PAPER • OPEN ACCESS

Using non-destructive testing to assess static elastic modulus of a limestone exposed to high temperatures

To cite this article: M E Garrido *et al* 2021 *IOP Conf. Ser.: Earth Environ. Sci.* **833** 012027

View the article online for updates and enhancements.

Using non-destructive testing to assess static elastic modulus of a limestone exposed to high temperatures

M E Garrido[1], V M-Ibáñez[2], C H Signes[1] and R Tomás[3]

[1] Department of Geological and Geotechnical Engineering. Universitat Politècnica de València. 4602 Valencia, Spain
[2] Department of Construction Engineering and Civil Engineering Projects. Universitat Politècnica de València. 46022 Valencia, Spain
[3] Department of Civil Engineering. Escuela Politécnica Superior de Alicante. Universidad de Alicante. P.O. Box 99, 03080 Alicante, Spain

egarrido@trr.upv.es

Abstract. The determination of static elastic modulus in the laboratory requires rock core extraction and the subsequent testing of the samples by means of standardised uniaxial compressive strength tests. However, this destructive procedure is not always suitable – as in the case of protected historic buildings. In these cases, the static elastic modulus can be obtained from the dynamic elastic modulus, in turn derived from the velocity of ultrasonic waves (a non-invasive and non-destructive test). The relationship between both the dynamic and static moduli of rocks has been extensively addressed in the scientific literature. Furthermore, several researchers have separately studied the evolution of static or dynamic elastic moduli of rocks exposed to high temperatures – although few studies have compared both values. It is well known that the dynamic modulus is generally higher than the static modulus, and the values diverge especially in rocks with a low modulus of elasticity. These differences can be mainly explained by the effect of porosity and the size of cracks in the determination of both parameters. In this research, the relationship between static and dynamic moduli for 'Borriol' limestone is studied for samples previously subjected to 200, 400, 600 and 800 ºC and then cooled slowly (in air) or quickly (immersed in water). The results show that the static modulus of samples heated up to 600 ºC decreased 80.9 and 79.1 % and dynamic modulus decreased 62.5 and 64.8 % for slow and quick cooling samples, respectively. For samples heated to 600 and 800 ºC, the static and dynamic moduli are similar. In general, no significant differences between both cooling methods are observed, even though static modulus shows more loss than dynamic modulus. Finally, linear models were used to correlate static and dynamic moduli, providing coefficients of determination of 0.99 and 0.97, for slow and quick cooling, respectively. It is also remarkable that the E_{dyn}/E_{st} rate was smaller than 1 for elastic moduli over 30 GPa (i.e., 105, 200 and 400 ºC) and greater than 1 for lower moduli (i.e., 600 and 800 ºC). The results obtained can be used to calculate the static elastic modulus of 'Borriol' limestone from dynamic modulus determined by non-destructive techniques.

1. Introduction

Strength and deformability are the two characteristics of rock most affected by fire. Deformability is assessed by the elastic parameters of the rock. The static modulus of elasticity (E_{st}) is the most frequently measured value because it simulates loading conditions similar to those of the material in service. This parameter is calculated from the load and deformation data recorded during uniaxial compressive strength tests (UCS). This is the most frequent method of assessing rock strength, although it requires many samples and is a destructive test. For protected historic monuments affected by fire, there are significant limitations to obtaining the samples necessary for these tests. Therefore, these tests are often replaced by non-destructive techniques – providing that reliable correlations can be established between the two. For the modulus of elasticity, the most developed non-destructive technique is based on ultrasonic propagation through the rock. These tests provide the dynamic modulus of elasticity (E_{dyn}).

