Focussed on Recent advances in "Experimental Mechanics of Materials" in Greece

Pre-failure indicators detected by Acoustic Emission: Alfas stone, cement-mortar and cement-paste specimens under 3-point bending

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ABSTRACT. Acoustic Emission (AE) is the technique most widely used nowadays for Structural Health Monitoring (SHM). Application of this technique for continuous SHM of restored elements of stone monuments is a challenging task. The co-existence of different materials creates interfaces rendering "identification" of the signals recorded very complicated. To overcome this difficulty one should have a clear overview of the nature of AE signals recorded when each one of the constituent materials is loaded mechanically. In this direction, an attempt is here described to enlighten the signals recorded, in case a series of structural materials (natural and artificial), extensively used for restoration projects of classic monuments in Greece, are subjected to 3-point bending. It is hoped that obtaining a clear understanding of the nature of AE signals recorded during these elementary tests will provide a valuable tool permitting "identification" and "classification" of signals emitted in case of structural tests. The results appear encouraging. In addition, it is concluded that for all materials tested (in spite their differences in microstructure and composition) clear pre-failure indicators are detected, in good accordance to similar indicators provided by other techniques like the Pressure Stimulated Currents (PSC) one.

KEYWORDS. Alfas stone; Mortar; Cement paste; Three-point bending test; Pre-failure indicators; Acoustic Emission.

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INTRODUCTION

For the restoration of fragmented structural elements of stone monuments a pioneering technique has been developed by the scientists working for the restoration of the Acropolis of Athens. According to this technique, the fragmented structural members are rejoined together by inserting threaded titanium bars into pre-drilled holes, which are then filled by a proper white cement paste [1]. However it has been observed that under specific loading conditions the adhesion between the cement paste and marble is sometimes not adequate permitting gradual or abrupt slip of the reinforcing bar from the body of the restored element, a phenomenon known as "pull-out". Taking into account that the construction of specimens in the form of structural elements of classical monuments is a difficult and costly task, an alternative experimental protocol was recently designed [2, 3], permitting laboratory investigation of the pull-out phenomenon (Fig.1). The specimens were made of Dionysos marble blocks drilled centrally throughout their length. The hole was filled with a cementitious material and a threaded titanium bar was driven into the hole while the filling material was still liquid. The experiments were implemented after a 28-day curing period. Using a properly designed metallic supporting system the marble was fully restricted (Fig.1c) and the bar was pulled-out under displacement-control mode (Fig.1d).

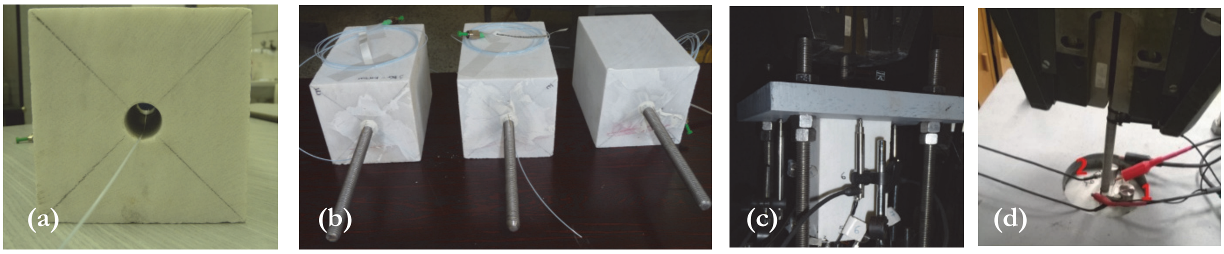


Figure 1: (a), (b) Typical pull out specimens; (c), (d) The experimental set up [2, 3].

The data obtained by the AE sensors during a typical pull-out experiment are somehow "chaotic" as it is seen in Fig. 2, where the AE recordings' duration is plotted (in conjunction to the marble-bar relative slip and also the force imposed by the frame) versus time. A direct interpretation of these data is difficult, especially concerning the sources of the emissions. In the direction of gaining a better insight it was considered that the AE technique should be first applied on specimens of simple geometry made of a single material. The protocol described here has proven that classifying the acoustic sources in such elementary tests is indeed a useful tool for the identification of the sources of acoustic signals in complex structures.

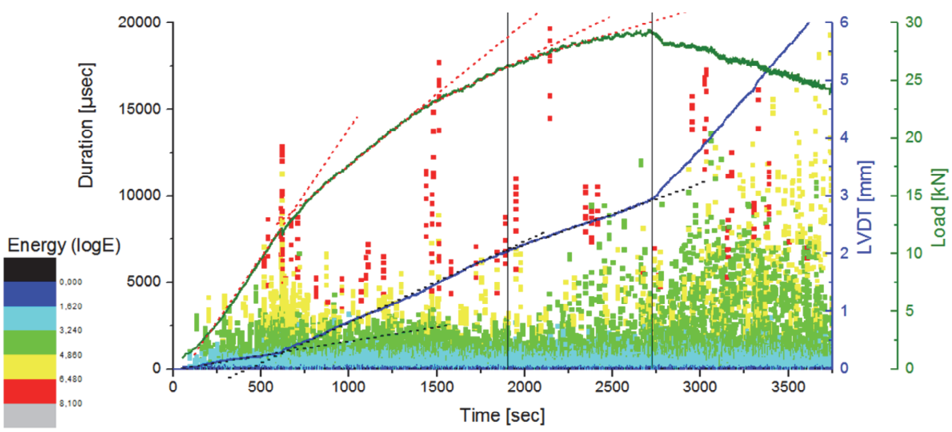


Figure 2: Time variation of the duration of AE recordings, the LVDT (in touch with the bar's lowest end) indications and the load [3].

