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Combined effects of cyclic load and temperature fluctuation on the mechanical behavior of porous sandstones

Fei Wang[a], Ping Cao[a], Yixian Wang[c], Ruiqing Hao[d], Jingjing Meng[e], Junlong Shang[b*]

[a] School of Resources and Safety Engineering, Central South University, Changsha, China

[b] Nanyang Centre for Underground Space, School of Civil and Environmental Engineering, Nanyang Technological University, Singapore

[c] School of Civil engineering, Hefei university of technology, China

[d] College of Mining Engineering, Taiyuan University of Technology, China

[e] Department of Civil, Environmental and Natural Resources Engineering, Lulea University of Technology, Luleå, Sweden

Corresponding author: jlshang@ntu.edu.sg, shangjunlongcsu@gmail.com (J. Shang)

Tel: +65 86499772

Abstract: Rocks in cold regions tend to experience exacerbated degradation under the combined effects of environmental and anthropogenic factors, which may arise from, for example, temperature fluctuation, mechanical excavation, and blasting. Activities related to rock support or open-pit slope optimization in cold regions require a complete understanding of the failure mechanisms of rock under the complex conditions. This paper quantitatively documents the impact of combined cyclic mechanical load and freeze-thaw cycles (i.e., the effect of stress "history") on the microstructural evolution and mechanical degradation of three porous sandstones with distinct porosity values (from 3.9 to 14.1%). The three sandstone samples were collected from different geological regions in China. The microstructural evolution of the tested samples was quantitatively analyzed using the low-field Nuclear Magnetic Resonance (NMR) technique. To investigate sample degradation arising from the impact of the stress "history", the cyclic-loaded and freeze-thaw cycled samples were eventually compressed to failure, during which an acoustic emission system was used to monitor microseismic activities. The results of the study show that the porosity of all tested sandstone samples was increased after cyclic load, with a much more rapid and further increase in porosity observed for samples being subsequently treated under the freeze-thaw cycles. More interestingly, the Chuxiong sandstone with relatively small porosity values were much more sensitive to the impact of cyclic load compared with the Linyi sandstone, exhibiting a somewhat larger increase rate in porosity. However, the Linyi sandstone with larger initial porosity values exhibited a relatively large increase rate in porosity under the multiple freeze-thaw treatments. The multiple freeze-thaw treatments mainly resulted in the development of relatively large pores. The results of the uniaxial compression tests show that the strength reduction of the samples being solely treated by freeze-thaw cycles was within the range of 5 - 10%, whereas it was within the range of 20 - 40% for those samples subjected to the combined cyclic load and freeze-thaw cycles.

Keywords: Porous sandstone; cyclic load; freeze-thaw cycles; porosity evolution; mechanical degradation

1 Introduction

Anthropogenic activities (e.g., blasting or mechanical excavation in open-pit mines) can cause the cyclic fatigue of pit slopes (Lin et al., 2019; Zhao et al., 2017; Lin et al., 2012). For open pits located in cold regions, the mechanically degraded pit slopes can be exacerbated when temperature fluctuation is pronounced (Bayram, 2012). The negative signature left within rock from the stress “history” will threaten open-pit mine vehicles, excavation facilities, and even human safety (Zhou et al., 2015; Yavuz, 2011; Wang and Wan, 2019). Besides which, the pore parameters (e.g., porosity or permeability) of rocks are suspected to exhibit different mechanical and elastic responses due to the effect of the stress “history”, thereby potentially affecting oil and gas recovery and anti-seepage performance. Therefore, it is important to understand the microstructural changes and degradation mechanisms of rock subjected to cyclic load and temperature fluctuation.

Many experimental and analytical studies have been carried out to investigate the impact of freeze-thaw cycles on the mechanical properties of rocks (e.g., Nicholson et al., 2001; Bayram et al., 2014; Han et al., 2016; Zhou et al., 2018). It has been demonstrated that the elastic modulus, uniaxial compressive strength, and cohesion exhibited an exponential reduction tend for many rocks being treated under freeze-thaw cycles (Wang et al., 2016). Earlier studies have also shown that the microstructural parameters of rock are affected by the freeze-thaw treatment and mechanical load

(Zhou et al., 2018; Bayram et al., 2014). The changes in rock microstructures tend to affect porosity and permeability (Head and Vanorio, 2016), as well as rock mechanical properties (Yavuz, 2011).

