The Ascertainment of the Presence of Damage Processes Using the Pressure Stimulated Current (PSC) Technique on Marble and Cement Samples

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Abstract. Current emissions due to externally applied stress were studied for various materials in the past. This work concentrates on comparative measurements conducted on marble and cement mortar samples. The current emitted and its correlation to the damage development in the samples bulk was studied. Specifically, several experiments were conducted by applying constant stress rate or deformation rate on marble and cement samples and the emitted Pressure Stimulated Current (PSC) was recorded.

The diagrams that correlate the emitted PSC and the stress, for both cement and marble samples show up three regions in which the PSC increases with the stress. On the basis of the above, using the form of the curves one may quantify the damages that have occurred within the bulk of the sample. The results proved that the form of the emitted current had the same characteristics for both materials showing this way that the underlying physical mechanism is the same.

Introduction

Mechanical damages in the structure of geomaterials lead to specific changes of various physical gradients. It has been experimentally verified that mechanical stress application on geo-material samples is accompanied by the production of weak electric variations. Several laboratory experiments have been conducted to study the behaviour of geo-material samples under stress and have showed electromagnetic activity as well as electric current emissions. More precisely, experiments have been conducted on rock specimens suggesting that the electric signals are produced by the piezoelectric effect due to presence of quartz [1,2], electrokinetic effect due to water movement [3,4], point defects [5,6], emission of electrons [7,8], moving charged dislocations (MCD) [9-11].

Recently, in a series of laboratory experiments conducted mainly on marble samples it has been confirmed that the application of a uniaxial stress on solid dielectric materials is accompanied by the production of weak electric currents that have been described by the term Pressure Stimulated Currents - PSC [11-16]. In the above experiments uniaxial compressive stress increasing either stepwise or at a constant low rate up to fracture was applied. Experimental results show that in each stepwise PSC emission the PSC peak becomes greater as stress reaches the vicinity of fracture. In the case of an increasing stress applied at a constant and low rate up to fracture PSC, is developed after the linear – nonlinear region limit of the mechanical behaviour of the sample.

According to the Moving Charged Dislocations (MCD) model, when a brittle material is uniaxially compressed with a time varying stress S, the Pressure Stimulated Current can be related with the following [11,13] :

$$I = c \cdot \frac{d\varepsilon}{dt} = c \cdot \frac{1}{Y} \cdot \frac{dS}{dt} \qquad (1)$$

where $d\varepsilon/dt$ is the compressive strain rate, c corresponds to a proportionality factor, dS/dt is the stress rate and Y is the modulus of elasticity (Young's modulus) that is constant in the linear-elastic behaviour region, varies in the non-linear deformation region of the composite material and in the localized failure crack zone that follows as a result of microcrack formation.

In this work for the very first time systematic comparative PSC measurements were conducted for hardened cement mortar sample and marble sample that were subjected to a time varying uniaxial compressive stress, up to fracture. At a first approximation, it is intended to check for similarities and differences in the behaviour of the two materials in the emission of PSC.

1. Materials and Experimental Technique

1.1 Materials

Marble and cement mortar samples have been used in this work. Marble belongs to the class of metamorphic rocks. Its structural inhomogeneities are due to either natural or man-made causes like the application of mechanical or chemical processing. On the other hand, Portland cement is a widely used material because of its ease of preparation, molding and low cost.

The marble samples were collected from Mt. Penteli, Attica. Marble is mainly composed of calcite (98%) and other minerals, such as muscovite, sericite and chlorite. Its content in quartz is very low (0.2%), while its density is 2.7 gr/cm^3 and its porosity is approximately 0.4%. Matrix rocks of the above origin were intentionally selected to be quasi single grained.

