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NMR research on deterioration characteristics of microscopic structure of sandstones in freeze−thaw cycles

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Abstract: In order to study the deterioration characteristics of the microscopic structure of sandstones in freeze−thaw cycles, tests of 180 freeze−thaw cycles were performed on sandstone specimens. The nuclear magnetic resonance (NMR) technique was applied to the measurement of sandstone specimens and analysis of the magnetic resonance imaging. Then, the fractal theory was employed to compute the fractal dimension values of pore development of rocks after different freeze−thaw cycles. The results show that the mass and porosity of rocks grow with the increase of freeze−thaw cycles. According to the NMR T_2 distribution of sandstones, the pore sizes of rock specimens increase after 180 freeze−thaw cycles, especially that of the medium-sized and small-sized pores. The spatial distribution of sandstone pores after freeze−thaw cycles has fractal features within certain range, and the fractal dimension of sandstones tends to increase gradually.

Key words: nuclear magnetic resonance (NMR); freeze−thaw cycles; deterioration of rocks; microscopic structure; fractal dimension

1 Introduction

The observation of the evolvement of microscopic structural deterioration of rocks is very important to the establishment of equations of deteriorating evolvement and the macroscopic constitutive relation of the medium of rocks. Current detections of microscopic deterioration of rocks are mainly conducted with traditional methods like the technique of CT scanning [1], method of scanning electron microscope (SEM) [2], technique of digital imaging treatment [3] and acoustic emission [4,5], yet these methods are not very satisfying. For example, results of CT scanning could not reflect the features of microscopic structure [6], and the experiment is costly; the method of electron microscope requires that the specimen is small, and it could only be used in real time observation; indices like frequency detected with the technique of acoustic emission could hardly be associated with the parameter of rock crack in a quantitative relation. Problems like these are many. As a new method for analysis and detection in physical tests of rocks, the technique of nuclear magnetic resonance (NMR) [7] could be used to obtain parameters like porosity, free fluid index (FFI), pore-size distribution, T_2 distribution of transverse relaxation time [8] and so on. It could be applied in experiments and detection researches on pore distribution, characteristics of the inner structure of rocks and so on [9,10]. What's more, the features of pore distribution in the solid porous medium could be presented visually with the magnetic resonance imaging (MRI), which provides a useful means for the research on the fractal characteristic of the microscopic structure of rocks.

There have been many scholars who adopt the NMR technique to conduct researches in the field of geotechnical engineering. LI et al [11] harnessed NMR to research the evaluation of pore structures, compared and analyzed the relation between the NMR pore spectrum of rock cores and the pore-size distribution with the mercury penetration method. WU et al [12] studied the pore structure and liquid flow velocity distribution in water-saturated porous media probed by MRI. NMR and constant-rate mercury intrusion experiments were carried out to analyze the influence of microscopic pore structure on physical characteristics

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and occurrence characteristics of free fluid [13].

The NMR technique was also employed in related fields in the aspect of rock freezing and thawing. DU et al [14] used NMR to verify the reliability of calibration curves by TDR in the frozen soil. ZHOU et al [15,16] analyzed the T_2 distribution of rocks, the changing features of T_2 spectral size and the characteristics of pore distribution in rocks with NMR. CAI et al [17] studied the characteristics of structural deterioration of pores inside rocks under the condition of cryogenic nitrogen freezing with NMR. However, there have been limited researches on freeze−thawing deterioration of rocks that are conducted with NMR.

Sandstones for the tests of freeze−thaw cycling were chosen, and NMR was adopted to test the characteristics of deterioration of frozen−thawed rocks. Then, the changes of the mass, porosity, T_2 distribution and MRI results of rocks after different number of freeze−thaw cycles were analyzed and discussed. In addition, the fractal theory was employed to compute the fractal dimension values of pore distribution in MRI figures, and the characteristics of deterioration of the microscopic structures of sandstones in freeze−thaw cycles are obtained.

