The influence of externally applied uniaxial stress on Isothermal Depolarization Current mechanisms in rock samples

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The technique of Isothermal Depolarization Currents (IDC) was applied on marbles collected from Mt. Penteli (Greece) in order to investigate the influence of previously applied uniaxial compressional stress on IDC relaxation processes. The applied stress varied from early elastic range up to fracture. After each stress application IDC measurements were conducted and the relaxation parameters concerning fast and slow depolarization processes were studied. Experimental results manifest a differentiation in the shape of the depolarization current curves for the stressed and un-stressed samples. Specifically, during the final stage of the depolarization current decays faster when the geo-material has suffered stress adequate to cause microcracks in its structure, than the unstressed samples. Additionally, for severely damaged samples the initial stage of depolarization, dominated by "*flip*" transition mechanisms, lasts longer. It is concluded that the stress level influences "*flip-flop*" transition mechanisms that dominate the final depolarization stage and has no influence on "*flip*" transition mechanisms of the very initial stages of dipole rearrangement.

1. Introduction

Mechanical stress upon rocks creates microscopic or macroscopic discontinuities resulting in changes in the mechanical behaviour of the solid [1, 2] and consequently, changes its electrical characteristics like conductivity [3–5] and stimulated charge flow [6, 7]. In the time domain, the isothermal polarization and depolarization with a long voltage pulse, is used to study the changes of the electrical properties of materials [8–10].

The phenomena of polarization and depolarization are followed by a current through the sample: polarization or depolarization current, respectively. These currents may show contributions from the transport of the charges from the bulk of the sample as well as the charges injected from the electrodes. In many structures localized states of different origin may occur. The localized states may influence atom interaction and carriers motion.

Time domain analysis of depolarization or decay current process can provide valuable information regarding the dielectric response of solids. The time dependent relaxation process following a sudden removal of a polarization field may be considered as a succession of four stages [8]. Each of these stages is dominated by a particular type of transition. The initial stage that is rather short ($t<10^{-12}$s) takes place immediately after removing the applied field. This stage represents the delay necessary to establish the *"flip"* processes [8]. During the three of the rest stages the depolarization current can be described by the following laws [8, 9]:

$$
\begin{array}{ll}
i_{\mathrm{d}} \propto t^{-n} & \text{for } t \ll 1/\omega_{\mathrm{p}} \\
i_{\mathrm{d}} \propto \exp(-\omega_{\mathrm{p}} t) & \text{for } t \cong 1/\omega_{\mathrm{p}} \\
i_{\mathrm{d}} \propto t^{-m-1} & \text{for } t \gg 1/\omega_{\mathrm{p}}
\end{array}
$$

The second stage that is dominated by the n factor is the result of the small *flip* transitions which is stimulated by great quantities of dipoles that attempt to relax and their interaction due to the forces among them. During this second stage the probability of small *flip* transitions to occur is much higher than "*flip-flop*" transitions (i.e. local dipole moment fluctuations which retain the average value of the total polarization [9]) on the account of the very large number of dipoles required to make a transition in the early stages of relaxation. The *flip* transitions dominate the relaxation process up to $1/\omega_{\mathrm{p}}$ which represents the delay time for the onset of a Debye-like large transitions dominating the relaxation process during the third stage. During the last stage(i.e. $t \gg 1/\omega_{\mathrm{p}}$), where polarization reaches equilibrium. the directional processes of the previous stages are replaced by the fluctuations of the *flip-flop* transitions during which there is no further change of the dipole moment but instead local redistribution of the energy occurs providing slow relaxation processes according to the t^{-m-1} power low. It must be clear that all three

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types of described transitions take place continually but at various rates during each stage.

The intensity of the electric field applied to the sample is one of the main factors determining the mechanism of polarization. For weak fields, the probability of nucleation is small and the polarization is mainly due to the motion of domain walls. The sample is polarized only insignificantly; however, the depolarization of the sample may again be reached only a long time after switching off the field. For very weak fields, the movement of the walls is reversible and the polarization disappears soon after removing the field. There is no remanent polarization. For fairly strong fields and for relatively long polarization time, the sample may achieve either a monodomain or come very close to a monodomain state. After switching off the field, domains randomize. The collapse process is rapid at the beginning of the depolarization state after removing the field, and becomes much slower in the final state of depolarization.

