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Thermal effect on the physical properties of carbonate rocks

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ABSTRACT

The effect of thermal damage on the physical properties of five carbonate rocks has been investigated. The tests were conducted on two marbles and three limestones, mainly composed of calcite but with different grain sizes, porosities, structural and textural characteristics. Cubic samples prepared from these rocks were gradually heated to a specific temperature level of 100, 200, 300, 400 and $500\,^{\circ}$ C, and gradually cooled down to room temperature without causing thermal shock in order to investigate the effect of heating temperature on physical properties such as microstructure, bulk density, effective porosity and P-wave velocity. Microscopic investigations from thin sections showed that damage in rocks at elevated temperatures was induced in different severity depending on grain size, porosity, structural and textural characteristics. Colour changes were also observed in porous limestones (Lymra and Travertine) due to organic material. In accordance with the degree of calcite dilation depending on heating temperature and in turn new microcrack occurrence, separation along intragrain and/or intergrain boundaries and widening of existing cracks, P-wave velocity decreased to various levels of the initial value, whereas porosity increased. Microscopic analyses and P-wave velocity measurements indicate that compaction of rock structure up to 150 °C occurred and induced calcite dilation had no significant damage effect on the rock material. Compaction of rock structure led to an increase in P-wave velocity and slight decrease in porosity. Most of the damage occurred within 24h of heating time and further heating treatments brought relatively minor changes in physical properties. Damage intensity was well explained with P-wave velocity and effective porosity values depending on temperature increase.

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1. Introduction

Rocks are composed of minerals, bounding matrix, and cracks and pores. The geometry and density of the cracks and pores are the main controlling parameters for the physical properties of rocks [1]. In engineering rock structures, temperature variation is one of the primary factors influencing the integrity and physical properties of rocks. It is responsible for the changes in microstructure of the rock by inducing new crack and microcrack development and so, for the increase in void space volume [2]. It is quite important to study the physical properties of rocks in the laboratory to evaluate the thermally induced microcracks at different temperatures.

The study of changes in the physical properties of rocks as a function of thermal cracking is relevant to various engineering applications. A nuclear waste repository is essentially an excavated underground cavity composed of tunnels and openings in which radioactive waste is emplaced [3]. This waste releases heat after placement and causes a long-term an increase in the

temperature of the surrounding rock [4]. Studying the physical characteristics of rocks under different temperature conditions is essential to assess the suitability of host rock for integrity of nuclear waste repository and to ensure the safe isolation of nuclear wastes. The other industrial application is geothermal energy extraction. The large changes in temperature associated with reservoir exploitation will affect the porosity and crack state of the rock, which will in turn affect flow and heat transport characteristics of the geothermal system [5].

Researchers studied many parameters to analyse the thermal damage in rocks. Darot and Reuschle [1] investigated the variations of compressional wave velocity and water permeability for thermally cracked granite at 510 °C under different ranges of confining pressures and pore pressures. They found a regular increase in compressional wave velocity and a decrease in permeability during effective pressure increase. Chaki et al. [2] studied the influence of thermal damage for the range of peak temperatures up to 600 °C on physical properties of granite rock through porosity, ultrasonic wave velocity and permeability measurements and found a good consistency between the results of measurements. Liang et al. [4] carried out a series of physical and mechanical tests on salt rock at different temperatures up to 240 °C and found that the mechanical parameters have different

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reactions to a changing temperature. Their tests showed that the ultrasonic velocity of samples decreases with temperature increase and both the uniaxial compressive strength and axial strain increase with temperature. Lion et al. [6] heated the Anstrude limestone at two temperature levels of 150 and 250 °C in order to evaluate possible thermal effects such as micro-cracking. They measured no notable differences in porosity and permeability and observed no visible cracks from microscopic observations. However, strength limit and poromechanical properties (measured with a non-nil pore pressure) varied after thermal treatment. Fredrich and Wong [7] investigated the effect of temperature on the structure of the three rocks: granite, diabase. limestone by SEM. They found an important increase of the crack density and the mode of propagation as different for grain boundary and intragranular cracks. Ferrero and Marini [8] determined the crack densities by means of microscopic analysis in limestone and marble samples heated up to 600 °C. A correlation between an increase in open porosity, due to new fractures, and crack density was found for the two rocks. David et al. [9] investigated the influence of thermal cracking in comparison to stress-induced cracking on physical properties of La Peyratte granite: porosity, acoustic velocity, attenuation, electrical conductivity and permeability. Good correlations were found between the evolution of these properties and the amount of damage induced. Jones et al. [10] conducted thermal cracking experiments to investigate the role of anisotropic thermal expansion of the constituent minerals of basalt, microgabbro and microgranite in changing the physical properties of the rocks. Most of the damage in basalt samples occurred above 300 °C, where there is a rapid increase of damage up to 600 °C, whereas the microgranite showed enhanced permeability due to thermal cracking at 200 °C, and the microgabbro at 600 °C. Martin et al. [11] found significant increase in thermal expansion of welded tuff up to 250 °C temperature and confining pressure had minimal effect on the thermal expansion for tuff.