The main processes affecting a rock exposed to high temperatures and reflected in its modulus of elasticity are: (1) thermal evaporation of water contained within the structure of the rock at temperatures of up to 400 °C results in increased porosity; and (2) the creation of micro-cracks as a consequence of the differing thermal expansions of minerals in the temperature range of 400 to 600 °C [1]. The deterioration of rock is related to the development of these micro-cracks and other mineralogical changes.
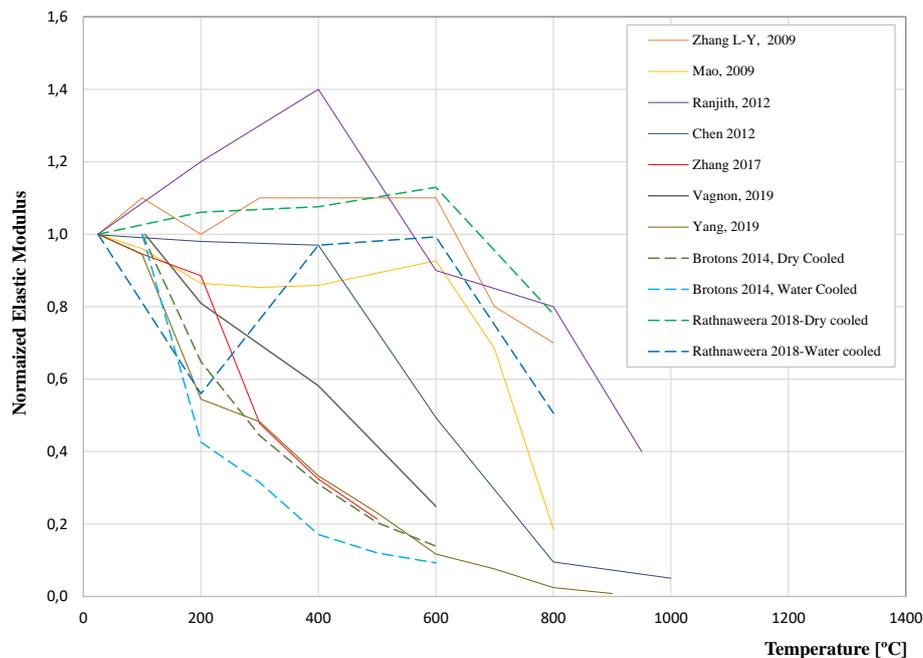
Figure 1. Relationship between the normalised elastic modulus and the target temperature for different types of rock.

There are many studies on changes in the modulus of elasticity in rocks exposed to high temperatures. The most studied rock types in relation to their exposure to high temperatures and their modulus of elasticity (figure 1) are granites, sandstones, marbles, and limestones. Mao et al. (2009) studied the variation in the modulus of elasticity in samples of a limestone from Xuzhou and observed that it decreased by 80 % at 800 °C. In this rock, it was determined that there is a critical temperature of approximately 600 °C and for lower temperatures the modulus of elasticity hardly changes; however, the modulus of elasticity decreases rapidly when this value is exceeded [2]. Zhang et al. (2017) tested a limestone from Shandong (Eastern China) and the decrease in the modulus of elasticity reached 89 % at 600 °C [3].

Similar results were published by Yang et al. (2019) on limestone rocks, also from Shandong, with a room temperature elastic modulus of 73.14 GPa. These authors showed that this modulus decreases significantly from 100 °C to 200 °C, followed by a slower reduction from 200 °C to 600 °C, and an even slower reduction for temperatures above 600 °C [4].

For the granites studied by Chen et al. (2012) at a temperature of 400 °C, the modulus of elasticity does not change significantly: but a sharp decrease is observed from this temperature upwards. At 1000 °C, the modulus of elasticity decreased by 90 % [5]. For sandstones, Zhang et al. [6] observed oscillations for temperatures below 600 ºC. Above that temperature, the value of the modulus of elasticity decreased by up to 35% at 800 ºC.

In the same type of rock Ranjith et al. [7] showed that the modulus of elasticity increases by 34 % at 400 °C relative to room temperature. For higher temperatures, there is a decrease of 74 % at 950 °C. Zhang et al. [6] also studied the variation of the modulus of elasticity of a marble and found that the modulus of elasticity decreases rapidly (with some fluctuations) up to 400 °C, and that the variation from this temperature is slower and finally shows a decrease up to 58 % at 800 °C. Vagnon et al. [8] examined an Italian marble and observed a decrease in the value of its modulus of elasticity up to 67 % at 600 ºC (compared to room temperature).

