Acoustic emissions and pressure stimulated currents experimental techniques used to verify Kaiser effect during compression tests of Dionysos marble

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ABSTRACT. The damage development due to externally applied mechanical stress is a hot topic of interest involving several applications of everyday life, like civil engineering, monument restoration, construction evaluation and health monitoring. Repetitive loadings of brittle materials cause internal damages that gradually extend, leading to inevitable failures. Such processes are studied under the concept of the materials' mechanical memory effect that is widely known as Kaiser effect. The Kaiser effect states that a structure will only suffer further internal damaging when exposed to applied stresses higher than previously encountered. Certain conditions lead to a violation of the Kaiser effect, known as the Felicity effect, quantitatively measured using the Felicity Ratio. This work presents the experimental results when repetitive mechanical load loops are applied on marble specimens, while concurrent Acoustic Emission (AE) and Pressure Stimulated Currents (PSC) measurements are conducted. The collected AE and PSC data are studied in combination with the mechanical data, like mechanical stress and strain, under the frame of the Kaiser effect. It is clearly seen that the Felicity ratio strongly depends on the stress range the material is subjected to, with regard to the elastic or plastic deformation region.

KEYWORDS. Acoustic Emissions; Pressure Stimulated Currents; Dionysos marble; Kaiser effect; Felicity Ratio.

Citation: Stavrakas, I., Acoustic emissions and pressure stimulated currents experimental techniques used to verify Kaiser effect during compression tests of Dionysos marble., Frattura ed Integrità Strutturale, 40 (2017) 32-40.

Received: 05.01.2017
Accepted: 06.02.2017
Published: 01.04.2017

INTRODUCTION

The scientific literature reports several studies and recordings of AE that are attributed to mechanical stresses adequate to cause microcracking phenomena in rocks [1]. Towards this direction, the experimental technique of AE detection has been evaluated and developed in order to constitute a valuable tool for the monitoring and interpretation of the underlying physical mechanisms of mechanical dynamic processes and the detection of an upcoming event of mechanical failure [2]. Further than the AE, the detection and recording of low-level electric signals provide reliable information, regarding the development of damage processes. Such electric signals are detected due to fracture processes in quasi-brittle materials following microcracks formation and growth. It is known that transient electric phenomena are often appearing when solid materials are subjected to stress and mechanical effects are taking place [3, 4].

Since 2000, the detection of the electric signals that are related to weak electric current emissions is conducted through an innovative experimental technique rendered under the term Pressure Stimulated Currents technique, while the recorded electrical currents are known as Pressure Stimulated Currents (PSC) [5]. The PSC are detected through the recording of a weak (low-level) electric current using a sensitive electrometer, when a pair of electrodes is attached properly on the specimen that is subjected to mechanical tests. The above-described experimental technique was initially applied when marble materials were subjected to axial compressive mechanical stress [6, 7].

The Kaiser effect is an AE phenomenon briefly defined as the absence of detectable acoustic emissions until the previously applied stress level is exceeded. This effect is based on the experimental discovery by Kaiser (1950), that metallic materials had the capability to remember the previous maximum stress level. The existence and the experimental verification of the Kaiser effect was also discovered in rocks and described in other works [8-10] and since then, the Kaiser effect is used to detect and assess the amount of damage that has been developed in rocks [11, 12].

The breakdown of the Kaiser effect can be represented quantitatively by the felicity ratio (FR) that is defined as the ratio of the AE-onset stress to the maximum stress of the previous cycle. It may be taken as a measure of the quality of the rock [13]. A high felicity ratio means that the rock is of good quality.

Regarding the PSC emissions, several works in the past demonstrate the ability of a marble specimen to “remember” previous mechanical loadings [5, 6, 14-16]. Specifically, the main properties of the PSC signal, which are affected by the existence of memory, converge to an inertial attitude of the material to the same stimuli and they are quite common with the properties of other fracture induced signals (i.e. AE). Namely, they are the following: (a) The PSC peak evolution over loading cycles is a changing signal property, with respect to the time interval between loadings, (b) The decrease of the dissipated electric energy during cyclic loading tests, (c) The PSC slower relaxation in each loading, quantified by the relaxation process parameters evolution, (d) The PSC signal initiates to show up at higher stress level after each next loading cycle.