Polarization field intensity, polarization time and temperature have been evaluated as factors that influence depolarization parameters (i.e. n, m and ω_p) [10].

In this work, damages in the structure of a metamorphic rock (marble) due to externally applied uniaxial stress were studied by applying the Isothermal Depolarization Current (IDC) technique. Microcrack and macrocrack fingerprints on the basic IDC parameters were studied. The samples were compressed up to various stress limits and the deformation was studied with respect to the depolarization power laws exponents. Specifically, the influence of the gradual increase of microcracks and their further development into macrocracks on the "time dielectric relaxation" of the samples was investigated. It was experimentally proved that structural changes affect depolarization and specifically the *flip flop* transition mechanisms that last longer and dominate the final depolarization stage.

2. Samples—Experimental setup

The experiments have been conducted using samples of Pentelicon marble (Dionisos) collected from Mt. Penteli, Attica. The samples are composed of 98% of calcite, and 2% of other minerals. The geo-material can be characterised as quartz-free since its content is less than 0.2%. Its porosity when the material has never suffered any stress is 0.371%.

Fig. 1 depicts the form of a representative stress-strain curve for the used marble samples. In Fig. 1 the stress axis is normalized to the ultimate compressional stress strength (i.e. S_{max}) described by the ratio s=S/S=$_{max}$. The mechanical behavior of marble during stress application can be described as follows. Initially, for low stress values and due to the existing discontinuities closing (Griffith cracks, [11]) the solid deforms plastically. This range is limited to the 0.1 approximately of the S_{max} of the sample. This is the maximum stress that can be applied on the sample before failure. Beyond this point and in the elastic range the applied stress is linearly related to the strain and this range extends up to $0.7S_{max}$ approximately. When stress becomes higher than $0.7S_{max}$, the sample deforms permanently. This range lasts up to fracture. Regarding the microcracks formation process, this does not occur for stress lower than $0.7\mathrm{S}_{max}$. From this point up to $0.98\mathrm{S}_{max}$ gradual increase of microcracks occurs until the first macrocrack takes place and forms the fracture plane. Beyond this point the fracture process becomes irreversible concerning the plane the sample will collapse. Stress-strain curve depends on the stress history, temperature, pore water, and anisotropy as well as viscosity and rigidity of the sample. Since all our samples come from the same rock mass and they were all maintained in a controllable environment, their stress-strain curves are not expected to deviate.

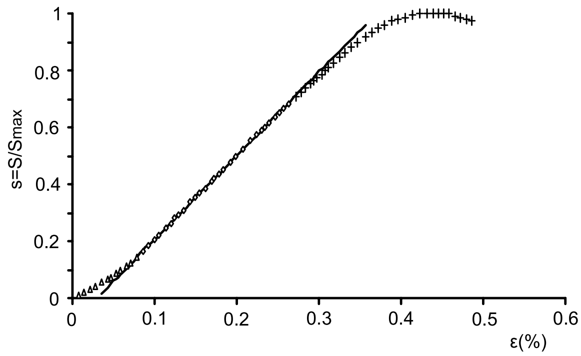

Figure 1 Experimental stress-strain curve for marble samples.

The used samples were cylindrical with diameter 30mm and height 10mm. Special care was taken to achieve constant temperature (295K) and hydration level of the order of 0.07% for all the used samples during all experimental procedure since these are factors that influence in the IDC .

The uniaxial compressional stress, S, was applied on the samples for time t_s = 300s and then it was removed and the samples remained unstressed for time t_r = 6 ks before performing the IDC measurements. The stressing system comprised a uniaxial hydraulic load machine (Enerpac–RC106) that applied compressional stress to the sample. The samples were classified in five different groups according to the uniaxial stress that they were about to suffer (see Table I).

In order for the depolarization currents to be measured the applied polarizing voltage to all samples was V_p=1000V reminent for polarization time t_p=30min. Fig. 2 shows the schematic diagram of a typical IDC measurement set-up. A Keithley 617 programmable electrometer was used to perform the measurements.

