The evolution of sandstone microstructure and mechanical properties with thermal damage

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Abstract
The physical and mechanical properties of rocks at high temperatures change considerably with geothermal exploitation, underground coal gasification, and nuclear engineering construction, posing a threat to the safety of underground engineering. To investigate the effect of temperature on micro- and macroscale damage of sandstone, a series of uniaxial compressive strength (UCS) tests were conducted using an MTS 815 mechanical testing system. Acoustic emission (AE) monitoring, scanning electron microscopy (SEM), and nuclear magnetic resonance (NMR) were also employed. Macroscopically, it was found that the physical and mechanical properties of sandstone change with treatment temperature, but these changes do not follow a monotonic trend. In addition, the brittle-ductile transition occurs at approximately 600°C, which is further confirmed by AE monitoring. Regarding the microstructural evolution of sandstone, the percentage of micropores shows a monotonically decreasing trend with increasing treatment temperature. The change in mesopores decreases slightly first, then shows a gradual increase, and finally decreases. The macropores first decrease and subsequently increase with increasing temperature. The decreasing trend of the meso- and macropores is attributed to thermal expansion at a relatively low temperature. However, the decrease in mesopores is due to their coalescence into macropores at higher temperatures. Furthermore, the integral value of the NMR spectrum first decreases and then increases with increasing treatment temperature, corresponding to the decrease in porosity from 25°C to 200°C, and then increases with temperature to 900°C. Finally, a constitutive model for the deformation and fracture of sandstone is established based on the effective medium theory and AE energy. The present study is helpful for improving the understanding of the process of thermal damage sandstone from both micro- and macroscale perspectives.

KEYWORDS
acoustic emission, microstructural evolution, nuclear magnetic resonance, sandstone, thermal damage
INTRODUCTION

Geothermal exploitation, underground coal gasification, and nuclear engineering construction have helped to reduce the consumption of conventional fossil resources. Generally, these geoengineering applications operate in subsurface geological formations and lead to the heating of the rock strata. As a result, the physical and mechanical properties of the original rocks deteriorate. Therefore, it is important to evaluate the thermal damage of rocks during heat treatment.

The thermal damage of rocks is currently a topic of interest in the fields of rock mechanics and engineering geology. Changes in the physical and mechanical properties of thermally treated rocks, including their mechanical properties, acoustic emission (AE) characteristics and wave velocity variations, and transmission properties (thermal conductivity, thermal diffusivity, permeability), have been extensively studied in laboratory tests. Significant variations in the physical and mechanical properties of rocks due to thermal damage have been observed in the abovementioned studies. Generally, with increasing treatment temperature, the strength and thermal conductivity of rock gradually decrease, while the AE events and permeability increase. Although extensive experimental studies have been conducted, the microstructural evolution of thermally treated sandstone remains unclear.

In addition to experimental investigations, many theoretical and numerical models have been proposed to investigate thermal damage of rock. For example, Sicsic and Bérest developed a fracture model to theoretically analyze the nucleation and propagation of salt, and Peng et al. used a phenomenological model to simulate the complete stress-strain curves of thermally treated coarse marble. Furthermore, Sirdesai et al. studied the effect of different temperatures and durations of thermal treatment on the mechanical characteristics of rocks and developed an effective numerical model.

The evolution of the mechanical properties of thermally treated rock is induced by microstructural changes in the rock matrix. Various techniques have been employed to reveal the evolution of microscopic cracks in rocks induced by thermal treatment. For instance, Ravalec et al. used gas adsorption and mercury injection methods to characterize the microstructure of granitic mylonite after undergoing different treatment temperatures. Using a coupled scanning electron microscopy (SEM) loading apparatus, Zuo et al. found that the threshold of thermal cracking of sandstone varied with the specific mineral content. Compared with the methods used in previous studies, nuclear magnetic resonance (NMR) provides quantitative data, which describe the microstructural evolution of coal and rock mass. Although it has an advantage in the visual observation of the surface topography at the microscale, SEM can only provide the local geometry of samples and cannot be used to derive quantitative information on the pore structure, especially inside the sample. Compared with SEM, NMR has been widely used to describe pores and fractures and determine the oil and water content and pore size distribution of water-saturated rock rapidly and accurately. In addition, as a nondestructive test method, NMR is advantageous for characterizing the pore structure of rocks.

Many scholars have discussed the physical and mechanical behaviors of sandstone exposed to various thermal treatment temperatures. However, studies of the microstructural evolution of thermally treated sandstone using NMR technique are scarce. Moreover, AE energy is used as a damage variable to characterize the deformation and fracture of thermally treated sandstone, which have not been fully understood in previous studies.

This paper reports the results of a series of uniaxial compressive tests on thermally treated sandstone conducted using the MTS 815 rock mechanics test system at a range of temperatures. Macroscale damage of the thermally treated sandstone was investigated first. Then, thermal damage of sandstone by single-polarization microscopy
sandstone was studied from the microscopic perspective using NMR. Finally, a constitutive model was established based on the effective medium theory and the damage variable defined by AE energy.