The aim of this work is to conduct a laboratory experimental investigation and verification of the Kaiser effect on Dionysos marble specimens combining the AE and PSC recordings. The specimens are subjected to compressive loading loops where the first loading is in the region where the material leaves the elastic region and enters the plastic deformation i.e., the stress-strain curve deviates from linearity.

TEST FACILITIES AND ARRANGEMENT

The marble specimens were subjected to three loading-unloading loops (see Fig. 1). The maximum stress level during the 1st and the 2nd loading was approximately 60 MPa, a value that corresponds to the 70% of the ultimate compressive strength of the material and leads to a Young’s modulus of 72 GPa (see Fig. 2). This stress level, according to preliminary tests on similar specimens, corresponds to the region that the material gradually enters the non-linear region regarding its stress-strain behaviour and initial plasticity effects take place, which is in accordance, also, to previously published data [17-19]. During the unloading processes the stress level was maintained at a value of 15 MPa, approximately. A third loading was attempted during which the specimen failed at a stress level of 82.7 MPa. The compressive stress was applied following load control at a rate of 320 kPa/s.

Fig. 1 shows the temporal variation of the loading path until the failure of the specimen and Fig. 2 shows the stress-strain curve during the complete cyclic loading. It must be noted that during the 1st loading the linear region was estimated to last up to a stress level of 58 MPa approximately.

The specimens (i.e. Dionysos marble) were prismatic with dimensions 35mm×35mm×75mm. The physical and chemical properties of this kind of marble have already been presented in bibliography [6, 20, 21]. The strain was measured using Kyowa strain gauges attached on the Microlink-770, 120Ω resistor bridge (see Fig. 3).

During the tests the AE events were monitored using a Physical Acoustic Corporation (PAC) Mistras Systems. The AEs transducer was the model R6 sensor provided from the Mistras S.A. that obtains a wide frequency range and was attached in the middle of the specimen’s height (see Fig. 3). The AE threshold for detecting acoustic events was set at 40 dB.

Concerning the PSC technique, the measuring system consisted of an ultra-sensitive programmable electrometer (Keithley, 6517A) resolving currents ranging from 0.1 fA to 20 mA in 11 ranges. The data of the electrometer were stored in a computer using a GPIB interface. The sensing system consisted of a pair of gold plated electrodes, installed in the middle of the specimen's height (see Fig. 3), enabling the collection of electric emissions as close as possible to any potential source of electric current (or equivalently to any point where damage occurs). Further details on the monitoring of the AE and PSC are described in earlier papers [22, 23].

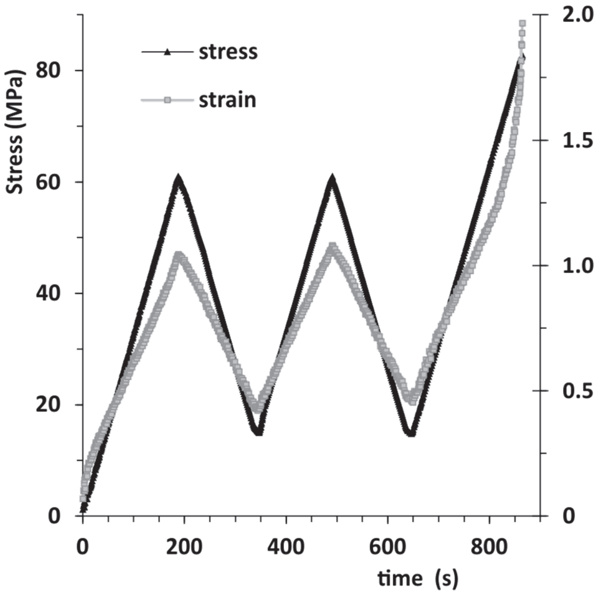

Figure 1: Temporal variation of the mechanical stress (black line), and the corresponding behaviour of the mechanical strain (gray line) during the complete cyclic loading.

