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*Influence of temperature on the tensile strength of a limestone and a marble*

# Influence de la température sur la résistance à la traction d'un calcaire et d'un marbre

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**ABSTRACT:** This paper aims to analyse the influence of temperature on the tensile strength of a Moleano limestone and a Macael Marble. Temperature is an essential parameter to study the brittle failure of rocks and their fracture assessment in applications where the rock is at a higher temperature than room temperature (e.g. geothermal energy).

The research is based on the results obtained from a systematic laboratory campaign focused on determining tensile strength of the chosen rocks through two different standardized methods: splitting tensile (Brazilian) test and four-point bending test. Both methodologies allow the sought parameter to be obtained indirectly, by using disc-shaped and parallelepiped specimens, respectively. The experimental program comprises 174 specimens tested under different heating conditions. The considered temperatures vary from room temperature (approximately 23ºC) up to 250ºC, being this latter quite common in high enthalpy geothermal applications. Half of the specimens have been preheated to the target temperature and tested under constant temperature conditions. The other half, have been cooled to room temperature before testing after reaching the target temperature.

Thus, this work studies the influence of temperature on the tensile strength of the selected rocks, both at constant temperature conditions and after a heating-cooling cycle. Likewise, the deviations in the measured tensile strength depending on the experimental procedures followed are also analyzed. Finally, microstructural features of the rocks are considered, particularly their porosity, which shows a direct influence on the mechanical behavior of rocks subjected to high temperatures.

**RÉSUMÉ:** Cet article a pour but d'analyser l'influence de la température sur la résistance à la traction d'un calcaire de Moleano et d'un marbre de Macael. La température est un paramètre essentiel pour étudier la rupture fragile des roches et l' évaluation de leur fracture dans les applications où la roche est à température supérieure à celle-là de la température ambiante (par exemple, l'énergie géothermique).

La recherche est basée sur les résultats d'une campagne systématique en laboratoire visant à determiner la résistance à la traction des roches choisies par deux méthodes standardisées différentes: un essai de traction par fendage (brésilien) et un essai de flexion en quatre points. Les deux méthodes permettent d'obtenir indirectement le paramètre recherché, en utilisant respectivement des spécimens en forme de disque et spécimens parallélépipédiques. Le programme expérimental comprend 174 échantillons testés dans différentes conditions de chauffage. Les températures considérées varient de la température ambiante (environ 23°C) jusqu'à 250°C, ce qui est assez courante dans les applications géothermiques à haute enthalpie. La moitié des échantillons ont été préchauffés à la température cible et testés dans des conditions de température constante. L'autre moitié a été refroidie à la température ambiante avant d'être testée, après avoir atteint la température cible.

Ainsi, cette étude analyse l'influence de la température sur la résistance à la traction des roches sélectionnées, tant dans des conditions de température constantes que après un cycle de chauffage-refroidissement. De même, les écarts de la résistance à la traction mesurée sont analysés selon des procédures expérimentales suivies. Finalement, les caractéristiques microstructurales des roches sont prises en compte, en particulier leur porosité, qui a une influence directe sur le comportement mécanique des roches soumises à des températures élevées.

**Keywords:** Tensile strength; Temperature; Rock; Limestone; Marble

## 1 INTRODUCTION

The strength of rocks and their fracture behaviour is governed by many variables such as temperature, pressure, stress history of the rock and by the presence of joints or other defects like microcracks, pores, etc. An accurate replication in the laboratory of all this cumulative factors is difficult. For this reason, it is common to study their individual effects on the strength of rocks.

In particular, temperature has a direct influence on the fracture resistance of geomaterials like rocks (e.g. Heuze 1983, Zhao 2016), and therefore, its study is of interest for several underground engineering fields where the thermal effect should not be neglected, for instance, in underground coal gasification, oil-gas exploitations, nuclear waste disposal or in geothermal energy. For example, projects dealing with the Enhanced Geothermal System (EGS) technology have broadly been developed and spread since the 1970s (McClure and Horne 2014). In EGSs, hydraulic fracturing is used to improve well productivity and injectivity in conventional geothermal resources where massive rock blocks are found at high temperatures up to approximatelly 250ºC.

Likewise, it is widely known that rocks have relatively low tensile strength compared to compressive or shear strength, which makes the tensile loading mode the easiest failure mechanism. In fact, the tensile strentgh turns out to be a key parameter for different rock fracture assessment methodologies as for example the Theory of Critical Distances (TCD) or the Strain Energy Density (SED) criterion. In both of them the accuracy of the rock fracture predictions depends on the good characterization of the tensile strentgh (e.g. Cicero et al. 2014, Justo et al. 2017, Justo et al. 2018).

