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CORRELATION OF STONE PROPERTIES USING STANDARDIZED METHODOLOGIES AND NON-STANDARDIZED MICRO-DESTRUCTIVE TECHNIQUES

Magdalini Theodoridou[1], Fabrice Dagrain[2] and Ioannis Ioannou[1]

[1] *Department of Civil and Environmental Engineering, University of Cyprus*
[2] *Department of Civil Engineering and Structural Mechanics, University of Mons*

Abstract

The characterization of masonry heritage materials is of utmost importance in the diagnosis of their pathology and state of conservation. Standardised experimental methods which demand controlled laboratory conditions are limited in their applications by the size of test specimens. This limitation is even tighter when experimentalists deal with heritage materials. Therefore, alternative techniques have recently been developed for the micro-destructive evaluation of certain stone properties, either in-situ or in the laboratory. In this study, the drilling and scratching micro-destructive tests have been used alongside a series of standardised laboratory tests aiming at the correlation of various stone properties (density, porosity, dynamic elastic modulus and uniaxial compressive strength). The results prove the potential of the two techniques in assessing the uniaxial compressive strength of the materials based on their intrinsic specific energy and drilling resistance. Furthermore, sound correlations are established between the mechanical properties of the test specimens and their respective densities and porosities. Thus, the two novel micro-destructive techniques are shown to be efficient in the physico-mechanical characterization of materials. As such, their use is strongly recommended for standardization and/or the evaluation of the characteristics of heritage masonry materials, which demand in-situ measurements or allow very limited sampling.

Keywords: stone characterization, micro-destructive techniques, DRMS, scratch tool

1. Introduction

Extensive literature referring to the physico-mechanical characterization of natural stones and possible correlations between their various properties exists and data that give a clear picture of the microstructure and the behaviour of different types of rocks have been reported (e.g. Yaşar et al 2010; Kasthurba et al 2008). Empirical equations relating the compressive strength and friction angle of various types of rocks to their physical properties have been proposed by Chang et al (2006) while the influence of porosity on the mechanical behaviour of stones has been examined by Palchik (1999) and Moh'd (2009). Moreover, research to determine the effects of the mineral grain size on the strength of certain building stones has also been undertaken (e.g. Yilmaz et al 2009; Yılmaz et al 2011).

Experimental methods for the physico-mechanical characterization of natural stones are mostly based on standardised techniques which allow the determination of the materials' properties under controlled laboratory conditions. When the main aim is the characterization of stones which are incorporated into existing structures, however, standardized laboratory methods are limited in their application by the size and number of samples that can be collected on-site. This can pose considerable problems when the structure under study is a monument and/or an object of significant cultural importance. Therefore, alternative experimental techniques have also been developed for the non- or micro-destructive evaluation of certain material properties. Non- and micro-destructive tests can be implemented either in situ or in the laboratory using very small samples.

One of the most widely used non-destructive methods aims at the assessment of the dynamic modulus of elasticity of stone using either ultrasonic pulse velocity or fundamental resonance frequency measurements. The methods of determination of stone sound speed propagation and dynamic modulus of elasticity using the fundamental resonance frequency have already been standardised (EN 14579 and EN 14146 2004). Two very promising non-standardised micro-destructive techniques developed recently are the drilling and scratching tests. These are mainly applied for the determination of the mechanical properties of materials. The general concept of both methods lies within the forces needed to destroy material by cutting or drilling mechanisms; these are related to the mechanical properties of the material and the technological parameters applied on the tool (Dagrain et al. 2010b). Therefore, for given testing configuration, the only parameter influencing the forces applied is the strength of the material.

For the purposes of this study, a wide collection of various types of natural stones were subjected to a series of standardised laboratory tests in order to determine their physico-mechanical properties: apparent density and open porosity, dynamic modulus of elasticity and uniaxial compressive strength. Additional results regarding the mechanical behaviour of the materials were obtained by the application of the two innovative micro-destructive testing methods mentioned above: scratching and micro-drilling. The experimental work has enabled the investigation of possible correlations between the various physical and mechanical properties of stones, as well as the evaluation of the applicability of the micro-destructive methods for the determination of natural stone properties.