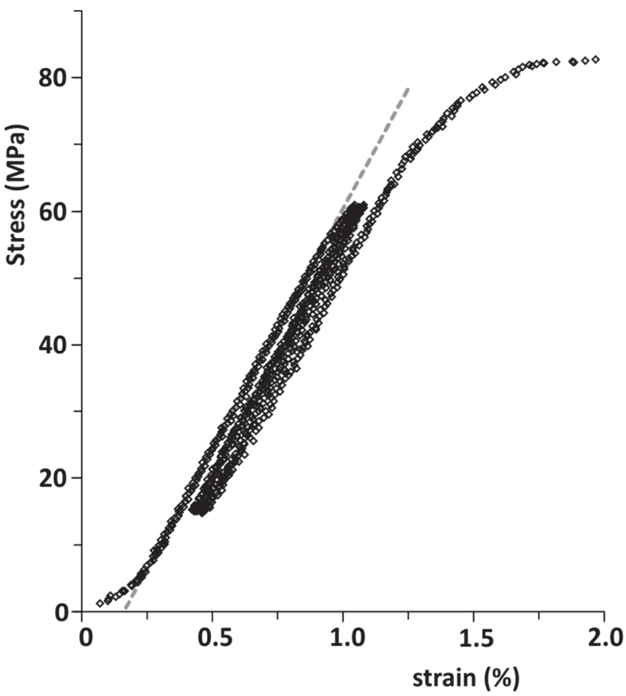

Figure 2: The stress-strain behaviour during the two loading / unloading loops as well as the final loading during which the specimen failed.

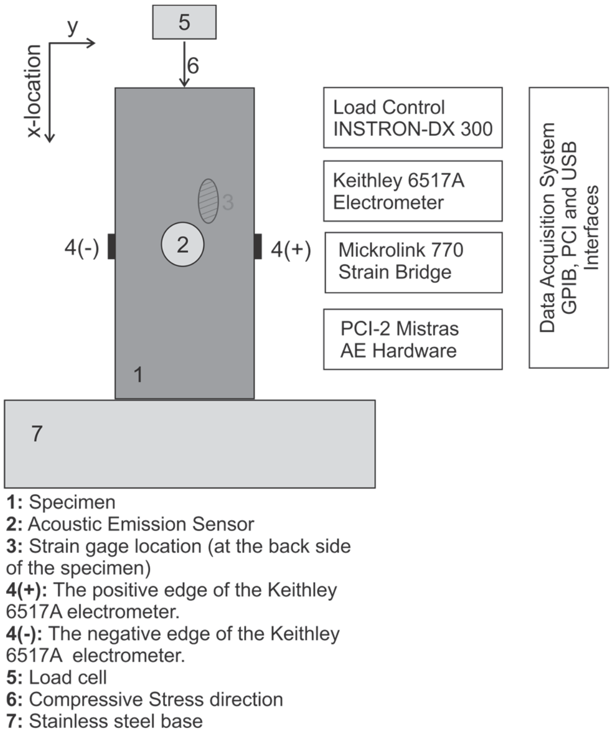

Figure 3: The schematic diagram of the experimental setup showing the physical locations of the sensors on the specimen, the geometry as well as the data communication interfaces.

VERIFICATION OF THE KAISER EFFECT

Acoustic Emission (AE) recordings

The AE activity during the complete experimental procedure is illustrated in Fig. 4. Specifically, the AE events per second are shown with respect to time. In the same figure, the temporal variation of the normalized applied mechanical stress is also presented. It may be clearly seen that AE were continuously detected during the entire process. Specifically, high AE rate is observed during the loading procedures while low AE rate is observed during the unloadings. It must be noted that despite the fact that the experimental apparatus is well isolated from external AE noise and the configuration of the AE hardware system significantly reduces the probability to record noise as AE events (i. preamplifier filters were applied and ii. background noise was continuously recorded and subtracted) the low values of AE rate may be attributed to such an external influence. During the first loading loop, a total number of 978 AE events is recorded while a significantly smaller number of AE events (289 AE events) were recorded during the second loading loop. It is noticeable that during the second loading of the specimen, practically no AE events were recorded until the load reached the 68% of the compressive strength (i.e. only 24 low amplitude AE events were recorded). The most of the AE events (i.e. 90% of the detected AE events) were recorded in the region between 68% and 73% of the compressive strength. This region corresponds to the upper limit of the linear behaviour of the material regarding its stress-strain curve. During the 3rd loading and before the stress reached the level of the previously applied stress, only 45 AE events were recorded while the 36 of them were recorded when the applied stress was in the region between 71% and 73% of the compressive strength. When the stress exceeded these values, 1237 AE events were recorded until the failure of the specimen. The AE records of the entire experimental procedure are plotted in a cumulative form, as shown in Fig. 5. These experimental results are in good agreement with the existing literature and specifically works published in [8-12].