THE EXPERIMENTAL PROTOCOL

The materials

The Alfas stone is a natural building material, quarried near the village of Alfas in the island of Crete. It was used as building material for quite a few monuments in Crete, both classical and modern, like for example the primary school at Scordilo village, built in 1884 (Fig.3a). Recently, during the construction of the Rethymnon-Ancient Eleutherna road, 128 ancient graves were discovered, all of them sculpted by Alfas stone. Nowadays Alfas stone is extensively used for the restoration of several monuments of great historical and cultural significance such as the

Monastery of Arkadi and the Fortezza fortress in Rethymno. It is also considered as a potential substitute stone for the restoration of the Epidaurus monuments.
When Alfas stone is mined it is relatively soft; however a short while after it is exposed to the sunlight it hardens. It is a compact sub-white stone of low porosity and rather homogeneous structure (Fig.3b). It is composed by 99% of calcite, 0.5% of quartz and 0.5% of aragonite [4]. Its main mechanical properties are recapitulated in Tab. 1 [4-7].

Figure 3: (a) Primary school at Scordilo village made of Alfas stone; (b) The compact texture of Alfas stone.

Modulus of elasticity	2.5-10.0 GPa
Uniaxial Compression Strength	15.0 - 36.8 MPa
Bending strength	7.4 - 10.4 MPa
Absolute density	2.45 g/cm^3
Bulk density	1.73 g/cm^3
Porosity	~30.0 (%)

Table 1: Mechanical properties of Alfas stone [4-7] (The values strongly depend on the point and depth of quarrying).

Two more materials were tested in the present protocol, i.e. a cement-mortar and a cement-paste. The specific materials are used as filling materials of the holes drilled and the grooves sculptured in the marble structural elements of the Acropolis of Athens monuments for the placement of metallic elements which either connect the epistyles to each other or restore the monolithic nature of fragmented structural members. These materials ensure the adhesion between marble and metal while in addition they protect the authentic building stone in case of overload [8]. According to the practice followed by the scientists working for the Parthenon's restoration project these filling materials are composed by coarse quartz sand (grain size: 1mm-2mm), fine quartz sand (grain size: 0.1-0.4 mm) and white Aalborg cement. The ratio for the mortar mix is 2 coarse quartz sand : 1 fine quartz sand : 1 cement. The quartz (silica) sand must be perfectly dry, clean and well graded. The sand's proportion of the containing SiO_2 is a factor of its quality. Quartz is known for its piezoelectric properties. The grains of the sand extracted from rivers are of perfectly spherical shape due to natural friction. Products composed by this sand exhibit excellent workability and elegant finished surfaces. The proper ratio of grain size ratio can provide several advantages to the final product such as the reduction of the gap between the grains, the reduction of the water absorption resulting to high water resistance, increased strength of the mortar and minimized cracking. In addition, quartz sand provides protection against corrosion due to poor environmental conditions. The Aalborg cement is a rapid hardening Portland cement with high early (2 days) and standard (28 days) strengths. It is produced of extremely pure limestone and fine-ground sand. It is characterized by its white color, high consistency, extraordinarily low content of alkali (Na_2O) and high sulphate resistance [9]. The composition for the cement-paste mix is 2.5 cement : 1 water, however it varies according to the needs of its specific application.

Acoustic Emission technique

Fracture is combined with release of stored elastic strain energy, consumed for the generation of new cracks. The elastic waves generated propagate in the material and can be detected by piezoelectric transducers mounted to the structure's surface by means of proper viscous materials [10]. The sensors' output is amplified through a low-noise preamplifier, filtered to remove any extraneous noise and further processed by proper electronic equipment. The AE method includes a wide range of applications such as laboratory experimental studies, field inspections, structural integrity evaluation, production quality control etc. The major difference between AE and other non-destructive techniques is that AE records signals due to the external application of load to the material. AE relies on energy produced by the material only under stress. The acoustic emission frequencies are in the range of 150-300 kHz, which is above the frequency of audible sound. Usually the sensors used for monitoring the response of structural materials have a recording ability in the 20 kHz -1 MHz range [7]. The basic advantages of the AE method are its high sensitivity, the early detection of defects and cracks and the real time monitoring at a relatively low cost. Several models are proposed to analyze acoustic emissions signals, such as the b-value and the improved b-value, the intensity analysis [11], the Ledeczi et. al. method, etc. Nowadays an alternative approach is widely used for the classification of the acoustic emissions' source based on the relation between the signals' average frequency with the RA (Rise Time/Amplitude) parameter (Fig.4). The results have proven very encouraging [12, 13]. The latter analysis model is adopted in the present study.