TABLE I Samples classification with respect to the previously applied mechanical stress

Sample code	Previously applied mechanical stress
MDNS	No stress
MDSL	Uniaxial stress in the linear range (elastic behaviour)
MDSLP	Uniaxial stress in the limits of elastic-plastic region
MDSP	Uniaxial stress in the plastic region
MDSF	Uniaxial stress in the vicinity of failure

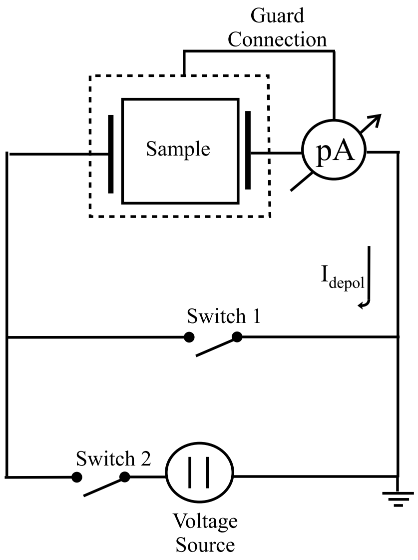

Figure 2 Schematic of the electric circuit used to perform the measurements demonstrating depolarisation processes.

3. Results—discussion

Fig. 3 demonstrates in log-log plot the recorded values of the isothermal depolarization currents with respect to time for four out of five classification groups (see Table I). The previously applied uniaxial stress of the referred samples is described by the normalized ratio s=S/S_{max} in Table II. The initial obvious results are the following: Time response of the decay current for all samples was characterized by two distinct branches of different slopes $-n$ and $-(m+1)$. The near-Debye process describes the transition part between the two branches. The samples (MDSP) that were stressed in the plastic range exhibit higher values of decay currents that become even higher in (MDSF) samples stressed in the vicinity of fracture. The decay currents of the samples (MDSL) that were stressed in the linear region are almost identical with those of the unstressed samples (MDNS). This is the reason for not demonstrating decay currents in the elastic region in Fig. 3. An obvious increase of decay currents is expressed for MDSLP samples in the limits of elastic and plastic range.

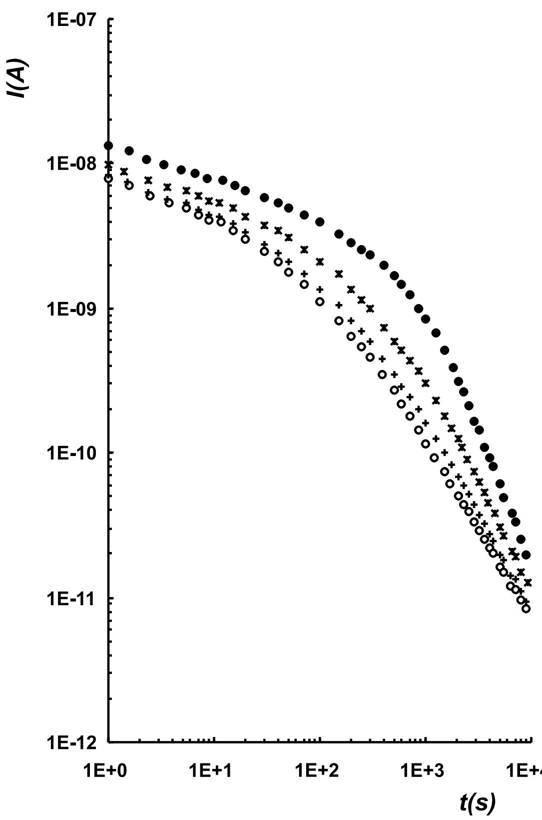

Figure 3 Representation of the depolarization currents in log-log diagram for the samples MDNS-02 (*open circles*), MDSLP-01 (+), MDSP (∗), MDSF (*solid circles*).