2 | MATERIALS AND METHODS

2.1 | Sample description and preparation

Sandstone blocks collected from Jiulongpo, Chongqing, China, were selected as rock samples in the experiment. Microphotographs of sandstone slices obtained by single polarization are shown in Figure 1. The studied sandstone is a granular cementitious material with medium-sized grains and an average grain size of approximately 0.2-0.3 mm. The apparent color of the in situ sandstone is blue-gray, and its density is 2346 kg/m³. To reduce the differences in the rock among the collected blocks, all the sandstone samples were cored from the same rock block. The dimensions of the rock samples were 50 mm in diameter and 100 mm in height. Next, the rock cores were trimmed, and the two ends of each cylinder were polished, using a lapping machine to create a height-to-diameter ratio of 2:1.24

Before the experiments, the P-wave velocity of the prepared sandstone samples was first measured using a rock parameter test (I-RPT) wave velocity apparatus to reduce sample heterogeneity. Samples with a similar P-wave velocity were selected for this study, and 18 rock samples were selected in total. Then, the prepared sandstone samples were thermally treated in the oven at the desired temperature for 3 hours so that the interior of the sandstone was uniformly heated.8 In the experiment, the 18 sandstone samples were divided into six groups of three specimens to improve test reliability. For each group, the treatment temperature was different, and the following temperatures were tested: 25°C, 200°C, 400°C, 600°C, 800°C, and 900°C. To avoid the collapse of the sandstone samples during the thermal treatment process, the heating rate was set to 5°C/min.8 After thermal treatment, the sandstone samples were slowly cooled to 25°C in the furnace. The apparent color of the thermally treated sandstone is illustrated in Figure 2. It is observed that apparent color of the sandstone did not change when the treatment temperature was less than 400°C. However, when the heat treatment temperature was higher than 400°C, the samples tended to be dark brown. As the temperature further increased, the apparent color of the sandstone gradually changed to brownish-red.

2.2 | Mineral composition of tested sandstone

The mineral composition plays a significant role in the evolution of the physical and mechanical properties of thermally treated rocks. Sandstone powders were tested using a D8 ADVANCE X-ray diffraction (XRD) device, made by Bruker AXS. The XRD spectrum of the sandstone is plotted in Figure 3. According to the XRD analysis, the composition of this sandstone is 41.2% quartz, 3.7% potassium feldspar, 34.5% plagioclase, 3.0% calcite, and 17.6% clay minerals.

2.3 | Experimental setup and procedure

In this study, a series of uniaxial compressive strength (UCS) tests were conducted on an MTS 815 servohydraulic testing machine with a maximum axial load capacity of 2600 kN. The machine comprises a control system, loading system, and data acquisition system. The control system is a servocontroller, the loading system is a hydraulic press, and the data acquisition system contains load and displacement sensors. The PCI-2 AE system was used to simultaneously capture the AE characteristics during the

**FIGURE 2** Apparent morphology of sandstone after exposure to different treatment temperatures
loading process. The AE sensor has a resonance frequency of 140 kHz, and a sensitivity of 115 dB, and its frequency range is 125-750 kHz. To reduce noise from the machines and the environment, the preamplifier and threshold values of the AE system were set to 40 dB and 45 dB, respectively. Moreover, to reduce the effect of end-friction between the machine and specimen, the two AE sensors were placed on the middle-upper and middle-lower parts of the sample. To better record the AE signals, Vaseline was applied as a coupling agent between the sandstone sample and the AE sensors.25

To investigate the pore characteristics and microstructure of the rock samples, NMR tests were performed on a MacroMR12-150H-1 rock core instrument and an associated core volume saturation device. The NMR parameters were as follows: 0.2 ms for the echo spacing (TE), 2048 for the maximum echo number, and 64 for the scanning number. The main magnetic field strength and radio frequency (RF) pulse were 0.3 T and 1.0-42 MHz, respectively. The P-wave velocity of the sandstone was measured by an I-RPT wave velocity apparatus, which had a sampling interval of 0.1-200 μs and five recording angles. The apparatus amplification gain was 82 dB, the emission pulse width was 0.1-100 μs, and the bandwidth was 300-500 Hz.

During the experiment, the mass, volume, density, and P-wave velocity of the sandstone were measured before and after heating.
after thermal treatment. The thermally treated rock block was then loaded into an MTS 815 servohydraulic testing machine with displacement control, at a loading rate of 0.05 mm/min. The AE signal was recorded synchronously during the uniaxial loading process. Subsequently, NMR, SEM, and magnetic resonance imaging (MRI) tests were carried out to investigate the microstructure of the rock samples after thermal treatment.

3 | RESULTS AND DISCUSSION

3.1 | Physical properties of sandstone after thermal treatment

Figure 4 shows the change in the mass, volume, density, and P-wave velocity of the sandstone before and after thermal treatment. Figure 4A shows that the change in mass decreases with the increase in treatment temperature, following a nonlinear concave-upward trend. Specifically, when the treatment temperature is lower than 400°C, the decrease in the mass is approximately 4%-8%. When the treatment temperature increases from 600°C to 900°C, the rate of decrease in mass slightly increases, and the decrease in mass is equal to or less than approximately 16%. This decrease in mass of the sandstone is related to water loss during the thermal treatment. Previous studies have shown that the internal water of sandstone is mainly adsorbed water and mineral-containing water. With the increase in treatment temperature from 0°C to 110°C, the absorbed water within the sandstone gradually evaporates. With a further increase in treatment temperature, the mineral-containing water starts to escape from the heated sandstone.\(^7\)\(^,\)\(^26\) In addition, the mineral constituents decompose at a high treatment temperature. The water starts to desorb from the clay minerals at a temperature ranging from 25°C to 220°C.\(^27\) The clay minerals and calcite decompose at 400-700°C and 700-830°C, respectively. At 573°C, the α-β transition occurs in quartz.