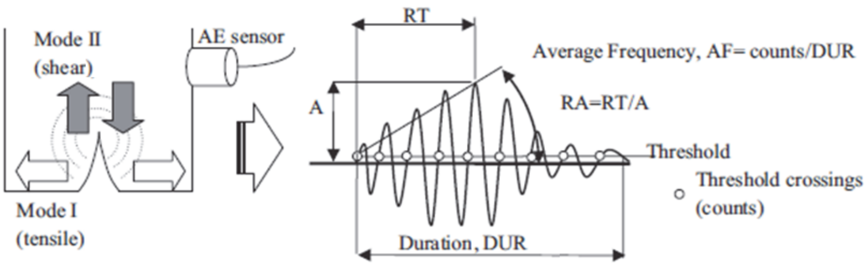


Figure 4: Cracking modes and typical acoustic emission signals [12].

The specimens and the experimental procedure

The specimens made of Alfas stone were of orthogonal parallelepiped shape of dimensions equal 22.5x10x2.2 cm^3. The artificial specimens made of mortar and cement paste were also of orthogonal parallelepiped shape and their dimensions were equal to 4 cm x 4 cm x 16 cm according to the ASTM C348 standard. The specimens were subjected to 3-point bending (3PB), under displacement-control mode and quasi-static loading conditions, using an INSTRON (300 kN) servo-hydraulic loading frame, with a 50 kN calibrated load cell. The load was applied monotonically up to the fracture of the specimens. A long series of preliminary experiments highlighted the crucial role of the loading rate on the results and indicated that for the specific materials a rate equal to 0.02 mm/s was the one allowing optimum sampling of the AE data.

For the Alfas stone specimens four acoustic sensors (R15α) were properly arranged around the critical region (Fig.5). For the mortar and cement paste specimens a single acoustic sensor (R15α) was attached in the middle of the specimens' span at their lowest side. The sensors were mounted on the specimen by means of proper silicone paste (Figs.5, 6 and 7).

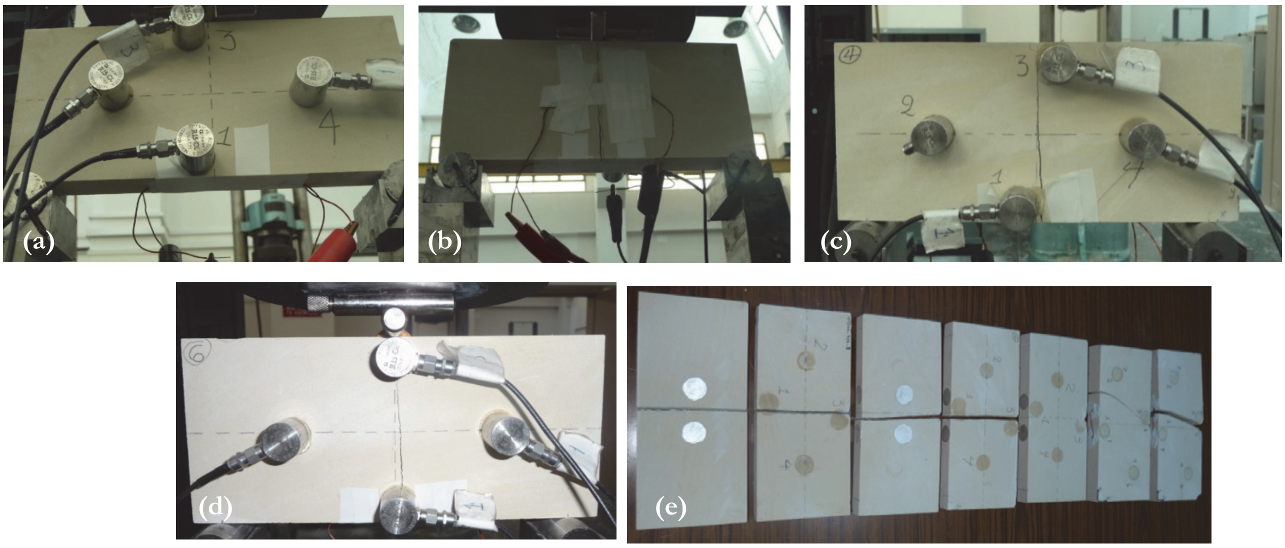


Figure 5: (a)-(d) Experimental set up for the Alfas stone specimens and the position of the AE sensors; (e) typical fractured specimens.

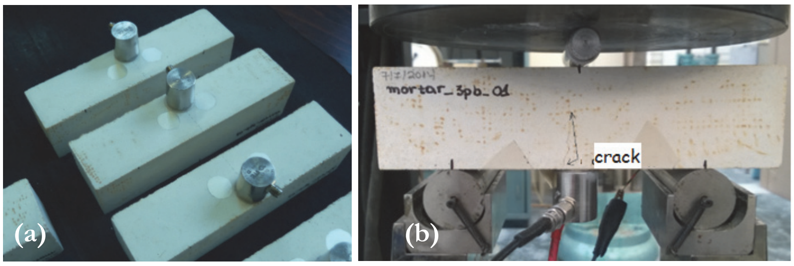


Figure 6: (a) The position of the acoustic sensor; (b) the experimental set up- and the initiation of the crack for a typical test with specimen made of mortar.