Thus, a comprehensive understanding of the variation of the tensile strength of rocks with temperature is necessary within several underground engineering fields. This work aims to study the influence of temperature on the tensile strength of two different isotropic rocks, namely a Moleano Limestone and a Macael Marble. The considered temperatures vary from room temperature (aprox. 23ºC) up to 250ºC, a common range of temperatures in geothermal applications.

Extensive research has been conducted to understand the effect of heat treatment on the tensile strength of rocks, which is mainly governed by the presence of pores, fissures and microcracks (e.g. Yin et al. 2015, Zhao et al. 2018). Two main trends are generally observed: rocks with low porosity and relative small presence of microcracks exhibit a gradual decrease in strength at the onset of heating, whereas rocks with high porosity and internal space for the inner expansion of the particles display an increase in strength up to a certain temperature followed by a decrease (Sirdesai et al. 2017). Homand-Etienne and Houpert (1989) and Dwivedi et al. (2008), for example, studied the thermomechanical behaviour of different igneous rocks like granites. They observed a persistent decrease in their tensile strength due to the low porosity and insignificant existence of microcracks within these rocks. The reduction of the tensile strength was caused by the creation of new microcracks as a response to uneven thermal dilation between adyacent grains with no space for expansion. By contrast, Rao et al. (2007) reported a case of a sandstone experimenting an increase in the tensile strength up to 250ºC, after which decreases. In this case, the grains can expand due to the preexisting pores and microcracks within the rock, which are filled at the onset of heating, leading to a compaction of the rock and, therefore, to an increment of the tensile strength. However, once the preexisting pores and microcracks are filled, the continuous expansion of the grains generate new fissures and reduce the tensile strength of the rock.

Different methodologies and testing methods are often used to obtain the tensile strength of the rocks: the uni-axial (Direct) test, the splitting tensile (Brazilian) test, the ring, the three- and four-point bending tests, etc. Many authors have tried to compare and critically evaluate these methodologies (e.g. Coviello et al. 2005, Langford and Perras 2014). However, there seems to be no general agreement within the scientific community on which test is best for its experimental determination. Among all the existing methodologies for the assessment of the tensile strength of rocks, the four-point bending tests and the Brazilian tests will be considered in this work.

With all this, this research aims to provide further information on the influence of temperature on the tensile strength of different types of rocks. Section 2 gathers a description of the analysed rocks. Then, Section 3 defines the followed experimental program and describes the two considered testing methodologies. Finally, Section 4 and 5 include the results and the conclusions of the research, respectively.

## 2 STUDIED MATERIALS

This work will focus on the results obtained for two different rocks with different characteristics and lithology: a Moleano limestone (C) and a Macael marble (M), from Portugal and Spain, respectively. Table 1 gathers some technical properties of the selected rocks, such as the bulk density, the open porosity and the mean grain size.

*Table 1. Some technical properties of the rocks*

| | **(C)** | **(M)** |
|---|---|---|
| Bulk density ($Kg/m^3$) | 2500 | 2715 |
| Open porosity (%) | 6.40 | - |
| Mean grain size ($\mu m$) | 100-300 | 100-200 |

### *2.1* Moleano limestone (C)

From a microstructural point of view, the Moleano limestone consists of several components: intraclasts, bioclasts, pellets and sparite crystals cementing the allochemical components. The average grain size is relatively uniform except for the sporadic presence of large intraclasts. Figure 1 includes a picture of some of the tested specimens and a microstructural image of the rock obtained with an optical microscope.

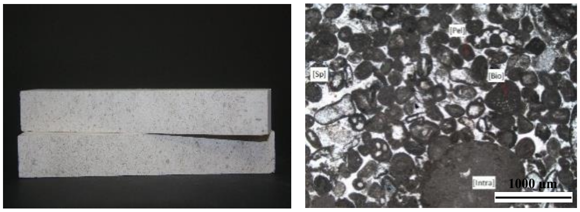


*Figure 1. Specimens and microstructure of the Moleano limestone (4x, crossed Nicols)*

### *2.2* Macael marble(M)

The Macael marble presents a well developed granoblastic texture with equidimensional idiomorphs leucocratic crystals (Figure 2). As shown in Table 1, there is no porosity within its internal microstructure.