Brotons et al. [9] studied the variation of the elastic modulus in a calcarenite, being one of the few studies in which samples are subjected to two types of cooling: quick and slow. At 200 °C, the decreases in the modulus of elasticity are approximately 35% (air cooling) and 58% (water cooling) and more than 85% at 600 °C for both types of cooling.

Rathnaweera et al. [10] also used two cooling methods, quick and slow, in addition to testing the samples at the target temperature. The elastic modulus showed a similar variation in the quick-cooled samples and those tested at the target temperature. At 200 ºC, the modulus decreased 55% and 62%, respectively, with respect to the initial value. For higher temperatures and up to 600 °C, the moduli increased until it returned to the room temperature value again and then decreased again to 49 % and 54 %, respectively, at the maximum temperature of 1000 °C. However, the slowly cooled samples showed a different trend, the modulus of elasticity increased by about 12% of its initial value at 600 °C, and then decreased to 65% at 1000 °C.

Therefore, exposure to high temperatures decreases the elastic modulus in every type of rock. Estimating the relationship between the static moduli obtained by destructive methods and the dynamic moduli obtained by non-destructive methods is important. This relationship can be analysed in terms of the ratio between the two values according to equation (1):

$$k = \frac{E_{dyn}}{E_{st}} \tag{1}$$

This ratio can vary between 0.5 and 2.1 [11] depending on the degree of alterability and weathering of the rock. The general trend shows k values above unity, which tend to converge to unity from 120 GPa [12]. This is because rocks with high elastic moduli are very compact, have low porosity, and no cracks, and these characteristics can affect dynamic and static tests differently [12].

The relationship between static and dynamic modulus has been extensively studied for intact rocks, but few researchers have studied rocks exposed to high temperatures. Linear, potential, logarithmic, and even relationships involving other rock properties (such as density or wave attenuation) have been determined [11], [12].

The experimental part of this work was undertaken with samples of a Cretaceous limestone belonging to the Benasal Formation and known as 'Pedra de Borriol' (hereinafter BL) and with which historical monuments in Spain have been built since the 17th century. The static and dynamic moduli of elasticity were determined in standard samples and others previously subjected to 200, 400, 600 and 800 ºC – and then cooled slowly (in air) or quickly (immersed in water). The change in these parameters with temperature, the influence of the cooling method, and the relationship between static and dynamic moduli have been analysed to find a consistent relationship between the two – and so estimate the deformability of this rock when exposed to high temperatures by means of a non-destructive test.

2. Sample preparation and test

The samples tested were supplied as prismatic blocks (measuring 100×140×300 mm) from a quarry located south of the town of Borriol in the province of Castellon (Spain). Cylindrical samples with a diameter of 55 ± 1 mm and a height of 140 ± 1 mm (slenderness ratio of 2.5) were extracted from these blocks. The end sections were checked to ensure that they were flat and at right angles to the axis of the sample and without apparent irregularities. Five of these samples were tested under standard conditions and ten samples were tested for each of the target temperatures of 200, 400, 600 and 800 °C. The heat treatment consisted of a 5 °C/min gradient heating until the target temperature was reached and then maintained for one hour. The samples were cooled in two ways: five samples were cooled slowly (air-dried to room temperature) and the other five samples were cooled rapidly (immersion in water for 10 min, then oven-dried at 70±5 ºC to constant mass) [9]. All tests were carried out 24 h after the end of the heat treatment at an ambient temperature of approximately 22 ± 2ºC.

A 10-kW muffle furnace with a maximum temperature of 1300 °C was used for the heat treatment. The interior temperature was recorded and monitored using mineral-insulated K and N type thermocouples connected to a PicoLog TC08 data acquisition module.

Dynamic tests (non-destructive) and then uniaxial compressive strength tests (destructive) were performed to determine the dynamic and static modulus of elasticity values, respectively. Tests were performed in accordance with the methods recommended by the ISRM (1979). To determine the static moduli of deformation, two pairs of strain gauges were used. On pair was placed parallel to the specimen axis and measured longitudinal strain; and the second pair was placed orthogonally to the specimen axis and used to measure transversal strain. The static modulus of elasticity was obtained by the tangent method, recording the variation of the axial stress and the axial strain corresponding to 50 % of the final axial compressive strength [13]. The dynamic moduli of elasticity were determined using the analytical solutions commonly accepted in rock mechanics [14].