Analytically, some researchers have explored the effect of freeze-thaw cycles on the mechanical properties of rock using damage models. Yavuz et al. (2006) proposed an equation for estimating the index properties of deteriorated carbonate rocks due to freeze-thaw cycles. A decrease in the index properties (i.e., P-wave velocity, Schmidt hardness) was noted for the freeze-thaw deteriorated rock. A damage constitutive model of rock under freeze-thaw and mechanical loading was established by Huang et al. (2018), and the model has been used to analyze the stability of a tunnel under the coupled thermo-hydro-mechanical condition in cold regions. Based on the single discontinuity surface theory (Jaeger,1960), a novel statistical model was proposed by Fu et al. (2018) to estimate the triaxial compressive strength of transversely isotropic rocks being freeze-thaw treated.

Some techniques have been used to investigate the changes in microstructures within rocks subjected to external loading. These techniques mainly include computed tomography (CT) scanning (Nasseri et al., 2011; Jia et al., 2013), scanning electron microscope (SEM) analysis (Zuo et al., 2015), and digital imaging treatment (Al-Shumaimri, 2012). As a new approach for characterizing the microstructures in rocks, the nuclear magnetic resonance (NMR) technique has been increasingly used in the laboratory to evaluate the porosity and pore-size distribution of rock (e.g., Baud et al., 2014; Shang et al., 2015; Li et al., 2016). Thanks to the NMR technique, the microstructural damage of rock caused by various loading conditions, including dynamic loading (Zhou et al., 2015), unloading (Zhou et al., 2018) and uniaxial compression (Shang et al., 2015), were investigated.

The above review shows that previous studies essentially focused on the pure effect of freeze-thaw cycles on the mechanical behaviour of rocks. The combined effect of cyclic load and freeze-thaw cycles on the microstructural and mechanical degradation of rocks is still not fully understood but is very important for rock engineering activities, especially in cold regions. We therefore report the results from a comprehensive experimental investigation that directly explored the microstructural evolution and mechanical degradation of sandstones under the combined cyclic load and freeze-thaw cycles. Three different porous sandstones with distinct porosity values were used in the study, which allows the effect of porosity on the microstructural and mechanical degradation of rock to be understood. The samples were first cyclically compressed using a uniaxial testing machine, and then degraded under multiple freeze-thaw treatment. The NMR technique was used to quantify the microstructural parameters of the tested samples. Finally, those degraded samples after the combined loading (i.e., cyclic load and freeze-thaw cycles) were uniaxially compressed to failure, during which an acoustic emission (AE) system was used to monitor microseismic activities for examining damage mechanisms.

2 Materials and methods

2.1 Sample preparation

To consider the influence of porosity and pore structure on the failure mechanisms of rock, three sandstone blocks with distinct porosity values were collected in three regions in China (Fig. 1a). They are Chuxiong sandstone (porosity ≈ 3.9%), Guixi sandstone (5.5%), and Linyi sandstone (14.1%). The Guixi and Linyi sandstones were collected from two cold regions, with the Chuxiong sandstone collected from a representative region in China where low porosity sandstones are available. The use of the three sandstones with distinct porosity values allows the role of microstructures (i.e., porosity and pore structure) to be highlighted and investigated in this study. The three sandstones were formed in different geological ages, which are Cretaceous, Devonian, and Cambrian, respectively. The Chuxiong sandstone (CS) was collected from the Liuju copper mine in Yunnan Province, China (Fig. 1b). The Guixi sandstone (GS) was collected from the Yinluling mine in Jiangxi Province, China (Fig. 1c). It contains ~75.4% fine quartz particles and is red in color. The Linyi sandstone (LS) was collected from the Linyi area in Shandong Province, China (Fig. 1d). It is yellow with a granular and tight micro-structure. Table 1 shows the mineralogy of the three tested sandstones. In the study, cylindrical samples having a size of ~ 50 × 100 mm (diameter × height) were prepared from the collected sandstone blocks, following the ISRM standard (ISRM 2007).