During the 2nd and the 3rd loading-unloading loops the Felicity Ratio (FR) can be calculated to be equal to 0.66 and 0.8 approximately, correspondingly (see Fig. 5). The FR values were calculated according to the following formula:

$$FR = \frac{load\ at\ onset\ of\ significant\ AE\ event}{previous\ maximum\ load}$$

The deviation from the optimum value of FR=1, may be justified due to the fact that the maximum stress values of the 1st and 2nd loading loops was selected to lay in the limits of the linear and non-linear region, regarding the stress-strain behaviour of the marble, and additionally to the quality of the used marble. It must be noted that during each loading loop the FR clearly depends on the maximum stress of the previous loop and decreases as the value of the maximum stress increases [13]. This fact clearly is not the case for the described loading protocol of this work since the FR seems to increase rather than decrease during each next loading. Such a behaviour may be attributed to two main reasons. Firstly, the two initial loadings were performed up to the same stress value, a fact that is not typical for the FR development and secondly the selected stress value of 60 MP lays in the limits between the linear and non-linear region of the stress-strain curve, so no further damages may be caused during the second loading leading, as expected at a higher FR value.

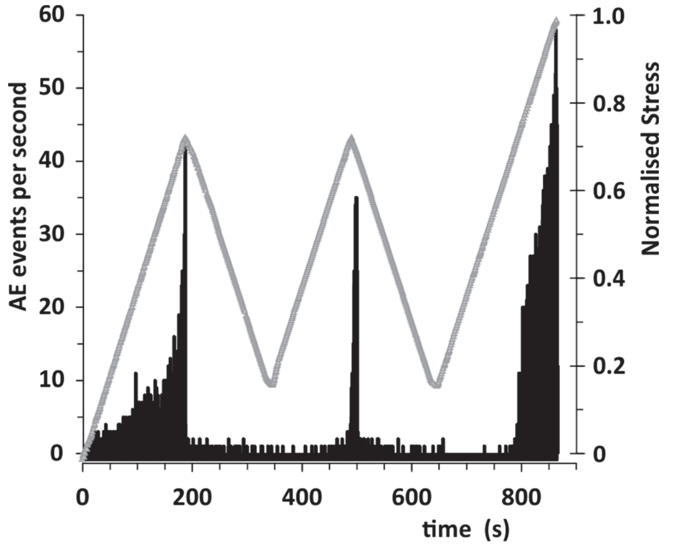

Figure 4: The mechanical stress temporal development during the complete experimental procedure (gray line) and the corresponding per second AE event rate (black bars).

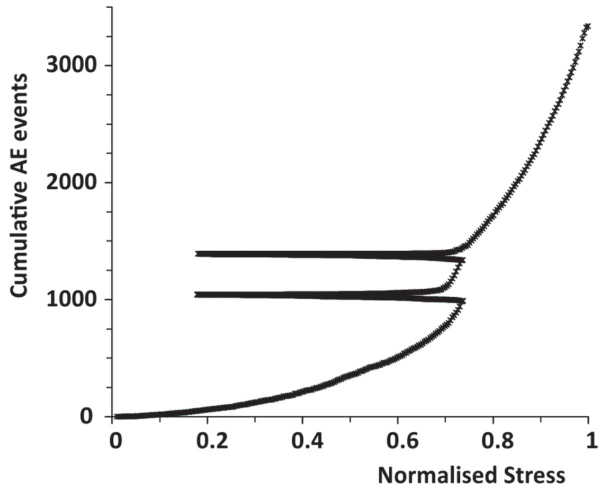

Figure 5: The behaviour of the cumulative number of the AE events with respect to the normalized value of the mechanical stress applied on a typical specimen.