3. Results

Table 1 shows the average values of the basic properties of Pedra de Borriol. These values are used as a reference to normalise the results of exposure to high temperatures.

Table 1. Mean value basic properties of *Pedra de Borriol.* Average value ± standard deviation.

Rock	Bulk density (kg/m^3)	Total porosity (%)	UCS (MPa)	Elastic modulus (GPa)	
				Static	Dynamic
Pedra de Borriol (BL)	2664 ± 12	2.4 ± 0.4	176.8 ± 6.7	66.8 ± 5.3	50.8 ± 2.1

3.1. Static elastic modulus

Figure 2a shows the results of the static modulus of elasticity for each temperature. A slight decrease is observed at 200 ºC, greater for quick cooling (18.1 %) than for slow cooling (6.3 %).
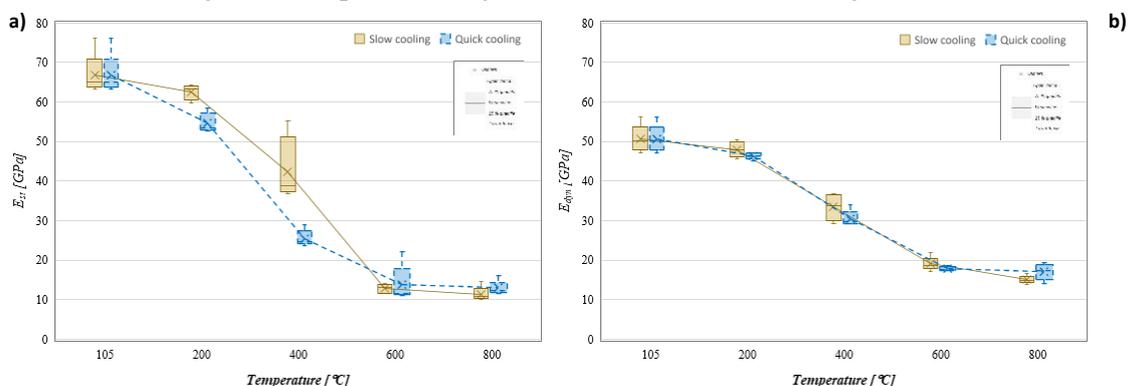
Figure 2. Relationship between a) static and b) dynamic elastic modulus and temperature.

A significant drop in this parameter is observed up to 600 ℃, 80.9 and 79.1 % for slow and quick cooling, respectively. Between 600 and 800 ℃ the decrease is almost negligible (i.e., less than 2 %).

The dispersion of the values is small except for the samples tested at 400 ℃ and air-cooled. At 200 and 400 ℃ the quickly cooled samples produce lower values than the slowly cooled samples – and this is reversed for the samples exposed to 600 and 800 ℃ (although in the latter two cases the differences between the two cooling methods are minimal).

Table 2 shows that the best fits for the relationship between static elastic modulus and temperature are exponential curves.

Table 2 Equations and determination coefficients for the best fitting static modulus and temperature.

Cooling	Fitting	Equation	R^2
Slow	Linear	$E_{st} = -0.09T + 77.1$	0.94
Quick		$E_{st} = -0.08T + 68.9$	0.88
Slow	Exponential	$E_{st} = 102.9e^{-0.003T}$	0.92
Quick		$E_{st} = 82.5e^{-0.003T}$	0.94

3.2. Dynamic elastic modulus

Figure 2b shows the results of the dynamic modulus of elasticity for each temperature. The variation in the data shows a similar trend to that of the static modulus. There is a slight decrease of less than 10 % between 100 and 200 ℃. Up to 600 ℃, a significant drop of 62.5 and 64.8 % is then observed for slow and fast cooling samples, respectively.

Between 600 and 800 ℃ the decrease is minimal, reaching 70.3 and 66.4 % for slow and quick cooling samples, respectively.

The dispersion of the values is minimal in all cases except for the samples tested at 400 ℃ and air-cooled, coinciding with observations for the static modulus. There is no appreciable difference between the samples cooled slowly or quickly, although the latter provides values in the lowest quartile (except for the samples exposed to 800 ℃ and rapidly cooled – some values exceed those of the same samples cooled slowly).

