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EVALUATION OF MARBLE THERMAL-INDUCED DECAY BY ULTRASONIC PULSE VELOCITY AND LEEB HARDNESS

Abstract - G. Pedrini, S. Pagnotta, M. Lezzerini, *Evaluation of marble thermal-induced decay by ultrasonic pulse velocity and Leeb hardness.*

Marble has a great importance in the field of cultural heritage and, therefore, it is important to quantify the decay level of the marble to implement at the right time restoration plans aimed at safeguarding as much as possible cultural heritages. The degradation of the marbles with consequent loss of mechanical properties is correlated to an increase in porosity; this increase in porosity is the result of the loss of cohesion among the grains and the formation of micro-cracks. When it is not possible to remove samples to be analysed in the laboratory or when are needed instant check, the role of non-destructive and operable in situ methods is fundamental. In this study, by using portable instruments, correlations were obtained between the variation in the ultrasonic pulse velocity (UPV), the variation in surface hardness Leeb (HL) in relation to the variation in porosity open to water. This porosity was obtained by subjecting two varieties of marble (from Apuan Alps, in Italy, and from Paros Island, in Greece) to artificial thermal degradation of varying intensity from 100°C up to 500°C, at ambient pressure. As the intensity of degradation increases, more and more porosities open to water are generated.

Key words - Apuan Alps, marble, decay, Paros Island, porosity, ultrasonic velocity, Leeb hardness

Riassunto - G. Pedrini, S. Pagnotta, M. Lezzerini, *Valutazione di marmi degradati termicamente mediante la misurazione della velocità di propagazione degli impulsi ultrasonici e della durezza superficiale Leeb.*

Il marmo ha una grande rilevanza nell'ambito dei beni culturali, e quindi è importante monitorarne il livello di degrado in modo da poter avviare tempestive pratiche di tutela e restauro. Il degrado dei marmi con conseguente perdita di proprietà meccaniche è correlato ad un aumento di porosità, tale aumento di porosità è il risultato della perdita di coesione tra i grani e della formazione di microfessure. Quando non è possibile asportare campioni da analizzare in laboratorio oppure quando si vuole fare una verifica istantanea il ruolo dei metodi non distruttivi e operabili in situ è fondamentale. In questo studio mediante l'utilizzo di strumenti portatili sono state ottenute correlazioni fra la variazione della velocità di impulsi ultrasonici (UPV), la variazione di durezza superficiale Leeb (HL) in relazione con la variazione di porosità aperta all'acqua. Questa porosità è stata ottenuta sottoponendo due varietà di marmo bianco (provenienti dalle Alpi Apuane, in Italia, e dall'isola di Paros, in Grecia), a degrado termico artificiale di varia intensità da 100 °C fino a 500 °C a pressione ambientale, con l'aumentare dell'intensità di degrado si sono cosi generate porosità aperte all'acqua sempre maggiori.

Parole chiave - Alpi Apuane, marmo, degrado, Isola di Paros, porosità, velocità impulso ultrasonico, durezza Leeb

Introduction

Since ancient times, marble has always been used as a building material for the most important buildings and monuments. Nowadays, several churches (Lezzerini, 2005; Ramacciotti *et al.*, 2015), theatres (Columbu *et al.*, 2018) (Taelman *et al.*, 2019), temples (Williams *et al.*, 1992) (Koralay & Kilinçarslan, 2016), other structures (Columbu *et al.*, 2014) (Skaggs *et al.*, 2019), and especially sculptures (Antonelli *et al.*, 2014; Pensabene *et al.*, 2012; Lezzerini *et al.*, 2017; Ouazaa *et al.*, 2013; Lezzerini *et al.*, 2012; Miriello *et al.*, 2012) of considerable historical value are made of marble. Since these man-made works are subject to processes of environmental and anthropic degradation, it is important to quantify the decay level of the marble to implement at the right time restoration plans aimed at safeguarding as much as possible cultural heritages. Generally, methods for analysing rocks are divided into destructive and non-destructive methods. In several recent papers marbles, stones and other building materials were analysed by using destructive methods such as OM, XRF, XRD, SEM-EDS (Franzini & Lezzerini, 2002, 2003b; Lezzerini *et al.*, 2018; Lezzerini *et al.*, 2013; Fratini *et al.*, 2015; Franzini *et al.*, 2010), but also some researches have made use of non-destructive methods such as light beam and geophysical methods (Biricotti & Severi, 2004; Yalçıner *et al.*, 2019; Boudani *et al.*, 2015) or by both of them (Baracchini *et al.*, 2005; Siegesmund *et al.*, 2021; Vagnon *et al.*, 2019; Sena da Fonseca *et al.*, 2021). Ultrasonic pulse velocity (UPV) method and Leeb rebound hardness (HL) test are very promising methods in the study of marble, stones and their decay (Ur Rehman *et al.*, 2022; Vasconcelos *et al.*, 2008; Gomez-Heras *et al.*, 2020).