The reactions listed above not only lead to the mass loss of heated sandstone samples, but also change the volume of the sandstone. This is reflected by the change in the volume of the sandstone samples, as shown in Figure 4B. To avoid uneven deformation of the sandstone sample in the heating process, the volume of the heated samples is measured by drainage. For treatment temperatures lower than 200°C, no apparent volume change occurs in the samples. With the increase in treatment temperature from 200°C to 400°C, the sandstone sample volume slightly increases. For greater treatment temperatures, the sandstone sample volume increases drastically. In the case of 900°C, the volume increases by 3.25%, which is two times higher than that at 600°C. This phenomenon can be explained by the increase in the distance between particles at higher temperatures.

Based on the mass and volume of the rock samples, the change in the density of the rock is obtained, as depicted in Figure 4C. According to the least squares fitting method, the rate of decrease in the density is 0.165 kg/m\(^3\)/K, and the density reduces by 3.17% at 600°C and 6.06% at 900°C.

Figure 4D shows the variation in the P-wave velocity of the sandstone sample with thermal treatment. The P-wave velocity of sandstone decreases with increasing treatment temperature, following a piecewise linear function. For treatment temperatures from 25°C to 400°C, the rate of decrease in the P-wave velocity is 0.753 m/s/K, and the average P-wave velocity decreases by 9.35%. However, when the treatment temperature increases from 400°C to 900°C, the rate of decrease in the P-wave velocity increases to 2.079 m/s/K. During the entire process of thermal treatment, the average P-wave velocity drops from 3306 m/s prior to thermal treatment to 1486 m/s at the treatment temperature of 900°C, a reduction of 55.05%. This rapidly decreasing trend in the P-wave velocity of the sandstone at treatment temperatures of 400-900°C is caused by the decomposition of the clay constituents, as mentioned previously, leading to microcracks.

3.2 | Mechanical properties of sandstone after thermal treatment

3.2.1 | Stress-strain curves of sandstone

Figure 5 shows the stress-strain curves of the sandstone after thermal treatments of 25°C, 200°C, 400°C, 600°C, 800°C, and 900°C. Clearly, the deformation and mechanical behaviors of the thermally treated sandstone deteriorated significantly. With increasing treatment temperature, the axial strain at peak stress increases markedly. This observation is consistent with the results of a previous study conducted by Xu et al.\(^28\) With the increase in treatment temperature from 400°C to 900°C, the rock exhibits ductile behavior near the
peak stress region. This phenomenon is mainly caused by the initiation and propagation of cracks during heat treatment. Notably, the UCS tests were conducted after the specimens cooled instead of during the heating process.4,29

3.2.2 | Strength and deformation behaviors of sandstone exposed to different temperatures

The mechanical properties of the thermally treated sandstone specimens are listed in Table 1. The crack damage stress ($\sigma_{cd}$) is defined as the axial stress when the volumetric strain of the sandstone switches from positive to negative.24,30 The crack initiation stress is defined as the stress at which the crack volume strain starts to dilate.31 The crack damage strain ($\varepsilon_d$) corresponds to the strain under the crack damage stress.

Poisson’s ratio $\mu$ is calculated by averaging the ratio of the radial strain to the axial strain in the elastic deformation phase.

The changes in peak stress, axial strain at peak stress, Young’s modulus, shear modulus, bulk modulus, and Poisson’s ratio are plotted in Figure 6. Figure 6A shows that the maximum Young’s modulus occurs at 200°C. The average peak stress increases by 14% when the temperature increases from 25°C to 200°C and decreases by 44.65% as the temperature increases from 200°C to 900°C. First, a gradual reduction in peak stress is observed as the temperature increases from 200°C to 800°C. Then, the average UCS decreases drastically when the temperature exceeds 800°C. At 400°C, the average UCS of the sandstone decreases to 65.05 MPa, which is approximately equal to the UCS at 25°C. In total, the strength decreases by 26.98% at 900°C compared with that at 25°C.

Figure 6B shows the evolution of the axial strain of the sandstone at the peak stress with increasing thermal treatment temperature. Overall, the axial strain increases slightly when the treatment temperature is less than 200°C. Nevertheless, for temperatures greater than 400°C, the rate of increase in the axial strain increases dramatically.

Figure 6C shows that the trend of Young’s modulus is similar to that of the peak stress with increasing thermal treatment temperature. Young’s modulus increases by 13.9% when the temperature increases from 25°C to 200°C and then decreases monotonically to 6.36 GPa at 900°C.