Table 3 shows that exponential curves are the best fits for the relationship between dynamic elastic modulus and temperature, although the difference is minimal with the linear fit.

Table 3 Equations and determination coefficients for best fitting dynamic modulus and temperature.

| Cooling	Fitting	Equation	R^2
Slow	Linear	$E_{dyn} = -0.06T + 56.9$	0.97
Quick		$E_{dyn} = -0.05T + 54.9$	0.93
Slow	Exponential	$E_{dyn} = 65.9e^{-0.002T}$	0.98
Quick		$E_{dyn} = 61.5e^{-0.002T}$	0.95

3.3. Ratio E_{dyn}/E_{st}

Figure 3 shows the relationship between the dynamic and static modulus of elasticity values obtained for each target temperature, represented by the k-value determined in accordance with equation (1). The values of k vary between 0.67 and 1.67.

As can be seen, the k values are less than unity for the standard samples, the samples exposed to 200 ℃ (for both cooling methods), and for the samples exposed to 400 ℃ with slow cooling (Table 4). The remaining samples present k values higher than unity and this coincides with values for the static and dynamic modulus of elasticity below 30 GPa (figure 3).

Table 4 k values. **μ** average value and **σ** standard deviation.

Sample group	k-value ($\mu \pm \sigma$)				
	Room temperature	200	400	600	800
Pattern	**0.76 ± 0.03**	-	-	-	-
Slow	-	**0.77 ± 0.01**	**0.80 ± 0.10**	**1.50 ± 0.13**	**1.33 ± 0.12**
Quick	-	**0.85 ± 0.03**	**1.20 ± 0.13**	**1.36 ± 0.31**	**1.33 ± 0.22**