2 Experimental

2.1 Rock specimens and equipments

The rock specimen was yellow sandstone, taken from Gannan area of Gansu Province, China. It has a nice property of homogeneity and integrity, and was mainly constituted by cemented sand. The yellow sandstones were processed into standard specimens of cylinder according to the requirement of height−diameter ratio of 2:1. The key experimental instruments included type TDS−300 freeze−thaw cycle testing machine manufactured by Donghua Testing Equipments Company, Ltd. in the city of Suzhou, and vacuum saturation machine and type AniMR−150 rock MRI imaging analysis system manufactured by Niumag Electric Technology Company, Ltd. in the city of Suzhou.

2.2 Experimental program and procedure

2.2.1 Freeze−thaw cycle

In accordance with the operation specifications for freeze−thaw cycle tests in “Codes of Rock Tests in Water Conservancy and Hydroelectric Projects (SL264−2001)”, and in consideration of the climate at the sampling site, the rock specimens were frozen for 4 h at the temperature of −30 °C and then thawed for 4 h in the water at −20 °C, that is to say, every freeze−thaw cycle lasted for 8 h, and the cycle was repeated in this way. A testing cycle included 20 freeze−thaw cycles. After every testing cycle, the specimens were taken out for the observation of their features change. They were also weighed for the comparison of mass changes before and after freezing and thawing. This procedure will not conclude until 180 freeze−thaw cycles are finished.

2.2.2 NMR tests

Before the freeze−thaw cycling tests, an NMR test was conducted once. Then, the rock specimens were taken out after every 20 freeze−thaw cycles. The specimens were wiped dry for NMR tests. The changes of porosity, T_2 distribution and MRI of rocks were thus obtained.

2.2.3 Experimental procedure

The procedure of the experiment is as follows: 1) Preparing for rock specimens; 2) Saturating the rock specimens with the vacuum saturation machine. The vacuum pressure was 0.1 MPa and the time for air exhaustion was 4 h. When the air was exhausted, the specimens were soaked in pure water for 24 h; 3) Conducting NMR relaxation measurement and MRI tests on rock specimens which have been treated with vacuum saturation, with AniMR−150 rock MRI analysis system (seen as 0 freeze−thaw cycle); 4) Experimenting all the specimens with 20 freeze−thaw cycles, and repeating Steps 2 and 3 successively; 5) Repeating Step 4. In this way, results for the 180 freeze−thaw cycle tests and NMR tests on rock specimens were obtained.

3 Results and discussion

3.1 Mass

Figure 1 shows the changes of the mass of rock specimens after different freeze−thaw cycles. It can be known that with the increase of the number of freeze−thaw cycles, the mass of every rock specimen grows gradually in a regular manner, which almost develops linearly. This means that during the freeze−thaw process, after the rock specimens are frozen, the water in the pores freezes and expands, producing

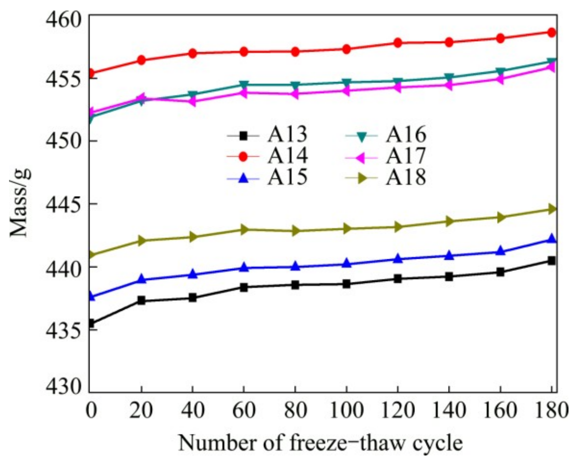

Fig. 1 Changes of mass of sandstones after different numbers of freeze−thaw cycles

extrusion force against the walls of pores, which leads to the expansion of micro-pores and formation of new micro-pores. While, at the stage of thawing, the water melted from ice would enter the newly formed micro-pores, and the moisture from outside would migrate into rocks through the passages of pores. The repetition of this process will lead to the increase of the mass of rocks.