2. Materials

A broad collection of 50 different natural building and decorative stones was used for the purposes of this study. The wide range of the physico-mechanical properties of the studied materials provides further value to the correlations deduced and, hence, to the evaluation of the applicability of the two micro-destructive techniques under study in the characterisation of natural stone.

3. Methodology

3.1 Standardised methodologies

The apparent density (ρ_b) and open porosity (ρ_o) of the stone samples were measured through vacuum assisted water absorption and submerged weighting, following the testing methodologies suggested by EN 1936 (2006). The dynamic modulus of elasticity (E_d) was estimated by a series of measurements of sound speed propagation, following the procedure described in EN 14579 (2004). Last but not least, a series of destructive tests was performed for the determination of the uniaxial compressive strength of all stones, taking into account the recommendations of EN 1926 (2006).

3.2 Non-standardised micro-destructive techniques

3.2.1 Scratching test

The scratching test, originally developed at the University of Minnesota, is based on the incremental formation of a shallow groove (depth of cut, $d = 0.01 – 2$ mm) on the surface of the material, using a sharp rectangular diamond cutter with a width, $w =$ 10 mm and a negative back rake angle of 15º moving at constant velocity, v. During the measurement, the tangential ($F^c{}_s$) and normal ($F^c{}_n$) components of the total applied force on the cutter are recorded (Figure 1). The output of the test is the evolution (log) of the magnitude of the two force components. According to Mitaim et al (2004) and Schei et al (2000), through the implementation of scratch tests, two strength parameters can be extracted for natural stones: (i) the intrinsic specific energy, which can readily be interpreted as the energy required for scratching a unit volume of a material (Suarez-Rivera et al 2002), and which can be related to the uniaxial compressive strength and (ii) the friction coefficient which is related to the materials internal friction angle.

It is worth noting that, at shallow depths of cut (typically less than 1 mm), the test specimen is intensively sheared ahead of the cutter and crushed at the tip. In this case, the scratching mode can be described as ductile (Dagrain and Germay 2006). Research (Almenara and Detournay 1992, Detournay et al 1995; Adachi et al 1996) has shown that, in the ductile regime, the cutting force F^c applied on the cutter face is proportional to the cross-sectional area of the trace that is formed during the test ($w{\cdot}d$). The tangential ($F^c{}_s$) force is equal to εwd, while the normal ($F^c{}_n$) force is equal to $\zeta\varepsilon wd$, where ε denotes the intrinsic specific energy (MPa) and ζ characterizes the inclination between the total force F^c and the direction of the cutter motion (Figure 1).

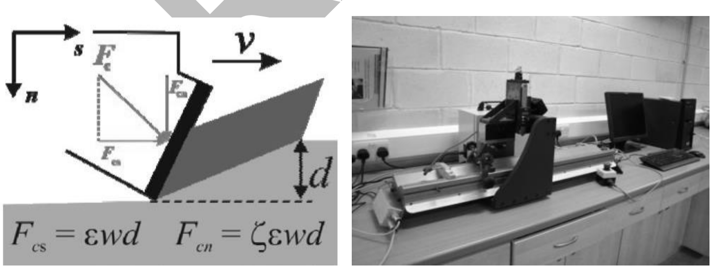

Figure 1. Schematic view of the cutting procedure during the implementation of a scratch test (Dagrain & Germay, 2006) (left). The Epslog Engineering WOMBAT Scratch Tool (right).