Between the standard samples and the samples exposed to 200 ºC, the difference is minimal, and the values are very homogeneous. The data dispersion is greater for the samples exposed to 600 and 800 ºC and quickly cooled.
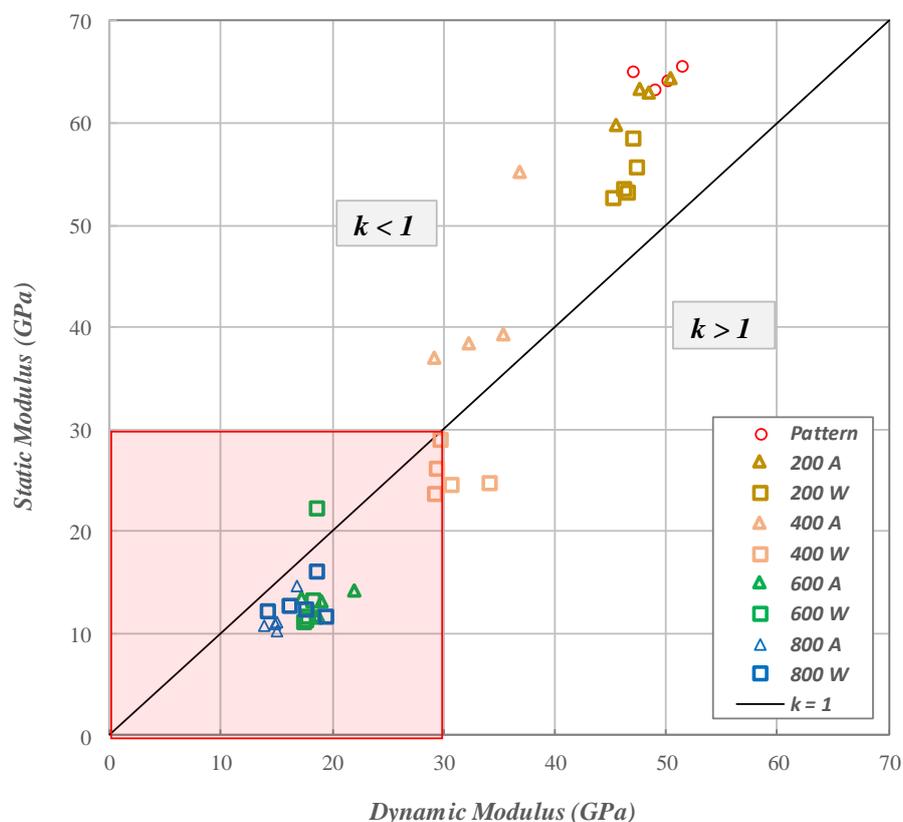
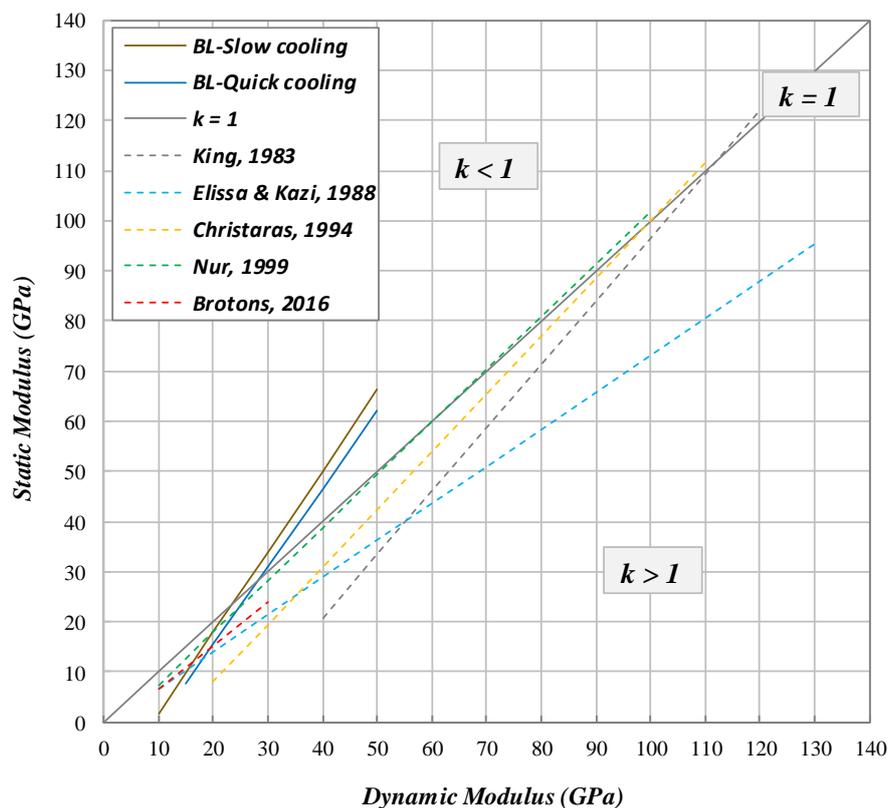**Figure 3**. Relationship between the dynamic and static elastic modulus for every target temperature.

4. Discussion
The limestone samples generally show a decrease in the modulus of elasticity of between 80 and 90 % [2]–[4], as shown for the BL. The evolution of BL resembles that of the limestone tested by Mao et al. (2009) and, like the latter, has a threshold temperature of 600 ºC. The calcarenite studied by Brotons et al. (2013) also shows a similar evolution, however, the threshold temperature is reached at 500 °C. The decrease recorded for the maximum temperature tested of 600ºC is 85 % and of the same order of magnitude as that observed for BL. Quick cooling decreases the modulus of elasticity by 10 to 20 % more than slow cooling [9], [10]. For BL, this difference does not exceed 10 % for the static modulus and 5 % for the dynamic modulus.

There is an excellent correlation between the modulus of elasticity value and temperature with better coefficients of determination for exponential correlations, and higher coefficients for dynamic moduli with 0.98 and 0.95 for slow and fast cooling, respectively.

If the values of the moduli of elasticity obtained by static and dynamic methods are compared, the latter are usually higher for most materials, and the divergence in the values obtained by both methods is greater the lower the modulus of elasticity of the material analysed [11], [12]. The general trend reveals values of k above unity that tend to converge to unity as the values of the moduli increase, so that from 120 GPa onwards, the rocks generally have identical dynamic and static moduli [12]. This is because the rocks with high elastic moduli are little altered and/or weathered and have little porosity and no cracks – all characteristics that can affect dynamic and static tests differently [12].

