TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF RED SANDSTONE

by

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In the underground rock diagenetic process, or artificial excavation in rock engineering, it is possible to make experience in the high temperature. The temperature dependence of Young's modulus of red sandstone was characterized by impulse excitation technology. It is shown that after heating-cooling cycle the Young's modulus of red sandstone decreases down to about 90% from the room temperature up to 800 °C of its initial value. The significant change of Young's modulus with temperature is attributed to thermal-induced damages in red sandstone, including the rock composition variation, mass loss, mineral phase composition change, porosity increasing, grain trans-granular, micro-cracks penetrated and disruption.

Key words: *red sandstone, impulse excitation technique, heating-cooling cycles, Young's modulus*

Introduction

With the industrial occupation of understanding space and exploitation of underground resources, for example, in underground chambers heat can be generated by the high temperature geothermal resources, geothermal heat extraction, geological CO_2 storage, underground disposal of nuclear waste, underground coal gasification [1-5] and so on. Moreover, high temperatures can significantly affect the mechanical behavior of rocks and thus further influence the reliability of relevant engineering structures [6, 7]. Up to now, many scholars have studied the variation of macroscopic rock properties caused by high temperature. Fredrich *et al.* [6] indicated that the thermally induced crack density is dependent upon the temperature, thermal expansion mismatch, thermal expansion anisotropy, initial crack porosity and grain size. Homand-Etienne *et al.* [7] showed that during the thermal treatment from 20-600 °C, the crack lengths barely evolved whereas their width increased with increasing with temperature. Ferrero *et al.* [8] found that two different types of marbles have been heated at different temperatures to induce micro-cracking at different temperatures up to 600 °C. Zhang *et al.* [9] studied the characteristics of mineral formation, micro configuration and inner micro thermal crack development have been made in micro scale in the laboratory under the different high temperature up to 810 °C. Zhao *et al.* [10] studied the effect of temperature on the damage in sandstone up to 600 °C by ultrasonic wave method and the damage factor will increase as the temperature increases. Zuo *et al.* [11] using in-situ SEM observation and they found two threshold temperatures of thermal cracking and three thermal cracking models in the sandstone. Ranjith

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et al. [12] found that the compressive strength and the elastic modulus of sandstone first increased with increasing temperature up to 500 °C and then decreased above 500 °C. Tian *et al.* [13] revealed that rock micro-structures changes significantly at high temperatures based on an extensive review of international literature. Sengun [14] revealed that the values of bulk density, P-wave velocity, uniaxial compressive strength, modulus of elasticity, Brazilian tensile strength and shore hardness decreased to a different extent, whilst apparent porosity increased under the influence of heat up to 600 °C. Rong *et al.* [15] found that 300 °C is the damage threshold temperature and in the range of 300-600 °C, significant damage of limestone would occur based on the SEM experiments, mercury injection experiments and P-wave velocity tests. Above all, so it is very important to study the physical and mechanical properties of rocks in the laboratory to evaluate the thermal damage at different temperatures.

As well known, the Young's modulus is one of the important engineering material parameters, which is commonly used in engineering design parameters. Determination of Young's modulus is of great significance on the Mechanical properties of metallic materials of various materials, optical materials, semiconductors, nanomaterials, polymers, ceramics and rubbers, but also for the field of mechanical parts and engineering geology design and so on. It's surprising that the temperature dependence of the Young's modulus of these materials is still fairly unknown. This study aims to fill the aforementioned gaps and tries to contribute to a more rational understanding of the differences on the Young's modulus of red sandstone under the high-temperature cycles. The related experimental data will be provided a reliable basis for the microstructural characterization of the materials and some additional key information for further research concerning the high-temperature up to 800 °C, hopefully leading to a more profound understanding of these complex materials.

Experimental set-up and characteristics method

The test rock material was a type of red sandstone, which is the most typical sedimentary rock and dark red in the natural state was collected from the eastern China hilly areas. All the samples (44 mm × 14 mm × 5 mm) were cut from a large block of red sandstone with the same orientation. The surfaces of the samples were carefully polished by abrasive paper (#800) according to the American Society for Testing and Materials (ASTM) standard 1876. Before measurement, the red sandstone brick specimens were suspended by thin metal wires in an IET furnace (IEMC-RF) as illustrated in fig. 1.