UPV is a measurement of the speed of ultrasonic waves passing through a body. It is an economic, fast, and non-destructive (which is not irrelevant in the studies of cultural heritage) test to determine the properties of natural rocks and other materials, like concrete and bricks either in situ or laboratory conditions. The method was inspired by the relationships

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between sonic and mechanical properties found in oil exploration; ultrasonic methods start developing in 1940's for the assessment of concrete strength (Jones, 1949) and later it was used for evaluations of rocks physical proprieties. P-wave propagation in materials is affected by bulk properties of the stone such as mineral composition, texture, density, and porosity. In monomineralic rocks, like marble, elastic wave propagation is mostly affected by texture, grains proprieties and porosity (Luque *et al.*, 2011; Åkesson *et al.*, 2006; Royer-Carfagni, 1999; Malaga-Starzec *et al.*, 2006; Shushakova *et al.*, 2011; Weiss *et al.*, 2002). UPV method is widely used in the field of cultural heritage, i.e. to evaluate the degradation of building materials used in historical architecture, such as mortars, brick and natural stones. Some studies shows that physical properties, such as water absorption and porosity have established good correlations with P-wave velocity for some carbonate rocks (Kahraman & Yeken, 2008), granites (Vasconcelos *et al.*, 2008), calcarenites (Rahmouni *et al.*, 2013) and sandstones (Gomez-Heras *et al.*, 2020). Another important direct application of ultrasonic pulse velocity measurements is on the assessment of the quality of marble (Weiss *et al.*, 2002). Several authors studied variation of porosity and UPV vs induced and natural thermal degradation, on porous calcarenite (Vasanelli *et al.*, 2021), carbonate rocks (Ur Rehman *et al.*, 2022, Yavuz *et al.*, 2010, Ugur *et al.*, 2014), and granites (Tomás *et al.*, 2021). The basic assumption for the quality assessment of a natural building stone based on ultrasonic measurements is that a decrease of UPV is correlated with a certain degree of degradation. Marble's quality is susceptible to be damage by heating, the entity of this damage is influenced by some relevant parameters as mineralogical composition, grain size, grain boundaries linearity, grain shape preferred orientation, lattice preferred orientation, number of adjacent grains and initial number of micro-cracks (Yavuz *et al.*, 2010; Luque *et al.*, 2011) and, due to it, every type of marble has its own "starting UPV", which is UPV measured on non-degraded samples and that "starting UPV" decrease according to the degree of degradation, therefore, to investigate the degree of degradation of marble it's important to know UPV values in advance. A verifiable and repeatable way of preparing degraded samples is to subject them to induced thermal degradation. Various authors studied variation of porosity and UPV vs inducted and natural thermal degradation, on carbonate rocks (Yavuz *et al.*, 2010, Ugur *et al.*, 2014, Tomás *et al.*, 2021) findings some correlations between UPV and porosity vs thermal degradation. However, porosity is an important factor in determining the effectiveness weathering due to thermal induced degradation (Franzoni *et al.*, 2013).

HL is another non-destructive method developed in 1975 to provide a portable hardness test, originally for metals and polymers. This test uses energy measurement principles in determining the hardness of material (Leeb, 1979; Boogaard & Van Dijk, 1989). Later its use was expanded to measure the surface hardness of rocks (Aoki & Matsukura, 2008). In previews studies LRH was usually correlated with UCS (Corkum *et al.*, 2018; Gomez-Heras *et al.*, 2020), HL and Smith hammer vs UCS (Çelik & Çobanoğlu, 2019; Aoki & Matsukura 2008), but only few studies correlates HL, UPV and porosity (Gomez-Heras *et al.*, 2020).

This study is aimed to investigate the porosity of marble induced by thermal decay evaluating it also by ultrasonic pulse velocity and Leeb rebound hardness test.

Material and methods

Two types of white marbles were analysed: 1) a fine grain-sized marble (MF), characterized by homeoblastic/granoblastic texture with straight boundaries and triple points at 120 degrees; 2) a coarse grain-sized marble (MC), characterized by heteroblastic/granoblastic texture with curve to lobate boundaries. MF and MC are from Apuan Alps (Italy) and from Paros Island (Greece), respectively. Six specimens for every selected marble, for a total of twelve samples, were measured before and after artificial treatments at 60 °C, 200 °C, 300 °C, 400 °C and 500 °C.

