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Marble Characterization by ultrasonic methods

Mohamed El Boudani^{a*}, Nicolas Wilkie-Chancellier^a, Loïc Martinez^a, Ronan Hébert^b,
Olivier Rolland^c, Sébastien Forst^c, Véronique Vergès-Belmin^d and Stéphane Serfaty^a

^a Laboratoire SATIE (UMR CNRS 8029), Université de Cergy-Pontoise, 5 mail Gay Lussac, 95031 Neuville Sur Oise, France

^b Laboratoire GEC (EA 4506), Université de Cergy-Pontoise, 5 mail Gay Lussac, 95031 Neuville Sur Oise, France

^c Etablissement Public du Château de Versailles, Atelier et restauration des sculptures, RP834, 78008 Versailles Cedex, France

^d Laboratoire LRMH (USR 3224), 29 rue de Paris, 77420 Champs Sur Marne, France.

Abstract

Ultrasonic techniques are increasingly being used in various fields such as mining, geotechnical, civil engineering, because they are non-destructive and easy to apply. These techniques are usually employed both in situ and in laboratory to characterize and determine the dynamic and physical properties of rocks. In cultural heritage, the characterization of marble statue by acoustic wave is a well-known non-destructive method. This paper undertakes a study of the Carrara marble that composes the statues exposed in the Château de Versailles gardens, in order to better understand the processes of deterioration affecting them. For this, 10 cm side square plates of Carrara marble are artificially aged through heating/cooling thermal cycles performed on one face of the samples. Acoustic waves such as compressive, shear and Rayleigh are generated by 1 MHz central frequency contact transducers excited by a voltage pulse generator. A laser vibrometry detection method is used in the case of Rayleigh wave detection. The transmission measurements point out a strong decrease of the wave speed as well as a dramatic decrease of the maximum frequency transmitted in conjunction with the weathering. The velocity of ultrasonic waves depends on the physical and mechanical properties of the stone (such as density, porosity, elastic constants, and structure). Therefore, correlating the changes in these properties with the measured ultrasonic velocity helps to provide classification schemes for evaluating the degree of stone deterioration Köhler (1991). Some classical tests such as drilling resistance and water absorption (contact sponge method and water drop test) were also performed in order to complete the results obtained by ultrasonic velocity method.

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* Corresponding author. Tel.: +33 1 47 40 21 13.

E-mail address: mohamed.el-boudani@u-cergy.fr

1. Introduction

Natural stone has been an important building material for thousands of years. A significant part of the world's cultural heritage is built of stone. In order to get the required knowledge for the preservation and conservation of stone, various investigation methods are applied. The traditional techniques already in use for the investigation of natural stone are generally destructive: uniaxial compression (L.N.Y. Wong and H.H. Einstein, 2009), triaxial deformation (MR. Ayling et al, 1994) and strength test (Mingqing You, 2011), etc. Among the non-destructive methods with most acceptance and applications in the study of the structural characteristics and state of conservation of stone materials in historic and artistic buildings is by determination of the propagation velocity of ultrasonic waves (Saez-Perez and Rodriguez-Gordillo, 2008). This research focused on the use of non-destructive ultrasonic technique as validated by traditional methods for the study of the degree of weathering of marble. For this, some aged samples were prepared artificially by a thaw-freeze cycles. Ultrasonic velocity seemed to correlate well with the degree of weathering in the marble.

2. Methods and equipment

2.1. Materials

Two samples of Carrara marble are tested I1 and G1 (10*10*3 cm). In order to simulate degradations encountered in Carrara marble statue in Chateau de Versailles, an ageing artificially protocol was developed and applied on the sample G1 (Fig. 1). The protocol consists in a 25 thaw-freeze cycles for different temperatures (100°C, 200°C, 300°C and 400°C). In this experiment the temperature cycles are exposed to one side of the sample. A ceramic hot plate (450°C max) and a recipient with ice cube are used.