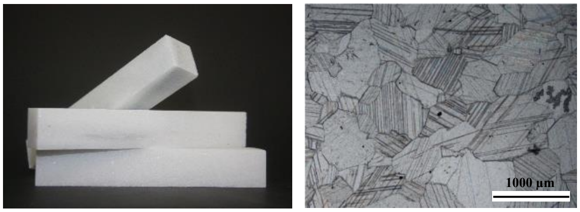


*Figure 2. Specimens and microstructure of the Macael marble (4x, crossed Nicols)*

Both rocks, together with others, are described more in detail by the authors at Justo et al. (2017).

## 3 EXPERIMENTAL PROGRAM

The tensile strength is defined as the rupture stress in a pure tensile uni-axial stress state. Therefore, from an strict point of view, the Direct tension test should be the most accurate option to determine the tensile strength of the rocks. The Direct test consists of a cylindrical specimen loaded till failure by a tensile axial force, so the stress state should be truly uni-axial and uniform by definition. However, this assumption is rather limited, since the specimen grips generate stress concentrations leading to early failure close to the grips. Besides, small geometrical imperfections and/or misalignments between the specimen and the loading frame introduce bending moments that generate a non-uniform stress state throughout the cross section (Coviello et al. 2005). For this reason and due to the tedious and expensive preparation of the specimens, the Direct test is not usually attractive and indirect tests are usually carried out in the laboratory.

This work will focus on two different indirect methodologies: the four-point bending test and the Brazilian test, which are described in detail down below.

### *3.1* Four-point bending tests

The four-point bending tests (4PBT) consists of simply supported parallelepiped specimens loaded perpendicularly to their axis in two points. This methodology is very popular in many civil engineering fields for determining the tensile strength of materials like rocks, natural or artificial building stones, cements, mortar, etc. due to their simplicity.

Many standards can be found dealing with the execution of these tests, as for example the EN 13161 and the ASTM C880. Basically, the tensile stress is assessed by means of continuum mechanics, applying the Navier's equation at the furthermost fiber from the neutral axis of failure (see Figure 3), where linear elastic behaviour of the material is assumed.

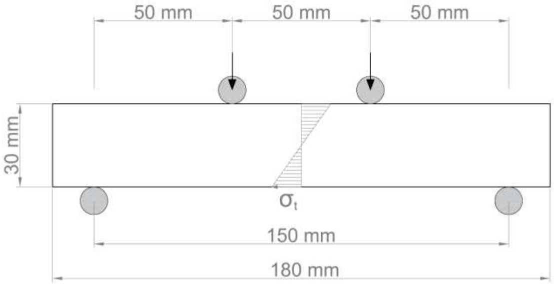


*Figure 3. Geometry of the 4PBT specimens*

In this work the European standards EN 13161 have been considered, using 180x30x30 mm size beams as shown in Figure 3. According to them, the tensile strength $\sigma_t$ (MPa) is defined by the following expression:

$$\sigma_t = \frac{F \cdot l}{b \cdot h^2} \qquad (1)$$

Where $F$ (N) is the failure load, $l$ (mm) is the span between the supporting rollers, and $b$ (mm) and $h$ (mm) are the depth anf the height of the specimen, respectively.

In total, 42 4PBT have been carried out considering 6 different temperatures varying from approximately 23ºC to 250ºC. Those tests conducted at a higher temperature than room temperature were performed inside an oven under constant temperature conditions, and the corresponding specimens were preheated to the target temperature during at least 24 hours before testing.

### *3.2* Splitting tensile (Brazilian) tests

The Brazilian test (BT) is also a widespread tension test among the scientific community due to the easy preparation of the specimens and the low dispersion of the experimental results (Coviello et al. 2005). It consists of disc-shape specimens diametrically compressed between flat or curved platens. The elastic stress fields induced by concentrated (flat platens) and distributed (curved platens) loads differ only in the outer region of the disc, where radial compressive normal stresses tend to infinite for the former case and are equal to the circunferential one (also compressive) in the latter (Hondros 1959).

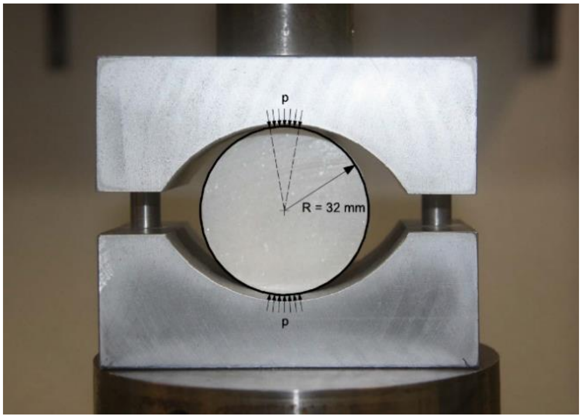


*Figure 4.Geometry of the BT specimens*

Here, curved platens will be used as shown in Figure 4. As reported by Colback (1966) and considering a modified Griffith's fracture theory that takes into account the friction in compressed cracks (McClintock and Walsh, 1962), the distributed contact forces of the curved platens will trigger an extension fracture anywhere in the central third of the disc. By constrast, in the case of the concentrated loads, the fracture would first occur at the disc-platen contact, which can affect the obatined results.

