room temperature (b) 200 ^{o}C (c) 400 ^{o}C



(d) 500 ^{o}C (e) 600 ^{o}C (f) 700 ^{o}C



(g) 800 ^{o}C

Fig.1 Stress-strain curves of sandstone after thermal treatment

Fig.1 shows that densification and elastic stages appear in the uniaxial compression of sandstone samples thermal treated. The yield strain before 400 ^{o}C is relatively small. Influenced by the loading mode, the residual deformation after peak is not obvious. With the increasing of temperature, especially after 400 ^{o}C, the strain of the densification stage gradually increases.

2) Peak strength and peak strain

After thermal treatment, the variation of sandstone peak strength is shown in Fig.2.

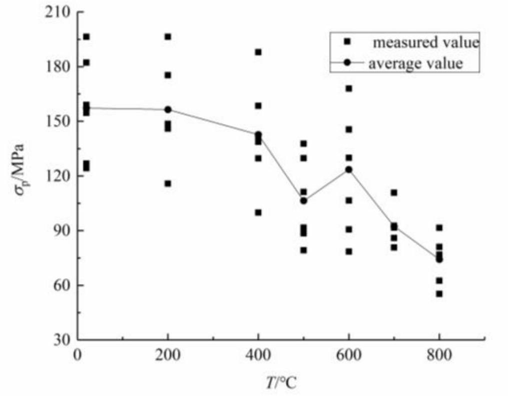


Fig.2 Peak strength variation curve of sandstone after thermal treatment

As demonstrated in Fig.2, with the temperature increase, the peak strength of sandstone thermal treated decreases in general. The variations in different temperature ranges are not the same, which can be divided into the following four stages:

Stage 1: from room temperature to 400 ^{o}C. In this temperature range, the peak strength of sandstone slowly declines with the growth of temperature. At room temperature, the average peak strength of sandstone is 157.21MPa. By contrast, the average peak strength at 200 ^{o}C and 400 ^{o}C decreases by 0.51% and 9.27%, respectively. Therefore, the heating treatment of 200 ^{o}C and 400 ^{o}C has very little and little influence on the peak strength of this sandstone.

Stage 2: 400 ^{o}C ~500 ^{o}C. In this stage, the peak strength drops sharply as temperature rises. Compared with that at 400 ^{o}C and room temperature, the peak strength at 500 ^{o}C decreases by 25.45% and 32.36%, respectively.

Stage 3: 500 ^{o}C ~600 ^{o}C. In this stage, the peak strength of sandstone stops to fall and begins to grow. Contrasted with the value at 500 ^{o}C, it increases by 16.13%, but still decreases by 13.42% as compared with that at 400 ^{o}C.

Stage 4: 600 ^{o}C ~800 ^{o}C. In this stage, the peak strength of sandstone drops rapidly. At 700 ^{o}C and 800 ^{o}C, it is 92.36MPa and 74.24MPa, decreasing by 41.25% and 52.78% compared to the values at room temperature.

After thermal treatment, the average peak strain variation is demonstrated in Fig. 3.

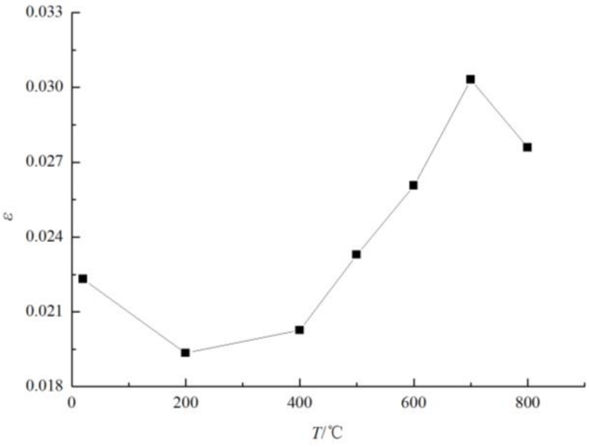


Fig.3 Average peak strain variation of sandstone after thermal treatment

As seen in Fig. 3, before 200 ^{o}C, the peak strain of sandstone becomes lower and lower with the rising of temperature. In contrast to the value of 0.223 at room temperature, the strain at 200 ^{o}C decreases by 13.31%. In the temperature range of 200 ^{o}C ~400 ^{o}C, the peak strain gradually increases, but it sees a sharp rise when the temperature grows to 400 ^{o}C ~700 ^{o}C. At 700 ^{o}C, the peak strain reaches 0.0303, increasing by 56.71% compared to that at 200 ^{o}C. Then when the temperature is 700 ^{o}C ~800 ^{o}C, the peak strain starts to fall.