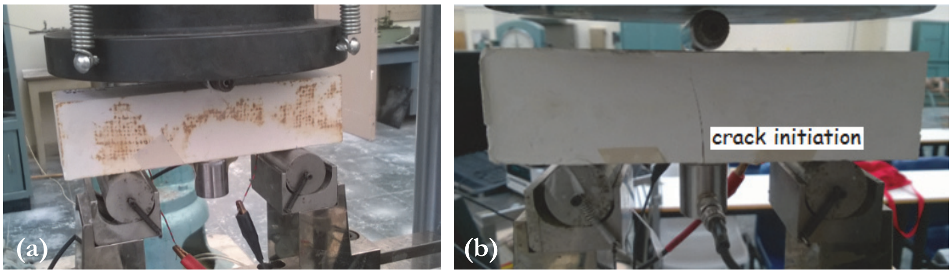


Figure 7: A typical test with a cement paste specimen (a) before and (b) after fracture.

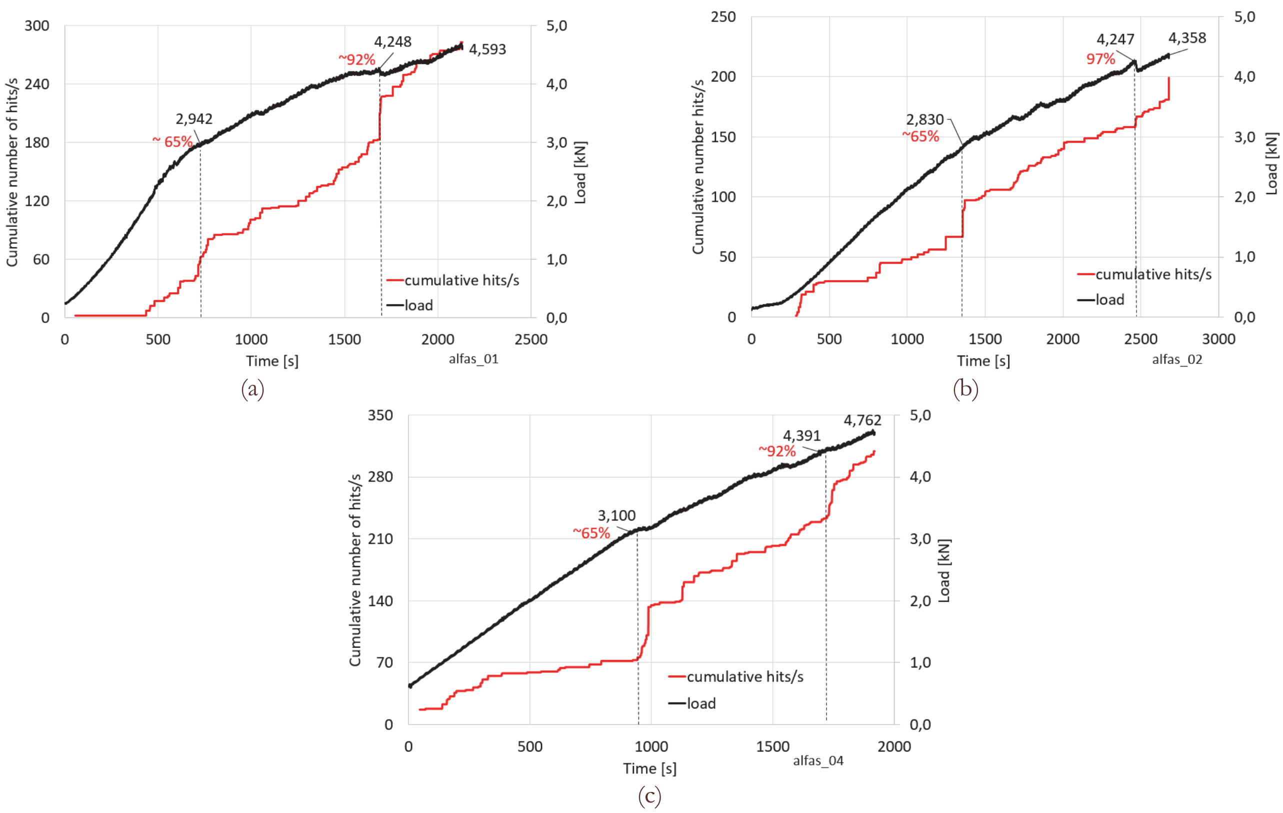


Figure 8: (a)-(c) The time variation of the cumulative hits/s for three characteristic 3-point bending experiments with specimens made of Alfas stone.

EXPERIMENTAL RESULTS

The time variation of the load imposed in conjunction with the respective variation of the cumulative number of hits/s recorded by the AE sensors are plotted in Fig.8 for three characteristic specimens made of Alfas stone. It is worth noticing that at about 65% and 95% of the maximum load abrupt increases of the hits/s appear systematically, in very good correlation with changes (either slope changes or slope discontinuities) of the respective load-time curve.