In this study, the specimens were rectified prior to testing using the machine's cutter. After levelling their surface, a series of 6 to 10 scratches at different depths of cut were traced on them in order to assess their intrinsic specific energy. The depth of cut was gradually raised incrementally from 0.1 mm to 1.2 mm. Data analysis was performed using Epslog Engineering software. The average values of the forces recorded during the formation of each scratch were computed and $F^c{}_s$ vs d diagrams were plotted. The intrinsic specific energy of each test specimen was calculated from the slope of the $F^c{}_s$ vs d diagram. Data that were not in context with the ductile scratching mode were excluded from the analysis. In all cases, data corresponding to at least four scratches conducted at different depths of cut were used for assessing the intrinsic specific energy.

In using the scratch test, particular attention has been given to the correlation of the intrinsic specific energy to the unconfined compressive strength of natural rocks. Previous work suggested that this is equal to 1:1 (Almenara and Detournay 1992; Detournay et al 1995; Adachi et al 1996). Due to the fact that the scratch test enables the monitoring of force signals along the profile of a material, it has also been used for the examination of materials that have sustained damage or have inherent inhomogeneity. It has been applied for the detection of faults and damage in refractory bricks and for the localized investigation of the properties along the matrix of concrete materials (Dagrain and Germay 2006). In addition, continuous scratch measurements have been used for the quantitative evaluation of rock heterogeneity (Dagrain and Germay 2006) and for the characterization of historic mortars (Dagrain et al 2010a). One of the latest applications of the scratching test is described by Modestou et al (2012) and refers to the ability of the technique to detect crystallised salts within porous materials.

3.2.2 Micro-drilling test

The determination of drilling resistance is carried out using an innovative non-standardized micro-destructive technique and a purposely made Drilling Resistance Measurement System (DRMS). The DRMS (Figure 2) is produced by SINT Technology (Italy). The instrument has been developed and validated within the European EC Hardrock Project (Tiano 2001). It is a cordless portable system and can therefore be used both in the laboratory and in situ. It comprises of the drilling device and a laptop, where the acquired data is transmitted, saved and presented, using a software developed with LabVIEW™. A special diamond drill bit is mounted on the system's drilling device. The system can measure the actual drill position, the penetration force, the rotational speed and the penetration speed.

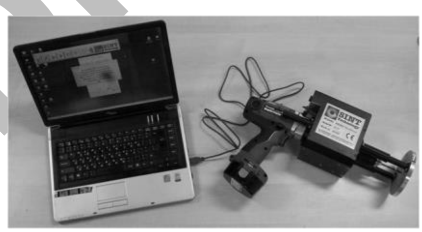

Figure 2. The Drilling Resistance Measurement System (DRMS).

For the determination of the drilling resistance, small holes, 5 mm in diameter, are drilled on the surface of the material examined. During the test, both the rotational speed and penetration rate are maintained constant. These rates are established by the operator before the initiation of the drilling procedure and can range from 20 to 1000 rpm and 1 to 80 mm/min respectively. During the penetration of the drill bit, the DRMS provides a continuous reading of the force required to penetrate a certain depth and records the time elapsed. The value of the applied force that can be measured by the system is between 0 to 100 N. The outputs of the test are given in x-y plots of the drilling force along the depth profile, while the data is registered in numeric values as well.

As mentioned by Exadaktylos et al (2000) the application of the drilling test can vary from the in situ assessment of stone quality, both at a quarry and on an existing structure, to the determination of weathering extent in depth on buildings. Research attempts have been made for correlating drilling resistance with the physico-mechanical properties of stones. Fratini et al (2006) indicated that the drilling resistance, as measured by the DRMS, can be correlated with the porosity and compressive strength of natural building stones. Exadaktylos et al (2000) have established a comparison between the uniaxial compressive strength and the drilling strength, while Wendler and Sattler (1996) have found indications correlating the drilling strength and the biaxial flexural strength. Comparisons between the drilling strength of minerals and Mors hardness have also been reported (Pamplona et al 2007). Furthermore, the drilling test has been applied for the evaluation of consolidating treatments in monuments (Tiano et al 2000) and for monitoring damage within the body of materials (Felicetti 2006). Drilling tests have also been used for the characterization of mortars (Dagrain et al 2010a), the evaluation of the compressive strength of ancient clay bricks (Fernandes and Lourenço 2007) and the micro-destructive mapping of salt crystallisation in stone (Modestou et al 2012).