Pressure Stimulated Current (PSC) recordings

Concurrently to the above described observations, PSC emission recordings were conducted. Fig. 6 shows the temporal variation of the recorded PSC with respect to the temporal variation of the mechanical stress. Observing the behaviour of the PSC, it may be clearly seen that during the first loading, where the mechanical stress reached a maximum value of 60MPa approximately, a strong PSC emission was recorded reaching a maximum value of 40pA approximately, while a gradual decay back to the background level occurs when the mechanical stress is relieved. It must be noted that the maximum value of the applied stress (i.e. 70% of the maximum stress) enters the specimen into the early nonlinear region regarding its stress-strain behaviour. During the second loading the applied mechanical stress reached approximately the level of the mechanical stress of the 1st loading (i.e. 60MPa) while the recorded PSC reached a maximum of 10pA which is significantly lower than the recorded PSC of the first loading. Such a behaviour may be attributed to the underlying physical mechanisms of the PSC generation and specifically the Moving Charged Dislocations model [4]. Since the PSC variation is attributed to the damage generation and extension it is expected that when a brittle specimen, like marble, is subjected to a mechanical stress in sequential loading cycles new damages occur only during the initial application of the stress. During each following stress application only minor extension of the already created damages is produced and consequently only weak PSC emissions are expected. The third loading was scheduled to lead the specimens to failure, and the mechanical stress was applied at the same rate of the two initial loadings. During this third loading a strong PSC emission was detected reaching a peak PSC value of the order of 100pA. It is worth noticing that a short time (i.e. 5 seconds) before the stress drop (due to the specimen's failure) the characteristic PSC decrease [5, 6] was observed clearly indicating the upcoming failure. The above experimental results are in good agreement with the corresponding results presented in other works [5, 6, 14-16].

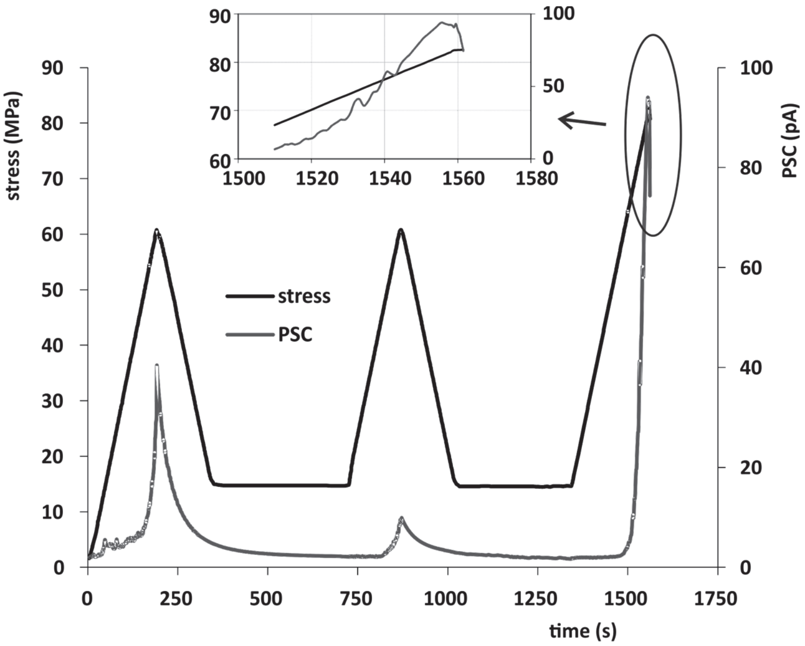

Figure 6: The temporal variation of the mechanical stress during the complete experimental procedure and the corresponding behaviour of the PSC emission. The inset figure constitutes a zoom in at the final stage before fracture making clear the characteristic PSC drop before the failure of the specimen.

