



Effects of multiple heating-cooling cycles on the permeability and microstructure of a mortar

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HIGHLIGHTS

- Real-time variation in the gas permeability and porosity of a mortar are measured.
- Multiple heating-cooling cycles will induce an increase in gas permeability.
- Thermal cycles enlarge the existing microcracks and generate new microcracks.

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ABSTRACT

Cement-based materials can be used as a sealing layer in compressed air energy storage (CAES) underground storage caverns to ensure airtightness. This experimental study investigated the effects of multiple heating-cooling cycles with variations in the gas permeability, porosity, and microstructure of a mortar sample. Three different temperature ranges, namely, 20–50 °C, 20–60 °C and 20–70 °C, were selected according to different operation conditions of the CAES plants. The results showed that the gas permeability decreases with an increase in temperature in the heating process within each temperature cycle due to a reduction in the pore volume. After the sample was subjected to multiple heating-cooling cycles, the gas permeability has an overall increasing tendency with an increase in cycle number, and the magnitude of the permeability increase is related to the maximum cycling temperature. The microstructure observed by SEM indicates that the temperature cycles will induce some microcracks, which are mainly distributed on the cement matrix surface and in the interfacial transition zone (ITZ) between the cement matrix and sand aggregate. The expansion of the existing cracks and appearance of new cracks caused by a temperature effect led to an increase in the gas permeability.

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1. Introduction

Power generation technologies with renewable energy sources have been developing rapidly in recent years. However, the generating capacities of renewable energy systems are limited by natural conditions that make them intermittent and unstable [1]. Energy storage technologies can overcome the problem of intermittency to produce more stable and reliable renewable energy sources. Compressed air energy storage (CAES) is a commercial and utility-scale technology that is suitable for providing long-duration energy storage [2]. In CAES, electrical energy is converted into a form of highly pressurized air and stored in underground

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rock caverns. The highly compressed air is then released through gas turbines to produce electricity when needed [3–5]. As the key component of CAES power plants, underground storage caverns normally have a very high requirement for airtightness. In engineering, there are two common methods for guaranteeing the airtightness of the CAES caverns. The first method is to construct the underground storage caverns in special rocks with extremely lower permeability, such as salt rock. To date, only two commercial CAES plants, namely, a 290-MW unit built in Huntorf, Germany in 1978 [6] and a 110-MW unit built in McIntosh, Alabama, USA in 1991 [7], were constructed by this method. The second method uses an artificial sealing layer within the caverns to ensure airtightness. In this case, cement-based materials, such as concrete or mortar, can be considered an optional material for constructing the sealing layer. However, because the working conditions in CAES caverns are very special, the gas permeability of cement-based materials under CAES operating conditions should

be investigated in detail before the consideration of its engineering application.

It is known that cement-based materials are non-homogenous materials that are composed of three parts: a cement paste matrix, aggregate particle inclusions, and a porous interface called the interfacial transition zone (ITZ) between the two [8–10]. Because the mixing proportions of cement-based materials can affect the microstructure and pore distribution and further influence the gas permeability, ample previous studies have focused on the influence of the mixture parameters on the gas permeability. The results indicate that the gas permeability of cement-based materials is related to the water to cement ratio, the aggregate content, and the aggregate volume content [10–15]. These factors can be regarded as the internal factors that affect the gas permeability and can be artificially modified to improve the performance of cement-based materials. In addition to the internal factors, some external factors, such as the environment temperature, will also affect the gas permeability and should be considered when investigating the gas permeability of cement-based materials.

During the operation of CAES plants, the temperature in the caverns will continuously fluctuate as a result of the frequent inflation and deflation of the compressed air. The range of temperature fluctuations, which depends on the operating conditions, normally ranges from 20 to 70 °C [16–18]. Under this circumstance, the cyclical change in temperature becomes an important external environment factor that affects the gas permeability of cement-based materials. Most previous studies focused on the effect of temperature on the mechanical properties of cement-based materials. It has been concluded that high temperature can cause significant changes in the pore microstructure, nucleation, and propagation of microcracks [19–21]. Moreover, the cooling process can also cause additional microcracking [22]. However, regarding the temperature effects on the gas permeability, only a few experimental studies have been carried out. Chen et al. [22,23] investigated the permeability of a mortar heated to 400 °C and found a significant increase in gas permeability after the heat treatment. Pei et al. [24] concluded that the permeability of a mortar increases by 2–3 orders of magnitude when the temperature was raised from 105 °C to 600 °C. Josipa et al. [25] measured the gas permeability of concrete at temperatures ranging from 20 °C to 300 °C and found a steady increase in permeability with increasing temperature. However, most of these experimental studies related to temperature effects were carried out under very high temperature, which does not correspond to the actual operating conditions of CAES.