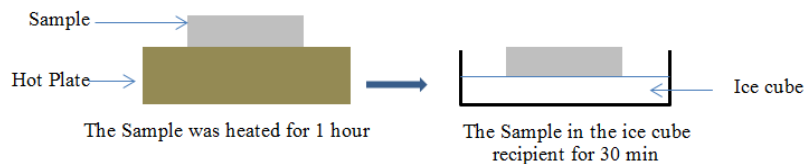


Fig. 1. Illustration of the thaw-freeze protocol.

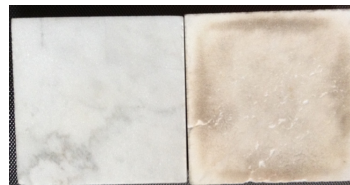


Fig. 2. Non-degraded sample I1 (left), artificial aged sample G1 (right).

2.2. Ultrasonic test

Ultrasonic method consists of propagating ultrasound waves through a material to measure the travel time and any change of intensity of the wave for a given distance. In the transmission method (the method that we use in this study), the ultrasonic transmitter and receiver are placed opposite to each other on either side of the marble sample and the time needed for the wave to travel along the distance between them is measured. The time of flight (TOF) allows calculating the shear and longitudinal wave velocities through sample. Acoustic waves such as compressive, shear and Rayleigh are generated by 1 MHz central frequency contact transducers excited by a voltage pulse generator. A vibrometry method is used in the case of Rayleigh wave detection. A coupling media, to improve the acoustic contact between the sample and the transducers was used (Fig. 3 and 4).

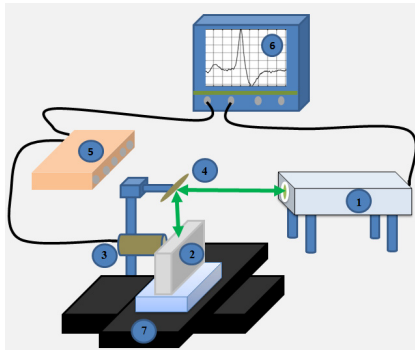


Fig. 3. Transmission Ultrasonic technique for Rayleigh wave.
(1) Vibrometer laser. (2) Marble sample. (3) Emitter transducer
(4) Mirror (5) Impulse generator (6) Oscilloscope
(7) Motorized linear stages .

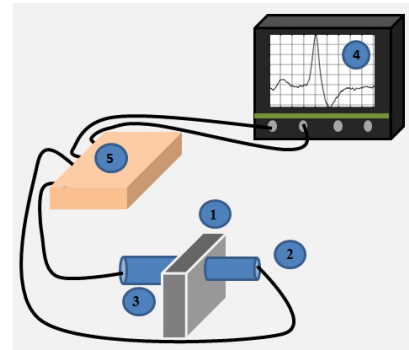


Fig. 4. Transmission Ultrasonic technique for P and S wave.
(1) Marble sample. (2) and (3) emitter and receiver transducer.
(4) Oscilloscope. (5) Impulse generator.

2.3. Classical tests

In order to validate results obtained by ultrasonic measurements, two kinds of classical tests have been performed: the estimation of the drilling resistance and the water absorbing behaviour.

The Drilling Resistance Measuring System (DRMS) is basically a power drill with constant feed and a force transducer that measures the thrust as a function of the drilling depth (JM Mimoso and D Rodrigues Costa, 2005). The force is continuously measured by a load cell and a graph showing the force versus depth is displayed after the test (Fig. 5). The fluctuations in the drilling resistance around the mean value can be attributed to, amongst other causes, non-homogeneities in the material, such as different sedimentation layers, different grain size and resistance, micro and macro cracks etc. (M Pamplona et al, 2007. E Valentini et al, 2008).

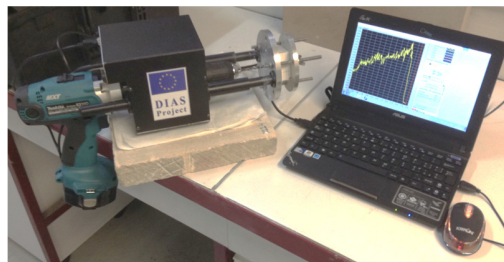


Fig. 5. Drill resistance test.

