Physical-mechanical properties of a granite used in the UNESCO World Heritage of the north of Portugal after high-temperature pretreatment

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ABSTRACT: This paper studies the evolution of physical and mechanical properties of 46 cubic samples of a granite widely used in the UNESCO World Heritage of the north of Portugal pre-treated at different temperatures (between 105 and 700 °C) and cooled under different conditions (i.e., air-cooled and water-cooled). The results showed that specific gravity and mineralogy did not exhibit significant changes after the thermal pre-treatment. In contrast, density, porosity, ultrasonic properties, uniaxial compressive strength and elastic modulus showed important variations, mainly at temperatures higher than 500 ºC. The SEM analyses clearly show an increase in the density of cracks, as well as in the width and length of the cracks from 500 ºC. Furthermore, it was observed an amplification of the damage in those samples cooled by water immersion. These results are of relevance for the evaluation of the integrity level of buildings built using this rock after a fire.

Keywords: thermal effect; physical properties; uniaxial compressive strength; elastic modulus; granite; cultural heritage.

1 INTRODUCTION

Fires are major threats for cultural heritage stone-built buildings. The high temperatures developed during a fire induce important changes in the properties of the stone used in the construction of the building (Chakrabarti, 1996; Gómez-Heras et al., 2010; Brotons et al., 2013; Ozguven and Ozcelik, 2014; Tomás et al., 2021). Consequently, it is of paramount importance to assess the impact of temperature on the physical and mechanical properties of the stone.

In this work, the variation of the mineralogical, petrographic, physical and mechanical properties of a medium-grained granite from the north of Portugal widely used for the construction of some cultural heritage buildings from the UNESCO World Heritage cities of Guimarães and Porto (UNESCO, 2018) are studied in detail. The results derived from this work will be of primary importance for the evaluation of the integrity level of granite-built UNESCO cultural heritage sites and other traditional constructions in the north of Portugal and Spain made of this granite after high temperatures caused by fires, and to define less damaging fire extinction methods.

2 METHODOLOGY

2.1 Preparation, heating and cooling of rock samples

The studied granites come from a quarry placed near the city of Vila Real (N Portugal). A total of 46 cubic samples of 10 cm side were used for the mechanical tests. It should be noted that rift, grain and hardway surfaces were identified in the quarry by means of a color code, and the tests were developed with riff plane parallel to the axial force direction.

Subsequently, the samples were subjected to a thermal pre-treatment on a programable furnace from 105 ºC up to the target temperatures of 300, 500, 600 and 700 ºC in one hour. Then, the target temperature was maintained constant for an hour and cooled in two different ways, at air (i.e., at laboratory temperature) and by immersion in tap water. Finally, the samples were stored in dry conditions until their testing.

2.2 Determination of mineralogical and petrographic properties

The composition of the studied granites at different temperatures after heating was determined by powder X-ray diffraction (XRD) and interpreted using the software package X Powder. Additionally, the characterization of the petrographic properties of the stone was carried out on thin sections, and the changes of the microstructure caused by heating and cooling (e.g., evolution of cracks length, opening and linear density) were studied by scanning electron microscopy (SEM).

A naked-eyed description of the color of the samples was also performed in order to detect potential variations related to internal mineralogical changes.

2.3 Determination of physical and mechanical properties

Total and open porosity were measured using weights, sample volume, dry unit weight and specific gravity according to the traditional techniques described in the standards (AENOR, 2007).

The measurement of ultrasonic P- and S- waves was performed along the three main direction of the samples according to the Spanish standards (AENOR, 2005), enabling the calculation of the dynamic elastic modulus (E_{dyn}) and Poisson's ratio (ν).

Finally, the static elastic modulus (E_{sta}) and the uniaxial compressive strength (UCS) were determined using a four-columns hydraulic press machine according to the suggested methods of the International Society for Rock Mechanics and Rock Engineering (ISRM, 1979). The longitudinal deformations were measured by means of two Linear Variable Differential Transformers (LVDT).

For more details about this methodology, we recommend reading Tomás et al. (2021).

3 RESULTS AND DISCUSSION

3.1 Evolution of the mineralogical and petrographic properties

The X-ray diffraction performed on samples after different temperature pre-treatments did not show any significant variation of the mineral composition of the studied granite.

