Investigation and Quantification of Damage in Geomaterials with the Technique of Dielectric Spectroscopy

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Abstract. The technique of Dielectric Spectroscopy (DS) or Impedance Spectroscopy (IS) was applied on marbles in order to investigate the influence of previously applied uniaxial compressional stress and three point bending up to fracture. Parameters to be measured were the ac conductivity (σac), the real (ε΄) and the imaginary (ε΄΄) parts of complex relative permittivity when an ac electric field at frequencies 10kHz and 100kHz was applied upon dry and saturated samples which were successively subjected to higher levels of mechanical stress.

The experimental results indicate that there are systematic declinations in the values of the above parameters between the state of stress and the state of relaxation. These declinations become more intense at higher stress values. It is concluded that the technique of dielectric spectroscopy constitutes a promising method of investigation and quantification of the damage that has occurred within the bulk of geomaterials.

Introduction

Marble is a metamorphic rock. Imperfections in its structure are usually due to either internal or external factors such as mechanical strain, chemical or physical processing and play an important role in the behaviour of the material. Mechanical stresses upon rocks create microscopic and macroscopic discontinuities resulting in changes in the mechanical behaviour of the material [1-3].

Various laboratory experiments on marble samples have been conducted recently in order to determine the way that stress and consequent mechanical damages influence the electrical properties of geomaterials. Such experiments include Dielectric Spectroscopy in the frequency domain [4,5] and Isothermal Depolarization Currents [6].

Dielectric properties are related with the capability of a material to be polarised under the influence of an electric field. The polarisability of a material depends on its structure and molecular properties and therefore dielectric measurements can provide information in this respect. Dielectric Spectroscopy (DS) is an electrical technique that has been used to characterize the rock microstructure and to provide useful information about the relationships governing microstructure, electrical properties, and chemical processes during hydration [7-10]. Complex relative permittivity ε^* (hereafter, referred to as complex permittivity for convenience) is defined as:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' = \varepsilon' + \frac{\sigma}{j\varepsilon_0\omega} \qquad (1)$$

where ε' and ε'' are the real and imaginary parts of ε^* respectively, σ is electric conductivity, ε_0 is vacuum permittivity, ω is the angular frequency ($\omega = 2\pi f$) and $j = \sqrt{-1}$. The real part of the complex permittivity is a measure of how much energy from the electric field is stored in the material. The imaginary part of the complex permittivity is a measure of how dissipative or lossy the matter is, in relation to the electric field.

In this work, marble samples suffered mechanical stress in two modes in order for defects to be created in their structure. These tests included uniaxial compressional stress up to fracture and three point bending. The measurements were conducted on both dry and saturated samples. Saturated are the samples that their pre-existing natural moisture is not removed by any laboratory processing. Consequent dielectric measurements were conducted at two distinct frequencies 10kHz and 100kHz in order to determine the change of the basic dielectric characteristics (real and imaginary parts of the complex relative permittivity as well as ac conductivity) due to the damages caused. The results indicated that dielectric measurements are a potential tool for Non Destructive Testing of materials.

1. Materials and Experimental Techniques

1.1 Materials

Marble 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. 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. The dimensions of the samples subjected to compression were 38mmx20mmx9mm, while the samples for bending had dimensions 100mm long and 26mmx10mm in cross section. Samples extracted from the same matrix rock were tested for gradually increasing uniaxial stress. In order to achieve acceptably dry material, the samples were heated for twenty four hours in a chamber of constant 105^{o}C temperature.

1.2 The Experimental Techniques

Uniaxial compression tests are used for characterizing the compressional strength of rock and rock-like materials such as building blocks, marble, concrete, etc. A series of uniaxial compression tests was carried out with prismatic marble samples measuring 38mmx20mmx9mm. A stiff loading frame (MTS-815) was used for performing uniaxial compression tests on the samples. Fig. 1 shows a schematic diagram of the loading apparatus used in these tests. In this apparatus, the spherical seat is incorporated in the upper loading platen of the frame. The load force was parallel to the longest side of the sample. The load on the samples was applied continuously at a displacement rate equal to 0.001mm/sec. At the beginning six sample beams were used to specify the mean value of the ultimate compressional strength of the samples which was found to be S_f =9.6kN.

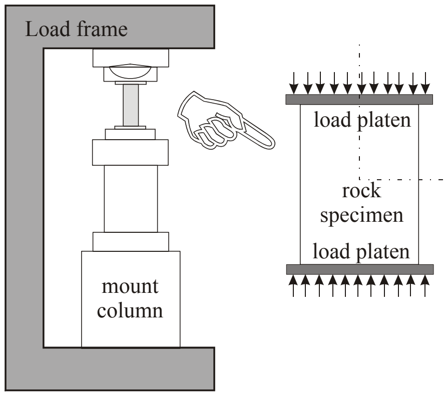

Figure 1: Typical rock testing system

Consequently, the samples were subjected to successive external uniaxial loadings. After each loading dielectric measurements were performed on both saturated and dry samples. Each new loading was at a higher level than the previous one. Thus, dielectric measurements were conducted initially for unstressed samples and then after loading for uniaxial compressional stress (S) corresponding to applied force values 3.2kN, 3.8kN, 4.2kN, 6.0kN 7.3kN and 8.5kN.