Fig. 7 demonstrates the behaviour of the PSC emission in logarithmic scale with respect to the mechanical stress during each loading cycle. Specifically, the blue, the green and the light grey lines correspond to the load increase from 0 MPa to 60 MPa of the first, the second and the third loading, respectively. The black line shows the behaviour of the PSC with respect to the mechanical load during the third loading, when the applied stress becomes higher than 60 MPa exceeding this way any previously applied level of stress. Observing Fig. 7 it is clear that during the three loadings and while the stress is below 60 MPa, the recorded PSC becomes significantly lower for each next loading. When the mechanical stress exceeded 60 MPa during the third loading, the PSC emission significantly increases reaching a peak of 100 pA, few seconds before fracture. The black line in Fig. 7 supports the dominance of the Kaiser effect during the experimental procedure and in addition that PSC may also be used to detect that a sample has suffered significant mechanical stress at earlier times and it "remembers" such a fact. Another observation is that during the second and the third loading the detected PSC and the corresponding PSC energy are both at the same low levels up to a stress value of 45 MPa. Beyond that point of stress and during the third loading the recorded PSC is maintained at a very low level. During the third loading and when stress becomes higher than 60 MPa strong PSC emissions and rapid increase is detected.

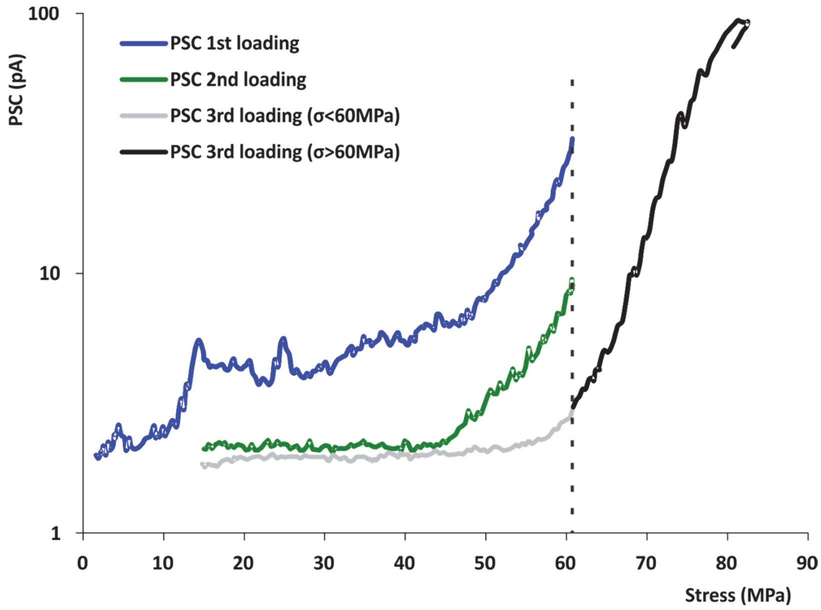

Figure 7: The behaviour of the PSC, shown in logarithmic scale, with respect to the value of the mechanical stress.

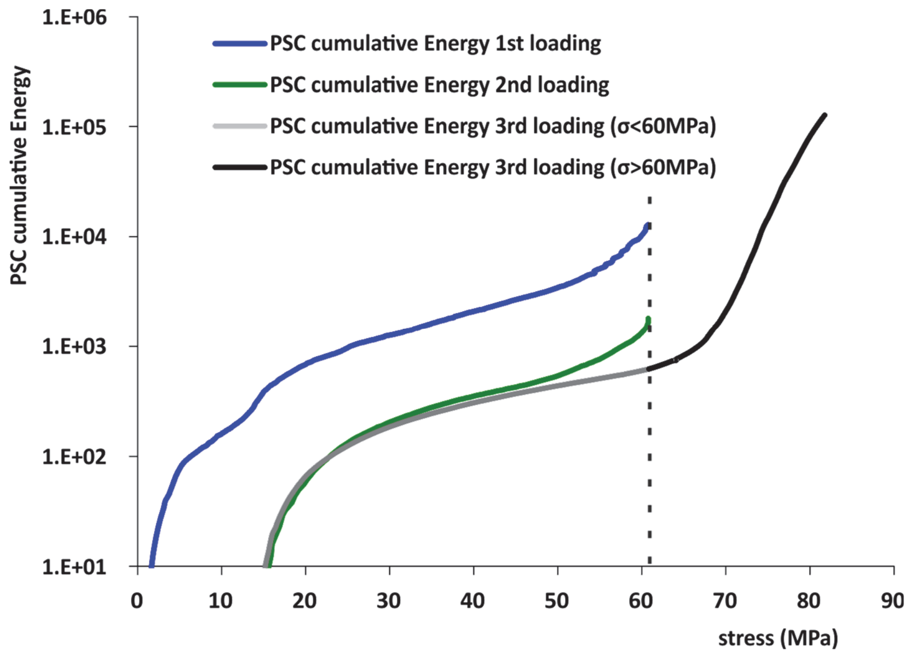

Figure 8: The behaviour of the PSC cumulative energy, shown in logarithmic scale, with respect to the value of the mechanical stress.