Real density, apparent density, porosity and water absorption at atmospheric pressure were determined according to the European Standards EN 13755 (EN 13755 2008; EN 1936 2006). The volumes of specimens were measured by using a hydrostatic balance on saturated samples (Franzini & Lezzerini, 2003a).

The ultrasonic pulse velocity (UPV) (Naik, Malhotra, Popovics, 2003) was measured by using a PROCEQ PUNDIT PL-200 instrument equipped with probes at frequency of 54 kHz. The measures were obtained for the three main directions of the cubic specimens and expressed as the mean of the three readings. UPV measurements were performed according to the European Standard EN 14579 (EN 14579 2004) on dry specimens at ambient temperature.

The measuring principle of Hardness Leeb (HL) is based on the dynamic rebound method. An impact body with a hard metal tip is projected by a preloaded spring against the surface of the samples. The impact causes a slight deformation of the surface, which leads to a loss of kinetic energy (Aoki & Matsukura, 2008). Speeds are measured by means of a permanent magnet in the impact body that generates an induced voltage in the firing pin probe. The identified voltage is proportional to the speed of the impact body. The HL values were obtained by using Proceq Equotip 550 with Type D device (HLD), the European Standard EN ISO 16859-1 (EN ISO 16859-1 2015) is specified for metal and not for rocks, so in this paper were follows recommendation for non-standardized test, averaging the hardness values taken at different points (Aoki & Matsukura, 2008).

Results and discussion

Table 1 shows for MF and MC Ultrasonic Pulse velocity UPV (m/s), Leeb Hardness measured with type D device (HLD) and type of thermal-induced degradation ordered by increasing open water porosity Ab_v (% by vol.). In MF samples an increasing of water open porosity from 0.27 to 3.84 (by fourteen time) leads to a decrease in the Ultrasonic Pulse Velocity UPV (m/s) up to 82% from 3865 to 696 and a decrease in Leeb Hardness HLD of 55% from 575 to 257. While, In MC samples an increasing of open water porosity Ab_v (% by vol.) from 0.20 to 3.37 (by sixteen time) leads to a decrease in the Ultrasonic Pulse Velocity UPV (m/s) up to 70% from 5011 to 1488 and a decrease in Leeb Hardness HLD of 65% from 347 to 121. For both marbles the increasement of porosity is correlated with increasement of intensity of thermally-induced decay as shown in other studies (Siegesmund, Menningen, Shushakova, 2021). The resistance to degradation change in function of the physical-mineralogical characteristics of the samples, such as: mineralogical composition, grain size, grain boundaries linearity, grain shape preferred orientation, lattice preferred orientation, number of adjacent grains and initial number of micro-cracks (Yavuz *et al.*, 2010; Luque *et al.*, 2011).
In the Figure 1, Ultrasonic Pulse Velocity UPV vs open water porosity Ab_v (% by vol.), MC and MF were well correlated by an exponential equations with coefficients of determination (R^2) respectively of: $R^2 = 0.9681$ for MF and $R^2 = 0.9494$ for MC, those equations confirms other what is shown in other studies (Siegesmund *et al.*, 2021; Gomez-Heras *et al.*, 2020). The correlation between open water porosity and HLD in marbles results less investigated from other studies (Çelik & Çobanoğlu, 2019). The Figure 2 shows an exponential correlation for both marbles with coefficients of determination (R^2) respectively of: $R^2 = 0.9613$ for MF and $R^2 = 0.9632$ for MC. The decrease of Ultrasonic Pulse Velocity and Leeb Hardness with the increase in porosity and, consequently, with thermal-induced decay are both significant and leaded by an exponential equation. Correlation between UPV and HLD shows a function that follows a linear trend for both marbles with high R^2: respectively 0.9367 for MF and 0.9579 for MC.

Table 1. UPV, LEEB and open porosity.

Samples		TD-A	TD-B	TD-C	TD-D	TD-E
MF	UPV	3865	1971	1233	978	696
	Std. dev.	*241*	*178*	*232*	*101*	*111*
	HLD	575	478	425	390	335
	Std. dev.	*20*	*21*	*16*	*14*	*11.1*
	Ab_v (% by vol.)	0.27	1.05	2.04	2.5	3.84
	Std. dev.	0.04	0.04	0.04	0.04	0.04
MC	UPV	5011	4153	3050	2629	1488
	Std. dev.	*347*	*322*	*242*	*147*	*121*
	HLD	487	458	416	356	318
	Std. dev.	*27*	*49*	*31*	*19*	*24*
	Ab_v (% by vol.)	0.2	0.98	1.04	1.92	3.37
	Std. dev.	*0.01*	*0.01*	*0.01*	*0.01*	*0.01*

TD-(A-B-C-D-E): Thermal Induced Deacys A, B, C, D, E were obtained using a muffle furnace by leaving the samples for 2 hours at the following temperatures: A = 60 °C, B = 200 °C, C = 300 °C, D = 400 °C, E = 500 °C. Measurements were taken once the samples cooled to room temperature (25 °C).