Table 1. Incorporates the characteristics of the samples including their geometric dimensions, the fracture limits and the applied stress and strain rates.

sample type & code	dimensions (mm)	maximum strength (MPa)	stress rate or strain rate
MD01 (Marble)	69.6x49.0x149.1	82±4	215 kPa/s
MD02 (Marble)	62.1x69.7x146.8	82±4	0.5μm/s
CM01 (Cement)	50.0x50.0x50.0	59±3	180 kPa/s

The mortar was composed of Ordinary Portland Cement (OPC), sand (SiO_2), and water at a ratio 1:3:0.5 respectively. The samples were measured 3 months after their construction in order to get aged properly and achieve an approximate 95% of their maximum strength. The diameter of the sand grains of the composition varied between 0.8mm and 2mm. Its density was 2.2 gr/cm^3 and its porosity was approximately 8%. Table 1 contains the dimensions of the used samples.

1.2 The Experimental Techniques

The experimental apparatus and technique have been duly described in previous works [14, 15]. The experiment was conducted in a Faraday shield to prevent from electric noise. Between each sample and the stressing system, thin teflon plates were placed in order to provide electrical insulation. The electrodes were attached to the samples, using conductive paste. Disk – like gold plated electrodes measuring 40 mm in diameter were used in all measurements in order that all PSC values be comparable. For PSC measurements a sensitive programmable electrometer Keithley 6517 has been used. All the recordings originating from the load cell and the electrometer were stored in a PC through a GPIB interface.

Two experimental techniques describing the stress development have been used to conduct the experiments. The technique LSR (Low Stress Rate) technique can be described as gradual increase of the stress with constant rate of the order of a few hundred kPa/s. This technique is in detail described in [15]. In the second technique the sample is subjected to deformation at a constant rate (Constant Rate Deformation – CRD technique). Both the stress rate and the strain rate are given in Table 1 as well as the ultimate compressive strength of the samples.

1.3 The Stress-Strain Curves of the Materials

The stress-strain curve provides information regarding the mechanical status of the sample. Fig. 1 depicts the stress-strain curves of the two used materials. The vertical axis corresponds to the relative compressive stress s which stands for the instant stress value S over the ultimate compressive stress S_{max} (i.e. $s=S/S_{max}$). Based on the above, the stress-strain curves can be subdivided in the following typical regions.

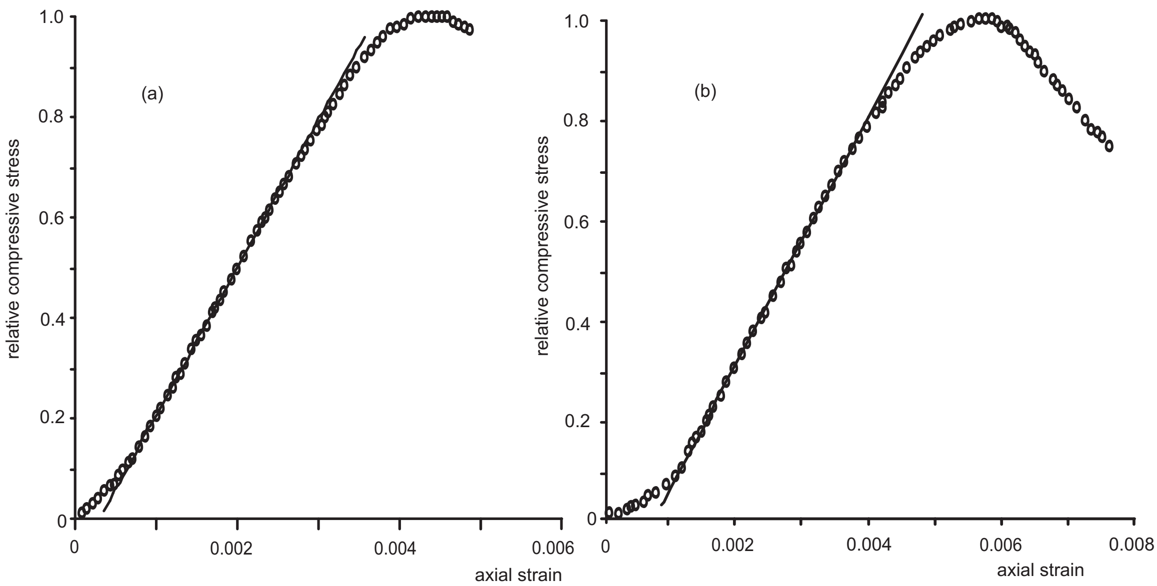

Figure 1. Stress–strain diagram of the marble sample (curve a) and cement mortar sample (curve b).