Poisson’s ratio is an important property, which represents the deformation of rocks, and its evolution with temperature is shown in Figure 6D. The change in Poisson’s ratio follows a generally decreasing trend with temperature from 25°C to 600°C and then switches to an increasing trend at higher temperatures. According to Kumari et al. and Greaves et al.32 such a change in Poisson’s ratio marks the brittle-ductile transition. The initial decrease in Poisson’s ratio is relatively small in magnitude, with a reduction of 72.5% from 25°C to 600°C. However, Poisson’s ratio increases by 389% from 600°C to 900°C. Poisson’s ratio is lowest at 600°C. The variation in Poisson’s ratio is consistent with that observed in

**TABLE 1** Strength and deformation parameters of sandstone at different temperatures

| Specimen | T (°C) | $\sigma_c$ (MPa) | $\sigma_{cd}$ (MPa) | $\varepsilon$ ($10^{-2}$) | $\varepsilon_d$ ($10^{-2}$) | $E$ (GPa) | $\sigma_i$ (MPa) | $\mu$ |
|----------|--------|------------------|--------------------|-----------------|-----------------|--------|---------|-------|
| A1       | 25     | 65.97            | 42.08              | 0.538           | 0.192           | 16.85  | 17.93   | 0.145 |
| A2       | 25     | 62.78            | 39.80              | 0.519           | 0.205           | 17.67  | 16.51   | 0.133 |
| A3       | 25     | 63.41            | 40.41              | 0.419           | 0.125           | 18.23  | 14.54   | 0.196 |
| B1       | 200    | 85.69            | 60.99              | 0.595           | 0.265           | 18.94  | 14.63   | 0.132 |
| B2       | 200    | 86.35            | 59.68              | 0.569           | 0.231           | 20.18  | 19.90   | 0.147 |
| C1       | 400    | 71.94            | 48.92              | 0.538           | 0.199           | 16.05  | 18.20   | 0.114 |
| C2       | 400    | 67.54            | 44.79              | 0.435           | 0.143           | 17.04  | 20.12   | 0.159 |
| C3       | 400    | 69.61            | 43.68              | 0.532           | 0.204           | 17.01  | 18.72   | 0.116 |
| D1       | 600    | 70.34            | 46.74              | 0.774           | 0.497           | 13.14  | 26.71   | 0.034 |
| D2       | 600    | 63.80            | 41.48              | 0.770           | 0.445           | 13.12  | 21.64   | 0.074 |
| D3       | 600    | 66.79            | 42.78              | 0.809           | 0.544           | 12.92  | 23.96   | 0.023 |
| E1       | 800    | 60.14            | 37.02              | 0.102           | 0.653           | 9.40   | 21.95   | 0.070 |
| E2       | 800    | 60.99            | 37.57              | 0.117           | 0.631           | 9.72   | 22.95   | 0.112 |
| E3       | 800    | 63.14            | 38.31              | 0.103           | 0.631           | 10.31  | 23.65   | 0.072 |
| F1       | 900    | 47.23            | 24.08              | 0.121           | 0.580           | 6.35   | 17.98   | 0.210 |
| F2       | 900    | 48.74            | 24.99              | 0.119           | 0.537           | 6.52   | 17.19   | 0.220 |
| F3       | 900    | 46.55            | 25.97              | 0.119           | 0.549           | 6.22   | 18.08   | 0.208 |

Abbreviations: $E$, Young’s modulus; $\varepsilon$, peak axial strain; $\varepsilon_d$, strain at the crack damage stress; $\mu$, Poisson’s ratio; $\sigma_c$, unconfined compressive strength; $\sigma_{cd}$, crack damage stress; $\sigma_i$, crack initiation stress.
a previous study carried out by Greaves et al.\textsuperscript{32} The main reason for the variation in Poisson's ratio is the \( \alpha-\beta \) transition in the quartz at 573°C.

As shown in Figure 6E,F, the trends of the shear and bulk modulus are similar to those of the peak stress with increasing thermal treatment temperature. However, the evolution...
trends of Young’s modulus and Poisson’s ratio with increasing thermal treatment temperature are distinctly different. Moreover, the shear and bulk modulus are related to not only Young’s modulus but also Poisson’s ratio. The trend of Young’s modulus is similar to that of shear and bulk modulus with increasing thermal treatment temperature. This similarity arises because the shear, bulk, and Young’s modulus all reflect the rock stiffness. Therefore, the stiffness should also first increase and then decrease with increasing thermal treatment temperature, and the reduction is much greater than the increase as the temperature increases from 25°C to 900°C. Therefore, the ability of sandstone to ultimately resist deformation under axial loading decreases, contributing to the final increase in axial strain prior to reaching the peak stress, as shown in Figure 6B. Furthermore, the bulk modulus exhibits a much less pronounced softening behavior than that of the shear modulus when the temperature exceeds the brittle-ductile transition, and stabilizes as the temperature continues to increase from 600°C to 900°C.

Figure 7 shows the evolutions of the normalized peak stress and Young’s modulus of sandstone obtained from this study and the studies of Rao et al. 33 Zhang et al. 34 and Ranjith et al. 5 Overall, the normalized peak stress increases and then decreases gradually with increasing temperature (Figure 7A). However, the change in normalized peak stress first decreases, then increases, and finally decreases with increasing temperature. 34 In addition, the deflection of maximum normalized peak stress occurs at different temperatures for various sandstones. These significant differences reflect the variation in the geological history, physical properties, and mineral compositions of the tested sandstones. Unlike the normalized peak stress, the normalized Young’s modulus results for the different sandstones present a similar trend (Figure 7B).