In this study, drilling resistance measurements were performed on all types of stone examined. The operating conditions during the tests were 600 rpm for the rotational speed and 10 mm/min for the penetration rate. The diameter of the diamond drill bit used was 5 mm and the total depth of penetration was 10 mm. As in the case of the scratch test, the direction of drilling was the same as the direction of application of compressive load during the implementation of uniaxial compressive strength tests, so that the results obtained could be comparable.

4. Results and Discussion

The densities of the stones under study ranged between 1515 – 2650 kg/m^3, while their porosities (P_o) lied between 1.5 – 43%. The dynamic modulus of elasticity (E_d) ranged between 5 – 58 GPa and the uniaxial compressive strength (R) between 6 - 125 MPa. The implementation of scratching test yielded estimates of the materials' intrinsic specific energies (ε) between 6 and 137 MPa. The average drilling forces (F) recorded during the drilling resistance measurements were between 0.7 – 83 N.

Correlation analyses were applied to investigate whether the individual properties measured (uniaxial compressive strength, dynamic modulus of elasticity, open porosity and apparent density) relate to the intrinsic specific energy determined by scratching and the drilling resistance measured by the DRMS and, hence, to evaluate whether these micro-destructive tests can be used in the physico-mechanical characterization of stone. The analyses were undertaken using Microsoft Office Excel. It should be noted that none of the results was excluded from the calculation of the mean values used in the analyses. The correlation coefficients (R^2 parameter values) and best fit curves were computed by the 'least squares curve fit' method. According to this method, a line is fitted through the points examined, so that the squared deviations of the points from that line are minimized. Linear and other type of relationships were considered between the materials properties. The results are presented in Figures 3 and 4. A possible correlation between the intrinsic specific energy and the drilling resistance was also examined and this is shown in Figure 5. All relations with R^2 parameter values > 0.7 were considered statistically significant, whereas those with $R^2 \geq 0.85$ were considered to show strong interdependence between material properties.