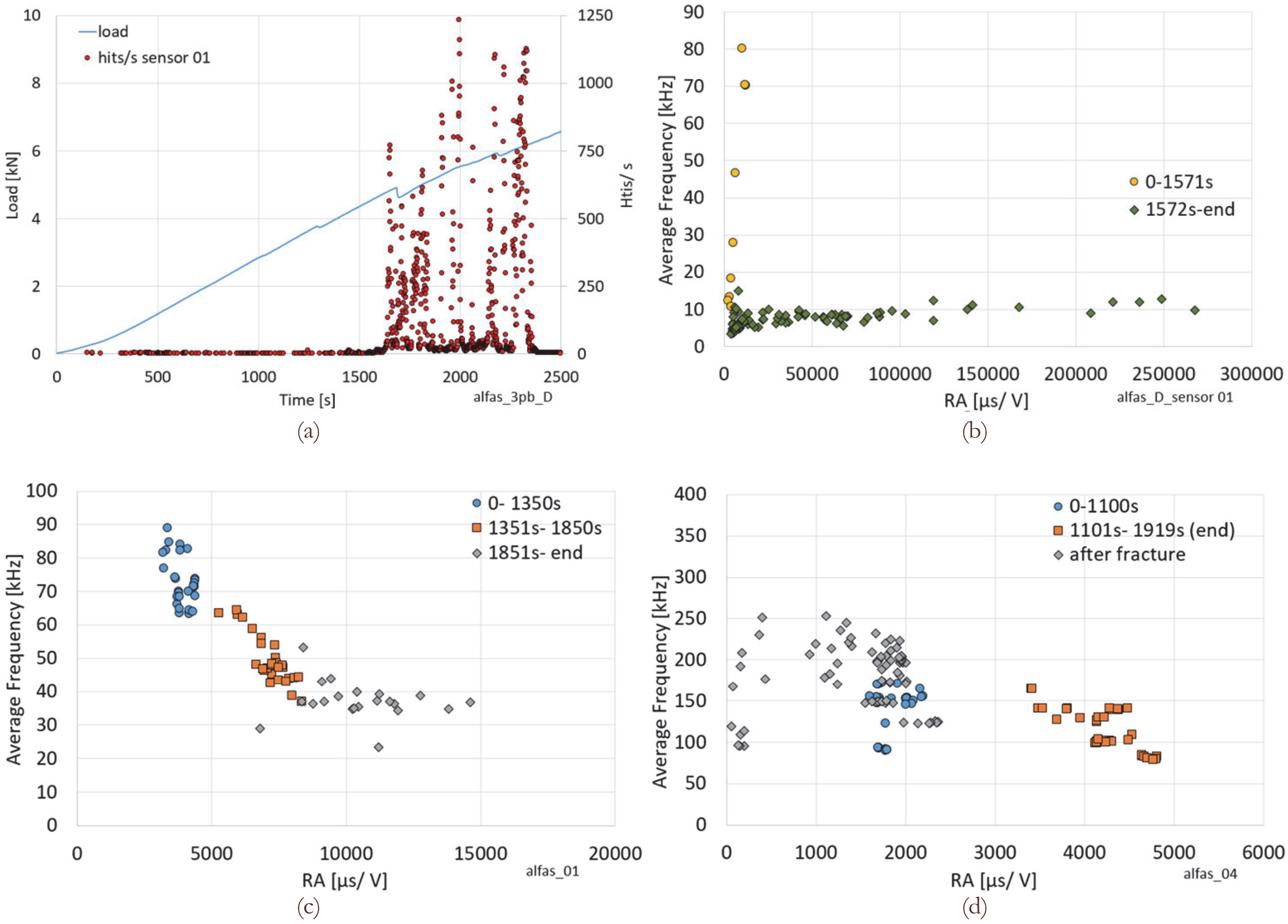


Figure 9: (a) The time variation of the hits/s and the load applied; (b) Average frequency versus the RA parameter (c) and (d) Average frequency versus the RA parameter for two additional characteristic experiments with Alfas stone specimens.

In an attempt to gain a deeper insight of the damage mechanisms activated within the mass of the Alfas stone specimens during loading, the data of sensor 01 (i.e., the one attached at the mid-section of the specimens' supporting length, or in other words the one closest to the critical area where the onset of macroscopic cracking is expected) are here analyzed further: The hits/s recorded by this sensor, are considered for two time intervals, i.e., before and after a critical time instant which, for example, for the specimen studied in Fig.9a is selected equal to $t_{cr}\approx 1570$s, for obvious reasons. For these two time intervals average values of the frequency and the RA parameter (Rise Time/Amplitude (μs/V)) are calculated. The mutual dependence of these two quantities is plotted in Fig.9b. A clear qualitative distinction of the acoustic signals recorded before and after the critical time instant is clearly visible. The signals recorded during the first time interval ($0<t<t_{cr}$) are characterized by very high frequency and relatively low RA, while the signals recorded during the second time interval ($t_{cr}<t<t_{fracture}$) are characterized by much lower frequency and extremely higher RA values. This classification of the recorded signals has been a concern of the scientific community for a long time and it is nowadays attributed to different modes of cracking producing elastic waves of different characteristics. According to the dominant explanation, AE with higher frequency and lower RA values are attributed to tensile cracking, while signals with lower frequency and higher RA

are attributed to shear- or mixed-mode cracking [12, 14]. According to this approach, it is concluded that for Alfas stone under 3-point bending the cracks produced before the critical time instant are of tensile nature and are followed by shear- or mixed-mode cracking, which leads eventually to catastrophic macroscopic fracture of the specimen.