### 3.2.3 Energy characteristics of sandstone after temperature treatment

The deformation and failure processes of rock are accompanied by energy dissipation and release 35,36. Therefore, energy theory is used to further reveal the mechanical mechanism of the deformation and failure of sandstone at different treatment temperatures. The total input energy $U_0$ comes from the external loading force. In general, the total energy includes the elastic energy $U_e$ and the dissipation energy $U_d$. Accumulated $U_e$ in a rock mass produces reversible elastic deformation, while $U_d$ produces irreversible deformation due to crack development.

In the process of uniaxial compression, the total energy, elastic energy, and dissipated energy of sandstone before the peak stress can be calculated as follows:

$$U_0 = AH \int \sigma_1 \, d\varepsilon_1 = A H \sum_{i=1}^{n} \frac{1}{2} (\sigma_{1i} + \sigma_{1i-1}) (\varepsilon_{1i} - \varepsilon_{1i-1})$$

(1)

$$U_e = A H \frac{\sigma^2}{2E}$$

(2)

$$U_d = U_0 - U_e$$

(3)

where $U_0$ is the total work performed by the axial loading before the peak stress, $U_e$ is the elastic energy accumulated before the peak stress, $U_d$ is the dissipated energy before the peak stress, $A$ is the loading area of the sandstone sample, and $H$ is the sample height.

The accumulated elastic energy and energy release of sandstone at different temperatures are calculated based on Equations (1)-(3). Figure 8 shows the change in elastic energy...
and dissipation energy of sandstone at different temperatures. The proportion of elastic energy gradually decreases, while the proportion of dissipated energy gradually increases with the increase in treatment temperature. At a lower temperature, the amount of elastic energy accumulated before reaching the peak stress of the sandstone is higher, indicating the relatively high integrity of this sandstone. For the energy dissipated under uniaxial compressive loading before peak stress, its proportion increases with increasing thermal treatment, indicating that the load bearing capacity of sandstone is weakened at higher temperatures.

### 3.2.4 | AE characteristics during uniaxial compression

The AE technique has been widely used to capture crack initiation, propagation, and coalescence in rocks under applied loading.\(^7,10,37\) Figure 9 shows the curves of the axial stress, AE energy, and accumulated AE energy with time at different treatment temperatures. The maximum AE energy of a single AE event generally initially increases and then decreases with increasing temperature from 25°C to 900°C. On the other hand, the AE event counts increase with thermal treatment temperature. During the entire process of unconfined compressive loading, the AE events show distinct characteristics at different treatment temperatures. Specifically, at a temperature of 25°C, the curve of accumulated AE energy vs loading time levels off before the peak stress and suddenly increases at the peak stress point. Accordingly, the AE energy release is negligible before the peak stress and increases dramatically afterward, thus reflecting the high brittleness of sandstone. With increasing temperature, the frequency of AE events intensifies near the peak stress region. In addition, a single AE occurs in the early loading stage, and the AE event frequency increases with temperature, especially for treatment temperatures greater than 600°C. Because the heat treatment leads to an increase in internal damage in the sandstone, the closure and propagation of microcracks will inevitably produce additional AE events during loading. Therefore, the AE events of sandstone are gradually enhanced by heat treatment.

Based on the AE characteristics of thermally treated sandstone, the entire loading process of the sandstone specimens is divided into four phases. The first phase, compaction phase, starts at the start of loading and continues to point A. In this phase, the UCS vs time curves of the sandstone show a nonlinear concave downward trend, irrespective of treatment temperature. Additionally, the AE features at temperatures of 25-400°C are different from those at temperatures of 400-900°C. During the first phase, at temperatures between 25°C and 400°C, few AE events occur. However, at treatment temperatures in the range of 400°C to 900°C, the number of AE events gradually increases with the treatment temperature. The second phase, known as elastic deformation stage, starts from point A and continues to point B. In this phase, at temperatures between 25°C and 600°C, very few AE events occur. However, the number of AE events increases when the temperature exceeds 600°C. The third phase is defined as stable crack propagation stage, which spans from point B to point C. In this phase, AE events occur more frequently; however, the magnitude of the AE energy of each AE event is low. Considering the deformation rule of rocks under uniaxial compressive loading, the AE events that appear in this phase may still be caused by regional microcracks, although the number of microcracks that appear in this phase is higher than that in the phase from point A to point B. Shao et al.\(^38\) took the stress at point B as the crack initiation stress threshold. The fourth phase is the unstable crack propagation stage, which ranges from point C to point D. In this stage, the heaviest and strongest AE events are obtained due to the evolution of microdamage into the macrofailure. Furthermore, the time required for the sandstone to fail increases with the treatment temperature at the same loading rate. Meanwhile, the tendency of the final dramatic increase in accumulated AE energy decreases with increasing temperature. Because the heat treatment increases the pore size of the sandstone, the elastic compaction stage gradually lengthens, and the concave-upward trend becomes increasingly obvious, causing the elastic stage to be further lengthened. Therefore, the time to specimen failure increases with increasing treatment temperature.