3) Derivation of the UCS test

After high temperature treatment, the variation of elastic modulus E (the straight line slope of stress-strain curve) and deformation modulus E_0 (the ratio of peak stress to peak strain) with regard to the temperature is demonstrated in Figs.4 and 5.

As illustrated, after high temperature, both the elastic and deformation modulus of sandstone experience a decreasing trend with the increase of temperature, indicating that the sandstone resistance to deformation is lowered after heating treatment. From room temperature to 400 ^{o}C, the elastic modulus of sandstone decreases slightly, compared with the 9.4GPa at 200 ^{o}C, the biggest percentage of reduction is 14.85%. Within the temperature range of 400 ^oC ~500 ^oC and 600 ^oC ~800 ^oC, the elastic modulus comes across two rapidly decreasing stages. At 500 ^oC, the value falls to 66.9% of that at 400 ^oC, and at 800 ^oC, it falls to 55.86% of that at 600 ^oC. Within 500 ^oC ~600 ^oC, the elastic modulus is relatively stable and rises a little. Within the range of room temperature to 200 ^oC, as the heating temperature increases, the deformation modulus gradually goes up while the deformation capacity goes down. The change of deformation modulus in other temperature ranges is basically in accordance with that of the elastic modulus. Comparing Figs.4 and 5, it is found that the tendency of elastic and deformation modulus variation is quite similar after 400 ^oC, and the major differences are before 400 ^oC.

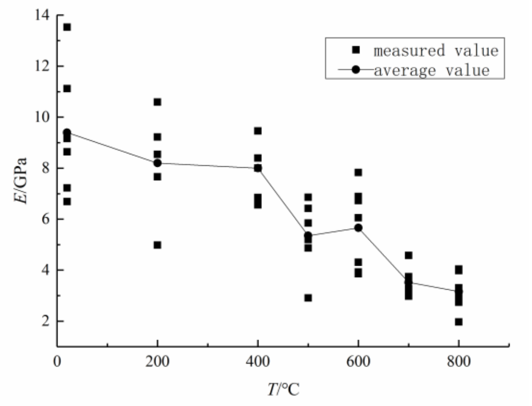


Fig.4 Elastic modulus variation curve of sandstone after thermal treatment

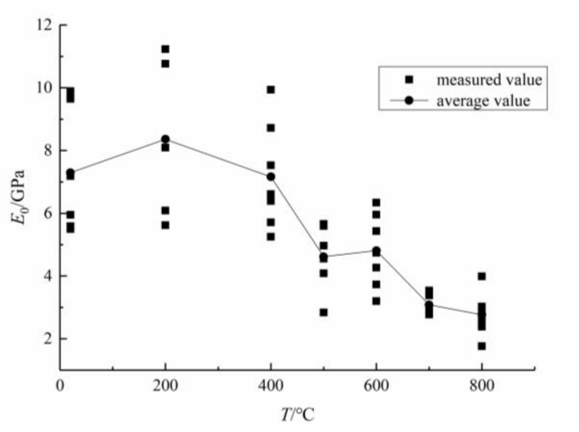
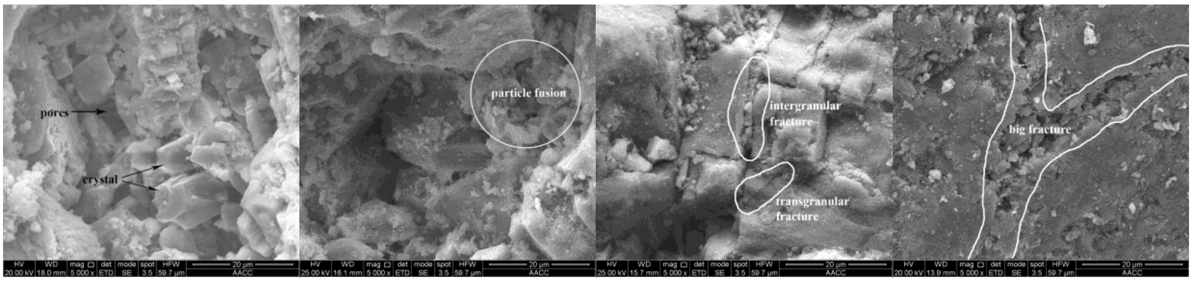


Fig.5 Deformation modulus variation curve of sandstone after thermal treatment

3.2 Sandstone pore structure

1) Microstructure and mineral composition

According to the sandstone uniaxial compression experiment result, samples treated under room temperature, 400 ^oC, 500 ^oC and 700 ^oC were taken for SEM analysis, to observe the micromorphology of sandstone samples after thermal treatment. The scanning magnification factors are all 5000, and the result is shown in Fig.6.



