IJCS
INTERNATIONAL JOURNAL OF CONSERVATION SCIENCE
ISSN: 2067-533X Volume 11, Special Issue 1, 2020: 271-276 www.ijcs.uaic.ro

ULTRASONIC NONDESTRUCTIVE TESTING OF BUILDING MATERIALS FOR THE DIAGNOSIS AND CHARACTERIZATION OF THE DECAY DUE TO SALTS CRYSTALLIZATION

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Abstract

The possibility to use ultrasonic nondestructive testing for the diagnosis and characterization of decay in building materials due to salts crystallization is here investigated. Salts crystallization is one of the main sources of decay for stone materials, especially for cultural heritage items underwater or exposed to water, and its early detection as well as a continuous monitoring is highly desirable. Currently, this often requires the use of destructive methods and the sampling of the items under inspection. This work shows promises to the successful use for this purpose of ultrasonic nondestructive testing.

Keywords*: Ultrasonic; Nondestructive evaluation; Salt crystallization; Building materials.*

Introduction

Calcareous rocks constitute the main type of stone used as building materials for World's architectural Cultural Heritage. Unfortunately, different degradation and alteration processes, such as salts crystallization, erosion, dissolution, biological attack, often affect these kinds of substrates. Between them, salt crystallization represents the amin cause of degradation of porous building stones [1]. Crystallization pressure of salt crystals is related to different alteration and degradation phenomena. In particular, from a macroscopic point of view, it is possible to recognize different degradation forms such as erosion, loss of material, cleavage, and exfoliation and, in some cases, even complete disaggregation of the material. The damage produced is linked to different factors: type and quantity of salt in the stone, the characteristics of the pores and the environmental parameters.

The evaluation of salts crystallization is therefore a fundamental element in the monitoring and diagnosis of built Cultural Heritage, especially those subject to aging and degradation processes. The techniques most commonly used for this purpose are: optical microscopy to define their mineralogical and textural features; colorimetric test to evaluate chromatic variations associated with salt crystallisation; mercury intrusion porosimetry and calculation of crystallization pressure in order to investigate the relations between the crystallisation pressures of salts and the intrinsic characteristics of stones. In this paper it is investigated the possibility to

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use ultrasonic nondestructive testing for the diagnosis and characterization of decay in building materials due to salts crystallization. The aim is to have a nondestructive, easy-to-use, and quantitative method capable of detecting salts crystallization in its early stage and of providing a measure of the extent of the phenomenon. There are some challenges to face in doing this, since porous materials are highly scattering for ultrasound propagation, so that a high attenuation of the ultrasonic beam inside the sample is found. To tackle this problem, the use of the pulse-compression technique is here exploited [2-4]. The work is developed in different sections: Section II introduces the theoretical background of the pulse-compression measurement scheme; Section III outlines the experimental setup and the specimens analysed; Section IV illustrates the results obtained and eventually Section V draws some conclusions.

Pulse compression fundamentals

Interesting features of the Sample Under Test (SUT) can be extracted by measuring its impulse response $h(t)$ over a mechanical excitation, here being at ultrasonic frequencies. In standard Pulse echo (PuE) method, the impulse response $h(t)$ is estimated by exciting the SUT with a short pulse $\tilde{\delta}(t)$ and then recording the system's response $\tilde{h}(t) = \tilde{\delta}(t) * h(t)$, where "$*$" is the convolution operator. If $\tilde{\delta}(t)$ covers uniformly the whole bandwidth of the transducers, the approximation can be considered very close to the true expected signals.

On the other hand, in a pulse-compression (PuC) measurement scheme an estimate $\hat{h}(t)$ of $h(t)$ is retrieved by processing the output of the system to a proper excitation. In more details, this is achieved by (i) exciting the system with a coded signal $x(t)$; (ii) measuring the output of the coded excitation $y(t) = x(t) * h(t)$; (iii) applying the so-called matched filter $\psi(t)$ to the output [2-5]. The result obtained at the end of the PuC procedure is mathematically described in Eq. (1):

$$\hat{h}(t) = \psi(t) * y(t) = \underbrace{\psi(t) * x(t)}_{\hat{\delta}(t)} * h(t) = \hat{\delta}(t) * h(t) \approx h(t) \qquad (1)$$

where the "pulse-compression condition" $\psi(t) * s(t) = \hat{\delta}(t) \approx \delta(t)$ has been exploited. As in most of the applications, in the present paper the matched filter is defined as the time-reversed replica of $x(t)$, $\psi(t) = x(-t)$, so that $\hat{\delta}(t)$ turns out to be the autocorrelation function of $s(t)$. In in the case of ultrasonic inspection, the most used waveform is the Linear Chirp (LC) that is the signal employed also for the present application.