Water absorption in stone materials is one of the main causes of accelerated deterioration. The WAB can be defined as the behaviour in which water is absorbed and retained into the stone by the characteristic pore structure and pore size distribution (D Vandevorde et al, 2012). The most commonly used non-destructive methods for analysis of the WAB are the contact-sponge method and water drop test.

2.4. The contact-sponge method

The Fig. 6 illustrates the method consisting of a sponge, enclosed in a contact plate. The contact plate is composed of two parts, a base and a cover. The diameter of the sponge corresponds with the inner diameter of the base, whereas the height of the sponge exceeds the vertical borders of the base. For the measurement the cover of the contact plate is removed, water is added to the sponge and the sponge enclosed in the contact plate is weighed (m_i). After weighing, the cover of the contact plate is removed and the sponge is pressed manually against the stone surface until the vertical borders of the base touch the stone surface. The contact sponge is removed and weighed inside the closed contact plate (m_f). (Tiano and C Pardini, 2004).

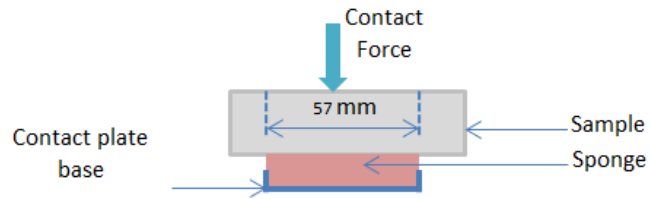


Fig. 6. Schematic illustration of the contact sponge test.

The water absorption coefficient is calculated according the next equation, where A equals the surface of the sponge (0.002376 m^2) and t_1 is the time contact:

$$W_a \left(\frac{g}{m^2} \cdot s \right) = \frac{\Delta m}{A \cdot t_1} = \frac{m_i - m_f}{A \cdot t_1}$$

2.5. Water drop test

The water drop test (Fig. 7) is especially suitable to investigate whether an unpolished stone, mortar or matt painted surface is water repellent or not (Bläuer Böhm, 2007).



Fig. 7. Water-drop test equipment.

A small drop ($5 \mu\text{L}$) of demineralized water is carefully applied on the surface of the material to be tested with the help of an Eppendorf micropipette. To quantify the observations a stop watch can be launched at the moment when the drop initially touches the surface and the time until the drop is completely absorbed by the material can be measured (C. Bläuer et al, 2012). 20 measurements were made and then their mean value was calculated.

3. Results and discussion

3.1. Ultrasonic results

We note that the travel time is shorter for I1 than G1 in both cases of P and S waves. Therefore, the P and S velocities are higher for I1 than G1. We note also a high difference in intensity for the waves in the two samples (for the shear wave in G1, we amplify so much to get a visible signal) (Fig. 8). That confirms a correlation between the state of degradation of rock and the velocity and intensity of the wave propagated in it.