3.2 Porosity

It is very important for the research on the characteristics of deterioration of rocks under the effect of freezing and thawing to describe the features of the structural changes of pores within rocks. Figure 2 displays the changes of porosity in the NMR of sandstones after different freeze−thaw cycles. It can be seen that with the increase of freeze−thaw cycles, the porosity of sandstones gradually grows with an average rate of 38.81%, which implies the appearance of essential changes of rocks under the effect of freeze−thaw cycles, such as the interior deterioration, expansion and connection of cracks, and the alteration of the mutual effect between grains. However, the different values of porosity change at different freeze−thaw stages indicate that the velocities and degrees of deterioration of rocks are various.

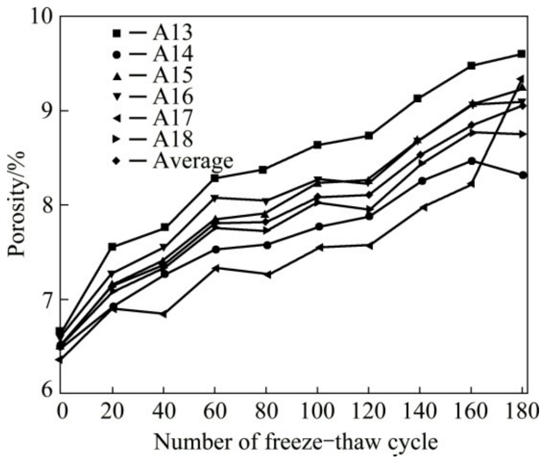

Fig. 2 Changes of porosity after different numbers of freeze−thaw cycles

According to the changes of the average porosity of rock specimens, the development of sandstone cracks shows certain rule after 180 freeze−thaw cycles.

1) After 20 freeze−thaw cycles, the average increase of porosity of sandstones reaches 10%, taking up 26% of the entire increase. This means that at the early stage of freezing and thawing, the freeze−grow force is much larger than the cohesive force between the crystal grains within the sandstone, which leads to a substantial increase of porosity.

2) At the stage of 20−120 freeze−thaw cycles, with the increase of freeze−thaw cycles, the growth of porosity of most rock specimens gradually slows down. It can be analyzed that during the freeze−thaw cycles, the grains within the rocks are compacted by the accumulating effect of the increase of pore mass, thus the cohesion between crystals gradually increases to offset some frost heave force, so the expansion or connection of the pores is limited. Besides, the entrance of water into the pores limits the rapid growth of pores to some extent, as the water under the static pressure in the pores produces extrusion force.

3) After 120 freeze−thaw cycles, the growth of porosity of rock specimens begins to increase, which shows that the pores in rocks start to develop and expand at a higher speed. This indicates that the repetitive accumulation of the frost heave force against the pores within sandstones has continually weakened the cohesive force between crystal grains, which causes the freeze−grow force to exceed the cohesive force between grains, thus the porosity increases sharply.

4) During the process of the increase of freeze−thaw cycles, there is a trend of “increasing→decreasing or constant→increasing” in the porosity of many samples, which shows that the expansion and connection of pores are not a process of continuous enlargement, but a process of recycling; the expansion of larger pores could produce a quasi-compaction effect on the smaller pores around, thus very little moisture could enter the compacted micro-pores, so the porosity generally decreased.