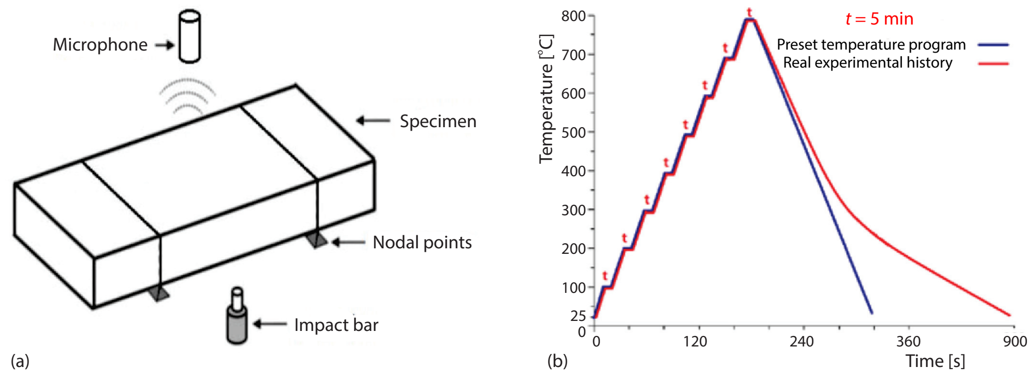


Figure 1. (a) Impulse excitation technique experimental set-ups for Young's modulus testing, (b) the pre-set and real heating-cooling cycle temperature program

During the test, the sound produced from the vibration of a sample, excited every 20 seconds by the impact bar, was transmitted along a rock bar and captured by a high-precision microphone outside the furnace. The Young's modulus can be calculated by the following equation:

$$E = 0.9465(mf^2)(L^3/wh^3)\Psi_{bar} \tag{1}$$

where m the mass of the bar, f – the fundamental flexural resonant frequency of the bar, L – the length, w – width, h – the thickness, and $\Psi_{bar} = 1 + 6.585\ (h/L)^2$ – the shape-correction factor. For the rock brick heating-cooling cycling test, the samples went through heating treatment from room temperature up to 800 °C at the rate of 5 °C/min. In addition, in the heating process the temperature will be held at 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, and 800°C for 5 minutes, respectively, shown in the fig. 1(b). In order to study the mass change after the different heating cycle condition of red sandstone, the different samples of the same rock heat treatment at 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, 700 °C, and 800 °C, respectively. Then investigate the mass loss after the heating-cooling cycle. Meanwhile, further to make an analysis of the porosity of red sandstone by the use of mercury experimental.

The red sandstone powder samples were analyzed with an X-ray diffractometer system PW3040/60 X'Pert Pro MPD in the range of 10° to 70° using Cu K-alpha radiation, various slit and filter geometries. The raw data were interpreted with X'Pert DIAMOND software, which can identify and quantify the mineralogy based on whole pattern fitting and Rietveld refinement methods. Two different scan settings were used, both with a step of 0.02 and a scanning velocity of 4° min^{-1}. Figure 2(a) shows the microscopic morphology of the rock sample at room temperature and fig. 2(b) shows the corresponding XRD analysis result. It is clear that this rock material mainly consists of the five mineral components, *i. e.* quartz, feldspar, calcite, clay and cuttings. The Keyence Model VK-X200 3-D laser scanning confocal microscope provides non-contact, micro/ nanometer-level profile, roughness and film thickness data on any material. This facility is mainly used for observing the surface morphology, roughness and crack growth dynamic change under different temperature cycles of the rock specimens.