(a) room temperature (b) 400℃ (c) 500℃ (d)700℃

Fig.6 SEM photos of sandstone

Fig.6 clearly shows the changing process of sandstone pores and fractures. At room temperature, the mineral particles are round and closely contacted, and the crystal structure is obvious (Fig.6a); after heating treatment of 400 ^{o}C the fusion of particles appears, with very few tiny fractures. The fissure width and length are both small, and the connectivity is poor (Fig. 6b); after heating treatment of 500 ^{o}C, a large number of intergranular and transgranular fractures appear, and their width and length increase significantly. Melted minerals fill the fractures and the connectivity of fractures are obviously strengthened, also, the recrystallization phenomenon appears(Fig.6c); after heating treatment of up to 700 ^{o}C, penetrating big fractures are found inside the sandstone samples. The fusion is evident, and the mineral particles cannot be identified so clearly (Fig.6d).

It can also be seen that affected by nonhomogeneous mineral particles, the thermal fractures of samples occur obviously at high temperature; the fractures are developed mostly in the temperature range of 400 ^{o}C ~500 ^{o}C; after 400 ^{o}C, minerals are melted and flocs are generated to fill the fractures incompactly; at about 500 ^{o}C, recrystallization happens, minerals melt to fill the fractures and their contact are rather close. The micromorphology change of sandstone may occur at a threshold temperature of about 400 ^{o}C ~500 ^{o}C. The mechanical property of sandstone may have significant changes in this temperature range.

To examine the mineral composition variation of sandstone after heating treatment, especially after 500 ^{o}C, X-ray diffraction analysis were conducted on the samples heated under room temperature and 500 ^{o}C. The results are listed in Table 1.

Table 1 Mineral composition of sandstone %

temperature	quartz	potassium feldspar	plagioclase feldspar	calcite	dolomite	clay minerals
room temperature	17.0	31.0	38.0	3.0	5.0	6.0
500℃	19.0	30.0	40.0	2.0	6.0	3.0

Experiments demonstrate that, after a thermal treatment of 500 ^{o}C, the mineral composition of sandstone does not change obviously, i.e., the quartz content remains in 20%; the content of feldspar remains at around 70%; the amount of calcite and dolomite remains unchanged; while that of clay minerals drops by half. So it can be inferred that heating treatment under 500 ^{o}C has limited influence on mineral composition, and the mechanical properties and mineral components are not closely correlated after thermal treatment.

2) Sandstone porosity

The heating treatment process of sandstone accompanies the closure, generation, extension and connection of fractures. The porosity keeps on changing, and directly affects the macro mechanical property of sandstone. The pores are categorized according to the following standard: nanopore (<100nm), micropore (100nm~500nm), mesopore (500nm~2500nm), macropore (2500nm~10000nm) and fractures (>10000nm) [11]. Mercury intrusion volumes of different pores are shown in Fig.7.