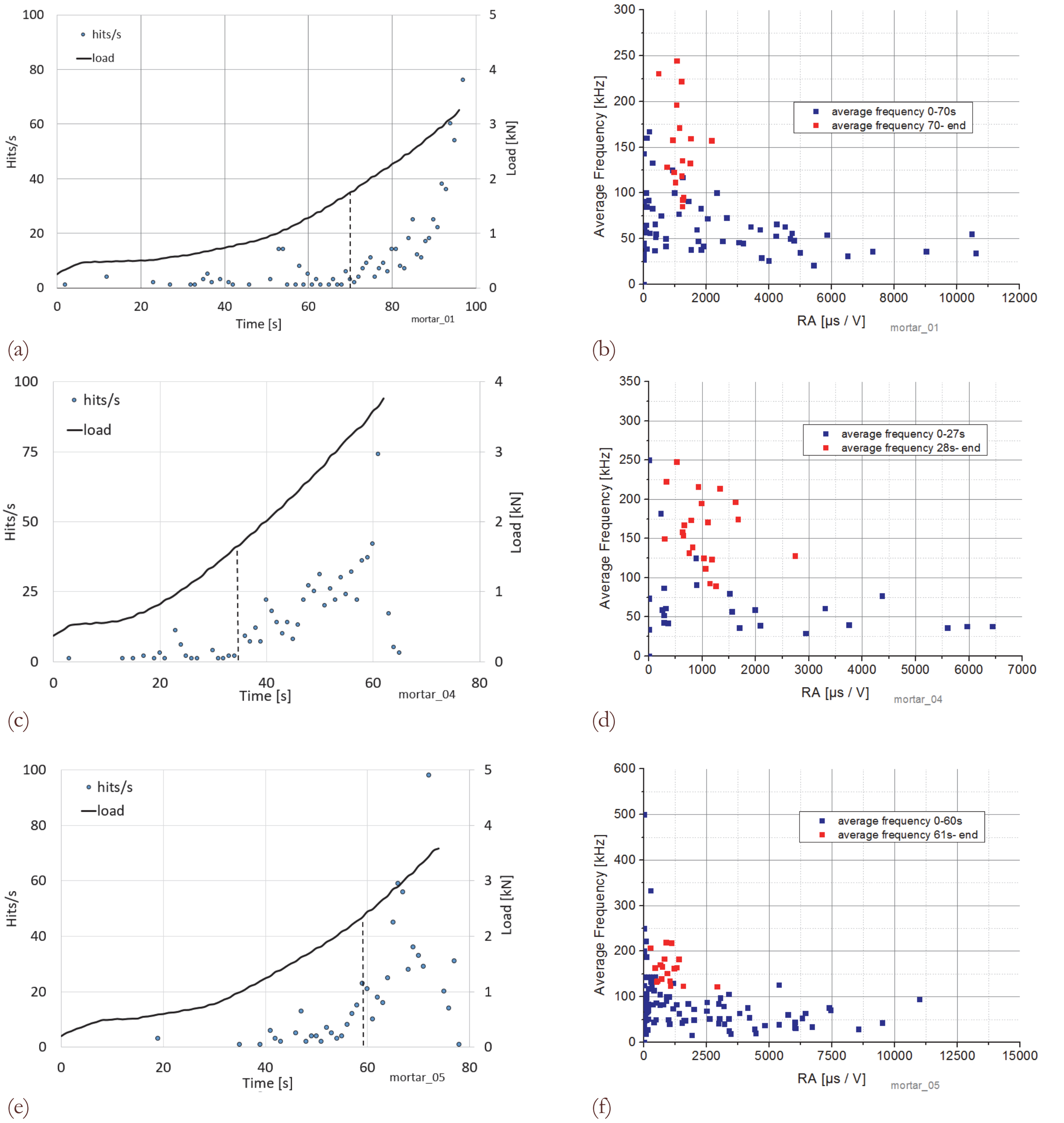


Figure 10: (a), (c), (e) Time variation of the hits/s and the load applied for three characteristic mortar specimens under 3-point bend-ing and (b), (d), (f) the respective average frequency versus the RA parameter.

For the specimens made of mortar and cement paste, the load-displacement curves did not provide any sign at all that the material approaches its critical stage and that catastrophic fracture is impending. In this context the acoustic activity appears to be an extremely valuable tool that can potentially provide clear "warning" of upcoming fracture or that the specimen has entered its critical stage. Along this direction the temporal dependence of the hits/s for three characteristic experiments with mortar specimens are plotted in Figs.10(a,c,e), in juxtaposition to the respective variation of the load applied. It is noticed that in all cases a critical time instant exists after which the number of hits/s starts increasing abruptly, indicating increased internal activity (micro-cracking). Following the procedure described for Alfas stone specimens the average values of the RA parameter and the average frequency of the signals recorded are calculated and plotted in Figs.10(b,d,f)

for the time intervals before and after the respective critical time instant. In all cases considered, the earlier signals $(0<t<t_{cr})$ are characterized by lower frequency and higher RA values (shear or mixed-mode cracking) while the signals recorded after the critical time instant $(t_{cr}<t<t_{fracture})$ are characterized by higher frequency and lower RA values.
The respective diagrams for a typical cement paste specimen are given in Figs.11(a,b). The RA values decrease as the load applied approaches its maximum value while the frequency does not exhibit noticeable changes. What is now much different, however, is that for some specimens (like the one considered in Figs.11(c,d)), after the maximum load is attained the specimen does not disintegrated abruptly but rather a post-peak region follows during which the load decreases gradually and the specimen collapses somehow "smoothly". The overall duration of the experiments of this class of cement paste specimens is divided into three intervals (instead of two). The respective diagram of the average frequency versus RA is plotted in Fig.11d. It can be seen from this figure that the signals recorded during the last time interval (i.e. after the peak load) have the same qualitative characteristics with those before the peak load.