2.2 Experimental scheme

All tests followed an experimental scheme consisting of the following five steps (Fig. 2):

(1) Conducting UCS test on the three sandstones. First, conventional uniaxial compression strength (UCS) tests were conducted on the three sandstones in air-dry condition to get their mechanical properties. Prior to the UCS test, two sets of strain gauges were attached to the surface of each tested sample to measure both strains in the longitudinal and transverse directions. The prepared air-dried samples were uniaxially loaded to failure with a small loading rate of 50 N/s by using a servo-controlled testing machine (Fig. 3a). In the meanwhile, an acoustic emission system was used to monitor microseismic activities. The mechanical properties of the three sandstones are shown in Table 2, where the Chuxiong sandstone had the minimum mean UCS of 48.8 MPa, and the Linyi sandstone had the maximum mean UCS of 75.9 MPa. Hence, a load of up to 10 kN (equivalence of 5.1 MPa given the sample size) was used in the later cyclic loading process, expecting that all tested samples can survive in the cyclic loading stage, and then can be used in the freeze-thaw treatment and the final uniaxial loading tests.

(2) Sample saturation and NMR test. For comparison purposes, the initial porosity values of the prepared sandstone samples were measured using the NMR technique. The samples were first saturated under a pressure of 100 kPa by using a vacuum saturation device for around 12 h. Distilled water was used as a liquid in vacuum saturation to eliminate the effect of physical and chemical reactions on test results. The signal decay of the hydrogen atoms in the fully saturated rock samples was then monitored using a low-field AniMR-150 NMR testing machine (Fig. 3c). The T_2 distribution curves, reflecting the magnitude of rock porosity, of all tested samples were obtained (see Section 2.3 for the principle of the NMR technique). The NMR measured porosity values of the three sandstones are shown in Table 3 (the third row). Following on from the description of the mechanical and petrophysical properties of the sandstones, the cyclic mechanical load and freeze-thaw cycles were applied on the samples, which are respectively introduced in (3) and (4).

(3) Samples were subjected to cyclic mechanical load (CML). A cyclic mechanical load of up to 10 kN was applied on the prepared samples (in air-dry condition) with a small loading rate of 20 N/s using the same servo-controlled testing machine as that used in (1). As described earlier, the use of the peak load (i.e., 10 kN) enables the cyclic-loaded samples to be re-used in the subsequent freeze-thaw treatment, to meet the purpose of this research. The relationship between cyclic load and time is shown in Fig. 4. After each CML test, the porosity of each tested sample was measured using the NMR technique; and the corresponding results are shown in Table 3 (the fourth row).

(4) The cyclic-loaded samples were subsequently subjected to freeze-thaw cycles (CML+FTC). All cyclic- loaded samples were re-saturated using the vacuum saturation device in the same manner as that described in (2), and then placed in the TDS300 freeze−thaw machine manufactured by

Donghua Testing Equipment Company, Ltd., China (see Fig. 3b). In this work, thirty freeze-thaw cycles were conducted on each sample and each cycle lasted around 10 h (Fang et al., 2018; Li et al., 2016; Zhou et al., 2015). Fig. 5 shows a schematic diagram of one freeze-thaw cycle, during which the samples were frozen at -20 °C for 5 h, and then thaw at 20 °C for an additional 5 h, simulating the temperature fluctuation in nature. Similarly, the porosity of each sample after the combined CML and FTC treatment was re-measured using the NMR technique, and the corresponding results are shown in Table 3 (the fifth row).

(5) Conducting UCS test on the samples being treated under CML and FTC. The samples after the combined cyclic load and multiple freeze-thaw treatments were first dried in room condition; the air-dried samples were then compressed to failure with a loading rate of 50 N/s. An acoustic emission instrument (model: PCI-8) produced by the Physical Acoustics Corporation (USA) was used to monitor the failure characteristics of the degraded samples (Fig. 3d). Four Nano30 sensors were attached on the surface of each sample using Vaseline as a coupling agent. To eliminate electronic or environmental noise, a threshold value of 45 dB was set in each test. The AE signals logged by the sensors were amplified by a gain of 40 dB with preamplifiers. Only those successfully logged data with high completeness were used in the latter data processing.