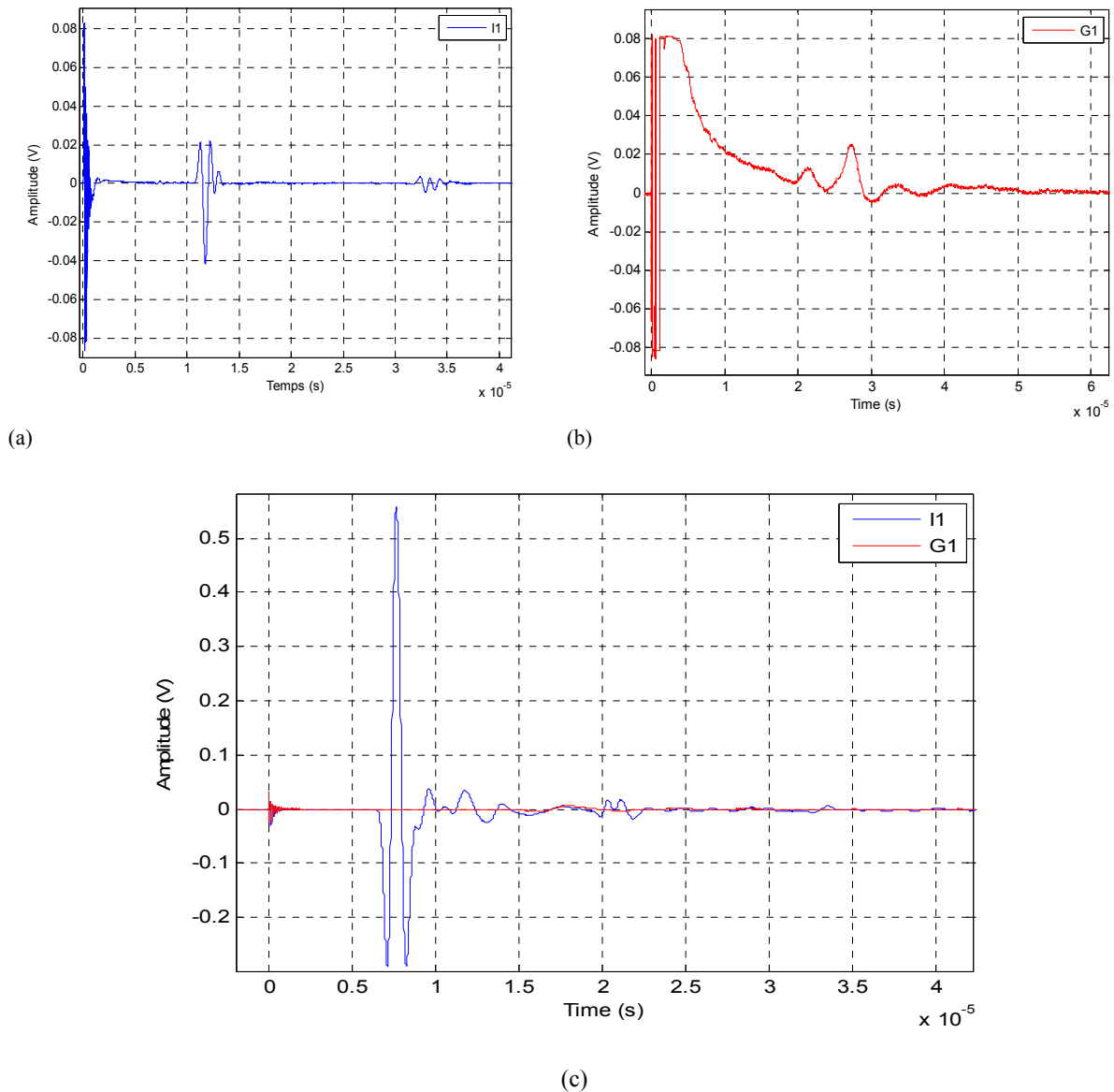


Fig. 8: Propagation of S waves in I1 sample (a); G1 sample (b); Propagation of P waves in I1 and G1 samples (c).

For Rayleigh wave propagation, we observe the apparition of a dispersive phenomenon in the degraded sample G1 and we can distinguee three zones with different slops (Fig. 9b). The sample G1 presents three degrees of degradation and non-homogeneity through depth : zone 1 (0 mm to 3 mm deeply), zone 2 (3 mm to 12 mm deeply) and zone 3 (12mm to 25mm deeply). Using the Fast Fourier Transform in 2 dimensions (Fig. 9 c and d), we get the experimental Rayleigh waves velocities, 2700m/s for I1 and 450m/s for G1.