LC is described by the expression [5]:

$$s(t) = A(t)sin\big(\Phi(t)\big) = A(t)sin\left(2\pi\left(f_1 t + \tfrac{f_2 - f_1}{2T}t^2\right)\right) = A(t)sin\left(2\pi F_c\left(\left(1 - \tfrac{B_{\%}}{2}\right)t + \tfrac{B_{\%}}{2T}t^2\right)\right) \qquad (2)$$

where T is the duration of the chirp signal, f_1 is the start frequency, f_2 is the stop frequency, $F_c = \big(f_1 + f_2\big)/2$ is the centre frequency and $B_{\%} = \big(f_2 - f_1\big)/F_c$ is the percentage bandwidth of the chirp. Note that T and B are not constrained by each other, so that the duration of the LC can be increased arbitrarily. $A(t)$ is a time-windowing function that modulates the amplitude of the chirp and $\Phi(t) = 2\pi F_c\left(\left(1 - \frac{B_{\%}}{2}\right)t + \frac{B_{\%}}{2T}t^2\right)$ is the chirp phase function that determines the instantaneous frequency of the signal accordingly with $f_{ist}(t) = {\Phi'(t)}/{2\pi}$.

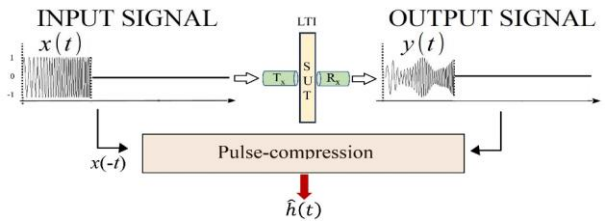

Fig. 1. Schematic diagram of the pulse-compression measurement procedure

Experimental setup and samples

The experimental setup used for the UT measurements consisted of: (a) a PC units equipped with Labview and MATLAB software used respectively for the managing of the data acquisition system and for the data processing; (b) a TiePie HandyScope 5 from TiePie Engineering used as Arbitrary Waveform Generator (AWG) and as Digital Oscilloscope (SCP); (c) an ultra-low noise variable gain-amplifier AD3881 from Analog Device with embedded a low-noise amplifier stage; (d) a pair of V109 Panametrics-NDT ultrasonic transducers from Olympus, with a 1/2 inch active element, a centre frequency of 5 MHz and an ultrawide bandwidth. The block diagram of the pulse-compression measurement procedure is illustrated in Fig.1: two probes were used in through-transmission configuration, to reduce attenuation with respect reflection pitch-catch configuration and to fit the samples dimensions, which were about 4cm × 4cm × 2cm, see Fig. 2 for the picture of the experimental setup and of a standard sample. A rubber plastic layer was placed on both transducers to act as coupling layer between transducers faces and samples. The input signal of the Tx transducer is a chirp signal, the estimate of the impulse response is retrieved by applying the pulse-compression procedure between the output and the input signal.

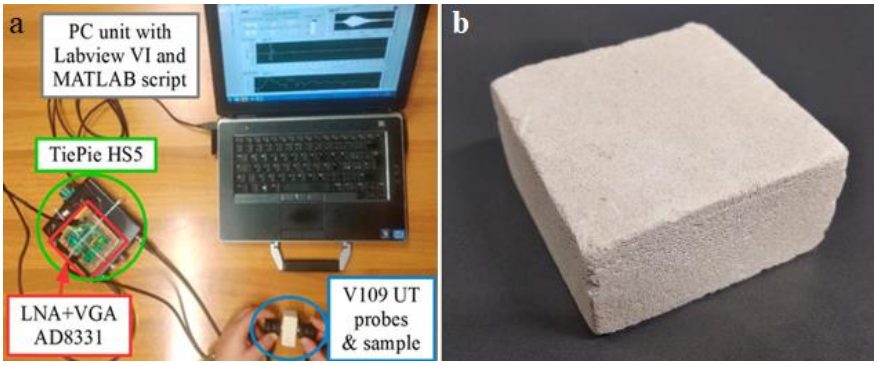

Fig.2. Experimental setup(a) and Representative specimen of "Pietra di Lecce" stone (b)