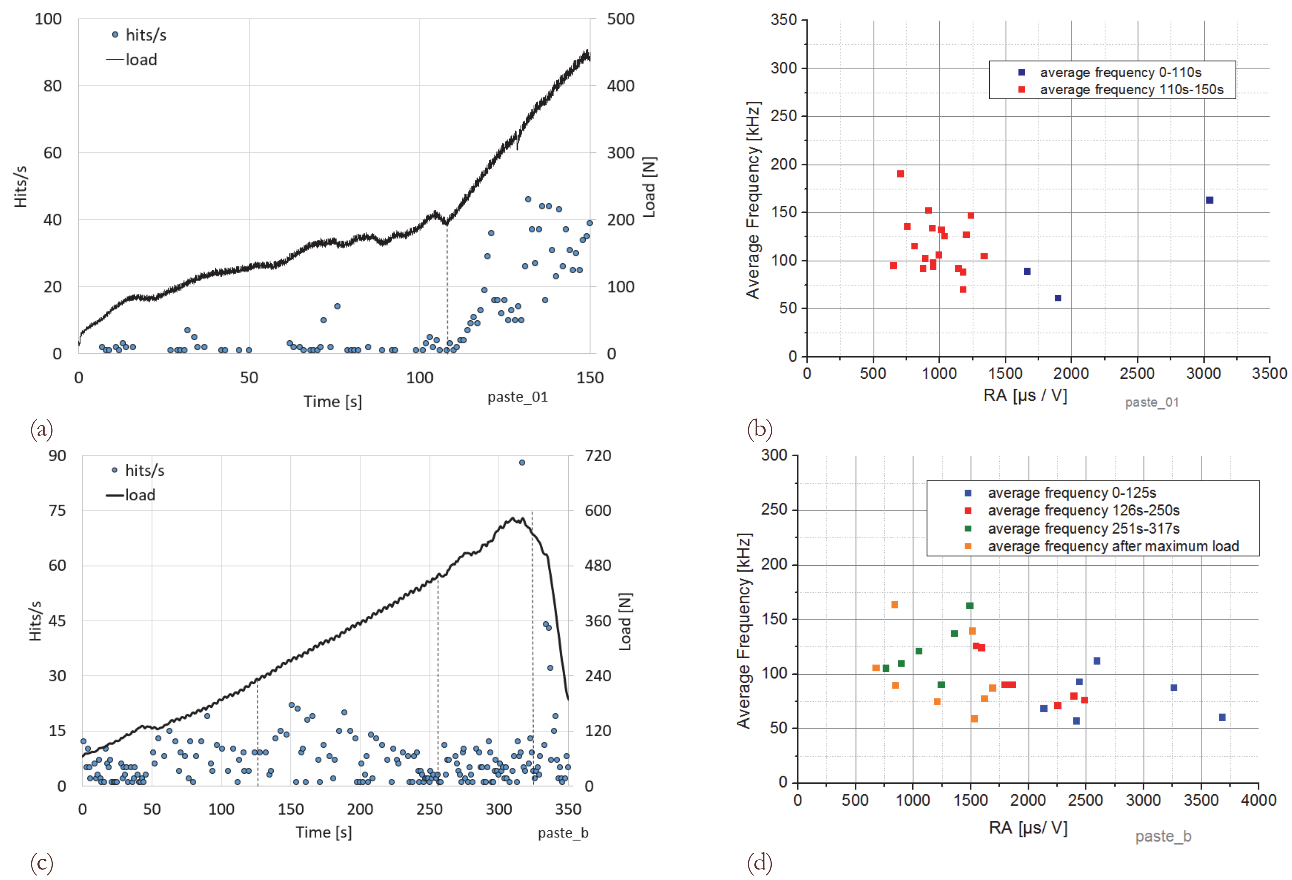
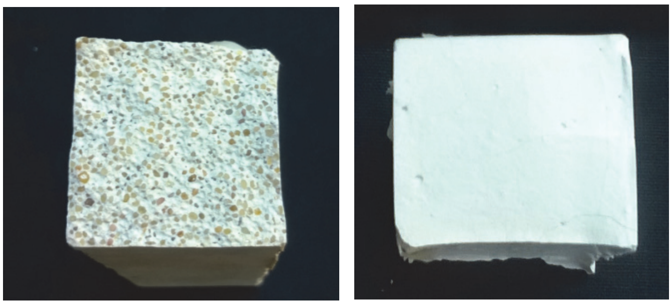


Figure 11: (a) Time variation of the hits /s and the load applied for a cement paste specimen and (b) the respective average frequency versus RA; (c) Time variation of the hits /s and the load applied for a cement paste specimen with a clearly distinguishable post-peak regime and (d) the respective average frequency versus RA.