Fig. 8 shows the behaviour of the released cumulative energy of the PSC in logarithmic scale with respect to the mechanical loading during the experimental procedure.
The square of PSC adjusted amplitude PSC(t), expresses an energy quantity [24] and may be calculated according to:

$$E_{PSC}\left(t_i\right) = PSC^2\left(t_i\right)$$

where t_i corresponds to the time instance of each PSC recorded sample.
The cumulative PSC energy CE_{PSC} during each loading is calculated according to the following formula:

$$CE_{PSC} = \sum_{i=1}^{n} E_{PSC}(t_i)$$

where n corresponds to the number of the recorded PSC values in the examined mechanical stress region.
It may be clearly seen that during the first loading a significant amount of energy is released (blue line). Additionally it is clear that the PSC emission initiated from early mechanical stress levels which means that the PSC is emitted even in low stress levels. During the second and the third loadings (green and gray lines respectively) the PSC energy initiates only when stress becomes higher than 15MPa approximately, while their cumulative energy levels are significantly lower than the corresponding of the first loading. Finally, during the third loading and when the mechanical stress became higher than the 60 MPa a significant increase of the released cumulative energy is recorded. This fact that also manifests Kaiser effect dominates the PSC emissions and the corresponding PSC cumulative energy.

Concluding Remarks

During the present experimental protocol two sequential compressive loadings and unloadings were applied on marble prismatic specimens up to a stress level that corresponds to early non-linear region regarding the stress-strain behaviour. During the third loading, the stress was not relieved but instead it was further increased until the sample mechanically failed. Concurrently to the loading AE and PSC emissions were recorded. The aim was to investigate and correlate the fingerprint of the Kaiser effect on the AE recordings and the PSC emissions as well as the corresponding cumulative energy. The tests were performed on series of marble prismatic specimens and representative experimental results of the carried out tests are presented herein.
Additionally, regarding the AE recordings, it is experimentally verified that for marble specimens the Felicity Ratio (FR) obtains high values (i.e. 0.66 and 0.8 approximately) during the two loading/unlading cycles when the applied stress lays in the region where the material is still near the limits of linear region regarding its stress-strain behaviour.
Regarding the behaviour of the AE events the number recorded is significantly large during the first loading while the number of AE events becomes fewer during the second load. AE events practically disappear during the third loading and more specific when the applied stress is in the range of the two previous loadings. When the stress was further increased significant AE events are recorded until the fracture of the sample.
Regarding the PSC emissions it is observed that the maximum value of the PSC emission becomes significantly lower during each next loading while the applied mechanical stress varies in the same stress limits. It is also important to notice that detectable PSC emissions show up at higher stress levels during the second and the third loading. During the third loading and while the applied mechanical stress becomes higher than the corresponding values of the previous loadings, intense PSC emissions are detected. The characteristic drop of the PSC is detected when the sample approaches failure.
All the above manifest that both AE and PSC emissions may be used to detect and qualitatively approach the Kaiser effect phenomena when Dionysos marble samples are subjected to mechanical stress.
Combining the experimental findings after applying the AE and the PSC experimental techniques when marble specimens are subjected to compressive mechanical stress it is observed that both experimental techniques may be used in order to detect an upcoming specimen failure. Further on qualitative comparison shows that the use of PSC and AE provide similar information regarding the existence of the Kaiser effect. Specifically, it was seen that when applying sequential loadings and unloadings on marble specimens the emitted PSC becomes lower during each loading cycle and the recorded AE events rate start to increase only when the applied mechanical stress gets values higher than the corresponding stress values during the previous loadings. Quantitative comparison is not yet possible and further experimental work is required in order to attempt such an approach.

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