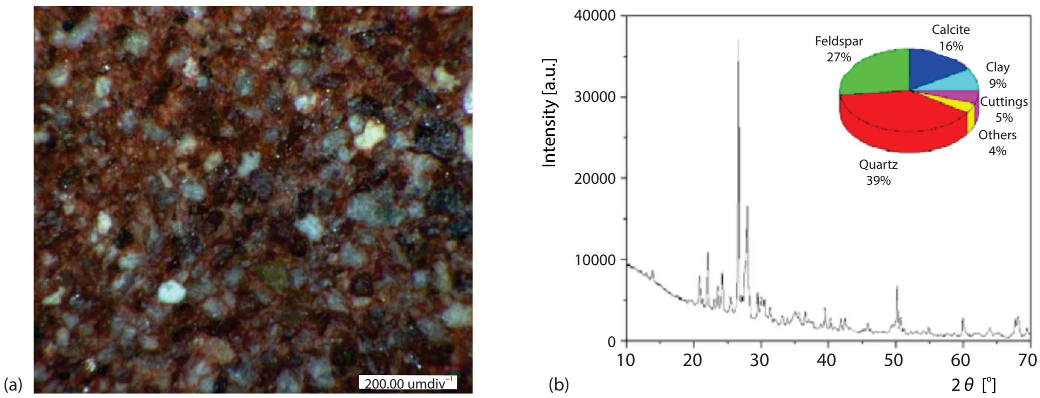


Figure 2. (a) the microscopic morphology, (b) the XRD pattern and mineral component of red sandstone at room temperature

Results and Discussion

Young's modulus and mass-loss with temperature changing

Figure 3(a) shows the temperature dependence of Young's modulus up to 800 °C in the air for the two specimens from the same block. At the initial experiment, Young's modulus of the sandstone was 13.5 ±0.8 GPa at room temperature. With increasing temperature, Young's value increases first very slightly up to 200 °C, and then in the range of 200-300 °C, there are similar hump fluctuations of Young's modulus values, forming the two narrow peaks and ending up in a maximum value of 15.6 ±0.9 GPa. Upon further heating, the maximum was: ed by a slowly level off at 400 °C with values of approximately 13.4 ±0.6 GPa which close to the original Young's modulus value. When the temperature goes up from 700 °C to 800 °C, the values remain unchanged and relatively balanced, fig. 3(b). In the cooling section, the high temperature of approximately 800 °C to 585 °C decreases linearly. Reduced by leaps from 585 °C to 550 °C by about 0.5 Gpa, then from 550 °C to room temperature, the Young's modulus value showed smooth and slow decreasing, and showing slowly level off to room temperature. During the cooling, the temperature dependence of the Young's modulus is decreasing smoothly according to S-shaped curved with varying curvature, until finally back to the room temperature minimum values of 1 ±0.2 GPa. Also, it is worth mentioning that the Young's modulus values are reduced steeply at about 575 °C.

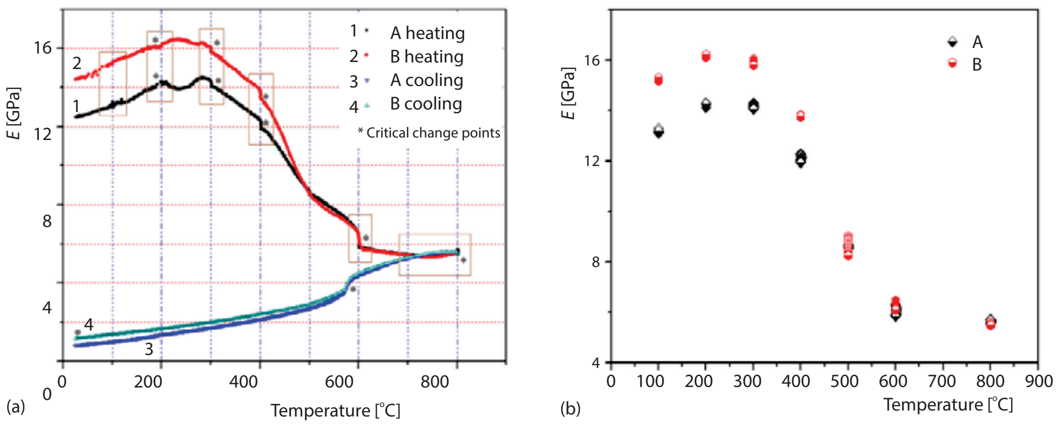


Figure 3. (a) Variations of Young's modulus with temperature for the heating-cooling cycles, (b) Variations of Young's modulus at different gradient temperature heated on for 5 minutes