TABLE II Exponent values with respect to s

Sample	$s = S/S_{max}$	Exponent values n	Exponent values $m+1$
MDNS-02	0	0.29 ± 0.05	1.21 ± 0.03
MDSLP-01	0.67 ± 0.03	0.26 ± 0.05	1.30 ± 0.03
MDSP-03	0.84 ± 0.03	0.24 ± 0.05	1.53 ± 0.03
MDSF-02	0.93 ± 0.03	0.23 ± 0.05	1.86 ± 0.03

It is important to notice the influence of the applied stress and the subsequent microstructural changes on the values of the two exponents (n and $m+1$) describing depolarization stages. The values of the exponent n that corresponds to the "short" times (i.e. second depolarization stage) do not exhibit major variations for all the measured samples. The calculated values of the exponent n are presented in Table II. The values of the exponent $m+1$ that correspond to "long" times (i.e. the fourth stage of the depolarization process) vary considerably depending on the previously applied mechanical stress. This can be attributed to the newly crack-originated boundaries. For a sample that is severely damaged and dominated by fast transitions from initial stage, the crack boundaries limit the number of *flip-flop* transitions (fourth depolarization stage) and current attributed to this mechanism decay faster. This limitation is introduced since the atoms around the crack break the chain of atom interactions and cannot further affect other neighbour atoms. The calculated values of the exponent $m+1$ are presented in Table II. It is also clear that the higher the stress is, the longer *flip* transition mechanism lasts. Since both *flip* and *flip flop* transition are additive mechanisms that exist during the whole depolarization process, each dominating a period of this process, it is expected that if one of these is weak then the other should dominate relaxation for a longer time. Thus, the weaker the *flip flop* transition is, the longer *flip* transitions dominate IDC relaxation process.

Fig. 4 demonstrates a correlation of the exponent $m+1$ with respect to the values of the normalised stress s. In the same figure the exponent $m+1$ yielded from the IDC recordings from more than ten samples is also illustrated. The samples used to construct this diagram were previously stressed in the range from the onset of the plastic deformation up to the vicinity of failure. It is evident that when s becomes higher than 0.8 the slope of the last branch of the depolarization process changes significantly. In this stress range major structural changes take place. It is indicative that this is

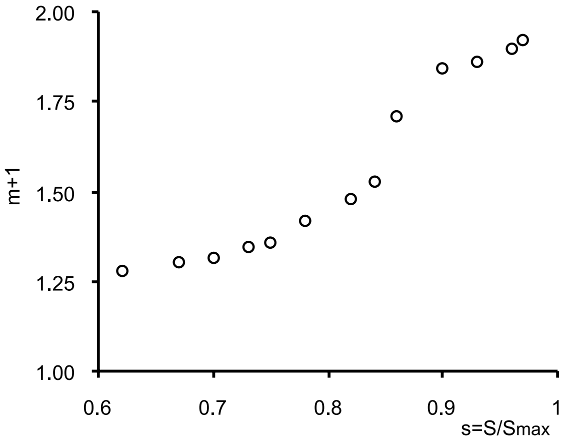

Figure 4 The representation of exponent $m + 1$ with respect to the stress ratio s.

the range that microcracks tend to develop into macrocracks guiding the failure plane.

4. Concluding remarks

In the light of the aforementioned results, the following conclusions can be drawn; When the samples were subjected to stress below the plastic range (as far as the mechanical behaviour of the material is concerned), no important changes were observed in the depolarization currents of marble samples. When the marble sample suffered stress adequate to lead it to the plastic range, the values of the depolarization currents were clearly different and higher than those measured when the sample was subjected to stress corresponding to the elastic range manifesting a clear change in the mechanisms that influence *flip-flop* processes.

The depolarization currents corresponding to "long" times, seem to decrease at a faster rate. This fact is related to the appearance of crack micro-structures and the way these microstructures interact with the polarized dipoles. The crack boundaries are considered responsible for the fast depolarization due to *flip-flop* transitions. When the applied electric field is removed initial fast *flip* transitions dominated depolarization process until the Debye-like stage, after which slow *flip-flop* transitions dominate IDC. Crack boundaries are considered as obstacles regarding atom interactions, thus, the current decay increased rate is attributed to them.

IDC measurements may be considered as a promising non-destructive testing method that can be easily adopted in the research for microcracks and structural imperfections not only in marble, but also in other geomaterials.

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