The samples investigated were made of "Pietra di Lecce" stone, from the Puglia region in the south of Italy. Starting from a unique block, various samples were realized and then they underwent to different treatments and aging procedures, as detailed in Table 1. The specimens were treated by using two consolidating products and then artificially aged by salt crystallization tests in order to define susceptibility to weathering.

Regard the products, two commercial suspensions were used: a) one of nanosilica (Nano Estel®), b) one of nanolime (CaLoSiL®). Each formulation has been applied on stone samples (5×5×2 cm) by brushing until they were getting saturation. During the test, untreated samples were used to make a comparison with treated specimens. Lastly, after the consolidation of the stones, the salt crystallization test was performed on untreated and treated samples in oder to

establish the resistance of materials to weathering [6-7]. The experimental set up used for salt crystallization test is described in the standard EN 12370:2001 [8], revised according to [7]., In particular, samples were subjected to several crystallization cycles consisting of: a) 2 h of immersion in a supersaturated solution of sodium sulfate (14 % w/w at 20°C) for 10% of their height, b) 8 h of drying in an oven at 45°C, and c) 16 h of cooling at room temperature. After consolidation treatment and salt crystallization test part of the specimens were stored in an environmental chamber where the temperature varied between 18 and 20 °C and the relative humidity between 55% and 75%.

Table 1. Experimental treatments on "Pietra di Lecce" stone

Sample code	Product	Aging test
PL1		Environmental test chambers (UV, Humidity and Temperature)
PL2	Nano silica	Salt weathering (Na_2SO_4)
PL3		Consolidated
PL4		Environmental test chambers (UV, Humidity and Temperature)
PL5	Nanolime	Salt weathering (Na_2SO_4)
PL6		Consolidated
PL7	Untreated	Untreated
PL8		Salt weathering (Na_2SO_4)

All the samples were firstly measured before any treatment/aging, then the measurements were repeated at any further step of sample processing (e.g. sample PL3 was measured just after the cutting, then after the treatment with Nano-silica, and finally after the aging with salt weathering). All the measurements were made by using the same parameters regulating the chirp generation, the VGA gain and the AWG output amplitude.

In particular, the chirp signal used had a centre frequency f_c of 1.5 MHz and a bandwidth equal to the 195% of f_c. The sample frequency was 20 MSamples/s and the length of the chirp was equal to 80000 samples, corresponding to a duration of 4 ms.

Experimental results

Three features were extracted by the UT measurements: (F1) the ultrasound velocity, see Fig.3, (F2) the centre frequency of the output signal spectrum, Fig.4, and (F3) the SNR of the 1st transmission peak through the sample at the centre frequency, Fig.5. The ultrasound velocity was estimated by fitting the 1st peak of the output signal envelope with a parabolic function. The centre of the parabola indicates the Time-of-Flight of the 1st passage in transmission.

At this value, the ToF measured with only the double rubber plastic layer was subtracted.

The velocity values measured are reported in Fig. 3. In general, the ultrasound velocity varied not so much among all the measurements, while a slight increase of the velocity seems to be found for samples treated with nanosilica, for which the velocity shows an increase in all samples and also after aging. Anyway, both the limited statistics and the slight increase evidenced that the ultrasound velocity doesn't seem a key feature to detect, monitor and estimate decay due to salt crystallization.

The centre frequency was evaluated by applying a Gaussian fit over the spectrum of the output signal. In the case of this feature, a clear difference between the values measured for the sample that underwent the salt weathering aging is evident as illustrated in Fig.4.

The same trend was found for the SNR feature, calculated as the difference in db scale between the amplitude of the 1st peak and the average noise level for $t \rightarrow \infty$, i.e. for a time at which any signal is exhausted, Fig.5. Please note that before the calculation of the SNR a bandpass filter was applied to all data, with centre frequency 1.5MHz and bandwidth of 300kHz. This was done to measure the SNR in a limited bandwidth. Indeed, the SNR is related to the signal attenuation, which in turn is frequency dependent. To have a clear definition of the SNR the bandpass filter

was then applied.