3.3 NMR T_2 distribution

The changes of NMR T_2 spectrum could reflect the structural changes of pores within rocks. The sizes of pores in rock specimens are in proportion to the fluid traverse relaxation time T_2. When T_2 is small, it means that the sizes of pores in rock specimens are small; the location of peaks in T_2 spectrum is directly related to the size of pores, and the peak value in T_2 spectrum reflects the concentration degree of the distribution of pore sizes within rocks. Therefore, the changes of T_2 spectrum could qualitatively describe the structural changes of pores inside the rocks. Figure 3 shows the T_2 distribution of rock specimens A13 and A15 after 180 freeze−thaw cycles. It can be seen that the T_2 of sandstones is mainly presented by 3-peak image. With the increase of the number of freeze−thaw cycles, the T_2 spectrum shows an apparent shift to the right, that is, skewing in the direction of T_2 of larger pores, which means that the intensity of NMR signals of T_2 spectrum of larger pores has increased. This also means that the freeze−grow effect of ice has caused the smaller pores to expand towards larger pores and connect with them. As the number of freeze−thaw cycles increases, the ranges of the 1st and 3rd peaks (counting from the left to the right) clearly extend, which means that new micro-pores have appeared and expanded in the sandstones, and water has entered the micro-pores. The three peaks in T_2 spectrum of rock specimens have grown larger, especially the 1st and 3rd ones on the left, which shows that under the freeze−thaw effect, new micro-pores have been continuously developed and produced inside the rocks, and have expanded into medium-sized and large-sized pores, segmenting the rock structures and causing freeze−thaw deterioration.

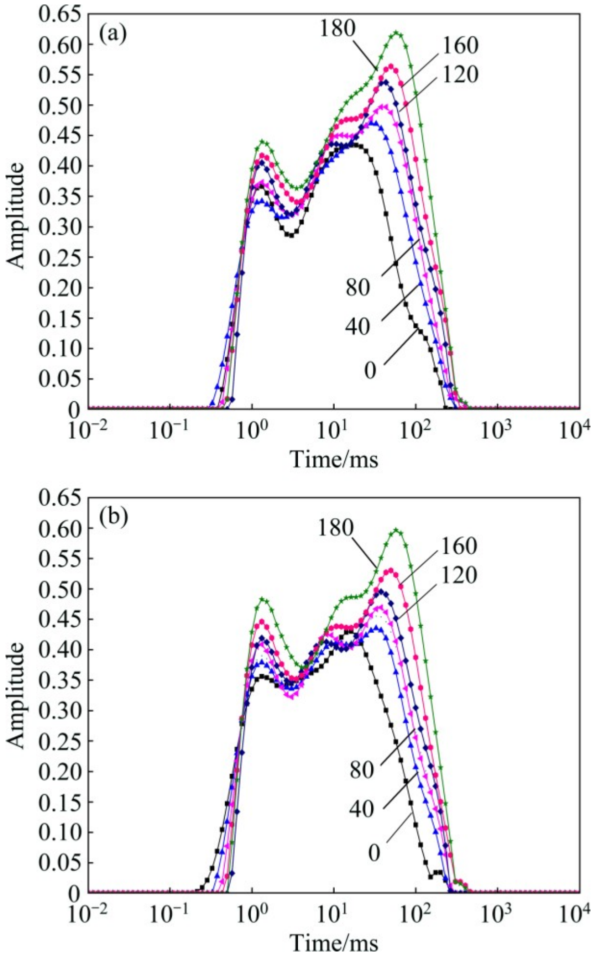

Fig. 3 Changes of T_2 distribution of typical rock specimens after different numbers of freeze−thaw cycles: (a) A13; (b) A15

It can be seen from the general form of T_2 spectrum, during the process from 0 to 180 freeze−thaw cycles, the relaxation time of sandstones mainly concentrate at 1−100 ms, which means that the most pores are micro-pores and small pores, and those large-sized pores such as voids and through cracks have not appeared inside the sandstones.