The first region shows up in the beginning of stress application (s<0.1). This region corresponds to the low stress transient part of the curve where the stress is not linearly related to the strain and occurs before the linear region shows up. Next comes the linear region which covers a region of relative compressive stress up to approximately 0.70 for both materials. The non-linear region follows, in which the Young's modulus continuously decreases up to failure.

2. Results and Discussion

2.1 Experiments with the LSR technique

Experiments with Low Stress Rate Technique were conducted on marble and cement mortar (codes MD01 and CM01).The applied uniaxial compressive stress rates were 215 MPa/s and 180 MPa/s respectively. Fig. 2 and 3 depicts the recordings of the PSC emitted in a logarithmic scale along with the relative compressive stress from marble and cement mortar samples respectively.

As long as the two samples are stressed uniaxially at low stress corresponding to relative stress values lower than 0.70 approximately, the PSC values are very small. Particularly in the limits $0<s<0.25$ the values of PSC do not exhibit significant variations and vary close to the electrometer noise background. As the applied stress increases ($s>0.25$) the values of PSC exhibit a small incremental tendency (for exponential increase), that can be described as follows:

$$I = I_O \cdot \exp(\alpha \cdot s) \qquad (2)$$

where I_O is a current constant and α is a characteristic exponent describing the magnitude of the PSC increase in the referred region. Computer fitting in the recorded values I with respect to s in the same region yields a value approximately equal to 2 for the exponent factor α for both samples (see Table 2).

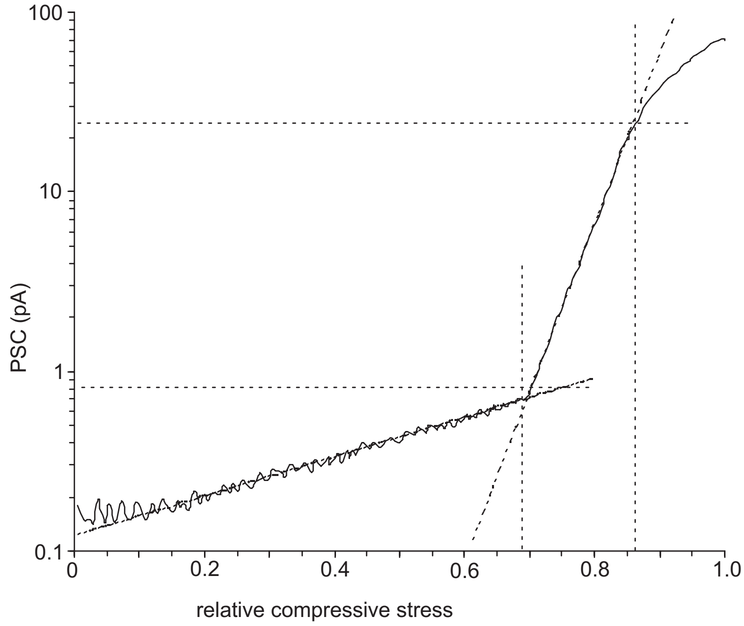

Figure 2. The PSC vs. s diagram for the marble sample.