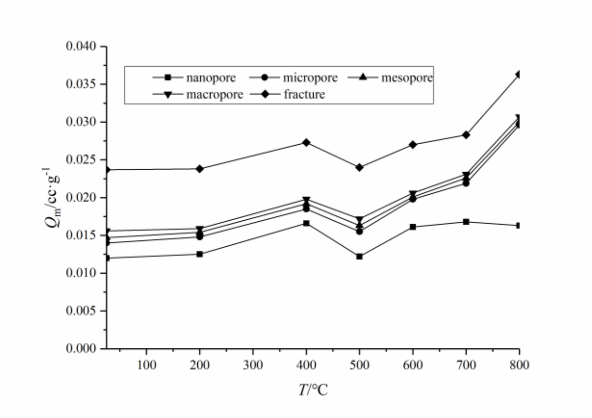


Fig. 7 Mercury intrusion of different sandstone pores

As shown in Fig.7, the total mercury intrusion increases evidently with the temperature rising. From room temperature to 800 ^oC, the total volume grows from 0.0237cc/g to 0.0363cc/g, increasing by 53.16%. This indicates that under the temperature effect, the number of pores of sandstone samples gradually increases, and the biggest contributor is nanopores and micropores. In comparison, the mercury intrusion of fractures in the sandstone declines slowly with the rising of temperature. Also, at room temperature and 800 ^oC, the mercury intrusion of sandstone fractures decreases from 0.0081cc/g to 0.0056cc/g, the percentage of which falls from 34.18% to 15.43%. In this process, the numbers of mesopores and macropores remain on a relatively low level at each temperature, and the proportion of mercury intrusion of nanopores at each temperature is about 50% of the total mercury intrusion of pores.

4 Discussions

Studies have shown that affected by thermal expansion, thermal fracture and mineral composition variation, the pore structure of rock under the effect of temperature would undergo obvious changes [12-13]. Generally, with the rising of temperature, the number of pores inside the rock will gradually increase [14-15]. Influenced by stress, heated rock is prone to form inner connective fracture network, so as to weaken its bearing capacity [16-17]. Experiments in this paper have confirmed that thermal treatment would change the mechanical property of sandstone, but the variation trend and degree at different temperature ranges are not the same, so as the dominant mechanism. The effect of temperature on rock mechanical property is basically reflected in the mineral composition, number and structure of pores, mineral fusion, recrystallization and crystal phase transition etc [18]. The crystal phase transformation is basically reversible, for example, quartz can experience anα-β phase transition at 573 ^oC, and then recover after cooling [19]. Therefore, for samples after heating treatment, phase transition has very limited effect on the mechanical property. The experiments in this paper have proven that after high temperature treatment, the mineral composition of sandstone samples does not change significantly. The mechanical property change is mostly caused by pore number and structure, and indirectly affected by factors such as thermal expansion, thermal fracture, fusion and recrystallization.

In the temperature range of room temperature to 200 ^{o}C, the thermal cracking of sandstone is not obvious, but the heating causes the expansion of mineral particles, leading to the closure of sandstone primary fractures and porosity reduction. During the cooling process, because of the nonhomogeneous thermal expansion of mineral particles, the contractions of particles are not uniform, and only some nanopores are generated. Due to the larger spatial freedom and expansion, bigger pores go through thermoplastic deformation, and some cannot recover after cooling. So, at 200 ^{o}C, the number of nanopores of samples relatively increases, while other pores become less, but the total porosity remains unchanged(Fig.7); the densification stage of stress-strain curve has no obvious change either(Fig.1); the peak stress keeps unchanged(Fig.2), and the decreasing of elastic modulus is not evident(Fig.4). On account of the thermal expansion, larger pores become partly closed, giving rise to the lower deformation capacity of samples and better resistance capacity to deformation, which is characterized by lower peak strain and higher deformation modulus, as shown in Figs.3 and 5. During this stage, the dominant factor of sandstone mechanical property is thermal expansion, while temperature has less effect. Previous research also showed that thermal fracture would not likely to happen within the temperature range of 150 ^{o}C ~200 ^{o}C [20].

When the temperature rises from 200 ^{o}C to 400 ^{o}C, the thermal strain exceeds the bearing capacity of mineral particles. The thermal cracking continues to strengthen, and the nanopore generation speed is much improved. In this case, the falling tendency of porosity above nanopores is curbed, and the total porosity is gradually increased. Therefore, in this stage, the peak stress, elastic modulus and deformation modulus all decrease slowly with the rising of temperature, but the peak strain keeps increasing. The major cause of sandstone mechanical property is thermal fracture, and the effect of temperature is getting stronger.

In the temperature range of 400 ^{o}C ~500 ^{o}C, the thermal fracture phenomenon of sandstone increases rapidly, while mineral particles are melted and recrystallized. As shown in Fig.7b, the 400 ^{o}C heating treatment leads to the melting of mineral particles, and some flocs of small particles stick to the crystal surface; at 500 ^{o}C, intergranular and transgranular fractures appear. Because of the thermal fusion of mineral particles, fractures are filled by melted minerals, but not cemented, and the filling is not so compact. On the one hand, the fractures expand; on the other hand, the fusion is intensified and the fractures are filled, so the porosity of sandstone samples is greatly lowered. The peak stress, elastic modulus and deformation modulus decline sharply, but the peak strain increases rapidly. In this stage, the mechanical property is affected by both thermal fracture and fusion, with the former taking a leading role.