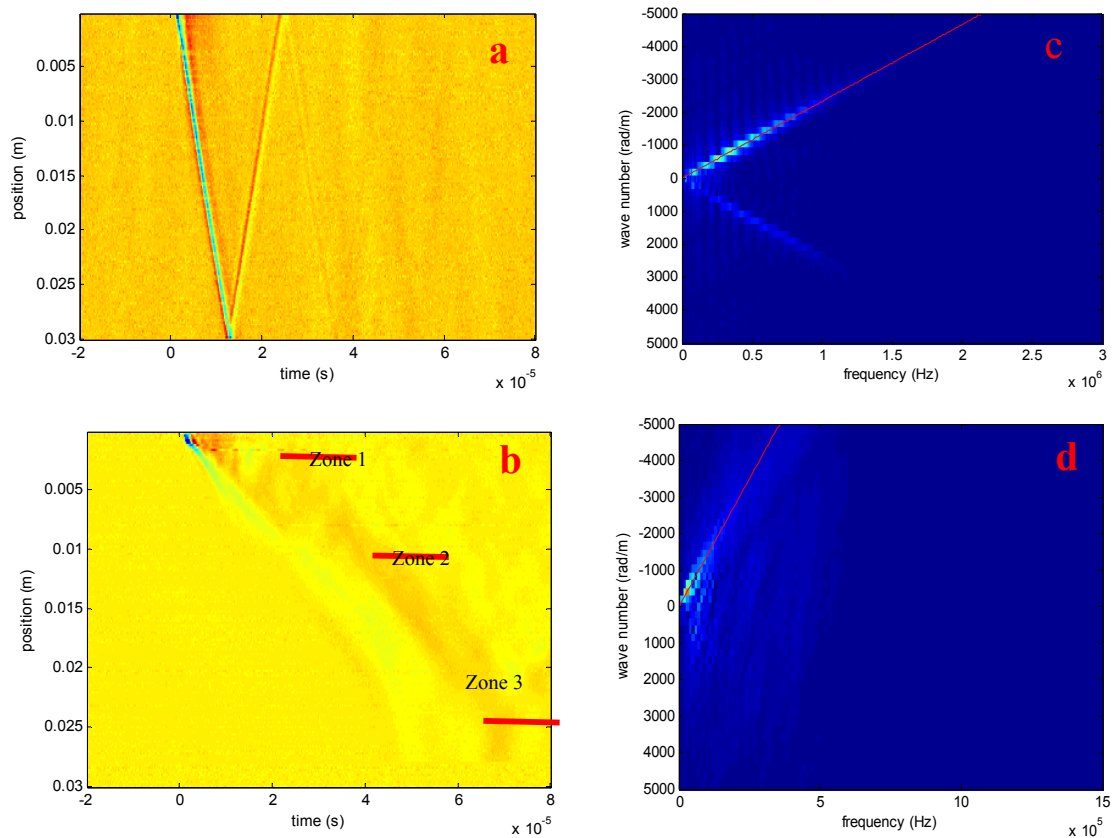


Fig. 9. Rayleigh waves vibrations in function of time and depth in I1 (a) and G1 (b). FFT2D and Rayleigh velocity asymptote (red line) in function of wave number and frequency of I1 (c) and G1 (d).

3.2. Drilling resistance results

The drilling resistance is higher and homogeny in the sample I1, while it is increasing vs. depth in G1. We can distinguish three different zones in the G1 drilling resistance curve (zone 1 : 0-3 mm; zone 2 : 3-12 mm; zone 3 : 12-25 mm) corresponding to the results obtained in Rayleigh wave vibration; the sample presents three degrees of degradation and non-homogeneity through depth (Fig. 10).

3.3. Contact sponge results

In I1, the sponge forms a wet patch with the same diameter of the sponge itself without any seepage in contrast to G1, in which the water penetrates into the material forming a wet patch that is bigger than the initial sponge diameter, and after a few seconds the water is absorbed completely. In Tab.1, we resume the computed absorption coefficient in the both samples: it's higher in G1 than I1. Therefore the porosity and the pores diameter are important in G1 than I1.

Table 1. Absorption coefficient Wa.