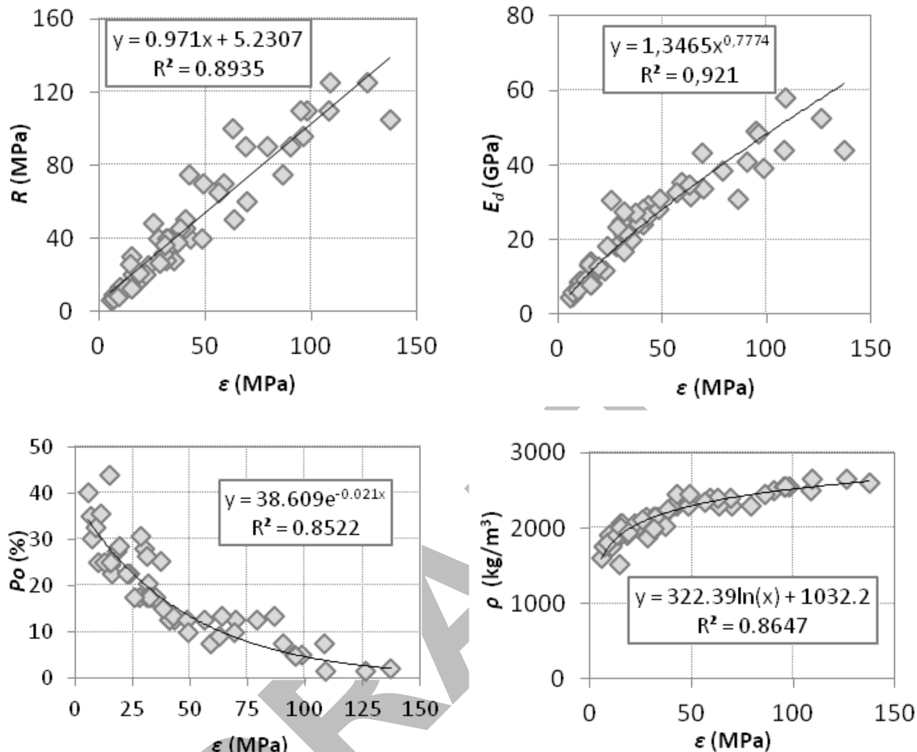

Figure 3. Correlation of the scratching test results, expressed by the intrinsic specific energy (ε), with uniaxial compressive strength (R), dynamic modulus of elasticity (E_d), open porosity (P_o) and density (ρ).

From Figure 3, it is obvious that there is a strong correlation ($R^2 > 0.85$) between the intrinsic specific energy and all other properties measured. A linear relationship very close to 1:1 is observed among the values of intrinsic specific energy and the mean values of uniaxial compressive strength. This agrees well with the literature (Almenara and Detournay 1992; Detournay et al 1995; Adachi et al 1996). The strong dependence of the intrinsic specific energy on the physical characteristics of stone, such as density and porosity, confirms that the resistance of a material to cutting is influenced, not only by the bonding between its grains (Huang and Detournay 2008), but also by the size and volume of pores within its structure. This also explains the good correlation found to exist among intrinsic specific energy and dynamic modulus of elasticity, evaluated from sound speed propagation measurements. Theoretically, the velocity with which ultrasound pulses are transmitted through rocks (and hence the estimated value of dynamic modulus of elasticity) depends upon the density of the material and its elastic properties (Goodman 1989). Taking into consideration that these parameters also affect the cutting strength of stone and bearing in mind that ultrasound and scratch tests were conducted along the same length of specimen, the relationship between intrinsic specific energy and dynamic modulus of elasticity may be deemed logical.