### 3.3 | Structural evolution of thermally treated sandstone

#### 3.3.1 | \(T_2\) spectrum of sandstone

To quantify the thermally induced microstructural change, low-field NMR was employed. Nuclear magnetic resonance measurement is based on the theory that the protons in fluid
molecules fill the pore space of a rock matrix. Three possible relaxation mechanisms include diffusion relaxation, bulk relaxation, and surface relaxation. Using diffusion relaxation, the NMR signal can be weakened when a low magnetic field and short pulse spacing are used. The NMR signal can also be weakened by employing bulk relaxation because of the presence of nonviscous water in the nonmagnetic rock samples. Therefore, the surface relaxation mechanism is used there. According to the surface relaxation mechanism of NMR, the solid-fluid interaction is the dominant relaxation mechanism and the relaxation rate can be expressed as follows:

\[ \frac{1}{T_2} = \rho \frac{S}{V} = \rho \frac{C}{R} \]

where \( S \) is the surface area of the pore, \( V \) is the volume of the pore, \( \rho \) is the surface relaxation intensity, \( R \) is the pore radius,
and $C$ is the shape factor (the $C$ values of spherical pores, cylindrical pores, and fractures are 3, 2, and 1, respectively). When the sample is saturated and the surface relaxation intensity parameters are known, the distribution of the relaxation time can be used to derive the distribution of the pore size with Equation (4). The distribution of the $T_2$ spectrum can be used to not only analyze the number, size, and peak position of the pore structure, but also interpret the degree of damage of the thermal treatment.

Figure 10 shows the evolution of the NMR $T_2$ spectra of thermally treated sandstone at different temperatures. According to Equation (4), assuming that the surface relaxation intensity $\rho$ is invariable, the water in smaller pores experiences a shorter relaxation time, as the smaller pores correspond to a larger surface-to-volume ratio. Here, the first peak of the $T_2$ spectrum corresponds to adsorption pores, and the second and the third peaks correspond to seepage pores. The multiple peaks of the $T_2$ spectrum imply that the studied sandstone has a complex pore structure. With the increase in treatment temperature from 200°C, the integral value of the $T_2$ spectrum increases gradually, indicating that the degree of damage to the sandstone increases. Overall, the sandstone experiences a slight decrease in porosity, while the treatment temperature increases from 25°C to 200°C, and the
sandstone porosity then increases linearly with temperature to 900°C.

According to the $T_2$ spectrum results, the area of each peak and the total number of peaks can be calculated. The peak area is calculated by mathematical integration. Figure 11 illustrates the percentage of each peak area and the total area of the $T_2$ spectra of sandstone samples after thermal treatment at different temperatures. The total area first decreases slowly and then increases significantly as the temperature exceeds 400°C. Meanwhile, the total number of peaks after different temperatures changes slightly. With increasing temperature, the percentage of the first peak area decreases sharply from 58.9% at 25°C to 0.02% at 900°C. However, within the same temperature range, the percentage of the second peak area increases slowly from 40.9% to 59.3%, and the percentage of the third peak area increases significantly from 0.2% to 40.7%.

### 3.3.2 Pore structure evolution of sandstone

Pores and microfractures are usually divided into micropores (<10^2 nm), mesopores (10^2-10^3 nm), macropores (10^3-10^4 nm), and microfractures (>10^4 nm). According to such a classification, Figure 12 shows the evolution of the pore throat size of the sandstone after thermal treatment.

Figure 12A shows the evolution of micropores before and after temperature treatment. Overall, the percentage of micropores follows a decreasing trend when the temperature increases from 25°C to 900°C. In contrast to the micropores, the percentage of mesopores shows a slight decreasing trend, and then increases gradually with increasing temperature until 800°C. After 800°C, the change in mesopores starts to decrease. This phenomenon can be explained by the mesopores gradually evolving into macropores at higher temperature. Figure 12C shows the trend of the variation in macropores. The macropores gradually decrease and then increase with increasing temperature.

To observe the pore structure of thermally treated sandstone, SEM (TESCAN MIRA3) at a magnification of 500 is used, as shown in Figure 13. Figure 13A shows the original pores of the sandstone at 25°C. When the temperature increases from 25°C to 200°C, closure of the original small pores occurs due to thermal expansion among the mineral particles. This finding is consistent with the observed evolution of the $T_2$ spectrum as shown in Figure 10. However, there is not enough void space for particle expansion when the temperature rises to 400°C, which results in the development of pores with a larger average size than that at 25°C. In addition, thermal expansion causes a gradual increase in the specimen volume, as shown in Figure 4B, and a slow decrease in the P-wave velocity, as shown in Figure 4D. When the treatment temperature increases to 600°C, microcracks are observed, as shown in Figure 13D, and intensify with a further increase in the temperature. At 573°C, the $\alpha$-$\beta$ transition occurs in quartz, leading to volume expansion. The $\alpha$-$\beta$ transition occurs in quartz, leading to volume expansion.
transition of the quartz constituent may lead to the initiation of microcracks, as the sandstone used in this study contains a significant amount of quartz (Figure 3). This transition also causes a decrease in the macropores, as shown in Figure 12C. With the continual increase in temperature, the thermal stress exceeds the tensile strength of the sandstone matrix, and cracks begin to propagate. As a result, a large number of isolated pores and cracks form in the specimens, as shown in Figure 13E. At 800°C, calcite decomposes and the bonding among particles further breaks and new cracks generate around the particles. Therefore, the evolution of mesopores and macropores into microcracks results in a decrease in the percentage of mesopores and macropores at higher treatment temperatures. The decrease in the percentage of macropores starts at 600°C, whereas the decrease in mesopores starts at 800°C. These changes can be expected because the coalescence of pores into microcracks starts with the larger pores. Eventually, the number of cracks and the connectivity of the cracks increase significantly, and crack coalescence is observed in the sample at 900°C, as shown in Figure 13F. The evolution of mesopores and macropores into microcracks is caused by the degumming of clay minerals, ultimately leading to a decrease in UCS, as shown in Figure 6A.