In contrast, the rock specimens experimented important color changes (Figure 1) from light pink into reddish hues from 105 to 700 ºC, respectively. These changes are mainly related to the increase of the number of fissures and the variation of the oxidation state of iron contained in the rock (Benavente et al., 2003). It should be noted that these changes do not mean any mineralogical transformation, in agreement with the X-ray diffraction observations.

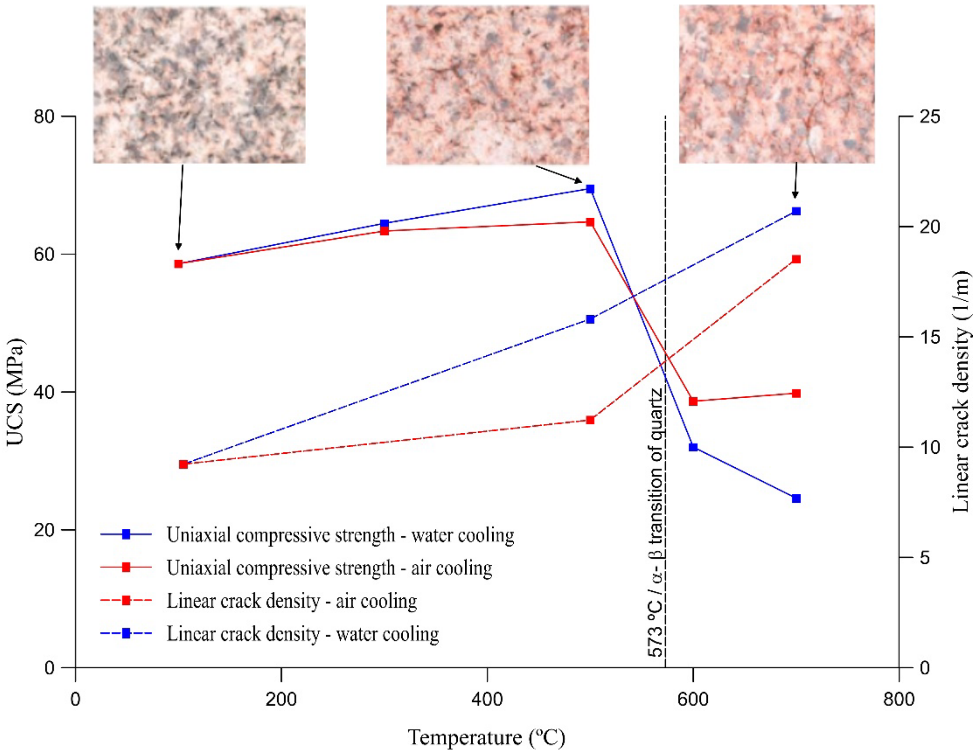

Figure 1. Evolution of the uniaxial compressive strength and the linear crack density after heating the samples at different target temperatures for air- and water-cooled samples. Color changes can be also recognized in the upper subfigures.

The linear density (i.e., the number of cracks per unit length) calculated from SEM images shows a gradual increase for both, air- and water-cooled samples (Figure 1). However, the density values are higher for those samples cooled by immersion on water due to the cooling shock.

The results also showed that the length of the cracks gradually increased, especially from 500 ºC. However, the crack width reduces up to 500 ºC and then, it considerably increases up to the doubled at 105 ºC.

As can be seen, the most important microstructural changes (i.e., increase of crack length, width and density) in the studied granite occurred from 500 ºC due to the induced stresses by thermal and cooling processes caused by differential anisotropic crystal expansion-shrinking, α-β transformation of quartz and so on (Vázquez et al., 2015; Fan et al., 2017; Hu et al., 2018).

3.2 Evolution of the physical and mechanical properties

Specific gravity experimented very small variations after thermal pre-treatment (Figure 2b). The observed variations (lower than 0.04) are probably related to the heterogeneity of the used portions of granite and the instrumental errors of the equipment confirming the mineralogical stability observed in the XRD. In contrast, dry unit weight is mostly constant for temperatures lower than 500 ºC. However, from this temperature, the dry density sharply falls (Figure 2b).