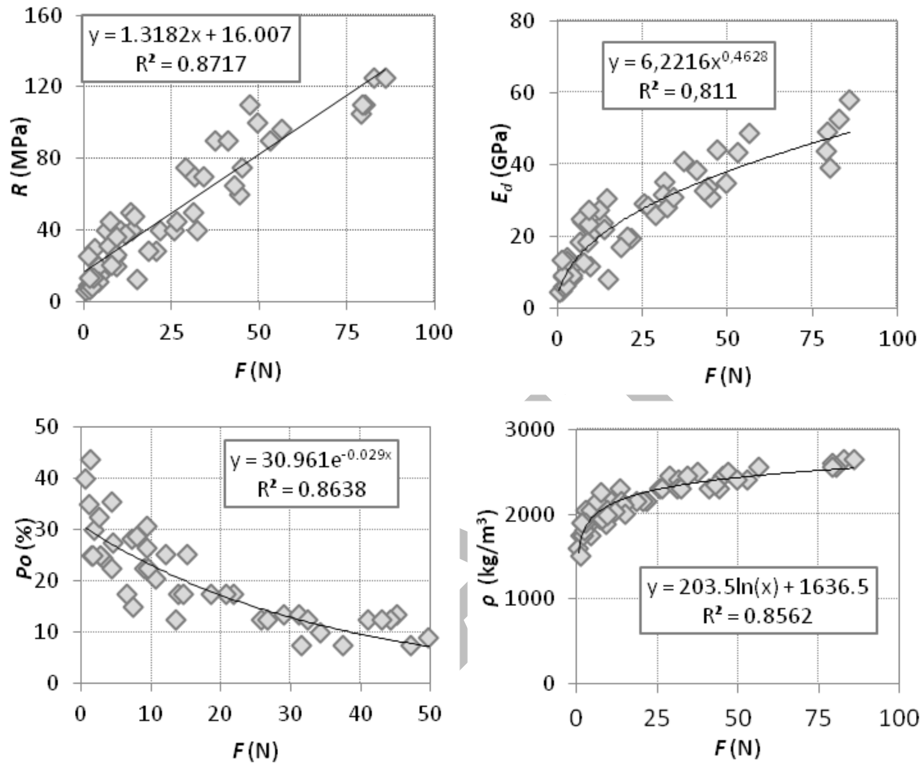

Figure 4. Correlation of the micro-drilling results, expressed by drilling resistance force (*F*), with uniaxial compressive strength (*R*), dynamic modulus of elasticity (E_d), open porosity (P_o) and density (ρ).

In Figure 4, a high correlation coefficient is observed for the relation between drilling resistance and uniaxial compressive strength. Therefore, sufficiently accurate predictions of compressive strength could be yielded via the portable, micro-destructive application of the micro-drilling test. Exadaktylos et al. (2000) have also managed to establish sound comparisons between uniaxial compressive strength and drilling resistance, while Fernandes and Lourenço (2007) have successfully used drilling measurements for determining the compressive strength of clay bricks. Strong associations are also shown between drilling resistance and the rest of the properties measured. This can be explained by the fact that, during the interaction of the drilling bit with the material's matrix, voids such as pores can affect the measurements taken. Similarly to the case of the intrinsic specific energy, linear and power fits were computed for the best correlations of drilling resistance with uniaxial compressive strength and dynamic modulus of elasticity accordingly, while exponential and logarithmic regression lines fitted better in the cases of open porosity and density respectively.

The comparison between the values of the two micro-destructive, non-standardised techniques (Figure 5) exhibits a correlation coefficient of about 0.84. This high interdependence of the two techniques can be explained by the relative nature of the results, which in both cases refer to the materials' resistance to cutting, even though the mode of cutting differs (scratching ≠ drilling).

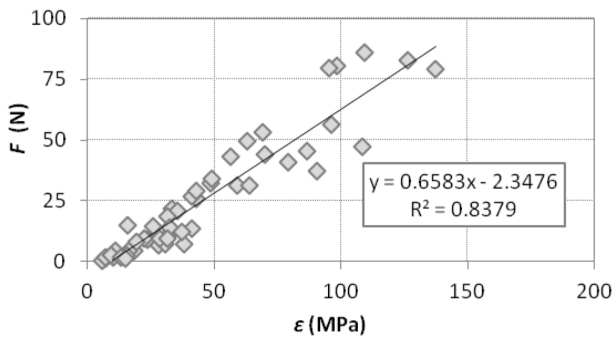

Figure 5. Correlation of the micro-drilling results, expressed by drilling resistance force (*F*), with scratching test results, expressed by the intrinsic specific energy (*ε*).

5. Conclusions

A complete knowledge of the physical and mechanical properties of natural stones is essential for full material characterization for various engineering applications. The use of non- and micro-destructive tests for the evaluation of material properties is of crucial importance in cases where the availability of test specimens is limited and/or the structure's fabric is of historic importance.

Within the framework of this study, laboratory testing was carried out on 50 different natural stones, aiming to examine the correlation of the results of two of the most innovative micro-destructive testing methods, the scratching and drilling tests, with various physico-mechanical properties such as apparent density, open porosity, dynamic modulus of elasticity and uniaxial compressive strength. Very strong interdependence was observed between the results of these two micro-destructive tests and the aforementioned properties determined by standardized laboratory tests. A further correlation of high statistical importance was detected between the results of the two micro-destructive techniques themselves. The observations made can clearly be considered as indications that the scratch tool and the portable drilling system could be employed for the evaluation of stone properties, especially in cases where sampling is limited (e.g. cultural heritage objects and architecture). Further research and enhancement of the database is expected to better validate the regression lines of all possible correlations. Finally, these very positive results bring up the need for standardisation of the two micro-destructive techniques; this will facilitate comparison of the experimental findings internationally.

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