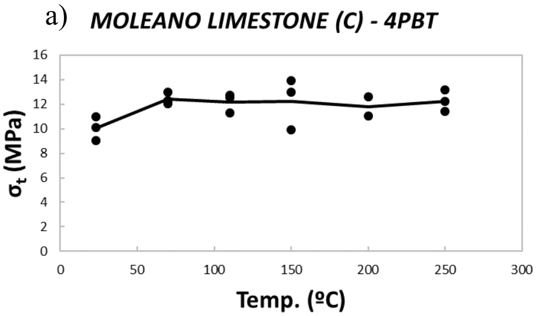
The followed experimental procedure is based on the Spanish standards UNE 22950-2, which are consistent with those suggested methods by the ISRM (1978). In this case, the formula for calculating the splitting tensile strength $\sigma_t$ (MPa) is as follows:

$$\sigma_\mathrm{t} = \frac{2 \cdot F}{\pi \cdot D \cdot t} \tag{2}$$

Where $F$ (N) is the failure load, $D$ (mm) is the diameter and $t$ (mm) is the depth of the disc. The depth of the tested specimens was equal to their radius ($t = D/2$). In this case, 6 tests have been performed for each of the two analysed rocks and six considered temperatures, which in total comprise 132 BT under different thermal conditions. Half of the specimens were preheated to the target temperature and tested under constant temperature conditions inside an oven, exactly the same as for the 4PBT. The other half, once subjected to the target temperature for at least 24 hours, were cooled to room temperature before testing.

## 4 RESULTS AND DISCUSSION

This section gathers the obtained experimental results for both the Moleano limestone (C) and the Macael marble (M). The results corresponding to each type of test will be analysed separately. First, Figure 5 shows the variation of the tensile strength ($\sigma_t$) of the rocks with temperature, based on the results of the 4PBT. Then, Figure 6 presents the same results for the case of the BT. In both figures dots correspond to the individual test results, while the solid lines represent the mean values. In the case of Figure 6 two different curves are depicted: the dots and solid lines in black correspond to the tests performed in the oven at the target temperature (hot tests), while the results in grey represent the tests that have undergone a heating-cooling cycle (cold tests) before testing.



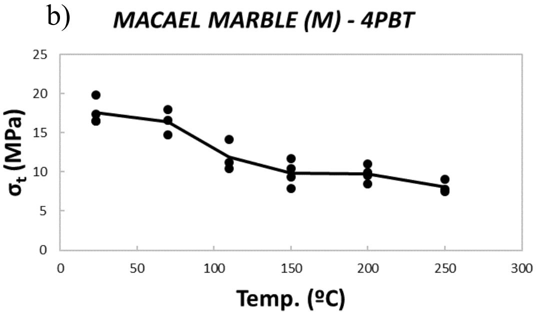
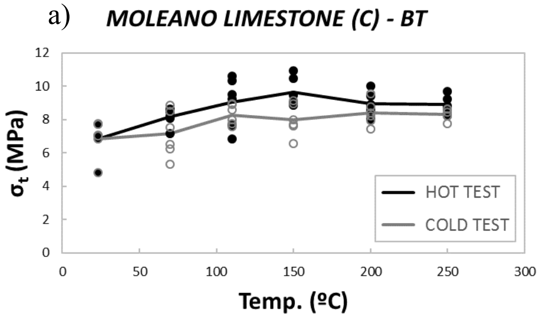


*Figure 5. Variation of the tensile strength with temperature of Moleano Limestone (a) and Macael Marble (b) using the 4PBT*

Two main trends are clearly distinguished at Figure 5. The Moleano limestone (Figure 5a) experiments an increase of the tensile strength at the beginning of the heating process, after which stays approximately constant up to 250ºC. By contrast, the Macael marble (Figure 5b) shows a gradual decrease of its strength all over the range of studied temperatures.