Figure 4 shows the linear relationship between dynamic modulus and static modulus represented by the k-value as defined in equation (1). As mentioned previously, this is usually greater than unity for low modulus of elasticity values, but as the modulus of elasticity of the rock increases, this value converges to 1 and even falls below unity. This has been observed in the literature for intact rocks with modulus of elasticity values above 50 GPa [15]–[17]. This, however, in the present study has been observed for rocks exposed to high temperatures where the modulus of elasticity values are above 30 GPa. In other cases, such convergence does not occur [18].

Figure 4. Relationship between dynamic and static elastic modulus from different authors (adapted from Brotons et al., 2014).

In linear relationships between static modulus and dynamic modulus, the wider the interval of modulus of elasticity considered, the lower the coefficients of determination. On specific kind of rocks, with medium to low modulus of elasticity values (Est < 50 GPa), the coefficients of determination are higher (Table 5).

Table 5. Linear fit between dynamic and static modulus of the reviewed rocks. E_{st}: static elastic modulus; E_{dyn} dynamic elastic modulus; R^2: coefficient of determination; and NA: not available.

Authors	Rock	Equation	R^2	E_{st} (GPa)
King et al. (1983)	Igneous-metamorphic	$E_{st} = 1.26E_{dyn} - 29.5$	0.82	40 - 120
Eissa & Kazi (1988)	All types	$E_{st} = 0.74E_{dyn} - 0.82$	0.70	5 - 130
Christaras et al. (1994)	All types	$E_{st} = 1.05E_{dyn} - 3.16$	0.99	25 - 110
Nur et al. (1999)	NA	$E_{st} = 1.153E_{dyn} - 15.2$	NA	> 15
Brotons et al. (2016)	Calcarenite	$E_{st} = 0.867E_{dyn} - 2.085$	0.96	5 - 30
BL – Slow cooling	Limestone	$E_{st} = 1.62E_{dyn} - 14.62$	0.99	10 - 70
BL – Quick cooling	Limestone	$E_{st} = 1.55E_{dyn} - 15.52$	0.97	10 - 70

5. Conclusions

This work studies the variation of the modulus of elasticity of a rock exposed to high temperatures, as obtained by static (i.e., destructive tests) and dynamic (i.e., non-destructive tests) means. A Cretaceous limestone (Pedra de Borriol and termed BL) was used for this research. This rock has been commonly used in the construction of historical monuments since the 17th century in the Valencia Region of Spain.

The relationship between the values estimated by both methods has been studied to establish if there is an acceptable correlation. The main conclusions are:

- The results of the tests on the BL show that the static modulus of the samples heated to 600 ℃ decreased to 80.9 and 79.1%, and the dynamic modulus decreased to 62.5 and 64.8% for slow and quick cooling samples, respectively. For those samples heated to 600 and 800 ℃, the static and dynamic moduli are almost the same.
- In the determination of the static modulus, differences were observed between slow and quick cooling at 200 and 400 °C, with a higher loss for quick cooling (around 10 % more). The differences are insignificant for the dynamic modulus over the entire temperature range tested.
- When using a linear regression model, an excellent correlation between static and dynamic modulus of elasticity is observed, providing coefficients of determination of 0.99 and 0.97 for slow and quick cooling, respectively.
- The ratio E_{dyn}/E_{st} ratio was less than 1 for elastic moduli above 30 GPa (i.e., for temperatures of 105, 200, and 400 ℃) and greater than 1 for lower moduli (i.e., for temperatures of 600 and 800 ℃).

The results obtained confirm that it is possible to reliably determine the static modulus of elasticity for the limestone rock, known as Pedra de Borriol, from the dynamic modulus. If a historical monument partially or totally built with this rock is affected by a fire, the necessary parameters to evaluate structural stability could be determined using non-destructive testing techniques such as ultrasound velocity.

References
[1]   A. Sygała, M. Bukowska, and T. Janoszek, "High Temperature Versus Geomechanical Parameters of Selected Rocks – The Present State of Research," *J. Sustain. Min.*, vol. 12, no. 4, pp. 45–51, 2013.
[2]   X. biao Mao, L. Zhang, T. zhen Li, and H. shun Liu, "Properties of failure mode and thermal damage for limestone at high temperature," *Min. Sci. Technol.*, vol. 19, no. 3, pp. 290–294,

2009.