2.3 Nuclear Magnetic Resonance

The Nuclear Magnetic Resonance (NMR) technique can detect the fluids within saturated rocks. This technique has been widely used to evaluate many physical parameters of rocks, such as pore size distribution, permeability, free-fluid index, porosity, etc (Freedman and Heaton, 2004). The NMR technique is attractive to researchers for its non-destructive nature of measurement, it is therefore used in the study for porosity measurement. Fig. 6 shows the principle of NMR, which relies on the interaction between the magnetic properties of H fluid nucleus (within water in the study) and the applied magnetic field. The resonance phenomenon is expected to happen during the interaction, thereby the relaxation characteristics of the H-containing fluid can be obtained (Chang et al., 1997; Kanters et al., 1998). As shown in Fig. 6a, the direction of nuclear spin is often disorganized in the absence of an external magnetic field, and the expected value of the macro magnetic moment of the spin system is 0. The orientation of individual nuclear spin will be affected after an external magnetic field is applied, and the spin system is expected to reach an equilibrium state, with a stable magnetization value (see Fig. 6a). Through emitting the RF pulses to fluid-saturated samples placed in the magnetic field, the H protons within the fluids will resonate and absorb the RF pulse energy. The RF energy absorbed by the H proton will be released after removing of the RF pulse, and the energy release process can be detected by a dedicated coil, which can be converted to the nuclear magnetic resonance signal (Fig. 6b).

It can be anticipated that fluid-filled pores with various sizes will yield different magnitudes of energy. The magnetization decay signal is featured by the relaxation time, which is associated with the type and properties of pore-filling fluids, as well as their interactions with pores, pore size distribution, and surface relaxivity. Generally, the smaller the relaxation time T_2 is, the smaller the pore size is. The T_2 can be expressed as (Anovitz and Cole 2015):

$$\frac{1}{T_2} \approx \frac{1}{T_{2F}} = \rho\left(\frac{S}{V}\right) \tag{1}$$

where T_{2F} is the transverse surface relaxation time (ms); ρ is the surface relaxivity, which is a factor for the intensity of the transverse surface relaxation (μm/ms); and S/V is the surface-to-volume ratio of a pore.

3 Experimental results

3.1 Microstructural evolution of sandstone

3.1.1 T_2 spectrum curve as an indicator of pore size distributions

Fig. 7 shows the T_2 spectrum curves of the tested sandstone samples subjected to the combined cyclic mechanical load (CML) and freeze-thaw cycles (FTC). For comparison purposes, the corresponding T_2 curves of the samples in the original state (OS), as well as after the cyclic mechanical load, are also shown in the figure. The porosity accumulation curves (blue line) of the samples under the three conditions (i.e., OS, CML, and CML + FTC) are also presented. Generally, the pore size increased with the increase in relaxation time. For the samples in the original state, the influence of lithology (sandstones with different geological formations) on the shape characteristics of the T_2 spectrum curves was pronounced.

The evolution of pore size distribution can be reflected by the changes in the shape of the T_2 spectrum curves. As shown in Fig. 7a, the T_2 spectrum curves of the LS-1 sample under the three different conditions all exhibited clear peak values, at around 100 ms. The presence of the peak values demonstrated that there existed large pores within the samples (Shang et al., 2015). Similar peak values were also observed for other samples (Figs. 7b-7c) but appeared at different times. As a general trend, the T_2 spectrum curves of all tested samples under CML moved upwards dramatically compared with that of the samples under OS, implying a clear increase in porosity of the tested samples after the cyclic mechanical load. A further increase in porosity was noticed when the combined CML and FTC were applied, which was probably due to the additional negative effect of expansion and contraction of pores as a result of the multiple freeze-thaw treatments.

The peak points of the T_2 spectrum curves of the GS-1 and CS-1 samples (Figs. 7b and 7c) moved to the top right after CML, and then moved to the top left after the combined CML and FTC. This observation indicated that the cyclic load probably led to the expansion of relatively small pores inside the samples, while the subsequently applied freeze-thaw force contributed to the generation of new pores. However, it is observed that the evolution of the peak points of the T_2 spectrum curves of the LS-1 sample (Fig. 7a) exhibited a reverse trend. It is suspected that the cyclic load caused a certain closure of large pores inside the sample, and the freeze-thaw force led to a continuous expansion of large pores.