3.4 Magnetic resonance imaging (MRI)

After applying an MRI test to the rock specimens which have undergone different freeze−thaw cycles, two-dimensional images of transverse cross sections are obtained. They are images of the same location in the axial direction of rock specimens. The images of transverse sections are in the shape of rock specimens, and the lightness of the images reflects the volume of water in specimens. Therefore, with this feature of images, the distribution of pores inside rock specimens could be discerned with MRI. Figure 4 shows the MRI results of rock specimen A15 after 160 freeze−thaw cycles, which show the structural changes of pores in the rocks that are processed with 0 to 160 freeze−thaw cycles. It can be seen from Fig. 4(a) that before freezing and thawing, the image is comparatively dark, and there is hardly any light spot inside the rock. This means that the original pores inside rock specimen A15 are mainly micro-pores. Besides, the brim of the image is apparently brighter than the central section, which shows that the pores on the surface of the rock contain much free water. Figure 4(b) indicates that after 20 freeze−thaw cycles, a few bright spots appear at the central section of the image, which shows that the freeze−thaw cycling effect has caused the pores inside the sandstone to change, and the micro-pores have started to expand into larger pores. After 40, 60, 80, 100 and 120 freeze−thaw cycles, the bright spots in the image gradually increase, the pores in the rock continue to grow larger and the large pores increase, too. These changes are presented in Figs. 4(c)−(g), which show that the freeze-thaw effect has reinforced the expansion of pores inside the rock and has consistently weakened the pore structures, which has led to the development and expansion of pores. When the number of freeze−thaw cycles reaches 140, the bright spots in the image gradually cover the entire transverse cross section, the brightness of the image becomes quite even, which shows that the interior of the rock is isotropic, as no through crack is found (see Fig. 4(h)). After 160 freeze−thaw cycles, large pores within the rock specimen increase, and bright spots continue to increase, together with tiny cracks, which shows that a weak structural surface has formed in the sandstone specimen and the interior of the sandstone has seriously deteriorated.

It can be seen from Figs. 4(a)−(i) that bright spots in the images increase and the brightness becomes evener, which show that with the increase of the number of freeze−thaw cycles, the freeze−thaw effect has caused the tiny cracks inside the rock to expand and connect with each other. It also implies that the size of pores in the sandstone specimen has evolved more even and concentrated.

3.5 Fractal dimensions of MRI image

Widely used researches on the characteristics of changes of microstructural deterioration of rocks, the fractal theory could reflect the process of microstructural