According to the MCD model as Eq. 1 indicates and given that in the LSR technique (dS/dt = const.), no PSC appearance should be expected in a region of relative compressive stress less than 0.7. The fact that a very weak but distinguishable PSC is recorded, must be related with the activation of some microcracks which might start from the interfaces between grains, especially in regions of the material near the surface of the compressed faces of the sample. When the relative compressive stress upon the sample becomes greater than 0.70 then, a very intense exponential increase of the PSC values is observed which is directly related with the fact that the material has been driven into the non linear deformation region (see Fig. 1). In this region, the Young's modulus gradually decreases and the PSC emission is also expected by Eq. 1 due to the occurrence of microcracks. This region ends with the appearance of localized failure cracks.

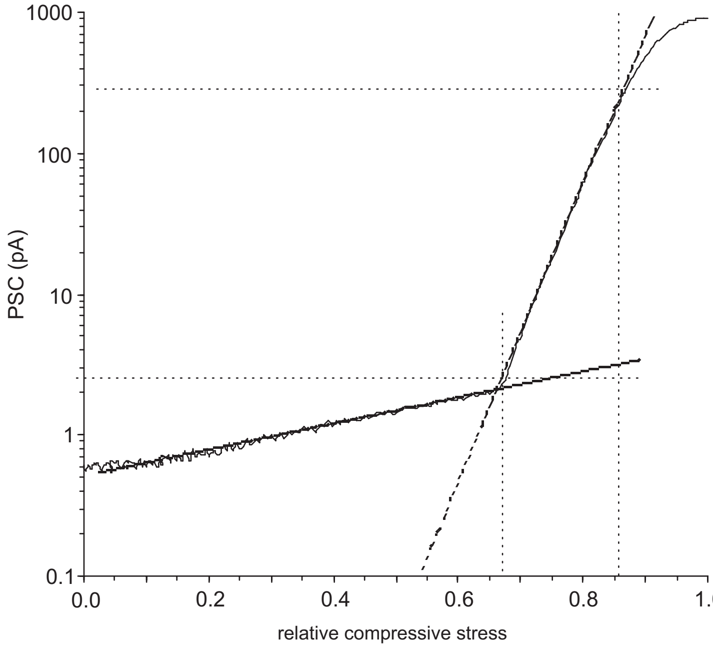

Figure 3. The PSC vs. s diagram for the cement mortar sample.

If the PSC values are correlated with the relative compressive stress values in the region (zone of nonlinear deformation: 0.7<s<0.85) are described by an exponential law of the same form with Eq. 2. The characteristic exponent a describing the magnitude of the PSC increase in the referred region, (after fitting) has a value $\alpha \approx 24$. The deviations of PSC values from the exponential law are evident when the relative compressive stress becomes greater than the value ($s \approx 0.85$), a value corresponding to a transition zone signaling the onset of unstable crack growth and the material enters a localized failure crack zone. In this region the PSC values keep on increasing intensely and as the ultimate strength value is reached, the PSC gets to a maximum.

Table 2. Fitting results for the exponent α based on the I - s measurements of the samples used with respect to Eq. 2.

sample	value exponent a	relative stress region	technique
MD01 (Marble)	2.5 21.7	0.15<s<0.70 0.70<s<0.85	LSR
CM01 (Cement)	2.1 24.3	0.15<s<0.65 0.65<s<0.85	LSR
MD02 (Marble)	2.4 23.5	s<0.65 0.70<s<0.85	CRD

The only difference between marble and cement mortar material samples that can be noticed after reading the PSC recordings from the experiments during all stages of the compression procedure is related with the PSC magnitudes. More precisely, the PSC values of the cement mortar are evidently greater than those of marble by approximately one order of magnitude. This fact can be a factor of differentiation between the properties of cement and those of rock materials. Table 3 shows the PSC magnitude for three characteristic values of relative compressive stress for all samples.

Table 3. PSC magnitudes for three characteristic values of relative compressive stress for all samples.

