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Technical Note

Experimental Studies on the Mechanical Behaviour of two Thermal Cracked Marbles

By

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1. Introduction

The specific aim of this paper is to find an experimental methodology which could measure crack density in rock and its influence on the behaviour of the rock material. The results of both experimental and numerical simulations showed that rock behaviour is mainly ruled by crack density within a specific rock volume. The behaviour of a rock with different crack densities has been studied by several authors, from the theoretical point of view and through experiments.

Experimental works (Sage, 1988; Rotonda, 1991; Franzini, 1995; Homard-Etienne and Houpert, 1989) have confirmed that a large, damaged area in a rock material induced by heating determines variations of rock properties that are closely connected to the degree of fracturing of the material. It has been shown that the overall moduli of elasticity and tensile strength are influenced by the presence of cracks (Yin and Ehrlacher, 1996; Wong et al., 1996; Laws and Brockenbrough, 1982; Bertagnini at al., 1993).

2. Experimental Studies

Specimens of two different types of marbles have been heated at different temperatures to induce micro-cracking.

The rock materials used in the testing campaign are two Italian ornamental stones:

- Ormea Black Marble, exploited in Piedmont. Its mineralogical composition is almost pure calcite (the medium crystal dimension is 0.6 mm) with a small percentage of oxides. In the following sections it is quoted as black marble.
- Perlato Sicilia, a whitish-pink limestone exploited in Sicily, composed of micrite and biogenic fragments. Its mineralogical composition is almost pure calcite

with a small percentage of dolomite. In the following sections it is denoted as white marble.

Micro-cracking was induced by slowly heating 3 lots of stone, each made up of five specimens, at the rate of 2.4 °C/min; the maximum temperature was maintained for one hour and for the subsequent cooling at an inverse gradient of 0.23 °C/min. The two materials were subjected to different maximum temperatures (400 °C, 470 °C, 600 °C for white marble and 230 °C, 400 °C, 500 °C for black marble) as they showed a different reaction to heating: the black marble was more sensitive to heating.

The physical and mechanical characteristics of the two rocks have been determined according to the Standards of the European Committee CEN/TC 246 Natural Stones and of the American Society for Testing and Materials (ASTM).

The degree and nature of fracturing were directly quantified through microscopic observation, while micro-hardness tests were performed to evaluate punctual rock hardness.

2.1 Strength and Deformability of the Cracked Rocks

The two marbles were tested in the laboratory to identify their mechanical features in natural and treated conditions.

For each lot of specimens non-destructive tests, such as open porosity (EN 1936) and ultrasonic wave velocity (both under dry and saturated conditions), were performed on the same cubic about 70 mm edge specimens, which were afterwards used for uniaxial compression tests (EN 1926). Young modulus was also determined during this test but, due to the geometry of the specimens, the obtained values should be considered only for comparative purposes (that is, to show the difference in Young modulus induced by heating). The permanent linear expansion was determined by measuring all the specimen edges before and after heating, with a precision of 0.01 mm.

Flexural tests were performed on prismatic specimens to determine the indirect tensile strength. To investigate the dynamic characteristics of the rocks and the changes induced by the thermal treatment, the ultrasonic wave velocity was determined both in dry and in saturated conditions by measuring the travel time of a pulse along the axis of unstressed specimens. Tables 1 and 2 show the main characteristics of the two marbles under natural conditions and after heating.

Figures 1a and b show the stress-strain curves obtained for specimens with different crack densities.

2.2 Microscopic Observations

A polarising optical microscope with a magnification range of 100–500 (as suggested by Nolen-Hoeksema and Gordon, 1987) was used. Two polished sections of each marble were thermally treated for each heating temperature, together with the cubic specimens utilised for the mechanical tests. Knoop hardness tests were

Table 1. Experimental data collected for white marble behaviour upon heating and cooling

Properties \ heating	None	400 °C	470 °C	600 °C
Apparent density (kg/m^3)	2693	2655	2650	2604
Compressive strength (MPa)	93	117	119	95
Tangent Young modulus E tan (MPa*10^4)	3.2	2.2	2.0	1.1
Secant Young modulus E sec (MPa*10^4)	2.1	1.3	1.3	0.8
Flexural strength (MPa)	18.7	12.4		7.3
Ultimate tensile strength (MPa)		9.600		5.790
P wave dry probes (m/s)	6498	4066	3659	
P wave wet probes (m/s)	6542	4229	4054	
Open porosity (% of volume)	0.11	1.43	1.75	2.51
Permanent linear expansion (%)		0.46	0.54	1.09
Knoop Micro-hardness HK 50 (MPa)	1830	1410	1200	1240
Microcracks count (mm^{-2})	< 5	140		232.5
Microcracks length distribution (μm)				
L25		17		13
L50		38		26
L75		82		48
Crack density (mm^{-1})		3.7		4.6