In this study, carbonate rocks were selected for the investigation of thermal damage. They are mainly monomineralic rocks and thermal cracking in these rocks is due to internal stress concentrations resulting from anisotropic thermal expansion of the calcite [6,12–14]. Depending on the temperature intensity, thermal cracking can occur between adjacent grains and/or within grains. Random orientation of the calcites contributes to predominantly high thermal stresses in the rock. Elevated

temperatures lead to expansion along the crystallographic *c*-direction and contraction along the direction perpendicular to *c*-direction [6,7,12–14]. It has also been demonstrated that the rate of expansion increases with the temperature [6,12–14]. Apart from the minerals on which rock sensitivity to temperature depends, other parameters are almost structural: granularity which can amplify differential expansions, porosity and cracks, both leaving free space for expansion at low temperatures [6]. The porosity and seismic velocity are two important parameters for the micromechanically based evaluation of the effects of the pores and cracks inside the rocks [15]. A detailed review of some of the numerous schemes proposed for estimating the effective elastic moduli of porous and cracked rocks is presented by Jaeger et al. [15].

This study investigates the effect of thermal damage on some physical properties of five different carbonate rocks. Different heating temperatures and heating time effects were studied. Thermal treatments were applied by heating the samples to a range of peak temperatures up to 500 °C for a period of up to 144 h. Ultrasonic wave propagation, bulk density and effective porosity measurements were performed since these tests are non-destructive and are more suitable to investigate the physical damage in rocks. Results of these tests are compatible and highly correlated with heat transport characteristics and mechanical properties of rocks [16,17]. In addition to porosity, bulk density and ultrasonic velocity measurements, a detailed petrographic study was performed to identify the thermal effect on the rock structure.

2. Description of rock samples

Five different carbonate rocks (two marbles and three limestones) were selected for testing in the laboratory to identify their physical features in natural and heat treated conditions. The names, types and origin of these rocks are given in Table 1. These rocks were chosen because they have different textural and structural characteristics although they have almost similar mineralogical composition (Table 1). A total of 75 cubic samples, 15 from each rock, were prepared from large blocks by sawing in 70 mm edge dimension. Attention was paid to prepare samples which were free from visible defects and flaws. The end faces of samples were parallel to each other and smoothened with no. 120 abrasive.

Table 1Name, type, class and petrographical description of studied rocks.

Rock name	Rock type	Rock class	Petrographical description
Finike Lymra (FL)	Limestone	Sedimentary	Intramicritic limestone in carbonate rock classification. Heterogeneous in mineralogical composition. Composed of mainly intraclasts (see "Int" in Fig. 6) cemented with micritic calcite. Size of intraclasts changes usually between 0.1 and 0.3 mm, rarely reaches to 1 mm. There are some clay matrix and scarce organic material. Homogeneous in structure and texture with common pores (see "c" in Fig. 6)
Denizli Travertine (DT)	Travertine	Sedimentary	Porous limestone (Travertine) in carbonate rock classification. Heterogeneous structure, contain a lot of pores (c) in irregular sizes and shapes. Homogeneous in mineral composition but heterogeneous in texture. Composed of 60% micritic calcites (Mc) and 40% sparitic calcite (Sp in Fig. 7) crystals settled on the surfaces of pores (Fig. 7)
Burdur Beige (BB)	Limestone	Sedimentary	Crystallised limestone in carbonate rock classification. Homogeneous in mineralogical composition with mainly sparcalcites (Sp) (> 0.004 mm in grain size), but heterogeneous in structure, contain too many micro-cracks (Cr in Fig. 8)
Afyon White (AW)	Marble	Metamorphic	Homogeneous in composition, texture and structure. Mineralogical composition is almost calcite (Ca in Fig. 9) with rare graphite and hematite. Crystal sizes range from about 0.1 to 1 mm. It has granoblastic texture (Fig. 9)
Mugla White (MW)	Marble	Metamorphic	Homogeneous in composition, texture and structure. Mineralogical composition is almost calcite (Ca) with rare graphite and hematite. Crystal sizes range from about 0.5 to 2 mm. It has granoblastic texture (Fig. 10)

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