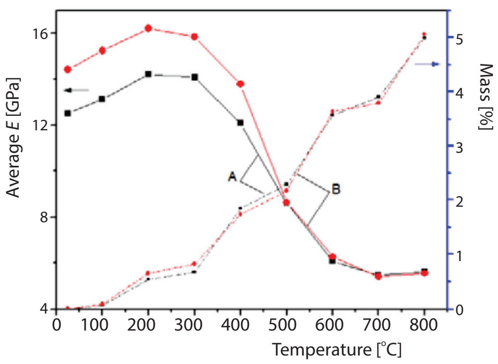


Figure 4. The average values of Young's modulus at different temperature and mass loss of red sandstone after the heating-cooling cycles (A and B is specimen A and B)

The mass loss of sandstone after the different heating-cooling cycles are shown in the same fig. 4, which indicated that the mass loss exhibited the volatility change and decreased by 4.58% from 300 °C to 800 °C after the different heating-cooling cycles. So it assumed that the entire volume of rock blocks remain balance after the heated process, the Young's modulus can be only regarded as proportional to the mass loss about 4.99% and 5.06%. After cooling from 800 °C to the room temperature, the Young's modulus reached a lower stable value, which is one reason by mass loss of rock. On the whole heating-cooling process, the Young's modulus values are decreasing by 56% and 62% during the heating process.

Phase composition analysis

From all the XRD patterns, the peaks of the main phase components grew much and much stronger, which almost increased fivefold. In the low temperature phrase, the composition of rock samples remained unchanged, however, only a slight increase in the peak intensity, fig. 5(a). From the fig. 5(b), some changes have taken place in some composition of rock samples. For the peaks of feldspar about albite and potassium aluminum silicon oxide, the peaks of feldspar grew stronger and stronger, and also the peaks of quartz and calcite also grew stronger and stronger, meanwhile the peaks of quartz gradually shifted to the left in the inserted image, fig. 5(b). When the high temperature heating-cooling cycles, fig. 5(c) showed that the diffraction peaks of quartz and feldspar show more and more clearance and which intensity is much stronger. The peaks of calcite and illite/cuttings become weaker and weaker, which occurred or lost because of decomposition. Obviously, the peaks of quartz also continue to shift to the left and the internal structure changes from the α-quartz to *β*-phase, see the inserted image in fig. 5(c).

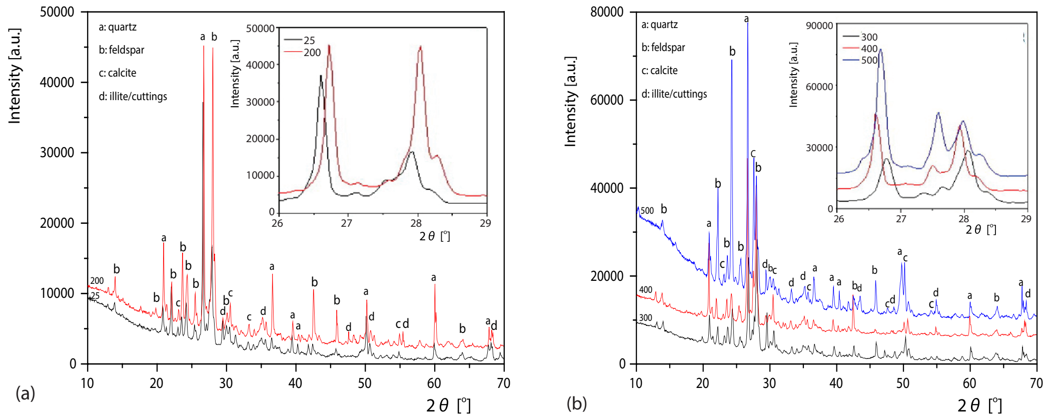
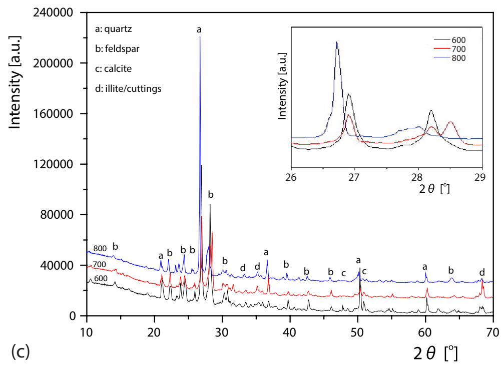


