Pressure Stimulated Current Emissions on Cement Paste Samples under Repetitive Stepwise Compressional Loadings

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The electric signals detection technique that is described here was applied on several geomaterials in the past and on cement based materials lately. In this work cement paste samples were studied regarding electric signal emissions during axial stress application processes and specifically when the samples were subjected to repetitive loadings and unloadings in the range where crack opening and propagation processes are established. It was observed that the electric signal was emitted in two stages. Initially, current was emitted simultaneously with the stress step in the form of a spike which gradually returned to its background level. A secondary current emission was recorded while the stress was maintained constant at the high level of each stress step.

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0 INTRODUCTION

The study of the properties of cement products has spurred scientific interest as they constitute the main structural materials. Several techniques have been applied in order to monitor the health of cement constructions. Some of them involve the electrical properties of cement. Since health monitoring does not provide the flexibility to extract cement samples from the constructions, non-destructive testing methods are the most suitable for application.

For many years it has been known that electric and electromagnetic (EE) signals can be observed when solids and especially non-metallic materials, are mechanically stressed. Such signals have been reported by [1] to [5]. Micro- and macro-cracking processes are often accompanied by these signals. Several mechanisms for the EE signal generation have been discussed in literature. Rapid movement of electric charges, separation of electric charges at crack formation and their recombination to form a miniature spark discharge, rapid movement of electric double layers under the action of the mechanical loading or piezoelectric phenomena are some which have been reported and studied by [2], [6] to [9].

In previous works, processes of electrical emission in rock samples like marble and amphibolite were studied by [10] to [14]. The emitted current during the temporal stress variation that leads to catastrophic processes in the bulk of the samples and finally to their fracture, has been rendered under the term Pressure Stimulated Current (PSC). The technique applied to detect and record such electric emissions is mentioned as the PSC technique. The relevant literature refers to electric signal emissions observed with similar techniques regarding electrical emission in mortars under low compressive loading [15] and the appearance of an electric current that increases nonlinearly with compressional stress [9].

During this work cement paste samples were subjected to stress adequate to lead them to the Crack Propagation Zone (CPZ). For the used samples this zone is estimated to be reached with compression at around 11 MPa. Consequently, repetitive mechanical loadings and unloadings were applied on the samples. Between each loading and unloading the stress level was maintained at its high value for a relatively long time. The emitted PSC during this process was measured and is presented and discussed here.

1 EXPERIMENTAL CONFIGURATION

A set of cement paste samples was prepared for the measurements. The dimensions of the samples were $40 \times 40 \times 40$ mm. The proportion of the contents of the OPC (Ordinary Portland Cement) was 2:1. The drying time of the

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samples was 90 days. Conducting preliminary systematic tests with various stress modes such as monotonically increasing stress at a constant rate, or maintaining high stress levels for a long time, or applying sequential stepwise stress increases up to a point of failure of the average ultimate compressional strength of the sample, was found to vary around 25MPa \pm 5 MPa. Consequently, samples prepared from the same mixture were used to conduct the experiments.



Fig. 1. A representative curve that describes the relative compressive stress with respect to strain for the used samples

Fig. 1 shows a representative relative compressional stress $(\hat{\sigma})$ – strain (ϵ) curve of the used samples. The relative compressional stress value is given as $\hat{\sigma} = \sigma/\sigma_{max}$ where σ_{max} corresponds to the ultimate compressional strength of the sample. It is evident that it can be

characterized by a linear behavior at least up to a stress of approximately 80%, of the ultimate compressional strength (i.e. $\hat{\sigma} = 0.8$). When $\hat{\sigma} > 0.8$ approximately, the material is driven to a range of non-linear deformation and eventually into the localized failure zone.

Fig. 2 shows the experimental installation. For the implementation of this experimental technique a pair of gold plated copper electrodes was attached at the perpendicular axis of the stress. The measurements were recorded using a Keithley electrometer (model 6514). Electric measurements were stored in a computer hard disk through a GPIB interface while the load cell and the strain gages bridge were guided to an A/D Keithley DAQ. The stressing system comprised a uniaxial hydraulic load machine (Enerpac– RC106) that applied the load to the samples. The experiments were conducted in a Faraday shield to prevent electric noise.