Table 2. Experimental data collected for black marble behaviour upon heating and cooling

Properties \ heating	None	230 °C	400 °C	500 °C	600 °C
Apparent density (kg/m^3)	2690	2674	2669	2663	
Compressive strength (MPa)	95	105	97	89	
Tangent Young modulus E tan (MPa*10^4)	3.0	2.3	1.9	1.2	
Secant Young modulus E sec (MPa*10^4)	2.1	1.6	1.4	1.0	
Flexural strength (MPa)	28.1		16.3		11.3
Ultimate tensile strength (MPa)	21.3		12		9.1
P wave dry probes (m/s)	6275	4693	5012	2800	
P wave wet probes (m/s)	6302	5701	5567	4911	
Open porosity (% of volume)	0.11	0.73	0.97	2.40	
Permanent linear expansion (%)		0.15	0.25	0.35	
Knoop Micro-hardness HK 50 (MPa)	1702	1150		800	
Microcracks count (mm^{-2})	< 5	97.5	235	315	
Microcracks length distribution (μm)					
L25		21	18	8	
L50		38	34	22	
L75		76	65	48	
Crack density (mm^{-1})		2.3	4.7	6.9	

performed on one of the polished sections. A grid with 1 millimetre spacing was engraved on a 7 mm × 7 mm square area on one other section. After heating, the permanent deformation of the grid was measured at the microscope. Figure 2 shows the deformed grid for the white marble. The number of cracks along the scanlines was also counted and their lengths measured. The crack length distribution is reported in Fig. 3, where F is the crack length relative frequency in percent.

It was therefore possible to calculate the crack density D, as defined by Homad-Etienne and Houpert (1989), as the ratio of cumulated crack lengths per unit area. It was expressed in millimetres per square millimetre.

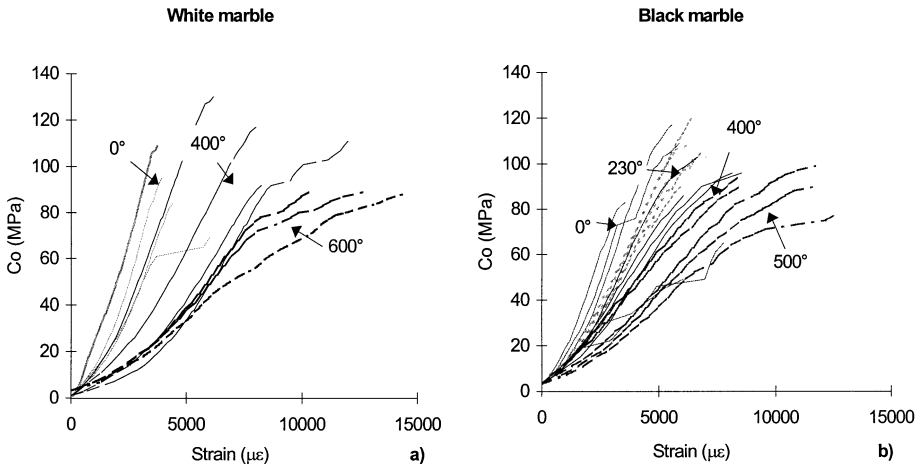

Fig. 1. Stress – strain curves obtained for white (**a**) and black (**b**) marble treated at different temperatures

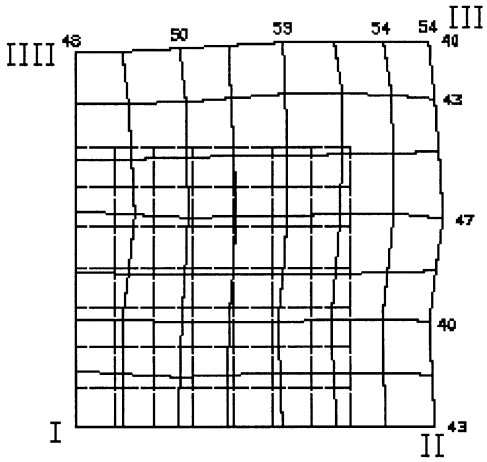

Fig. 2. Interrupted line: grid with 1000 micro meter spacing engraved on a polished section of the white marble (on 7 mm × 7 mm square area). Continuous line: deformed grid upon heating at 600 °C. Expansion is exaggerated and sides I–IIII and I–II supposed to remain straight. Numbers refer to the overall expansion in μm of the base lines, whose initial length was 7000 μm

2.3 Experimental Results

It was observed that compressive strength is not affected by micro-cracking after only one cycle of heating, whilst Mahmutoglu (1998) showed that compressive strength strongly decreases with cyclic heating at higher temperatures.