Figure 5. The XRD pattern of red sandstone after the heating-cooling cycles; (a) the heating-cooling cycles at 100 °C, 200 °C, (b) the heating-cooling cycles at 300 °C, 400 °C, and 500 °C (c) the heating-cooling cycles at 600 °C, 700 °C, and 800 °C



The XRD results showed that when the cycle temperature blow 600 °C, the obtained peaks of *α*-quartz has not changed, however, when that higher above 600°, the significant changes from *α*-phase into *β*-phase quartz. That is corresponding to that in theory, *α*-quartz will transform into hexagonal *β*-quartz at 573 °C at normal pressure trigonal. The results showed that thermal decomposition of calcite starting at 700 °C follows the equation, $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$. Therefore, the factor also plays important roles in sandstone mass losses. The SiO_2, Al_2O_3 and other ingredients in different combinations constitute quartz, feldspar, calcite and clay/cuttings minerals*, etc.* These chemical constituents in different forms exist in different particles, physical and mechanical properties of red sandstone have different effects, mainly exchange cations Ca^{2+}, $Fe^{2+/3+}$ is adsorbed on the surface of the particles, the $CaCO_3$ in the form of the cement particles cemented together or granules wrapped up, and prevent red sandstone from swelling phenomenon, but the porosity of the rock will be increased, structure lose and strength decreased with the cycle temperature elevated.

Morphological evolutions

From fig. 6, rock specimens at room temperature 25 °C, rock grains are cemented closely and almost no micro-cracks could be observed by the 3-D laser microscopy, fig. 6(a). When the sandstone brick specimens were heated to 200 °C, the effective porosity showed a slightly increasing trend by mercury test. This stage does not produce any obvious new micro-cracks, therefore, the effective porosity had a little change, but greater volatility in the rock roughness occurred in the range of 100-200 °C, fig. 6(b). During the heating process, the weakly bound water dehydration, some swelling clay mineral disintegration occur and fill the existing gaps, thereby reducing the effective porosity based on the XRD results.

When the temperature rose to 300 °C, this stage the strong binding water continued to dehydrate and clay minerals disintegrated more thoroughly but limited to the original throat blockage, meanwhile effective porosity rebounded and continued to increase, fig. 6(c). When the heating temperature rose to 400 °C, the number of micro-cracks in the rock samples increased