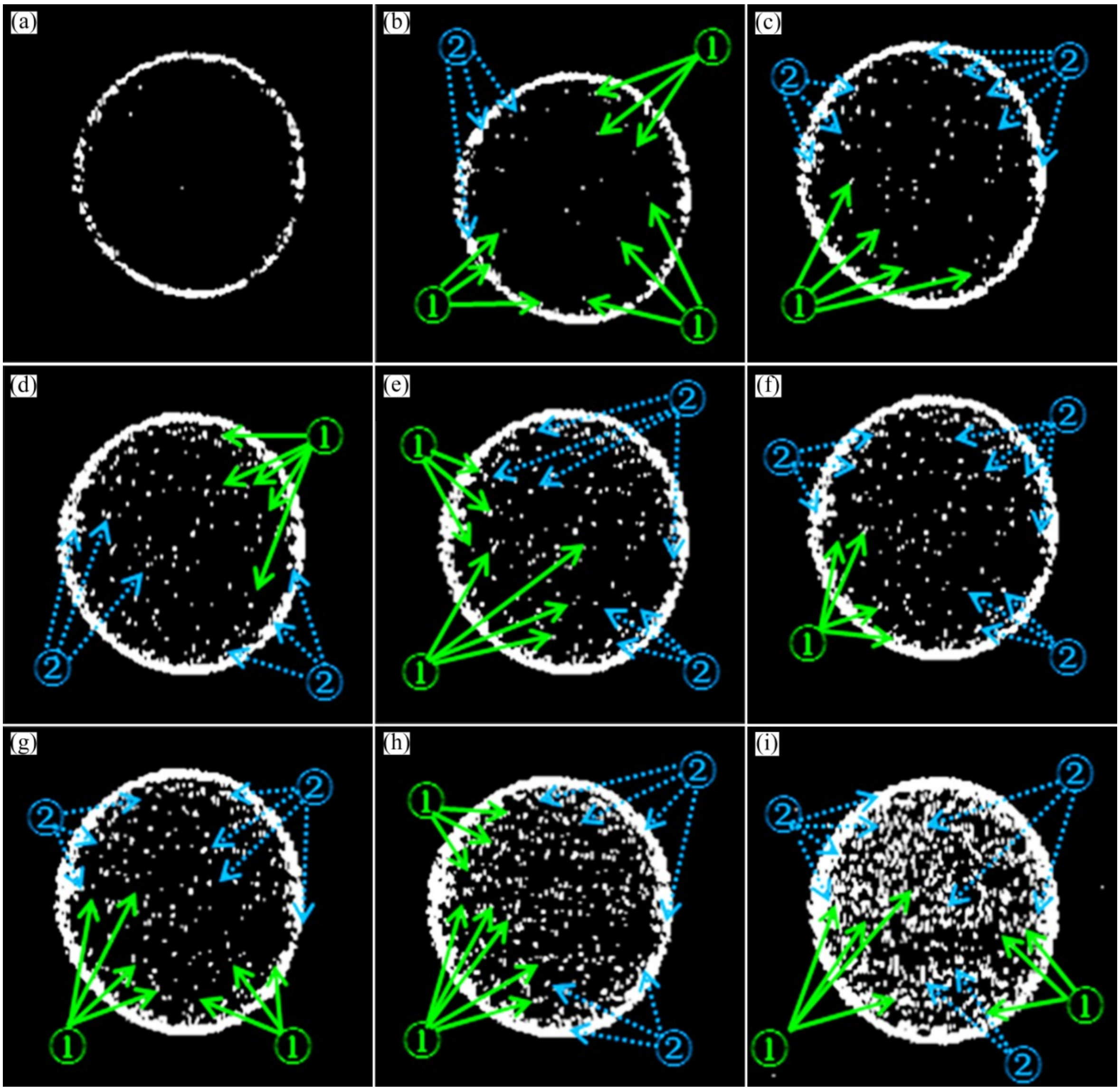

Fig. 4 MRI images of sandstones after different numbers of freeze−thaw cycles: (a) 0; (b) 20; (c) 40; (d) 60; (e) 80; (f) 100; (g) 120; (h) 140; (i) 160 (Two different kinds of pore deformation are approximately labelled by ① which indicates new pores generated, and ② which indicates pores deformed to larger size)

deterioration of rocks. The process from microscopic deterioration to macroscopic fracture of rocks shows apparent statistical self-similarity [18], and fractal features are shown by numerous mechanical quantities and geometrical senses, such as the distribution of cracks, density of cracks, and fracture toughness [19,20]. The MRI image of frozen−thawed rocks reflects the authentic features of pore development, thus the fractal theory could be adopted to describe the influence of features of crack distribution on the mechanisms of freeze−thaw effect and rock deterioration and fracturing. With regard to the fact that the MRI image reflects a “body” of 3D image, the method of triangular prism was adopted to characterize the fractal features of pores of frozen−thawed rocks [21].

According to the MRI images in Fig. 4, the fractal dimensions are computed with the method of triangular prism in the fractal calculation procedure. The results are listed in Table 1.

It can be seen from Table 1 that after different numbers of freeze−thaw cycles, the fractal dimensions D of pores of every rock specimen are greater than 2, and the relevant coefficients of fractal dimensions of every MRI image are greater than 0.98, which shows that the spatial distribution of pores in rocks during the development under the freeze−thaw effect has fractal features in the statistical sense within certain range.

