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Physical and mechanical behavior of claystone exposed to temperatures up to 1000 °C

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A B S T R A C T

The behavior of claystone exposed to temperatures up to 1000 °C has been investigated experimentally at laboratory scale. Uniaxial and triaxial compressive testing and other measurements of geometric and density changes were performed on the specimens after thermal treatment. These experiments showed that compared to untreated specimens, bulk density increases with increasing temperature, while total porosities of those exposed to 200 °C and above dramatically increase. Deformation modulus and compressive strength of the specimens exposed to thermal treatment at 800 °C and below always increase, but decrease after 1000 °C treatment, compared to those exposed at room temperature. In addition, thermal treatment has little influence on the types of stress–strain curves and failure under conditions of confining pressures up to 20 MPa and at temperatures up to 1000 °C. Micro-crack closure in rocks due to thermal expansion is the main contributor to the increase in strength below 200 °C, and baking effects contribute to increase in strength up to 800 °C, whereas significant fracturing causes a decrease in strength at 1000 °C.

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1. Introduction

With the increasing utilization of underground space by modern rock engineering applications such as geothermal heat extraction [1,2], underground coal gasification [3,4], underground disposal of radioactive waste [5,6] as well as in-situ combustion enhanced oil recovery [7,8], there is a growing demand for knowledge on rock behaviors at and after high-temperature conditions to provide a basis for deformation, stability and safety analyses of the corresponding projects. Under the impact of high temperatures, rock microstructures may change significantly [9], new micro-cracks may be developed [10], and pre-existing ones may be extended and/or widened [11]. Moreover, it is well established that high temperatures cause various physical and mineralogical changes within the rocks [12,13]. Consequently, the rock behavior exposed to high temperatures can be quite different from those under conventional (room temperature) conditions.

In the last decades, extensive laboratory research has been carried out on rock specimens exposed to high temperatures to investigate temperature influences on physical [14–18], thermal [19–21] and mechanical [22–25] properties, microstructures [13,26,27], failure mechanisms [28–30], thermo-mechanical models [31–33], etc. Usually, these experiments were performed on specimens either under high-temperature conditions or at room temperature but with experience of slow heating and cooling. However, the results from testing on slow cooling of a preheated rock in the air or the furnace chamber are almost equivalent to those conducted on a heated rock [14,34]. These research efforts indicate that rock properties can be highly dependent on temperature, and the characteristics of temperature dependency vary with rock types, rock initial features such as micro-cracks and structure, temperature gradient, etc. For example, elastic modulus and compressive strength of granites [35,36] and marbles [37,38] generally decrease with increasing temperature, while these properties can increase for sandstone at temperatures below a certain temperature and subsequently decrease [25,39]. In addition, even for the same types of rocks, temperature-dependent rock behavior shows great variations when they are under different geological and stress conditions [40].

Even though plenty of investigations have been conducted for crystalline rocks such as granites and marbles and sedimentary rocks such as sandstone and limestone, limited data are available for argillaceous rocks such as claystone and mudstone. In order to extend the available data, claystone material was sampled to study physical and mechanical characteristics of claystone exposed to

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high temperatures through a suite of laboratory tests involving thermal treatment up to 1000 °C, uniaxial and triaxial consolidated undrained compressive tests under confining pressures of 5, 10, 15, 20 MPa, etc.

2. Experimental methods

2.1. Specimen characterization

Rock specimens under study are homogeneous and isotropic claystones retrieved at a depth of about 1190–1210 m in the Auguste-Victoria hard coal mine, North-Rhine Westphalia, Germany. Cylindrical specimens, for all tests in the study, with nominal diameter of 38 mm and length to diameter ratio of two were prepared by dry cutting.

To quantify physical and mechanical properties of the intact claystone specimens, a series of laboratory tests were first performed on the ones that had experienced two-week air drying. The testing program included uniaxial and consolidated undrained triaxial compression tests as well as the measurement of geometric and physical properties. The corresponding results are summarized in Table 1. X-ray diffractometry revealed that the claystone tested is composed of 38.97% illite, 16.84% kaolinite, 5.69% chlorite and 38.50% quartz (by weight percent) before thermal treatment.