In the temperature range of 500 ^{o}C ~600 ^{o}C, the thermal fracture of mineral particles continues with a higher intensity. The width and length of fractures are getting larger, and the fracture connectivity is enforced. Meanwhile, the particles fusion is increasing, and the recrystallization accelerates. So, after cooling, the nanopores and micropores are open, and the porosity is improved. Affected by fusion and recrystallization, pores larger than micropores are filled, and the densification is relatively improved. In this temperature range, the peak stress is higher than that at 500 ^{o}C, but is still lower than that at 400 ^{o}C. This is also confirmed by other studies [21-23], but none of them have offered a clear explanation. The test results of He Guoliang et al [21] indicate that the wave velocity of sandstone samples increases after heating treatment of 100 ^{o}C ~200 ^{o}C, decreases after 400 ^{o}C, and drops rapidly after 600 ^{o}C, suggesting that there is a threshold temperature between 400 ^{o}C and 600 ^{o}C, which exactly confirms the interpretation of mechanical property change mechanism of sandstone after heating treatment of 600 ^{o}C in this paper, as indicated in Fig.9.

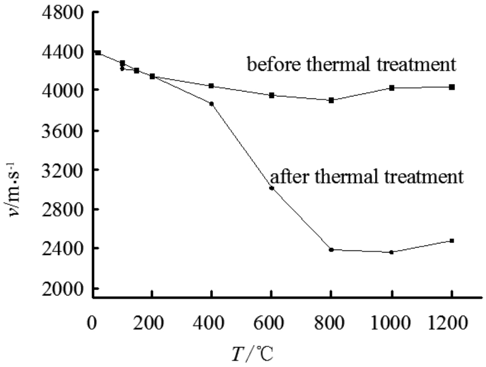


Fig.8 Average P-wave velocity of sandstone samples

In the temperature range of 600 ^{o}C ~800 ^{o}C, thermal fracture, fusion and recrystallization of particles get intensified. The fusion and recrystallization penetrate into the crystal and fractures continue to develop. The porosity is increased in further, while the peak stress, elastic modulus and deformation modulus decrease rapidly. In this stage, the dominant influence on sandstone mechanical property is thermal fracture and fusion.

From the above analysis, it can be concluded that 200 ^{o}C is the threshold temperature of the transition from thermal expansion to thermal fracture; 400 ^{o}C is the threshold temperature of thermal cracking from gentle to drastic and the minerals particle fusion; 500 ^{o}C is the threshold temperature of particles recrystallization after fusion. At these three temperature points, the sandstone mechanical properties are changed.

5 Conclusions

After thermal treatment, mechanical behaviors of the arkosic sandstone will change to a certain extent. In general, with the treatment temperature rising, the strength, elastic modulus and deformation modulus all decrease slowly, but the peak strain and porosity increase gradually, which is basically affected by thermal expansion, thermal fracture, fusion and recrystallization etc. Roughly speaking, 200 ^{o}C is the threshold temperature of the transition from thermal expansion to thermal fracture for arkosic sandstone; 400 ^{o}C is the threshold temperature of thermal fracture from gentle to drastic and mineral particle fusion; 500 ^{o}C is the threshold temperature of particle recrystallization after fusion. When the treatment temperature is below 200 ^{o}C, the major factor is the thermal expansion of mineral particles, and the mechanical properties change a little at this stage; when the temperature is 200 ^{o}C ~400 ^{o}C, thermal fracture is the dominant factor, and temperature begins to exert larger influence on the mechanical property; when the temperature is 500 ^{o}C ~600 ^{o}C, mineral particles are partly melted and filled into the fractures, and the sandstone strength is thus restored to some extent; when the temperature is higher than 600 ^{o}C, thermal fracture, fusion and recrystallization jointly affect the mechanical properties, and the thermal fracture has larger influence at this stage. Arkosic deposits in deep high geothermal engineering are supposed to represent the similar mechanical behaviors when they are artificially disturbed and cooled.

Acknowledgments

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Nomenclature

σ	—the stress, [MPa]
ε	—the strain
T	—the temperature, [^oC]
σ_p	—uniaxial compressive strength, [MPa]
E	—the elastic modulus, [GPa]
v	—the longitudinal wave velocity, [ms^{-1}]
Q_m	—the volume of mercury intrusion, [ccg^{-1}]

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