Strong decrease in Young's modulus was observed in both marbles: 50% of the original value for white marble, and 35% of the original value for black marble.

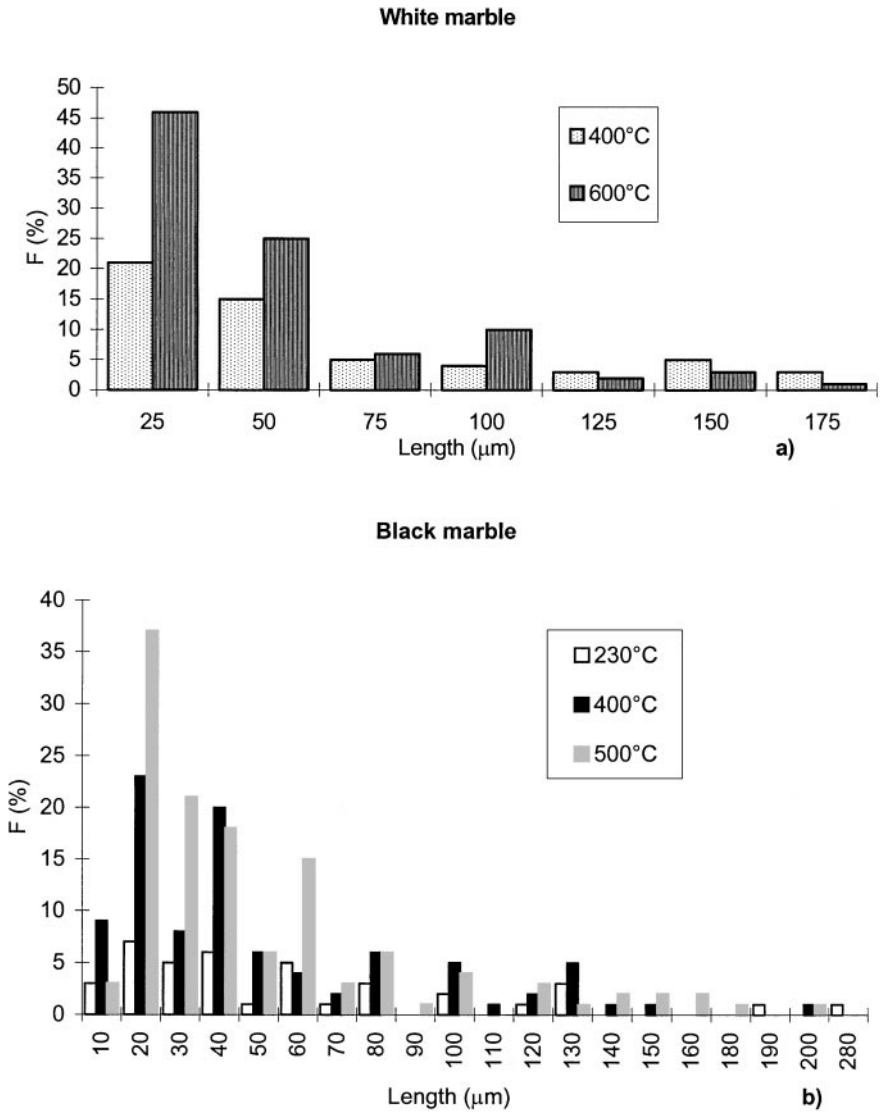

Fig. 3. Crack length distributions measured on polished sections of white (**a**) and black (**b**) marble treated at various temperatures

Both the flexural and the tensile strengths are greatly affected by heating. An increase in open porosity is evident in both marbles. The connection between the increase in porosity and new crack formation is determined in the next section. Experiments show that wave velocity in dry specimens is very sensitive to micro-cracking. The wave speed measured on dry probes markedly decreases with heating and, consequently, with increasing crack density. The wave speed of saturated probes shows the same trend even if it is less affected.

