Engineering Geology 200 (2016) 47–57

Contents lists available at ScienceDirect

Engineering Geology

journal homepage: www.elsevier.com/locate/enggeo

Ultrasonic and X-ray computed tomography characterization of progressive fracture damage in low-porous carbonate rocks

J. Martínez-Martínez [a,b,*], N. Fusi [c], J.J. Galiana-Merino [d], D. Benavente [a,b], G.B. Crosta [c]

[a] Dep. de Ciencias de la Tierra y del Medio Ambiente, Universidad de Alicante, Campus Sant Vicent del Raspeig, AP 99, 03080 Alicante, Spain
[b] Laboratorio de Petrología Aplicada, Unidad Asociada UA-CSIC, Spain
[c] Dip. di Scienze dell'Ambiente e del Territorio e di Scienze della Terra, Università degli Studi Milano-Bicocca Piazza della Scienza 4, 20126 Milano, Italy
[d] I.U. Física Aplicada a las Ciencias y las Tecnologías, Universidad de Alicante, Campus Sant Vicent del Raspeig, AP 99, 03080 Alicante, Spain

ARTICLE INFO

Article history:
Received 8 January 2015
Received in revised form 16 November 2015
Accepted 25 November 2015
Available online 3 December 2015

Keywords:
Damage evolution
Crack initiation stress
Crack propagation
Uniaxial compression test
Limestone
Marble

ABSTRACT

This paper studies the fracturing process in low-porous rocks during uniaxial compressive tests considering the original defects and the new mechanical cracks in the material. For this purpose, five different kinds of rocks have been chosen with carbonate mineralogy and low porosity (lower than 2%). The characterization of the fracture damage is carried out using three different techniques: ultrasounds, mercury porosimetry and X-ray computed tomography. The proposed methodology allows quantifying the evolution of the porous system as well as studying the location of new cracks in the rock samples.

Intercrystalline porosity (the smallest pores with pore radius < 1 μm) shows a limited development during loading, disappearing rapidly from the porosimetry curves and it is directly related to the initial plastic behaviour in the stress–strain patterns. However, the biggest pores (corresponding to the cracks) suffer a continuous enlargement until the unstable propagation of fractures. The measured crack initiation stress varies between 0.25 σ_p and 0.50 σ_p for marbles and between 0.50 σ_p and 0.85 σ_p for micrite limestone. The unstable propagation of cracks is assumed to occur very close to the peak strength. Crack propagation through the sample is completely independent of pre-existing defects (porous bands, stylolites, fractures and veins). The ultrasonic response in the time-domain is less sensitive to the fracture damage than the frequency-domain. P-wave velocity increases during loading test until the beginning of the unstable crack propagation. This increase is higher for marbles (between 15% and 30% from initial vp values) and lower for micrite limestones (between 5% and 10%). When the mechanical cracks propagate unstably, the velocity stops to increase and decreases only when rock damage is very high. Frequency analysis of the ultrasonic signals shows clear changes during the loading process. The spectrum of treated waveforms shows two main frequency peaks centred at low (~20 kHz) and high (~35 kHz) values. When new fractures appear and grow the amplitude of the high-frequency peak decreases, while that of the low-frequency peak increases. Besides, a slight frequency shift is observed towards higher frequencies.

1. Introduction

The origin of mechanical cracks (i.e. the location of the fractures and the moment when they appear) constitutes an important issue, widely studied at first in homogeneous materials and then in non-homogeneous materials, such as rocks. At the beginning of the last century, Griffith (1920, 1924) demonstrated that ruptures in glass rods are started in the flaws, and concretely in this case, in the notches. His conclusions have been applied to other materials, in particular to rocks, and since then it is commonly thought that fractures are developed from material flaws. Moreover, several authors prove that the initiation and propagation of cracks under stress are highly dependent upon the mineralogical and textural characteristics of rocks (i.e. content of soft mineral grains, inclusions, grain boundaries, cleavage plans, cavities and preexisting micro-cracks) (Germanovich et al., 1994; Dyskin et al., 1995, 2003; Zabler et al., 2008; Rigopoulos et al., 2011).