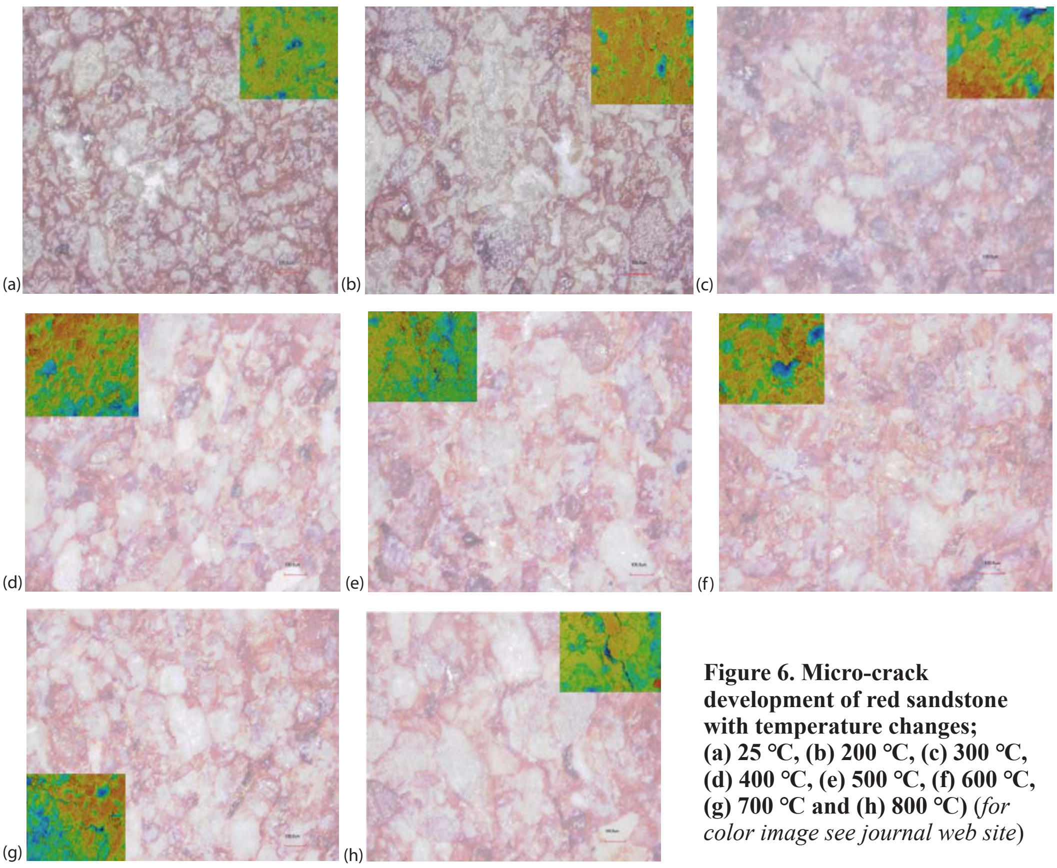


Figure 6. Micro-crack development of red sandstone with temperature changes; (a) 25 °C, (b) 200 °C, (c) 300 °C, (d) 400 °C, (e) 500 °C, (f) 600 °C, (g) 700 °C and (h) 800 °C) (*for color image see journal web site*)

and also effective porosity showed an increasing trend. Micro-cracks are mainly concentrated in the edge of the grain particles and the crack width is about ~0.01 μm, fig. 6(d). When the heating temperature rose to 600 °C, effective porosity presented an increasing trend and the number of micro-cracks continued to increase. Rock bridge micro-cracks penetrated and a sharp increase in the number. Also the width of the crack was increased to 1 μm, fig. 6(f), the large numbers of cracks in the rock surface appeared a larger increase of about 15% (based on the statistical 3-D microscopy per units square centimeter). When the heating temperature rose to 700 °C, a red powder appeared on the surface of the sandstone specimens, some of which conglomerated, fig. 6(g). When the sandstone samples were heated to 800 °C, most particles had become fractured with only a few unbroken crystal ribbons left. Obviously, cracks ran through the rock sample and it's worth mentioning that gently squeeze the rock sample changed into a red powder, fig. 6(h). Meanwhile, the surface roughness and porosity of sandstone increased dramatically, fig. (7).

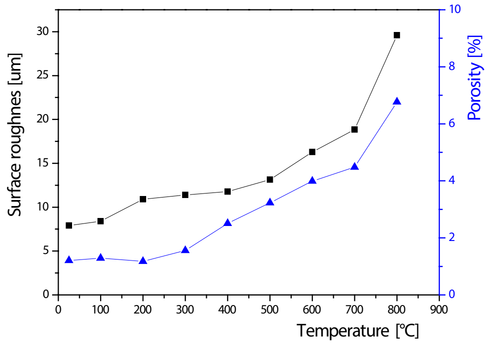


Figure 7. surface roughness of red sandstone with temperature changes

The fact that after heating to 800 °C and subsequent cooling from high temperature back to room temperature, the Young's modulus is essentially continued to change and reduce, confirms that there is a large amount of damage accumulation. Therefore, this finding also indicated that there is an important relationship between the Young's modulus and heat accumulation damage. Thermal treatment could cause the changes in the microstructure of rocks, including porosity, micro-cracks, trans-granular and disruption, which also have a very important influence on Young's modulus of rocks.