As for the effects of the temperature cycle, Chen et al. [22,23] found that the gas permeability of mortars irreversibly increased after one heating and cooling cycle. Trilok et al. [26] studied the effect of a heating-cooling process on rubberized concrete, and the results showed an increase in permeability after cooling. However, in the above tests, the heating-cooling process was limited to only one cycle. Lin et al. [27] investigated the effect of multiple heating-cooling treatments on the macro-mechanical property and permeability of a mortar, and the test results showed a gradual increase in the porosity and permeability with an increase in the maximum heating temperature and the number of heating cycles. However, the heating-cooling cycles in Lin et al.'s tests [27] were carried out in an oven, which means that the permeability measurements only occurred after cooling the specimens to room temperature rather than performing the permeability measurements during the heating and cooling process.

In conclusion, considering that the operating condition in CAES is very special, there is almost no specialized experimental study that has investigated the real-time permeability and porosity variation in cement-based materials subjected to multiple heating-cooling cycles in a relatively low temperature range (within 20–70 °C). In this work, different multiple heating-cooling cycles in a

relatively low temperature range were applied on mortar samples to simulate the actual temperature variation. Different maximum temperatures were selected to correspond to different operation conditions of the CAES plants. During the experiment, the real-time variation in the gas permeability and porosity of mortar were measured. This study attempts (1) to investigate the variation law of gas permeability, porosity, and microstructure under multiple heating-cooling cycles of a mortar lining within a CAES gas storage cavern, and (2) to propose recommendations on the operation condition of CAES plants according to the variation law under different temperature ranges.

2. Experimental procedures

2.1. Materials and specimen preparation

The experiments were carried out using a mortar made from China commercial P II 52.5 Portland cement and ISO standard silica sand (0–2 mm in diameter) with a water/cement (w/c) ratio of 0.4. The mixing proportion of the mortar was determined by a series preliminary experiments considering the w/c ratio and sand volume content. In this study, mortar samples were prepared with a 0.4 w/c ratio and a 0.5 cement-sand ratio. The cement-sand ratio in the formulation is larger than that of standard mortars, which produces mortar with a relatively low permeability and reasonable mechanical strength [10,12]. The cement and mortar compositions are shown in Tables 1 and 2, respectively.

The mixing procedure comprised the following steps according to the Chinese Standard for test method of performance on building mortar (JGJ/T70-2009) [28]:

- (1) The sand and cement were mixed within 30 s, and then water was added, and mixing was continued for 2 min.
- (2) The obtained mixtures were placed into an annular Teflon mold with inner diameter of 50 mm and height of 10 mm.
- (3) The molds were vibrated until no significant air bubbles escaped from the top surface of mixtures. The intensity and duration of the vibration was adjusted according to the workability of each sample.
- (4) The samples were demolded after 24 h and then cured in a sealed desiccator at a constant temperature of 20 ± 2 °C and a relative humidity (RH) of 90%.
- (5) After 28 days curing, the samples ($\Phi 50 \times 10$ mm) were taken out from the desiccator and could be used for the tests.

Before measuring the gas permeability and microstructure, a procedure to remove sample moisture is necessary. In this study, the samples were slowly dried in an oven at 40 °C for approximately 15 days until the mass of the samples was stable. Previous studies indicate that this temperature does not modify the pore structure [29,30].

2.2. Measurements of the gas permeability and porosity

2.2.1. Gas permeability measurement

The measurements of the gas permeability and porosity of the mortar samples subjected to thermal cycles were carried out using a permeability and porosity test system developed by the authors [31]. The schematic diagram of the test system is shown in Fig. 1. The confining pressure is provided by a water pump. The temperature is controlled by a water bath that can maintain a constant temperature between 5 and 95 °C. Water at a certain temperature flows through a copper tube surrounding the sample to heat or cool down the confining water. The temperature of the confining water was measured by a thermocouple which was located near

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