However, it is a hard task to know when, how and where cracks appear and develop in rocks under compression. One of the main problems is the fact that the opacity of most of the solid materials makes impossible to observe directly the cracking process. Several authors found solutions to this problem by means of different ways: 1) studying transparent materials, such us glass, resin and ice (Hoek and Bieniawski, 1965; Bieniawski, 1967; Horii and Nemat-Nasser, 1985; Cannon et al., 1990; Germanovich et al., 1994; Dyskin et al., 1995; Schulson et al., 1999; Dyskin et al., 2003). 2) Measuring changes in petrophysical properties of rocks such us volumetric strain, acoustic emission, ultrasonic wave propagation velocity, etc., during compression test and correlate them with the progressive fracture damage (Xue et al., 2014).

* Corresponding author at: Dep. de Ciencias de la Tierra y del Medio Ambiente, Universidad de Alicante, Campus Sant Vicent del Raspeig, AP 99, 03080 Alicante, Spain.
E-mail address: javier.martinez@ua.es (J. Martínez-Martínez).

http://dx.doi.org/10.1016/j.enggeo.2015.11.009
0013-7952/

3) Using specific techniques that can observe the internal rock texture and structure, such us computed tomography (CT) (Klobes et al., 1997; Zang et al., 1998; Keller, 1998; Ohtani et al., 2001; Vervoort et al., 2003; Hirono et al., 2003; Ketcham, 2005; Crosta et al., 2010; Fusi and Martínez-Martínez, 2013).

Computed tomography seems to be the best technique for studying the progressive fracture damage because of the possibility for observing the inner part of rocks. However, the problem is focused on the methodology for the continuous rock scanning while the rock is loaded. For soft rocks, Viggiani et al. (2004) developed a manual triaxial apparatus that could be placed in the X-ray beam. In this way the specimen can be scanned under loading without any position changes, with a maximum loading of 7.5 kN. Zabler et al. (2008) tested stiffer rocks, with a maximum σ_c of 50–100 MPa, loading the sample step by step and scanning it after each loading step. Unfortunately, this method presents the problem of repositioning the specimen in the CT scanner in the same position after each loading steps.

The aim of this paper is to study the progressive fracture damage of low porous and hard rocks during uniaxial compression tests. Three different kinds of marbles and two micrite limestones were selected, all of them are quarried and commercialized as ornamental and building stones. Characterizing the cracking process in this kind of rocks is difficult because of the reasons exposed above. As a consequence, a new methodology was employed that includes: uniaxial compressive test, ultrasounds, mercury porosimetry and X-ray micro-CT.

2. Materials

Five commercial varieties of Spanish carbonate rocks were selected, taking into account their low porosity and relatively high uniaxial compressive strength. The criteria of rock selection were: a) carbonate rocks (calcite or dolomite as main minerals); b) low porosity (mostly <2%); and c) each rock must present a different crystal size and a particular structural complexity (Fig. 1). Acronyms were established according to lithotype (Mb = marble; L = limestone), mineralogy (c = calcite; d = dolomite) and/or fabric aspects (m = micrite). In order to avoid confusion between similar rocks (there are two cMb and two mL) a suffix was added in reference to their colour in polished–finished conditions (G = grey; W = white; Y = yellow; R = red and O = orange).

The crystal size of these rocks was measured by image analysis using a petrographic optical microscope and scanning electronic microscope. Connected porosity was performed according to the European Standard (UNE-EN, 1936).