2.2. Testing equipment and procedure

In this study thermal treatment was performed in an electrical furnace using the following procedure. Cylindrical specimens were carefully selected, subjected to a two-week air drying first, and then slowly heated at a rate of 50 °C/h at atmospheric pressure to a predetermined temperature in the furnace chamber. These specimens were then maintained at the temperature for 2 h and subsequently cooled down in the chamber at the same rate to room temperature. After thermal treatment, the specimens were conserved in a desiccator until mechanical testing. The predetermined temperatures were 80 °C, 150 °C, 200 °C, 300 °C, 400 °C, 600 °C, 800 °C and 1000 °C.

Height, diameter, and mass of each specimen were measured both before and after thermal treatment, whereas the geometric precision was 0.01 mm and weight precision 0.005 g. Mechanical testing on the treated specimens included uniaxial and consolidated undrained triaxial compression tests, following the corresponding standards in the German Industry Standards (DIN) published in 1990. The former was performed at a loading rate of 0.2% of the specimen height per minute, and the latter at a loading rate of 0.055 mm/min in a servo-controlled testing system. The number of specimens tested after thermal treatment at each temperature level and confining pressure is listed in Table 2.

Table 1
Physical and mechanical properties of claystone under the air-dried condition.

Parameters	Values	German Industry Standards
Bulk density (10^3 kg/m^3)	2.60 ± 0.06	DIN 18125
Grain density (10^3 kg/m^3)	2.81	DIN 18124-KP
Water content (%)	1.40 ± 0.22	DIN 18121
Total porosity (%)	8.65 ± 2.45	DIN 18125
Elastic modulus (GPa)	1.64	DIN 18136
Uniaxial Compressive Strength (MPa)	17.24	DIN 18136
Cohesive strength (MPa)	4.66	DIN 18137-CU
Friction angle (°)	16.73	DIN 18137-CU

Table 2
The number of specimens tested at different temperatures and confining pressures.

	σ_3=0.1 MPa	σ_3=5 MPa	σ_3=10 MPa	σ_3=15 MPa	σ_3=20 MPa
Air-dried	3	3	3	3	3
80 °C	3	3	3	3	3
150 °C	3	3	3	3	3
200 °C	2	3	3	3	3
300 °C	3	3	3	3	3
400 °C	3	3	3	3	3
600 °C	3	3	3	3	3
800 °C	2	1	1	1	1
1000 °C	2	–	–	–	–

3. Experimental results

3.1. Apparent shape

Thermal treatment on rocks causes not only a color change but also other external signs such as cracks and volume expansion [41]. Tian et al. [42] observed color changes from gray to reddish brown appearing on two kinds of claystone specimens after treatment at 1000 °C . For the claystone tested in this study, color changes of the specimens subjected to thermal treatments below 800 °C are very slight, but become quite obvious above that temperature. As shown in Fig. 1, the air-dried specimen is dark gray; after treatment at 400 °C and 600 °C the color turns light gray, but light and dark reddish brown after the treatments at 800 °C and 1000 °C, respectively. The color change may be caused by chemical reactions of minerals.

No visible fractures were observed on the specimens after different high-temperature treatment at below 1000 °C (Fig. 1), but slight variation of the volume was measured (Fig. 4a). However, six specimens treated to 1000 °C were all cracked or even collapsed (Fig. 2), which is consistent with the observation from Tian et al. [42], who found sandy claystone cracked after treatment at 1000 °C . In addition, if a specimen containing a certain amount of water is used for thermal treatment, it will collapse or break into pieces during the heating process at 600 °C or even lower (Fig. 3), for moisture can freely escape from the specimen.

3.2. Volume, mass and bulk density

Since all specimens tested are of cylindrical shape, it is easy to calculate the volume of a given sample from the height and diameter measured and bulk density from the mass determined and volume calculated. Thus, the variation of volume, mass and bulk density of a specimen before and after thermal treatment can be easily obtained, and respectively represented as volume increase rate (v_v), mass decrease rate (v_m), and density decrease rate (v_ρ) as

$$v_v = \frac{V_2 - V_1}{V_1} \times 100\% \quad (1)$$

$$v_m = \frac{m_1 - m_2}{m_1} \times 100\% \quad (2)$$

$$v_\rho = \frac{\rho_1 - \rho_2}{\rho_1} \times 100\% \quad (3)$$

where V_1, V_2, m_1, m_2, ρ_1 and ρ_2 are the volume, mass and bulk density of a specimen before and after thermal treatment, respectively.

Figs. 4 and 5(a) show the relations between v_v and v_m as well as v_ρ and treatment temperature, expressed by the calculated and

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