3.1.2 Porosity evolution under combined CML and FTC

Table 3 shows the porosity of the tested samples under different conditions; the data are also plotted in Fig. 8 with the percentage increments highlighted. The initial porosity of the LS-1 sample was 14.1% (Table 3), while it was only 3.9% for the CS-1 sample. The porosity values of all tested samples increased clearly after CML (Fig. 8, red bars), compared with that of the samples in their original state (green bars). A further increase in porosity was noticed after the subsequent multiple freeze-thaw treatments (blue bars). The increase rate in porosity for the three sandstones in response to cyclic mechanical load was different. It is 33.3% for the CS-1 sample with relatively smaller initial porosity, while much smaller increase rate in the porosity was noticed for the more porous GS-1 and LS-1 samples (which are 20.8% and 2.6%, respectively, see Fig. 8). The observed difference in the mechanical load response in terms of porosity increment can be due to the pores' buffering effect (Liu et al., 2012), which states that the application of cyclic mechanical load can result in the compaction of relatively large pores inside rock.

After the subsequent freeze-thaw treatment, the porosity values of the GS-1 and LS-1 samples were increased further by 18.7% and 15.3%, respectively, while the corresponding porosity increment of the CS-1 sample was only 9.9% (Fig. 8). This observation demonstrated that the freeze-thaw treatment can have much more significant impact on porous sandstone in contrast to tight ones, because the former is expected to experience more significant expansion and contraction of water in internal rock pores after being fully saturated, leading to marked degradation of rock (Baud et al., 2014). As such, different lithology types are expected to have different tolerance to the same frost heave (Huang et al., 2018).

3.1.3 Alteration of pore parameters: a micro-scale insight

In this section, we report the alternation of the pore parameters (reflected by the amount of free fluid and bound fluid) and permeability of the tested sandstones under combined CML and FTC. It is known that the fluid inside the pores in a rock can be divided into free fluid and bound fluid (Hurlimann et al., 2002). The T_2 cutoff value of 10 ms is often used for sandstone to distinguish free fluid and bound fluid (Fan et al., 2018), and this scheme was followed in this study. As shown in Fig. 9, the T_2 cutoff value (10) divides the T_2 spectrum distribution into two parts, with the left area representing the bound fluid porosity (BVI) and the right part indicating the free fluid porosity (FFI). The permeability of the tested rock can be readily available in the NMR post processing stage, which is calculated based on the results of NMR experiments and the Coates permeability model (Rezaee et al., 2012):

$$K = \left(\frac{PHI}{10}\right)^{2}\left(\frac{FFI}{BVI}\right)^{-2} \tag{2}$$

where K is the permeability; PHI is the porosity.

The calculated pore parameters of the tested samples are listed in Table 4, in which the bound fluid saturation of the LS-1 and GS-1 samples were 38.52% and 91.91%, respectively. The results also implied that there existed many relatively large pores in the Linyi sandstone, whereas smaller voids are more prevalent in the Guixi sandstone. Fig. 10a shows that the amount of bound fluid within all tested samples, which was decreased under the combined cyclic mechanical load and freeze-thaw cycles.

Fig. 10b shows that the increase rates in permeability of all tested samples were larger than 1

(100%), with the maximum value of 18.49 for the CS-1 sample. This observation indicated that the permeability of all tested samples increased significantly after the combined CML and FTC; and the most pronounced increment in permeability was occurred in the Chuxiong sandstone with the lowest initial porosity. It is also noted that the ratio value was only 2.99 when FTC was solely applied on the CS-2 sample (Fig. 10b, blue bar).

3.1.4 NMR imaging

A 10-mm-thick sheet along the longitudinal direction of the core samples was used in the NMR imaging analysis. The position for capturing NMR images was fixed for the same sample under the different loading schemes. Fig. 11 shows the NMR images of the tested rock samples under different loading conditions. The brightness of the NMR images of the samples at the original state (OS) was closely related with their initial porosity values. In the original state, the NMR image of the LS-1 sample was the brightest compared with that of other samples, indicating that the initial porosity of the LS-1 sample was the largest (this has been tested in Table 3). Clear layers were also observed within the LS-1 sample (Fig. 11).