2.1. cMb-W

This rock corresponds to a white homogeneous calcite marble with low development of metamorphic foliation. Although the main constituent of this marble is calcite (99%), other accessory minerals can be found, such as pyrite, chalcopyrite, apatite, dolomite and micas. cMb-W presents a fine-grained homeoblastic texture consisting of a mosaic of equidimensional calcite grains with an average size of the order of 0.15–0.45 mm, but punctually some bigger crystal can be found (up to 1.5 mm). This rock presents an open porosity of around 0.4%, and the type of pores are predominantly intercrystalline.

2.2. cMb-G

cMb-G is a grey banded calcite marble. The main mineral constituent is calcite (99%) and pyrite, micas, quartz and ilmenite are observed as accessory minerals. Texture is homeoblastic with xenoblastic crystals which define a shape preferred orientation. Average crystal size is around 0.6 mm, but some crystals up to 2 mm can be found. Porous content of this rock is lower than 1%, and, as in the previously described marble, the type of pore is mainly intercrystalline.

2.3. dMb-Y

This rock is a yellow dolomite marble (dolomite content >90%) with abundant fissures partially filled with both calcite and iron/manganese oxides/hydroxides. Other accessory minerals found in this rock are micas, apatite and ilmenite. Texture of this rock is similar to the other studied marbles (homeoblastic texture) and in this case, crystal size ranges between 30 to 110 μm. Crystals are xenomorphic and present a shape preferred orientation. The average value of porosity in these rocks varies around 2%, and the porous system is constituted by both intercrystalline pores and fractures.

2.4. mL-R

A red micritic limestone without allochems. This rock presents a high number of white calcite veins, fractures and stylolites. Open porosity of this kind of rocks range from 0.7 to 2%, and it is mainly associated to open fractures and stylolites.

2.5. mL-O

mL-O is an orange-cream micritic limestone (99% calcite) with abundant stylolites. Texture of this rock is very similar to the previous one (mL-O) being micrite the main component with the absence of allochems. Open porosity varies between 0.4 and 1.4%.

3. Methodology and techniques

Uniaxial compressive test has been carried out in order to induce sample fracturing on at least three cores, 20 mm in diameter and 30 mm in length, for each type of rock. Samples from banded marbles have been obtained perpendicular to its structure.

Two kinds of compression tests have been carried out at a constant axial displacement rate (0.08 mm/min): 1) simple UCS test; and 2) multi-stage UCS test. The aim of simple UCS test is the definition of the stress–strain behaviour of rocks as well as their ultrasonic response under uniaxial loading. On the other hand, the multi-stage UCS test is focused on checking the evolution of rock texture during uniaxial compression and to know when and where microcracking and complete failure occur.

In the simple test, the specimen is continuously loaded in uniaxial compression until rock failure occurs. Ultrasonic transducers and rock strain measurement sensors are attached to the sample in order to monitor the loading and unloading behaviour.

In the multi-stage test, the samples are loaded to different percentages of the maximum strength, obtained from the previous simple test. When load reaches the pre-established value, unloading is carried out and the sample is removed from the loading frame. Then, the sample is scanned in the micro-CT system checking for internal changes. Core position inside micro-CT apparatus is accurately checked in order to maintain the same orientation and numbering of micro-CT slices throughout the cores after different loading cycles. Once the rock has been inspected, the core is put again in the loading frame and load is increased to the subsequent fixed loading step. Stress–strain behaviour has been recorded in these tests during every stage. In order to obtain a precise analysis of the crack propagation process, samples are scanned by micro-CT after Hg impregnation in a porosimeter. Combination of both tests (micro-CT and Hg porosimetry) guarantees a better characterization of small defects inside low-porous rocks than employing solely micro-CT (Fusi and Martínez-Martínez, 2013). Due to the reduced dimensions of sample holder in Hg-porosimeter, cores have been cut with a non standard height-to-diameter (h/d) ratio of 1.5. In this way, one cycle in the step-by-step test is composed by: 1) Hg impregnation of the sample with mercury porosimeter, 2) scanning with micro-CT system, and 3) uniaxial compression load. Details of techniques and procedures are given in the following section.