The sample under test was slowly loaded up to a value of approximately 50% of the compressional ultimate stress strength. Consequently, a stress increase was applied at a relatively high rate and the stress maintained its high value for 10 min. Afterwards the stress was removed until the level of 50% of the ultimate stress strength was reached. This procedure was repeated three times. Consequently, the stress was further increased in the vicinity of the fracture and after some time the sample failed without further increasing the stress. During this entire process the emitted PSC was recorded.



Fig. 2. Schematic diagram of the experimental setup.

2 RESULTS AND DISCUSSION

Fig. 3 shows the temporal variation of the three repetitive mechanical loadings and the corresponding emitted PSC. Specifically, the upper plot (a) corresponds to the applied compressional stress and is graded in MPa, while the three lower (b,c and d) are graded in pA and correspond to the temporal variation of the emitted PSC during the three repetitive loadings. Two kinds of PSC emissions can be seen in Fig. 3. Specifically, a primary current that is emitted simultaneously with the stress increase from the lower to the higher level is observed. This current is restored relatively fast. A secondary current emission that takes place while the stress is maintained practically constant is also observed. It is obvious that both PSC emissions during each following loading become weaker.

The primary PSC emission is attributed to the crack formation and propagation processes

that are measured by means of the corresponding deformation. The reduction of the peak value of the PSC can be attributed to the electric emission memory effect that has already been discussed and interpreted in previous works that refer to PSC emissions from rock samples like marble [14] and [16] and amphibolite [13].

Fig. 3 also shows the secondary PSC emissions that take place after stabilizing the stress at the corresponding high level of its stress increase. The stress level that was maintained after each stress step was approximately 16.5 MPa. It becomes obvious that despite the fact that there is no stress variation, a significant PSC is emitted. This can be attributed to the fatigue of the sample due to the opening of new cracks formation or propagation of the existing ones since the material is already in the Crack Propagation Zone (CPZ).



Fig. 3. a) Plot of a representative stress step which after a 3-fold repetition produced primary and secondary PSC emissions during the first; b) the second; c) and the third; d) stress step, respectively

Fig. 4 depicts the last stress step (Fig. 4a) that was performed from 16.5 MPa up to 22 MPa approximately. The sample suffered this loading for 5 min and consequently it failed due to fatigue. During this compressional stress step a significant PSC emission was observed and lasted until the sample failed. During this final stress step the primary PSC emission cannot be distinguished from the secondary and they seem to overlap. The fact that the duration of the emissions is long (from the stress change to fracture) and has a high magnitude in combination with the fact that the primary emission was never restored, are the factors that predict the fracture of the sample which finally took place at the time $t_f = 270$ s (see Fig. 4a). Another observation is that before the sample fracture the secondary emission has a brush-like form introducing the upcoming fracture. This prior-to-fracture PSC emission (Fig. 4b) is put in

contrast to the PSC emission of the first stress step (Fig. 4c) so that the changes in the form of the emitted PSC slightly before fracture become clear. The deformation, after the application of a compressional stress step, continues to increase (hysteresis). This phenomenon becomes more intense as the sample reaches the ultimate stress strength.

The above findings become obvious in Fig. 5 where the temporal variation of the emitted PSC (Fig. 5a) and the corresponding temporal development of the deformation are depicted (Fig. 5b). Here the deformation continues to increase despite the fact that the stress is maintained practically constant.

During this process the PSC becomes more intense due to the fact that the strain increases. Slightly before failure and while the strain increases at a gradually higher rate a brushlike PSC indicates the upcoming failure.



Fig. 4. a) The final stress step in the vicinity of fracture and b) the corresponding emitted PSC; (c) the PSC emission during the initial stress step at lower stress level in order to observe the time window between the primary and the secondary PSC emissions



Fig. 5. *a)* The temporal variation of the PSC during the last stress step and b) the corresponding temporal recording of the deformation

3 CONCLUDING REMARKS

Cement paste samples were studied using the PSC technique. The recorded PSC showed a primary emission detected during the stress increase and a secondary one recorded while the stress was maintained constant at the higher level of each stress step. The PSC emissions are attributed to the crack formation and propagation processes and the consequent deformation. The experimental results were discussed according to this theoretical background. The primary PSC emission was simultaneously with stress attributed to the deformation increase and the secondary emission was attributed to the deformation hysteresis mechanisms. Another experimental observation was a lower value that the PSC reached after each stress application and this result was put in contrast to similar previous results recorded and discussed for geomaterials like marble and amphibolite. In conclusion, the PSC technique and the qualitative characteristics of the emissions observed can become a significant factor in monitoring the health state of cement paste using a non destructive method.

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