The NMR images of all samples became much clearer after CML and FTC. This was due to the fact that more larger pores were generated within the samples being loaded. It is worthy to note that the increase in bright spots after FTC was not obvious for some samples (e.g., CS-2), because of the nature of their porosity values which are very small.

3.2 Rock fatigue degradation due to cyclic mechanical load

To understand the fatigue degradation of the tested sandstone samples after the cyclic mechanical load, the three cyclic-loaded samples were re-tested under one more cycle of uniaxial loading and unloading. The corresponding stress-strain curves (red in Fig. 12) were logged and compared with the stress-strain hysteresis loops obtained during the cyclic load process. Under the cyclic load, the maximum strains of the LS-1, GS-1 and CS-1 samples were around 0.012, 0.009, and 0.008, respectively. The strain for the three different sandstone samples was a function of their initial porosity, i.e., the samples with relatively large porosity values exhibited somewhat larger strain under the cyclic mechanical load. Fig. 12 also shows that the stress-strain curve of the samples after the cyclic mechanical load exhibited a hysteresis loop. The sizes of the hysteresis loops were clearly different for different sandstones. The size of the hysteresis loop was proportional to the porosity increase rate.

For example, the CS-1 sample with relatively large porosity increase rate yielded a hysteresis loop with somewhat larger size. It can also be seen that the compaction stage still existed for all samples under the secondary load (red curves), although the samples have been cyclically compacted.

Another important observation was that the slopes of the elastic portions of the cyclic-loaded curves (black) and their corresponding secondary loaded curve (red) remained essentially the same. The stress-strain curve of the LS-1 sample (red curve in Fig. 12a) under the secondary load cycle shifted to the left, whereas the corresponding curves of the CS-1 and GS-1 samples (Figs. 12b and 12c) did not show a clear movement. This was probably due to the difference in the porosity values of the three tested samples. The LS-1 sandstone sample exhibited a much larger initial porosity (Table 3), with relatively large pores dominated in the rock (Fig. 7). Therefore, under the cyclic mechanical load, the compaction phase for the LS-1 sample was suspected to be reduced more significantly compared with that of the CS-1 and GS-1 samples. We acknowledge that although the cyclic mechanical load was expected to be applied within the elastic deformation stage, the fatigue degradation of the rock samples still occurred, as demonstrated by the NMR imaging analysis (Fig. 11).

3.3 Failure characteristics of samples being treated under CML and FTC

The degraded sandstone under the combined CML and FTC were uniaxially compressed to failure. The mechanical test results are shown in Table 5 and Fig. 13. As shown in Fig. 13, the UCS values of the CS-1, GS-1 and LS-1 samples after the CML and FTC loading were decreased by 38.9%, 32.1% and 27.1%, respectively. The UCS value of the CS-2 sample was only decreased by 7.3% when the sample was solely subjected to FTC (Fig. 13, blue bar). As shown in Table 5, the evolutionary trend in the elastic modulus was similar to that of the UCS of the tested sandstone samples, and the Poisson's ratio was increased after the freeze-thaw cycles but decreased after the combined cyclic load and freeze-thaw cycles.

The stress-strain curves of the tested samples in the study can be broadly divided into four stages (Fig. 14): micro-crack compaction stage, elastic stage, plastic failure stage, and post-destruction stage. The stress-strain curve at the compaction stage was non-linear and the AE count was relatively small. In the elastic stage, the samples were elastically deformed, with no clear macrocracks observed. Despite this, the formation of microcracks or the changes in micropores within the rock still occurred

(Fig. 11). During the plastic failure stage, the peak strength appeared, and the AE count gradually increased and finally reached to the maximum. During the post-destruction stage, the stress suddenly dropped, showing a brittle failure nature of the sandstones. In the meanwhile, the acoustic emission count decreased continuously.