The experiments have shown a permanent linear expansion due to heating. Tables 1 and 2 report the average values obtained on the specimens, since they did not show anisotropy at the specimen scale. At a microscopic scale, instead, the deformations (Fig. 2) are strongly influenced by anisotropic behaviour of crystal.

Tables 1 and 2 also report the crack length computed at the 20%, 50% and 75% (L25, L50, L75) cumulative frequencies respectively and the measured crack densities. These values show that the average and the maximum crack lengths are not much affected by the heating temperature, whilst the crack densities obtained with heating at various temperatures always show an increase with an increase in the temperature of heating. The micro hardness values on the polished sections treated at different temperatures show an evident decrease of hardness due to thermal treatment.

3. Double Effects of Heating on Marble

The experiments have shown how heating determines an increase in porosity due to two effects: the matrix porosity increase, due to rock degradation, and the increase in porosity, due to induced fractures. Porosity variation can be a good indicator in the estimation of crack density to predict the mechanical behaviour of cracked rocks. To experimentally measure these two components one should proceed as follows:

1. Measure the apparent volume of each specimen (V_1)
2. Measure the mass of each dry specimen (M_1)
3. Compute the apparent density ($\gamma_a = M_1/V_1$) (1)
4. Experimentally determine the real density γ_v of the rock using the pycnometer method
5. Compute the total porosity as: $n = 1 - \dfrac{\gamma_a}{\gamma_v}$ (2)
6. Repeat points 1–5 for the heated specimen and determine the total porosity of the heated rock (n_h).
7. The difference between the total porosity of the natural and heated specimens ($n - n_h$) gives the increase in porosity that is due to heating.

As far as marbles are concerned, the difference between the apparent and the real density is in fact too small to be measured with sufficient precision. Moreover, the determination of the real density is a destructive test and consequently cannot be performed on the same specimens under natural conditions and after heating.

Consequently, the specimen volume and mass variation due to heating were measured according to these three hypotheses:

1. The apparent and the real densities are equal and known with sufficient precision. The real density does not show significant changes after heating. This hypothesis appears realistic since the rocks are basically made up of pure calcium carbonate with small percentages of magnesium, and the heating temperatures are much lower than the carbonate dissociation temperature (900 °C).

Moreover, the calcimetric analysis performed on the marble on both natural and heated specimens did not show any variation.
2. Permanent linear deformations can be measured with sufficient precision.
3. Mass variation is measurable and very small.

The mass variation is possibly due to the evaporation of water contained within the specimens, although they were dried at 90 °C to constant mass.

Finally, both the linear deformation and mass variation are small in comparison to the size and mass of the specimens.

With these hypothesis one can define:

$$\varepsilon = \frac{\Delta l}{l}, \tag{3}$$

where Δl is the side elongation.

$$\Delta M = \frac{M_i - M_f}{M_f}, \tag{4}$$

where M_i is the mass of the specimen in the natural state and M_f is the mass of the heated specimen.

The void volume increase is therefore due to the sum of two terms:

1. The pure volume increase of the specimen which is equal to 3ε
2. The volume corresponding to the mass decrease which is due to water evaporation $\Delta M/\gamma_w$

Consequently, the total porosity increase expressed as a percentage (A) is:

$$A = (3^*\varepsilon + \Delta M/\gamma_w)^*100 \tag{5}$$

The open porosity was measured on each specimen both under natural conditions (p_{oi}) and after heating (p_{oh}), according to EN 1936.

The increase in open porosity ($B = p_{oh} - p_{oi}$) can be compared to the increase in total porosity (A). The difference between the total porosity increase and the open porosity increase ($A - B$) indicates the amount of non-connected voids. B can be explained by the formation of new fractures or by the extension of old ones, ($A - B$) represents a mass deterioration at a smaller scale, which could be related to the decrease in rock hardness.

This procedure was applied to both rocks, and the results (a and b in Fig. 4) show their different behaviour. No measurable difference was detected between total and open porosity for the black marble even after heating and curves A and B coincide, while, for the white marble, the difference between total and open porosity after heating is greater and can be easily measured. These results are in accordance with the different grain sizes of the two rocks: in the black marble (composed of crystallized calcite) the volume increase is only due to new fracture formations, whilst in the white marble (composed of microcrystalline calcite), the rock deterioration at a small scale is evident and induces a greater increase in the rock volume (55%).