Conclusion

In this work, the temperature dependence of Young's modulus of the red sandstone has been studied using the impulse excitation technique and interpreted on the basis of quantitative composition data obtained by X-ray diffraction, the Archimedes method, the microscopy-based image analysis and mercury intrusion experimental. The temperature dependence of Young's modulus of red sandstone with porosities around 4.7% has been investigated by impulse excitation up to 800 °C. The results showed that at room temperature the Young's modulus of the samples are very similar (9-12 GPa). The temperature variations induce micro-cracks in red sandstone because of its mineralogical components heterogeneity or phase transition of some components. In this study, it has been shown that red sandstone has the unusual property that the degree of damage accumulation is more critical when the materials are heated to higher temperatures. Other components have caused the breakdown or decomposition of mineral grains. Various rock engineering was relevant to high-temperature condition and thus thermal cracking undoubtedly had a certain influence on the progressive failure of rocks.

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References

[1] Class, H., *et al.*, A Benchmark Study on Problems Related to CO_2 Storage in Geologic Formations, *Computers* and *Geosciences*, *13* (2009), Dec., pp. 409-434
[2] Sundberg, J., *et al.*, Modelling of Thermal Rock Mass Properties at the Potential Sites of a Swedish Nuclear Waste Repository, *International Journal of Rock Mechanics and Mining Sciences*, *46* (2009), 6, pp. 1042-1054
[3] Roddy, D. J., *et al.,* Underground Coal Gasification with CCS: A Pathway to Decarbonising Industry, *Energy and Environmental Science*, *3* (2010), 4, pp. 400-407
[4] Gischig, V. S., Natural Hazards Cracking Cliffs Feel the Heat, *Nature Geosciences*, *9* (2016), 5, pp. 344-345
[5] Collins, B. D., *et al.,* Rockfall Triggering by Cyclic Thermal Stressing of Exfoliation Fractures, *Nature Geosciences*, *9* (2016), Mar., pp. 395-400
[6] Fredrich, J. T., *et al.,* MicroMechanics of Thermally Induced Cracking in Three Crustal Rocks, *Journal of Geophysical Research*, *91* (1986), B12, pp. 12743-12764
[7] Homand-Etienne, F., *et al.,* Thermally Induced Microcracking in Granites: Characterization and Analysis, *International Journal of Rock Mechanics&mining Sciences and GeoMechanics Abstracts*, *26* (1989), Feb., pp. 125-134
[8] Ferrero, A. M., *et al.,* Experimental Studies on the Mechanical Behaviour of Two Thermal Cracked Marbles, *Rock Mechanics and Rock Engineering*, *34* (2001), 1, pp. 57-66
[9] Zhang, Y., *et al.*, Process of Sandstone Thermal Cracking, *Chinese Journal of Geophysics*, *48* (2005), Mar., pp. 722-726
[10] Zhao, H. B., *et al.*, Experimental Study on Effect of Temperature on Sandstone Damage, *Chinese Journal of rock Mechanics and engineering*, *28* (2009), June, pp. 2784-2788
[11] Zuo, J. P., *et al.*, The SEM in Situ Investigation on Thermal Cracking Behaviour of Pingdingshan Sandstone at Elevated Temperatures, *Geophysical Journal International*, *181* (2010), 2, pp. 593-603
[12] Ranjith, P. G., *et al.*, Transformation Plasticity and the Effect of Temperature on the Mechanical Behaviour of Hawkesbury Sandstone at Atmospheric Pressure, *Engineering Geology*, *151* (2012), Nov., pp. 120-127
[13] Tian, H., *et al.*, Physical Properties of Sandstones after High Temperature Treatment, *Rock Mechanics and Rock Engineering*, *45* (2012), 6, pp. 1113-1117
[14] Sengun, N., Influence of Thermal Damage on the Physical and Mechanical Properties of Carbonate Rocks, *Arabian Journal of Geosciences*, 7 (2014), 12, pp. 5543-5551
[15] Rong, H. R., Expermimental Research on Mechanical Properties and Microstructure Change Law of Red Sandstone after Different Temperatures, *Rock and Soil Mechanics, 36* (2015), pp. 463-469

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