Figure 14 illustrates the change in porosity of the sandstone at different treatment temperatures. The change in porosity decreases as the temperature increases from 25°C to 200°C. The average change in porosity drops by 3.448% as the temperature increases from 25°C to 200°C. This variation trend is similar to that of the total peak area of the $T_2$ spectrum, as shown in Figure 10. This phenomenon can be explained by the fact that the compact structure of mineral grains results from thermal stress. However, as the treatment temperature exceeds 200°C, the change in the sandstone porosity starts to increase linearly with temperature. As the temperature increases from 400°C to 900°C, the average percentage in porosity increases from 0.39% to 21.46%, implying that large numbers of microcracks form and thermal deterioration occurs in the sandstone. At 900°C, the average porosity of the sandstone reaches a maximum of 16.17%, which is an increase of 21.46% than that of its initial state. A comparison of Figures 6 and 14 shows that the macroscale mechanical property evolution of sandstone is consistent with the pore structure change induced by thermal treatment. Although the micropores, mesopores, and macropores show different trends at different stages of thermal treatment, the

![Figure 14](image1.png)  
**Figure 14** Change in porosity with treatment temperature

![Figure 15](image2.png)  
**Figure 15** Unified mapping of MRI images for sandstone samples at different treatment temperature: (A) 25°C; (B) 200°C; (C) 400°C; (D) 600°C; (E) 800°C; and (F) 900°C
thermal damage is almost irreversible at higher treatment temperatures, which causes the rapid decrease in the UCS of the sandstone.

### 3.3.3 | MRI and fractal characteristics of sandstone at various temperatures

According to the different energy release rates, images of the internal structure of sandstone can be investigated. Based on the principle of unified mapping of MRI images, the brightest image of all the MRI images is taken as the reference for unified mapping. Then, normalized image is obtained by unifying the original MRI image using image processing software. Finally, the deterioration characteristics of the internal microstructure can be directly observed by comparing the unified mapping of the images. Along the axial direction of the cylindrical sandstone specimens, two-dimensional (2-D) transverse cross-sectional images are obtained. Figure 15 shows the unified mapping of the MRI images of the sandstone sample at different temperatures. The bright area is the sample image, and navy blue is the background color. The brightness gray-scale level of the image directly reflects the amount of water in the sample. According to the principle of NMR, Figure 15 indirectly reflects that the NMR signal intensifies gradually with increasing temperature. The change in the signal intensity of the MRI image presents a trend that is essentially consistent with the change in porosity. At 25°C, the image is relatively blue compared with the images at other temperatures, and there are only a few blue spots in the image. At 400°C, yellow spots gradually occur in the image, indicating that the

| $T$ (°C) | $\sigma_{ucs}$ (MPa) | $E$ (GPa) | $\epsilon_c$ | $\alpha$ (%) | $\beta$ (MPa) | $R^2$ |
|---|---|---|---|---|---|---|
| 25 | 63.41 | 18.53 | 0.000407 | 0.448 | 47.94 | 0.999 |
| 200 | 86.34 | 19.99 | 0.000829 | 0.330 | 25.60 | 0.999 |
| 400 | 69.61 | 17.84 | 0.000908 | 0.323 | 20.22 | 0.997 |
| 600 | 63.80 | 13.15 | 0.002518 | 0.557 | 16.28 | 0.998 |
| 800 | 63.13 | 10.31 | 0.003949 | 0.749 | 13.64 | 0.999 |
| 900 | 46.56 | 5.62 | 0.003698 | 1.149 | 19.76 | 0.997 |
deterioration of the internal microstructure of sandstone gradually increases. With further increases in temperature, more red spots appear in the MRI cross-sectional image, indicating the development of new internal pore structures due to the thermal treatment. At 900°C, the red spots nearly cover the entire cross section, demonstrating that severe deterioration occurs in the sandstone (shown in Figure 13F).

Fractal theory has been used to study the characteristics of irregular pore distributions in rocks. The fractal dimension can be used to evaluate the surface roughness and interior structural irregularity of pores. The fractal dimension of pore geometry characteristics can be calculated by using the box-counting method. Here, the 2-D box-counting method is employed to calculate the fractal dimension of the MRI images. The fractal dimension can be defined as follows:

\[
D = \lim_{\delta \to \infty} \frac{\log N(\delta)}{\log \delta}
\]  

where \(D\) is the fractal box dimension, \(\delta\) is the box size, \(\alpha\) is a proportionality constant, and \(N(\delta)\) is the number of self-similar parts.