Figure 5 shows the open porosity due to new fractures versus crack density. One can observe that the same increase in open porosity indicates a different new

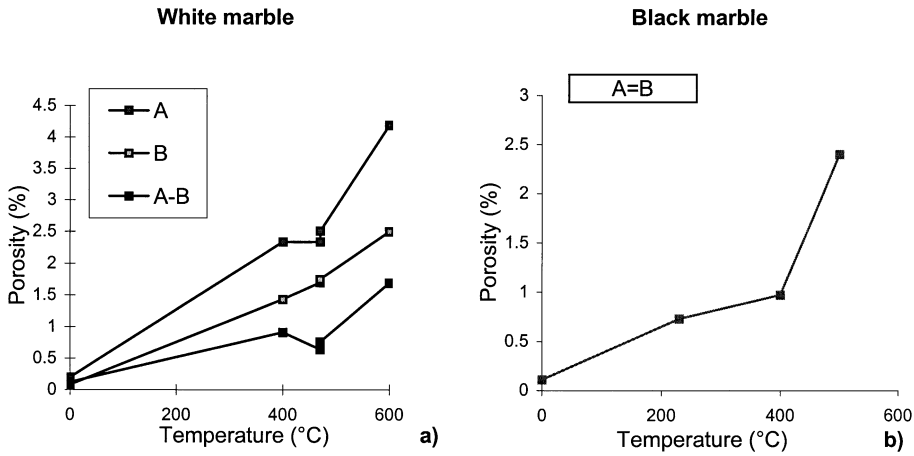

Fig. 4. Porosity increase with temperature in white (**a**) and black (**b**) marble respectively: *A* total porosity, *B* open porosity due to the formation of new cracks, $A - B$ volume increase due to the deterioration at smaller scale. In black marble, *A* and *B* curves coincide

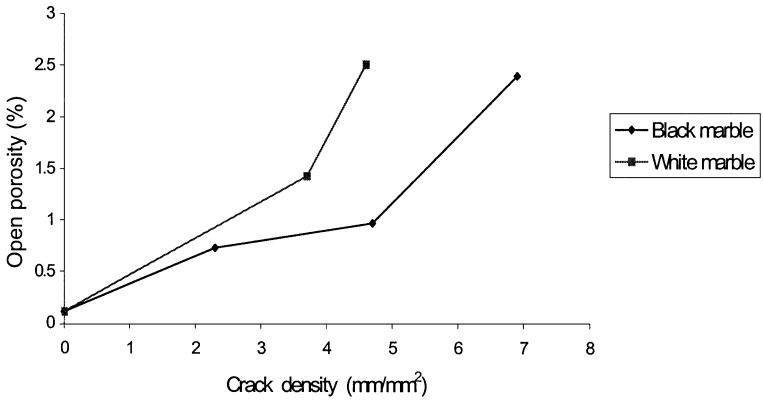

Fig. 5. Open porosity due to new fractures vs. number of cracks per square millimetres

crack density for the two marbles. This fact is probably due to a difference in the mean values of the fracture apertures for the two rocks. The black marble seems to be characterized by a lower mean value of crack aperture than the white marble.

4. Conclusions

The behaviour of two marble upon heating has been studied with the aim to identify the two main effects: the formation of new cracks and matrix deterioration. The formation of new cracks (and consequently an increase in crack density)

can be measured directly by means of microscopic analyses with measuring and counting of the cracks, or indirectly, by means of simple open porosity tests. A correlation between an increase in open porosity, due to new fractures, and crack density has in fact been found for the two marbles.

The relationship between open porosity crack density varies according to the rock types; consequently, direct reading at the microscopic scale should be performed to calibrate the relationship for each rock to be studied. Matrix deterioration can be measured as the difference between the total porosity increase and the open porosity increase ($A - B$). As the total porosity of marbles under natural conditions is very small and not easily measurable, the total porosity increase is indirectly estimated by the increase in apparent volume due to rock deformation. Heating had different effects on the two rocks: in the black marble (composed of crystallized calcite) the volume increase was only due to new fracture formations, whilst in the white marble (composed of microcrystalline calcite) the rock deterioration, at a small scale, is evident and induces the greater increase in the rock volume (55%). The effects of heating are conditioned by the mineralogic and textural features of the rock: in crystalline marbles (black marble) the formation of new cracks is much more important than matrix deterioration, while the reverse happens for micritic limestone (white marble).

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