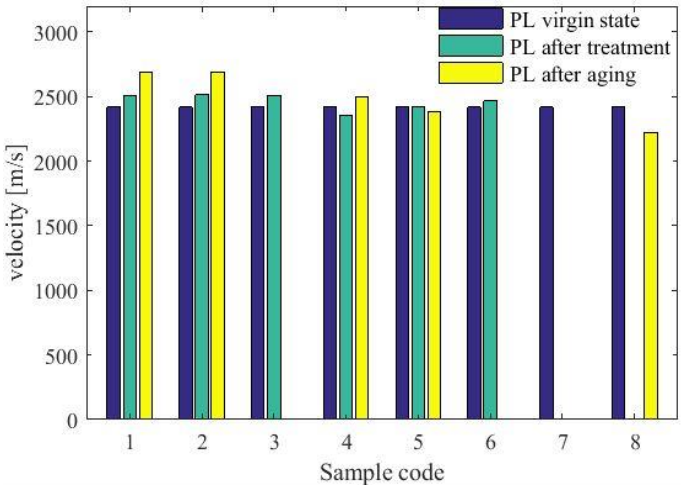

Fig. 3. Velocity values measured with the setup depicted in Fig.2

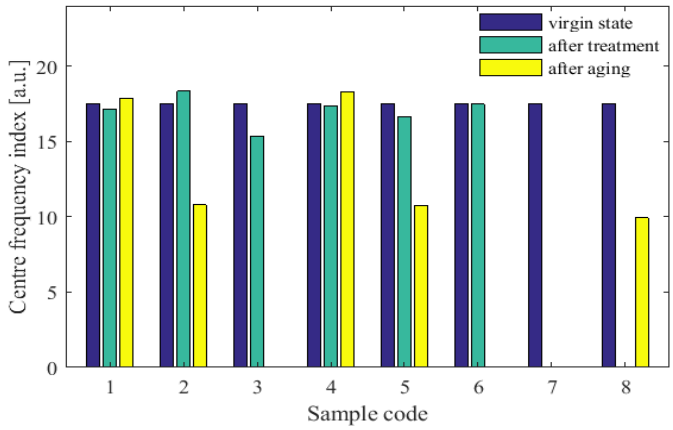

Fig. 4. Centre frequency of the output signal spectrum

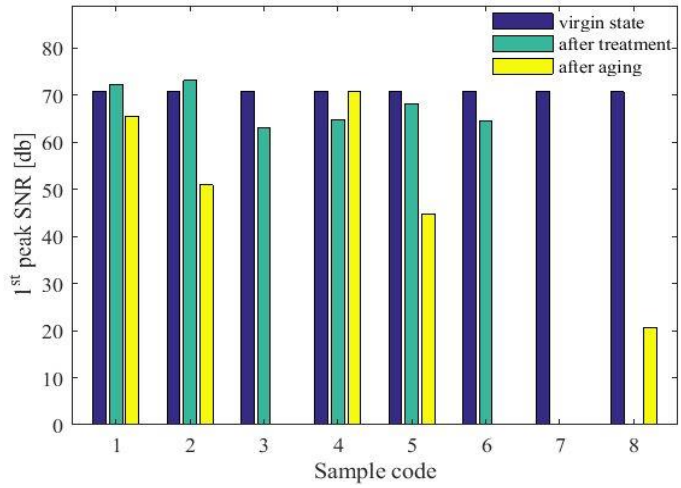

Fig. 5. 1st peak SNR value with respect noise background at $t \to \infty$

Conclusions

The results of the analysis of the ultrasonic data, although preliminary, seem indicate that spectrum analysis and SNR evaluation can provide a clear indication of the onset of salt crystallization in the porous material here investigated. Further analysis will be undertaken to increase the statistics of the data and to repeat the analysis after various stages of ageing treatments in salt weathering.

The extension of the research to use at other different treatments will be also considered [9-11].

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Received: December 7, 2019
Accepted: Febryary 24, 2020