[3]   Y. Zhang, Q. Sun, L. Cao, and J. Geng, "Pore, mechanics and acoustic emission characteristics of limestone under the influence of temperature," *Appl. Therm. Eng.*, vol. 123, pp. 1237–1244, 2017.

[4]   J. Yang, L. Y. Fu, W. Zhang, and Z. Wang, "Mechanical property and thermal damage factor of limestone at high temperature," *Int. J. Rock Mech. Min. Sci.*, 2019.

[5]   Y. L. Chen, J. Ni, W. Shao, and R. Azzam, "Experimental study on the influence of temperature on the mechanical properties of granite under uni-axial compression and fatigue loading," *Int. J. Rock Mech. Min. Sci.*, vol. 56, pp. 62–66, 2012.

[6]   L. Zhang, X. Mao, and A. Lu, "Experimental study on the mechanical properties of rocks at high temperature," *Sci. China, Ser. E Technol. Sci.*, vol. 52, no. 3, pp. 641–646, 2009.

[7]   P. G. Ranjith, D. R. Viete, B. J. Chen, and M. S. A. Perera, "Transformation plasticity and the effect of temperature on the mechanical behaviour of Hawkesbury sandstone at atmospheric pressure," *Eng. Geol.*, vol. 151, pp. 120–127, 2012.

[8]   F. Vagnon *et al.*, "Effects of thermal treatment on physical and mechanical properties of Valdieri Marble - NW Italy," *Int. J. Rock Mech. Min. Sci.*, 2019.

[9]   V. Brotóns, R. Tomás, S. Ivorra, and J. C. Alarcón, "Temperature influence on the physical and mechanical properties of a porous rock: San Julian's calcarenite," *Eng. Geol.*, vol. 167, pp. 117–127, 2013.

[10]   T. D. Rathnaweera *et al.*, "Experimental investigation of thermomechanical behaviour of clay-rich sandstone at extreme temperatures followed by cooling treatments," *Int. J. Rock Mech. Min. Sci.*, vol. 107, no. September 2017, pp. 208–223, 2018.

[11]   J. Martínez-Martínez, D. Benavente, and M. A. García-del-Cura, "Comparison of the static and dynamic elastic modulus in carbonate rocks," *Bull. Eng. Geol. Environ.*, vol. 71, no. 2, pp. 263–268, 2012.

[12]   V. Brotons *et al.*, "Improved correlation between the static and dynamic elastic modulus of different types of rocks," *Mater. Struct. Constr.*, vol. 49, no. 8, pp. 3021–3037, 2016.

[13]   ISRM, "Suggested Methods for Determining the Uniaxial Compressive Strength and Deformability of Rock Materials," *Int. Soc. Rock Mech.*, no. December, pp. 0–3, 1977.

[14]   A. Aydin, "Upgraded ISRM Suggested Method for Determining Sound Velocity by Ultrasonic Pulse Transmission Technique," *Bull. Eng. Geol. Environ.*, vol. 74, no. 4, pp. 1499–1500, 2015.

[15]   M. S. King, "Static and dynamic elastic properties of rocks from the Canadian shield," *Int. J. Rock Mech. Min. Sci. Geomech. Abstr.*, vol. 20, no. 5, pp. 237–241, 1983.

[16]   A. Nur and Z. Wang, *Seismic and acoustic velocities in reservoir rocks*, no. 10. Society of Exploration Geophysicists, 1998.

[17]   B. Christaras, F. Auger, and E. Mosse, "Determination of the moduli of elasticity of rocks. Comparison of the ultrasonic velocity and mechanical resonance frequency methods with direct static methods," *Mater. Struct.*, vol. 27, no. 4, pp. 222–228, 1994.

[18]   E. A. Eissa and A. Kazi, "Relation between static and dynamic Young's moduli of rocks," *Int. J. Rock Mech. Min. Sci. Geomech. Abstr.*, vol. 25, no. 6, pp. 479–482, 1988.