Total porosity slightly reduced from 0 to 300 ºC and increases from 300 to 500 ºC, considerably increasing from 500 ºC (Figure 2a).

P-wave velocity exhibits a gradual and continuous reduction with temperature (Figure 3b) with a clear fall between 500 and 600 ºC, and a stabilization from 600 to 700 ºC.

Uniaxial compressive strength (Figure 1) and static Young's modulus (E_{sta}) (Figure 3a) show similar trends. UCS exhibits a slight improvement up to 500 ºC followed by a sharp decrease from 500 to 600 ºC. Similarly, the static Young's modulus shows an increase between 105 and 300 ºC, a small fall between 300 and 500 ºC and a drastic decrease between 500 and 600 ºC.

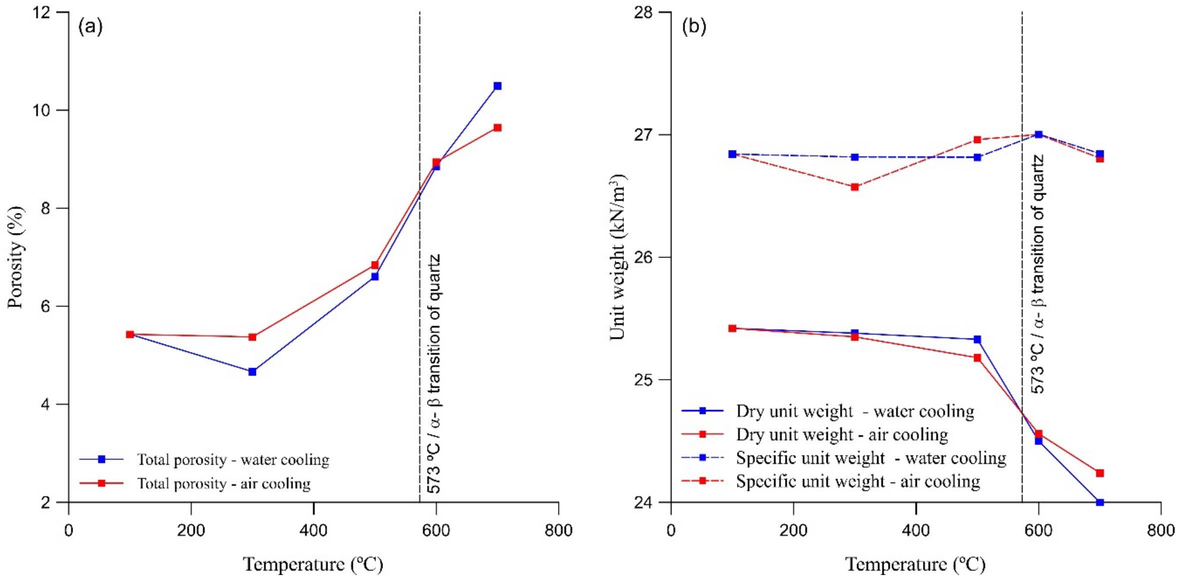

Figure 2. Evolution of the (a) total porosity; and the (b) specific and dry unit weights after thermal pre-treatment of the samples at different target temperatures.