Besides above, the influence of porosity and combined loading was pronounced. It can be seen from Figs. 15a that the strain of the LS-1 sample was the largest in the compaction stage (~ 0.015), compared with that of the other three samples (all within 0.01). This was because the LS-1 sample had a much larger porosity (Table 3), for which the rock tends to be compacted under external loading. The AE activities clustered at the plastic failure stage for the samples LS-1 and GS-1 with relatively higher porosity values, whereas for the much tighter samples with low porosity (i.e., CS-1 and CS-2), scattered AE signals were monitored during the whole UCS test. It is suspected that the tight sandstone matrix was much easier to be ruptured locally (at the microscale) without the help of pore collapse, thereby exhibiting high frequency of AE activities. Also, it clearly can be seen that the UCS values of the stress degraded samples were much smaller compared with that of their respective ones in the original state (Table 6 and Fig. 14), which matches with the findings in Shang et al. (2015), although water weakening effect may be at play (Baud et al., 2000). It should be noted that we can only quantify the UCS reduction arising from the combined loading (CML + FTC), given the testing scheme used in this research (Section 2.2).

4 Discussion

4.1 Water weakening effect

In this study, the prepared sandstone samples were first saturated with distilled water and then treated under freeze−thaw cycles in the laboratory, thereby simulating in-situ temperature fluctuation (Section 2.2). It has been understood that the presence of water may affect the physical and chemical properties of rocks, causing a water weakening effect (Baud et al., 2000; Shang et al., 2018). Despite this, it is suspected that the water weakening effect can be very weak in this particular study or within an acceptable range, given that the samples used in this study contained very small fractions of soluble clay minerals (below 0.8%, Table 1). On the other hand, quartz dominated the mineral composition (75.4 – 92.3%) of all tested samples, the solubility of quartz in water can be neglected considering the temperature (-25 – 25 °C) and pressure (room condition) used in the study (Futera et al., 2017). A quantitative analysis of water weakening effect on UCS reduction would be required for rocks with larger fractions of clay minerals which is beyond the scope of this study.

4.2 Role of microstructures and minerology on the strength

The mechanical properties of rocks are closely related to their micro-structural and mineralogical properties (Nicholson, 2001; Li et al., 2016; Shang et al., 2016; Aliyu et al., 2019). As shown in Table 1, the three sandstones tested consisted mainly of quartz (75.4 - 92.3%), which is one of the common hard minerals in the Earth. The quartz content in the sandstone samples was positively correlated with their UCS values (Table 2), which is in good accordance with Price (1966). Under the cyclic mechanical load, the porosity of all tested samples increased. It was found that the rate of increase in porosity was negatively related to initial porosity values, i.e., samples with relatively large initial porosity values exhibited somewhat smaller rate of increase in porosity. This observation is consistent with previous studies (Shang et al., 2015; Adnan and Adrian, 2013; Geraud et al., 1998). The changes in porosity of the samples under the cyclic mechanical load was due to the combined effects of pore generation, micro-crack expansion, and pore collapse (Shang et al., 2015). The large pores within the LS-1 sample were expected to be compacted under low external stress, considering that the sample had a large porosity (14.1%, Table 3). This hypothesis was demonstrated by the slightly right movement of the red curve compared with the black curve (Fig. 7a). For the GS-1 and CS-1 samples with smaller porosity values (Figs. 7b and 7c), the porosity increment was mainly due to the generation and expansion of new micro-cracks, which were reflected by the right shifting of pore size distribution curves. Similar observations in the evolution of pores under stress were reported by Li et al. (2016) on an unidentified sandstone, and by Adnan and Adrian (2013) on Gosford sandstone. By comparing the secondary loading stress-strain curves and the cyclic loading curves in the compaction phase (Figs. 12b and 12c) for the samples GS-1 and CS-1, we can infer that there was a clear reversible compaction for the samples.