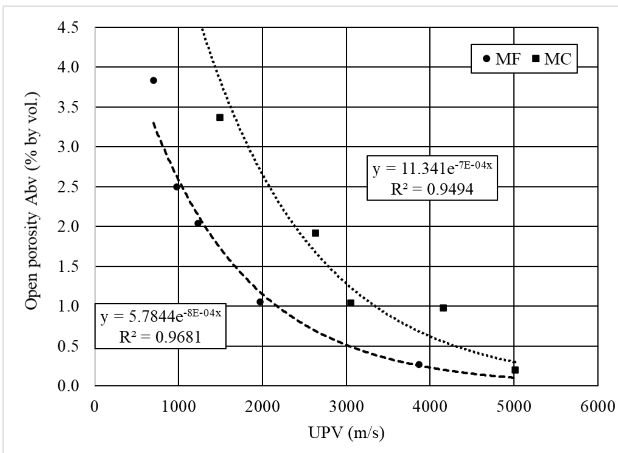

Figure 1. Ultrasonic Pulse Velocity UPV vs Open water porosity Ab_v (% by vol.).

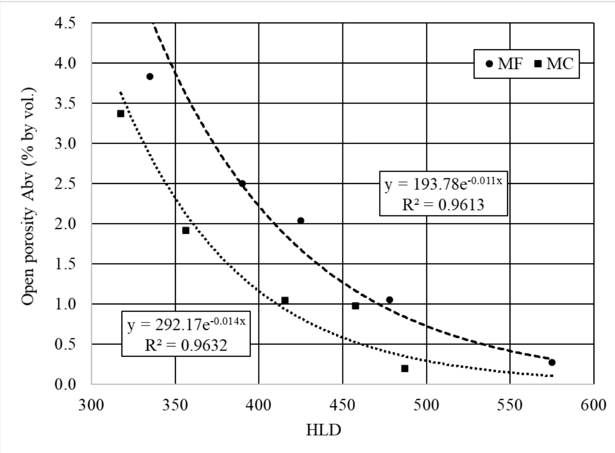

Figure 2. Leeb Hardness HLD vs Open water porosity Ab_v (% by vol.).

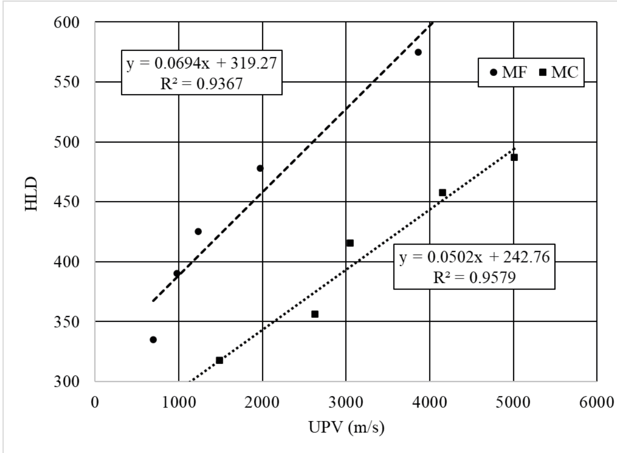

Figure 3. Ultrasonic Pulse Velocity UPV vs Leeb Hardness HLD.

Conclusions

The present study confirms that thermal treatments generate increasing porosity in marble. Porosity is confirmed to be an excellent parameter for evaluating the artificial marble decay. Each marble reacts differently to the thermally induced degradation, essentially due to the different petrographic characteristics, showing different porosity values for the same intensity of thermal degradation in different samples. UPV method is useful to determine the increase in porosity in a non-destructive way. A decrease in UPV speed is attributable to an increase in porosity. UPV measurements results a powerful and sensitive method for measuring degradation in marbles. Also, HLD method can be used for determining the increase in porosity in a non-destructive way, and the decreasing in hardness is strictly related to an increase in porosity. In conclusion NDT as UPV and HLD can be considered valid tools for the quick and in situ study of marbles decay.

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