The evolution of the fractal dimension of the MRI images with increasing treatment temperature is shown in Figure 16. The fractal box dimension of the MRI images is negatively correlated with the increase in thermal damage. The fractal box dimension increases slowly at first (the fractal box dimension peaks at 200°C), and then decreases gradually with increasing temperature until 900°C. This change is due to the development of mesopores and macropores, which results in decreases in the structural irregularity and surface roughness of the pores, leading to a reduction in the fractal dimension.

The microscopic observations show that the thermal expansion of mineral grains increases the strength of rock by closing the original small pores as the temperature increases from 25°C to 200°C. When the temperature increases to 600°C, microcracks are observed in the sandstone, indicating the brittle-ductile transition of the sandstone. However, as the temperature increases to 900°C, the coalescence of boundary cracks and transgranular cracks leads to a significant reduction in the UCS of the sandstone.

3.4 | Damage evolution and modeling of sandstone

3.4.1 | Effective medium theory

According to the effective medium theory proposed by Gueguen and Sarout, axial strain is composed of crack axial strain and matrix axial strain. Therefore, the stress-strain curve can be divided into two parts at the crack closure stage. The crack closure model at the crack closure stage can be calculated as:

\[
\varepsilon = \varepsilon^m + \varepsilon^c
\]

where \(\varepsilon^c\) is the crack axial strain, and \(\varepsilon^m\) is the matrix axial strain.

In order to simulate the crack closure stage of the stress-strain of thermally treated sandstone, crack axial strain and matrix strain are calculated using the following formulas:

\[
\varepsilon^c = a \left[ 1 - \exp \left( -\frac{\sigma}{\beta} \right) \right] \]  

\[
\varepsilon^m = \frac{\sigma}{E}
\]

where \(a\) is the maximum crack closure strain, \(\beta\) is a stress constant, \(\sigma\) is the axial stress, and \(E\) is the Young’s modulus.

The fitting parameters of \(a\) and \(\beta\) at different temperatures are shown in Table 2. The crack closure stage under different temperatures is simulated using Equation (7). The experiment results and the simulated results with of Equation (7) are shown in Figure 17.

3.4.2 | Damage variable and modeling

Acoustic emission events have been widely used to capture crack initiation, propagation, and coalescence in rocks from initial loading to final instability and failure. Previous studies have shown that damage variables are usually defined based on the AE counts. In the present study, the damage variable \(D\) is defined based on the cumulative AE energy. The damage variable is defined as:

\[
D = \frac{E_d}{E_0}
\]

where \(D\) is the damage variable, \(E_d\) is the cumulative AE energy in each stage, and \(E_0\) is the cumulative AE energy in the entire stage.

The damage of rock under thermal treatment is a gradual progressive process. The constitutive equation can be given as:

\[
\sigma = (1-D) E (\varepsilon - \varepsilon^c)
\]

By combining Equations (6), (7), (8) and (10), the constitutive model of thermally treated sandstone can be given as:

\[
\begin{cases}
\varepsilon = \frac{\sigma}{E} + a \left[ 1 - \exp \left( -\frac{\sigma}{\beta} \right) \right], \varepsilon \leq \varepsilon^c \\
\sigma = (1-D) E (\varepsilon - \varepsilon^c), \varepsilon > \varepsilon^c
\end{cases}
\]

The experimental results and the theoretical results of Equation (11) are illustrated in Figure 18. It is found that the
predicted results and the experimental results show a similar trend.

4 CONCLUSIONS

In this study, the microstructural evolution and thermal damage of sandstone were experimentally investigated by conducting uniaxial compressive tests. Based on the experimental results, the following conclusions can be drawn:

1. According to the results obtained from NMR, the integral value of the $T_2$ spectrum increases gradually, when the treatment temperature exceeds 200°C, indicating that the thermal damage of the sandstone increases at an increasing rate. The integral of the $T_2$ spectrum first decreases and then increases, indicating a decrease in porosity from 25°C to 200°C and an increase from 200°C to 900°C. With the increase in treatment temperature, the variations in the percentage of micropores, mesopores, and macropores show different trends, and the micropores show a monotonic decreasing trend with increasing temperature. The mesopores first decrease, then increase, and finally decrease. The macropores gradually decrease and then increase with increasing temperature. Using the box-counting method, the fractal box dimension shows great potential as an index to describe the thermal damage of sandstone.

2. Regarding the macroscale mechanical properties, the peak stress and Young’s, shear, and bulk modulus of the sandstone increase with treatment temperature from 25°C to 200°C, and then decrease up to 900°C. The Poisson’s ratio generally decreases as the treatment temperature increases from 25°C to 600°C but increases as the temperature further increases. At a lower temperature, the sandstone samples present typical brittle failure characteristics, and the brittle-ductile transition occurs at approximately 600°C. As the treatment temperature increases from 25°C to 900°C, the maximum AE energy of a single AE event first increases and then decreases, and the frequency of AE events intensifies around the peak stress region.

3. Combining the effective medium theory and the damage variable defined by AE energy, a constitutive model was established to simulate the deformation and fracture of thermally treated sandstone. The theoretical results and the experimental results present a similar trend.

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