Sample	Technique	Times PSC (pA)		
		s≈0.70	s≈0.85	s≈1
MD01 (Marble)	LSRT	0.8	25	85
MD02 (Marble)	CRD	0.4	15	60
CM01 (Cement)	LSRT	25.0	300	970

2.2 Experiments with the CRD technique on marble

Experiments using the CRD techniques were conducted on marble samples, the results of one of which (coded MD02) are presented here. The experiments were conducted at a continuously increasing uniaxial compressive stress such as to keep a constant deformation rate equal to 0.5μm/s. Fig.4 depicts simultaneously the temporal recording of PSC (curve b) and the temporal recording of uniaxial compressive stress s (curve a). When the material enters the nonlinear deformation region (s>0.7), the stress rate continuously decreases and diminishes at s=1, when the sample fails. As can be seen in Fig. 4, the PSC peak at fracture is clearly distinguished. The PSC emission behaviour of the sample with respect to relative compressive stress s is similar to the one shown up with the LSR technique. The benefit of this method is that it shows up the PSC peak at failure in detail, due to the slow stress rate.

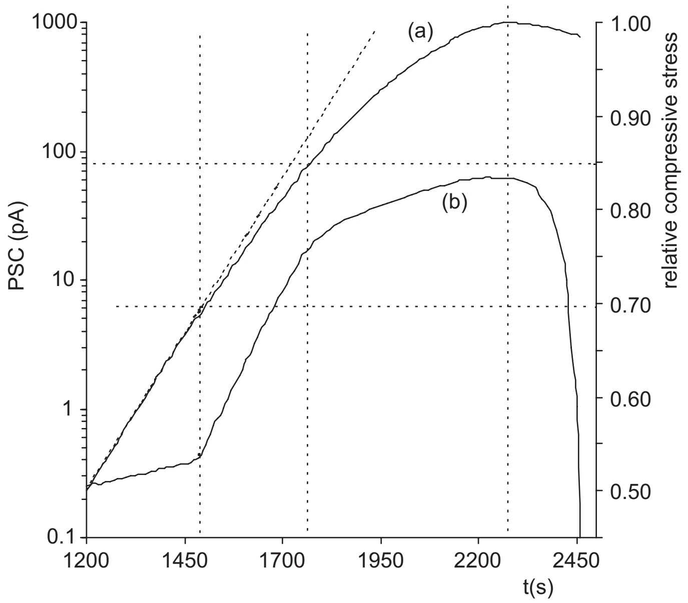

Figure 4: Detailed representation of the temporal variations of both PSC (curve b) and relative compressive uniaxial stress (curve b) applied on a marble sample while the deformation rate is kept constant.

If computer fitting is employed on a PSC vs. s diagram, based on Eq. 2 for regions s<0.70 and 0.70<s<0.85 then, the yielded exponent α values are comparable with those of the other material samples (see Table 2). The smaller PSC values of the MD02 marble sample with respect to MD01 are expected [15] because the MD02 sample was compressed at a lower stress rate. In the region s<0.7 (linear region) it keeps the constant value 55 kPa/s approximately, a value well below 215 kPa/s at which the sample MD01 is compressed.

3. Conclusions

In this work for the very first time systematic comparative PSC measurements were conducted for hardened cement mortar sample and marble sample that were subjected to a time varying uniaxial compressive stress, up to fracture. PSC emissions for both cement and marble samples show up three regions in which the PSC increases with the relative stress value s. For the first two regions (s<0.70 and 0.70<s<0.85) and before the unstable crack growth region an exponential law of the form $\mathrm{PSC} \propto \exp(\alpha \cdot s)$ seems to dominate but with different values of the exponential parameter α. For both samples, the values of the exponential parameter α in the mentioned ranges are comparable ($\alpha \approx 2$ and $\alpha \approx 23$ respectively). In the third region (s>0.85) which is characterized by the stable growth of localized failure cracks and up to sample failure there is a considerable PSC increase with a tendency to reach peak values at failure where the crack creation and propagation processes are unstable.

It should also be noted that the experimental results are comparable irrespectively of the material sample being stressed at constant stress or constant strain. The real difference in PSC recordings of the two material samples has to do with the magnitude of the cement mortar PSC which is at least one order greater than that of marble in all regions.

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