DISCUSSION AND CONCLUDING REMARKS

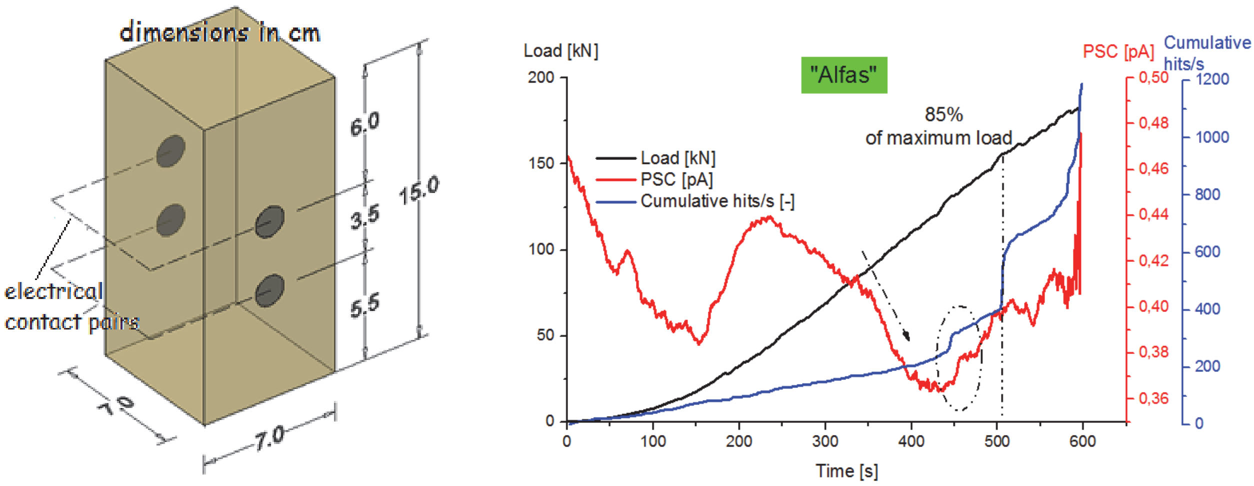
Specimens made of either a natural building stone (Alfas stone) or mortar or cement paste were tested under 3-point bending. The main target of the study was to assess the AE signs recorded during the experiments and attempt to classify them according to the nature of the source from which they were emitted. Considering as critical threshold the time instant at which the number of hits/s exhibits an abrupt increase, the duration of the experiments was in general divided into two intervals (excluding the specimens with clear post-peak regime). For each interval the average frequency was plotted against the RA value, in order to apply the classification criterion for the source of the AE emissions [12-14]

according to which the emissions are due to microcracking or friction depending on the combination of frequency and RA values.
It was concluded that a clear difference between specimens made from artificial materials and specimens made from natural stone exists: Indeed, for Alfas stone specimens the failure mechanisms during the primary loading stages are governed by tensile micro-cracking while the last stages before macroscopic fracture are governed by shear- or mixed-mode cracking. On the contrary, for the specimens made of artificial materials the succession of mechanisms is inverted: Shear- or mixed-mode cracking prevails during the primary loading stages and it is then followed by tensile micro-cracking at the loading stages before the final disintegration of the specimens. Differences appear, also, between the mortar and the cement paste specimens concerning mainly the existence or not of a clearly distinguishable post-peak regime which characterized some specimens made of cement paste. This difference can be attributed to their different microstructure, which is clearly visible in Fig.12.



(a) (b)

Figure 12: Cross section of typical specimens made of (a) mortar and (b) cement paste.



(a) (b)

Figure 13: (a) A typical prismatic specimen made of Alfas stone subjected to uniaxial compression and (b) the respective time variation of load, cumulative hits/s and Pressure Stimulated Currents [15].

Another critical conclusion that can be drawn from the present experimental protocol is that the signals recorded by the AE sensors are characterized by some indicators that can be considered as warnings that the specimens change their state and enter critical stage, well before the peak load is attained. Due to its significance the specific conclusions must be further studied with specimens made of different materials and subjected to other loading modes. In this direction, an experimental protocol is in progress, with Alfas stone specimens subjected to uniaxial compression (Fig.13a) [15]. The results of this protocol are very encouraging. In Fig.13b the data concerning the cumulative hits/s for a typical compression experiment of that protocol are plotted versus time in juxtaposition to the respective variation of the load imposed. It is seen from this figure that at about 85% of the peak-load finally attained the cumulative hits/s exhibit an abrupt jump

equal to about 40% of its value before the sudden jump. This discontinuity can be again considered as a clear indicator that the specimen enters its critical stage and macroscopic fracture is impending.
Another interesting conclusion drawn from Fig.13b is that the pre-failure indicators provided by the AE technique are in very good agreement with similar ones obtained by another sensing technique known as Pressure Stimulated Currents (PSC) [16-20]. Indeed it is seen from Fig.13b that just before the "jump" of the graph of the cumulative hits/s the PSC recorded by a pair of electric contacts starts increasing gradually. The same signal (PSC) exhibits a very steep "jump" just before the macroscopic fracture of the specimens. Considering the data of these two protocols it can be concluded that a combined use of the AE and PSC techniques may be proven valuable in the direction of further enlightening the internal damage process of specimens made of building materials as well as in the direction of providing safe pre-failure indicators.

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