Sample	m_i (g)	m_f (g)	Δ_m (g)	Wa (g/m ² .s)
I1	15.84	15.80	0.04	0.14
G1	16.16	15.16	1.00	3.50

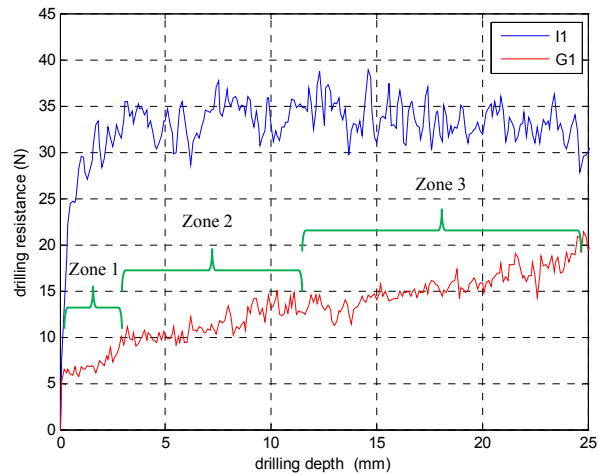


Fig. 10. Drilling resistance in the samples I1-G1.

3.4. Water drop results

For the Sample I1, the drop forms a nearly perfect spherical shape without any spread on the surface; the surface is highly water repellent. However, for the Sample G1, The drop penetrates quickly forming a much bigger wet patch than the diameter of the initial drop near the surface (0-5 mm deeply); the material is any more water repellent but underneath there is a layer of material that does not allow water to be sucked up well. In the zone 15-25 mm deeply, it penetrates into the material forming a wet patch that is not much bigger than the initial drop size; the material is porous and capillary. We note also, that the absorption time of the drop is much higher in I1 than G1 and it increase with the increasing of the distance to the surface, which was heated of G1; the sample G1 presents different degree of alteration in depth (Fig. 11).

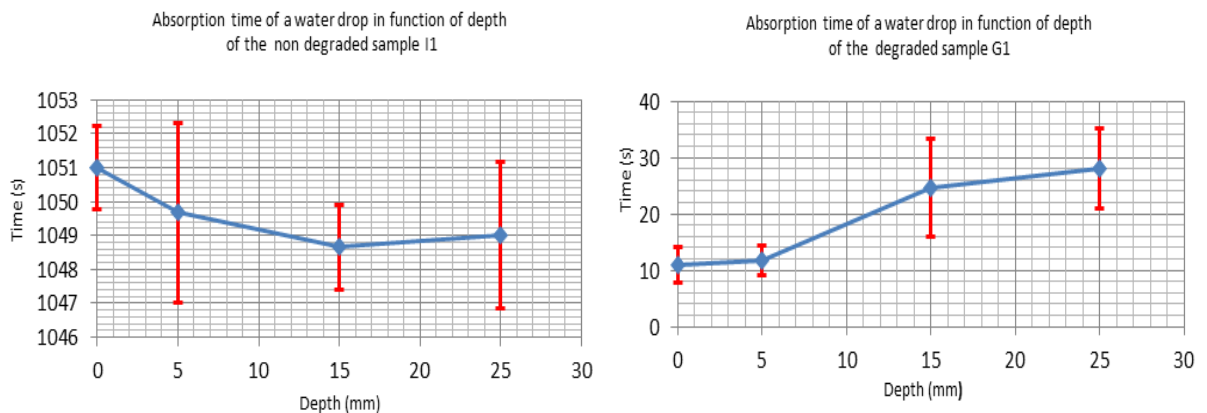


Fig. 11. Absorption time of a water drop of the non altered (left) and altered samples (right).

4. Conclusion

The P and S wave propagations, using the transmission method, provides information about changes in travel time and intensity “attenuation” that’s correlate with degree of the stone degradation. The change of the vibro-acoustic behaviour of Rayleigh wave in a stone depth gives information able to determine the depth of different weathered layers and the damage intensity.

We noted the existence of three different zones with different degrees of degradation and non-homogeneity through depth in the sample G1 (zone 1 : 0mm to 3 mm; zone 2 : 3mm to 12 mm; zone 3 : 12mm to 25 mm). These results were confirmed by some classical tests.

The next step of this work, will be a best comprehension of the Rayleigh wave dispersion in altered layers, and determine dynamics and physicals properties of rocks.

Acknowledgement

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