The porosity increase rate of the LS-1, GS-1 and CS-1 samples after the multiple freeze-thaw treatments were 15.3, 18.7 and 9.9%, respectively. The freeze-thaw damage made in the samples, in the aspect of porosity increment, was suspected to be caused by the repeated initiation and dissipation of pore ice pressure (Park et al., 2015; Freire-Lista et al., 2015; Matsuoka and Murton, 2008). When the pressure caused by volume expansion reaches the tensile strength of rock, the pores will be enlarged, resulting in the formation of new microfractures (Chen et al., 2004). It is argued by Park et al. (2015) that the changes in porosity of rock subjected to freeze-thaw treatments are the result of the combined impact of original porosity and strength. This perspective has been further testified by our data, for example the Linyi sandstone had the largest original porosity (~14.1%), the porosity increase rate of this sandstone however was not the largest among the three tested sandstones due to its relatively high UCS compared with that of Guixi and Chuxiong sandstones.

Table 6 shows the porosity increment and associated strength degradation of the tested sandstone samples. The strength degradation of all tested samples was positively correlated with their porosity increase rate. For example, the porosity of the CS-1 sample increased 46.4% and its strength decreased 38.9%. The porosity of CS-2 sample increased by 8.1% and its strength decreased 7.3%. Similar relationship between porosity increment and strength degradation was reported by Dunn (1973) on sandstone, by Yavuz (2011) on andesite stone, and by Tugrul (2004) on various rocks.

5 Conclusion

This paper documents the results of an extensive laboratory investigation of the combined effects of cyclic mechanical load and freeze-thaw cycles on the microstructural evolution and mechanical degradation of three sandstones with distinct porosity values. The NMR technique allowed the inspection of the microstructural properties of the tested sandstones, in terms of the evolution of pore size distribution, porosity, and permeability, which are responsible for the bulk properties of the sandstones. The mechanical strength reductions of the degraded sandstone samples were quantified and characterized based on the UCS tests, as well as the AE measurements. From the experimental results, the following conclusions can be drawn:

(1) The porosity of all tested sandstones was increased after the cyclic mechanical load. Significant increases in porosity were also observed after the subsequent freeze-thaw cycles.

(2) The sandstone samples with relatively small porosity values were more sensitive to cyclic load, in terms of the increase rate in porosity. It was also observed that the cyclic load led to the enlargement of relatively small pores and the generation of new pores, whereas the freeze-thaw cycles mainly resulted in the development of relatively large pores.

(3) The final UCS test results show that the UCS of the tested samples after the freeze-thaw cycles was decreased within the range of 5 - 10%, while it was within the range of 20 - 40% for samples under the combined cyclic load and freeze-thaw cycles.

(4) The Poisson's ratio of the tested sandstone samples was increased after the freeze-thaw cycles, but it was clearly decreased when both cyclic load and freeze-thaw cycle were applied. The AE results show that the larger the internal porosity of the sample, the more active the AE activity during the compaction stage. The peak acoustic emission count was positively correlated with the peak strength of the tested sandstone samples.

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Fig Captions

Fig. 1 Geological locations of the three sandstones used in the study.

Fig. 2 Flowchart of the experimental scheme.

Fig. 3 Experimental apparatus. (a) Servo-controlled testing machine, (b) TDS300 freeze-thaw machine, (c) low-field NMR testing machine (model: AniMR-150), and (d) UCS test system.

Fig. 4 Relationship between cyclic mechanical load and time.

Fig. 5 Schematic diagram of one freeze-thaw cycle.

Fig. 6 The principle of NMR. (a) Protons in applied field, and (b) magnetization vector changes under the effect of RF pulses (after Friebolin, 1991).

Fig. 7 T_2 pore size distribution and porosity accumulation of the tested sandstone samples under different loading conditions.

Fig. 8 Porosity evolution of the tested samples under different loading conditions.

Fig. 9 Schematic diagram of a T_2 spectrum division.

Fig. 10 NMR test results of the pore parameters of the samples. (a) Bound fluid saturation, and (b) permeability.

Fig. 11 Longitudinal NMR imaging of samples saturated with water under different conditions. The light spots represent the pore spaces that have been filled with water, while the black areas denote rock matrix.

Fig. 12 Stress-strain curves (black line) of different sandstone samples under cyclic mechanical load and secondary uniaxial loading and unloading (red line).

Fig. 13 UCS reductions of the tested sandstone samples subjected to cyclic load and freeze-thaw cycles.

Fig. 14 Stress versus strain measured in the UCS tests, together with the measured acoustic emission count.

Conflict of interest

We have no conflict of interests to declare

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