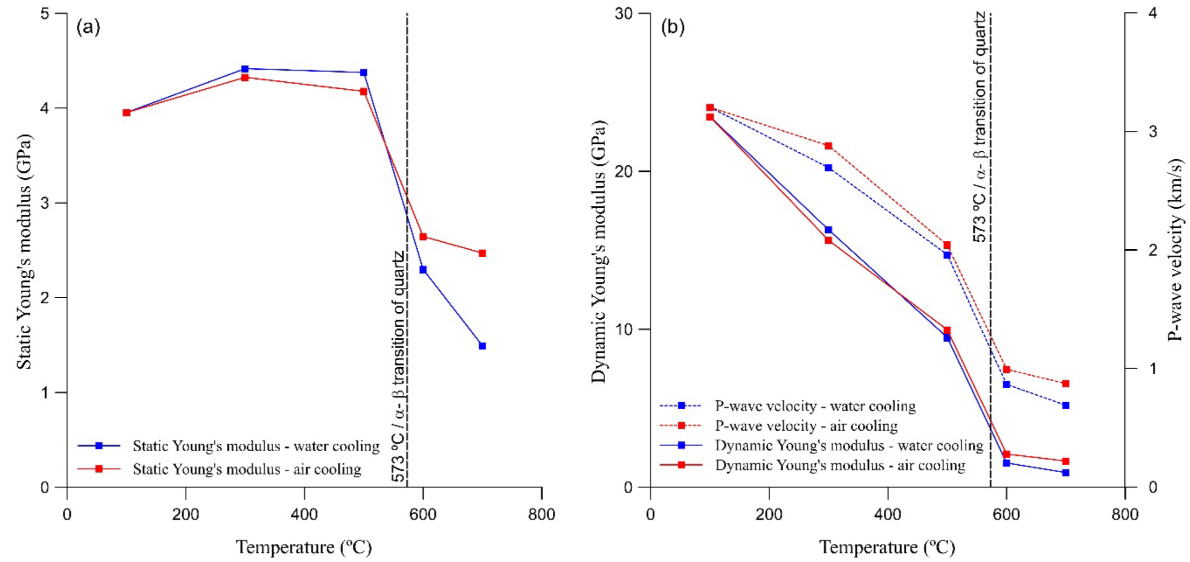

Figure 3. Evolution of the (a) static Young's modulus; and the (b) P-wave velocity and the dynamic Young's modulus after heating the samples at different target temperatures.

3.3 Discussion

Although mineralogy and specific unit weight show a clear stability at different temperatures, the rest of properties exhibit clear changes, mainly between 500 and 600 ºC when temperature increases. The observed changes are mainly caused by the enlargement of existing cracks and the generation of new ones due to different causes, which increase the level of damage of the stone samples (Vázquez et al., 2015; Fan et al., 2017; Hu et al., 2018).

UCS and E_{sta} and total porosity results suggest that microfissures play a doble role in the properties of the studied granite (Zhang et al., 2020; Rao and Murthy 2001). On the one hand, the increase of temperature causes thermal hardening. On the other hand, the increase of temperature degrades the rock samples as shown by P-wave velocity. The predominance of one of these two effects strongly influences the properties of the rock. For temperatures lower than 500 ºC thermal hardening predominates, closing microcracks, and subsequently increasing UCS, E_{sta} and reducing total porosity. However, after 500 ºC, the thermal degradation of the rock predominates, the length, aperture and density of microcracks increase and UCS and E_{sta} considerably decrease. The great expansion caused by α-β transformation of quartz is the main cause of the sharp changes observed between 500 and 600 ºC for most of the properties.

The cooling method also plays an important role in the properties of the studied granite. For temperatures lower than 500 ºC, water-cooled specimens exhibit a better mechanical behavior than air-cooled samples. However, for higher temperatures the high gradients induced by water immersion strongly damage the samples reducing their properties.

4 CONCLUSIONS

In this work, the physical-mechanical properties of a granite used in the UNESCO World Heritage of the north of Portugal after high-temperature pretreatment have been studied. To this aim, 46 cubic samples of 10 cm side have been heated at different temperatures and tested after cooling by immersion in water or at room temperature.

The results show that some properties as mineralogy and specific gravity did not significantly change at different temperatures, indicating that the mineralogical composition of the samples remained constant. The color of the samples experimented naked-eye changes due to iron oxidation and the increase of the density of microcracks.

Most of the studied parameters experimented thermal-induced changes, mainly between 500 and 600 ºC due to α-β transformation of quartz grains. Static Young's modulus and uniaxial compressive strength slightly increased for heating temperatures lower than 500 ºC and fell abruptly after this temperature. A closure of microcracks was also measured from the analysis of SEM images up to 500 ºC, followed by a sharply opening of the microcracks after this temperature. These observations suggest that the studied rock undergo a hardening up to 500 ºC, although after this temperature, the thermal damage, enhanced by the α-β transformation of quartz grains, predominates against hardening, considerably reducing the static Young's modulus and the uniaxial compressive strength.

The cooling method also play an important role on the thermal damage of this granite. For temperatures lower than 500 ºC water-cooled samples experience a hardening, while after 500 ºC, the rocks immersed in water considerably worsen their physical and mechanical properties.

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