Figure 5 shows the relation between fractal dimension and the number of freeze−thaw cycles for every rock specimen. It can be seen that with the increase of freeze−thaw cycles, fractal dimension gradually increases. It can be explained with the fractal theory that

Table 1 Results of fractal dimensions of MRI images of rock specimens

Number of freeze−thaw cycle	Fractal dimension	Relevant coefficient
0	2.199	0.986
20	2.260	0.986
40	2.288	0.986
60	2.319	0.985
80	2.340	0.985
100	2.348	0.985
120	2.335	0.985
140	2.404	0.984
160	2.433	0.984

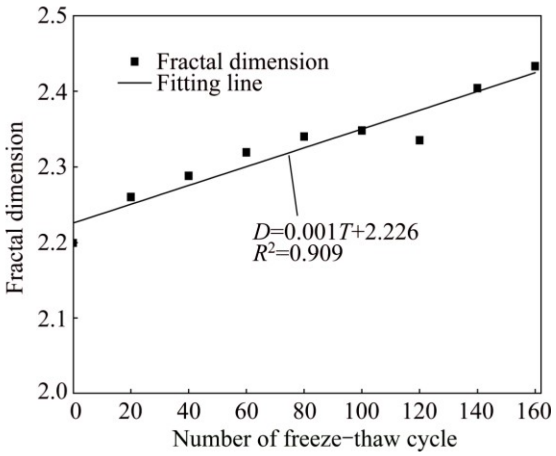

Fig. 5 Relation between fractal dimension of sandstones and number of freeze−thaw cycle

the greater the fractal dimension of the distribution of pores in rocks is, the more the pores have developed and the more uneven the distribution of pores has been. This means that under the freeze−grow effect of ice, cracks have kept developing and expanding, causing the micro-pores to expand into large pores, thus fractal dimension tends to increase gradually. The results also show that the development of pores inside the frozen−thawed sandstones is quite self-similar, therefore, it is pragmatic to use the value of fractal dimension to characterize the degree of pore development in the frozen−thawed rocks.

4 Conclusions

1) With the increase of freeze−thaw cycles, the mass and porosity grow. It shows that under the effect of freeze−thaw cycles, deterioration occurs to rock specimens, and the cracks keep developing and expanding with the increase of freeze−thaw cycles. The structural features of pores in rocks clearly affect the freeze−thaw deterioration of rocks.

2) The NMR T_2 distribution of sandstones is mainly represented by 3 peak images. From 0 to 180 freeze−thaw cycles, all of the T_2 distributions of rock specimens grow, and the growth of that of medium-sized and small-sized pores is especially obvious. This shows that under the freeze−thaw effect, micro-pores in rocks keep developing and expanding, and freeze−thaw deterioration occurs to rock specimens.

3) The MRI results of rock specimens after different numbers of freeze−thaw cycles show the evolvement of freeze-thaw deterioration of the microscopic structure of rocks in a dynamic way. And the spatial distribution of pores inside sandstones after freeze−thaw cycles has fractal features within certain range. The greater the fractal dimension of pores is, the more developed the pores are, the more uneven their distribution is, and the greater the freeze−thaw influence will be.

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砂岩细观结构冻融损伤特征的核磁共振研究

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摘　要：为了研究砂岩细观结构的冻融损伤特性，对砂岩共进行了 180 次冻融循环实验，利用核磁共振技术（NMR）对冻融循环后的岩样进行了检测和核磁共振成像分析，并采用分形理论计算了砂岩经历不同冻融循环后孔隙发育的分形维数。研究结果表明：随着冻融循环次数的增多，砂岩的质量、孔隙度、核磁共振弛豫时间 T_2 分布均会增大；核磁共振 T_2 谱分布表明，经历 180 次冻融后，砂岩的孔隙尺寸均会增大，尤其是中、小尺寸孔隙增大明显；在冻融作用下，砂岩的孔隙结构演化，在空间分布上具有分形特征，其分形维数出现逐渐增大的趋势。

关键词：核磁共振(NMR)；冻融循环；岩石损伤；细观结构；分形维数

(Edited by Xiang-qun LI)