On the other hand, in case of analysing the results corresponding to the BT (Figure 6), the observed tendencies remain practically the same. In the limestone (Figure 6a) the initial increment of the tensile strength is better captured than in the case of the 4PBT. Performing additional 4PBT at high temperatures would probably help to better define this trend in Figure 5a. Conversely, the marble (Figure 6b) displays once again a continuous drop of the tensile strength with temperature.

With all this, two key micromechanisms can be identified: firstly, the Moleano limestone (C) has significant porosity and, therefore, sufficient space for internal expansion of the particles with temperature. For this reason, an increment of the tensile strength is developed due to the closure of the preexisting pores or microcracks up to a certain critical temperature, which is better appreciated in Figure 6a. However, the continuous thermal increment generates new microcracks, so from a certain point onwards the strength reduces. On the contrary, the non-porous marble directly experiments a decrease of the tensile strength because of the lack of internal space for the uneven dilation of grains.



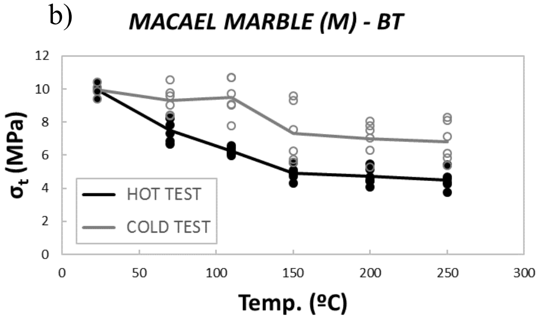


*Figure 6. Variation of the tensile strength with temperature of Moleano Limestone (a) and Macael Marble (b) using the BT*

Likewise, the observed tendencies in Figure 6a and 6b for those BT performed at room temperature conditions after a heat cycle (cold tests) are gentler than those corresponding to the hot tests, even though the increase and decrease patterns are maintained in both cases. These smoothing of the curves is due to a partial recovery of the inner microstructural conditions of the specimens after cooling.

One of the most important aspects to highlight is the significant difference of the tensile strength values obtained from both the 4PBT and the BT. As stated by Colback (1966), in the latter, the Griffith's criterion ensures that the computed tensile stress at failure roughly coincides with the uni-axial tensile strength obtained from the Direct tests. Similarly, Mellor and Hakes (1971) concluded that, although Direct tension tests cannot be fully substituted by indirect ones, the BT offers reasonable estimations of the tensile strength provided that compressive loads are distributed on the disc surface, as for example by curved platens (Figure 4). However, the 4PBT tends to overestimate the tensile strength as shown in Figure 5. One of the main reasons explaining these higher estimations is the so called stress gradient effect and the non-linear behaviour of the rock near bending failure. Large stress gradients imply a small volume of the material being subjected to tensile stress, which reduces the probability of including material flaws in comparisson to the Direct tests. Besides, from a strict point of view, the two loaded sections of the 4PBT should be spaced at least two heights (and the fracture should develop in that central part of the specimen) so that the Navier's equation can correctly describe the variation of the normal stress in elastic materials. Indeed, in the de Saint Venant solid, the Navier's equation does not apply for sections which are closer than a height to concentrated loads (Coviello et al. 2005), so the appliaction of Equation 1 is limited and the linear stress distribution (Figure 3) considered in the 4PBT will lead to overestimations of the tensile strength.

## 5 CONCLUSIONS

Several conclusions can be underlined from the interpretation of the obtained results. In the first place, the influence of temperature on the tensile strength of the Moleano Limestone (M) and the Macael marble (C) is considerable in the range of studied temperatures (up to 250ºC), which are quite common in geothermal applications. The reported results are consistent with those found in the bibliography (e.g., Sirdesai et al. 2017, Zhao et al. 2018) for other rocks with comparable properties. In this sense, those rocks with porosity and preexisting microcracks develop an increment of the tensile strength at the onset of heating due to their closure, and once a critical temperature is reached, thermally induced microcracks arise and the tensile strength decreases. By contrast, in the case of non-porous rocks, the grains have no space for uneven expansion and, therefore, a gradual decrease of the strength is observed from the beginning of the thermal treatment. Furthermore, when exposing the rocks to a heating-cooling cycle, the observed trends are soften as a consequence of a partial recovery afer cooling of the microstructural conditions.

Finally, the 4PBT leads to higher estimations of the tensile strength compared to the BT. These higher values are mainly ascribed to the stress gradient effect, as well as to the limitations of the Naviers's equation in the considered specimens. Conversely, as demonstrated by many authors (e.g. Colback 1966), the BT provides good estimations of the tensile strength compared to the Direct test.

## 6 ACKNOWLEDGEMENTS

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