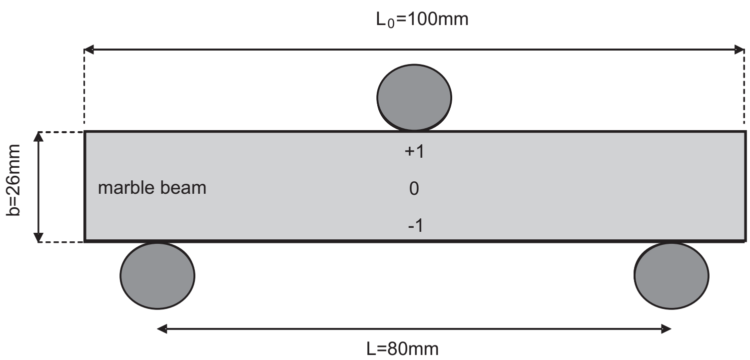

Figure 2: Schematic describing sample beams used for the 3PB tests

A series of three point bending (3PB) tests was carried out with prismatic samples 100mm long and 26mmx10mm in cross section (see Fig. 2). The tests were conducted with a 3PB test system (Tri - Scan 50kN). The samples were placed on two steel rollers at a distance of 80mm from each other. The load was applied at the middle of the upper surface of the beam. In typical testing procedures, the load on the sample is applied continuously at a displacement rate of 0.06mm/sec as recommended by Standard Test Methods (ASTM). The lower zone surface (area: -1, see Fig. 2) was tensed, though the upper zone surface (area: +1, see Fig. 2) was compressed. Between the upper and the lower zones there is a layer which is called natural zone (area: 0, see Fig.2). The length of this natural layer of the beam does not change. The first five beams were used to specify the mean strength of the samples which was found to be 0.97kN approximately. Bending loads ℓ were applied successively on the samples at 0.4kN and 0.5kN. Between each loading dielectric measurements were conducted, on both saturated and dry samples, at the three predefined areas (+1, 0, -1).

The experimental arrangement for conducting the dielectric measurements has been described in previous work [5]. The dielectric measurements were conducted using an LCR meter (Agilent model 4284A), accompanied by the dielectric test fixture (Agilent model

16451B) and further supported by a computer for data recording, storage and analysis. The dielectric test fixture that was used to hold the sample was protected by a cabin providing constant temperature (298K), inert atmosphere by continuous effusion of inert gas and also low humidity. It is important to mention that although the mentioned rock is characterised by its low porosity which is a necessary condition especially when dealing with dried samples as the existing ac conductivity mechanisms depend determinately on the water content of the sample [11].

2. Results and Discussion

For the first set of marble samples, that were subjected to uniaxial compressional stress the ac conductivity was measured in the frequency range 1kHz up to 1MHz. The results presented here correspond to the frequency of 10kHz since it is the most sensitive to show up ac conductivity value variations for loading changes. More specifically, the percentage of the ac conductivity change, at 10kHz, is presented with respect to the continuously increasing applied stress. As reference conductivity we consider the value measured without having applied any stress.

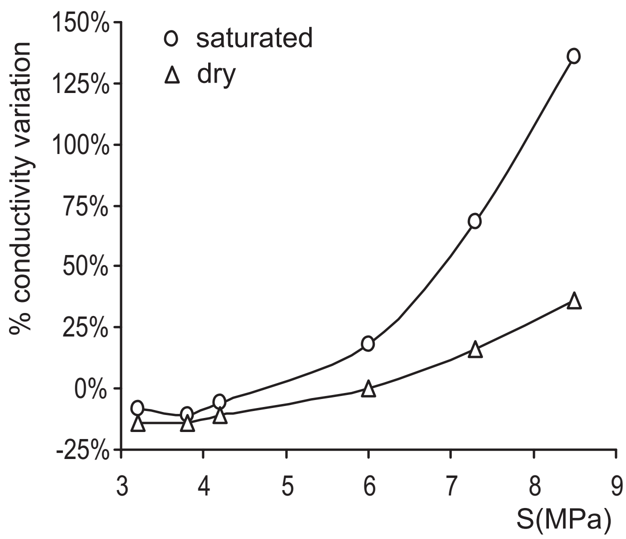

Figure 3: The percentage of the ac conductivity change with respect to the applied stress.

Fig. 3 shows the percentage change of ac conductivity with respect to the applied uniaxial stress for both dry and saturated samples. It is noted that the experimental values of Fig. 3 correspond to the mean values of ac conductivity for a series of measurements on eight tested samples. Fig. 3 also shows that for saturated samples and for stress values smaller than 5MPa the ac conductivity values are slightly smaller with respect to the one of the unstressed samples. For stress greater than 5MPa an intense increase in conductivity becomes obvious. This is due to the greater freedom of charge motion favoured by the creation of extended crack micro-structures. For stress larger than 5MPa microcrack generation and propagation process start in the samples bulk. Regarding the dry samples the applied stress does not cause such intense changes in the ac conductivity values even for high stress values. The above observation supports the hypothesis that the increased conductivity for heavily stressed samples can be attributed to the existence of water that penetrated the sample through surface cracks.

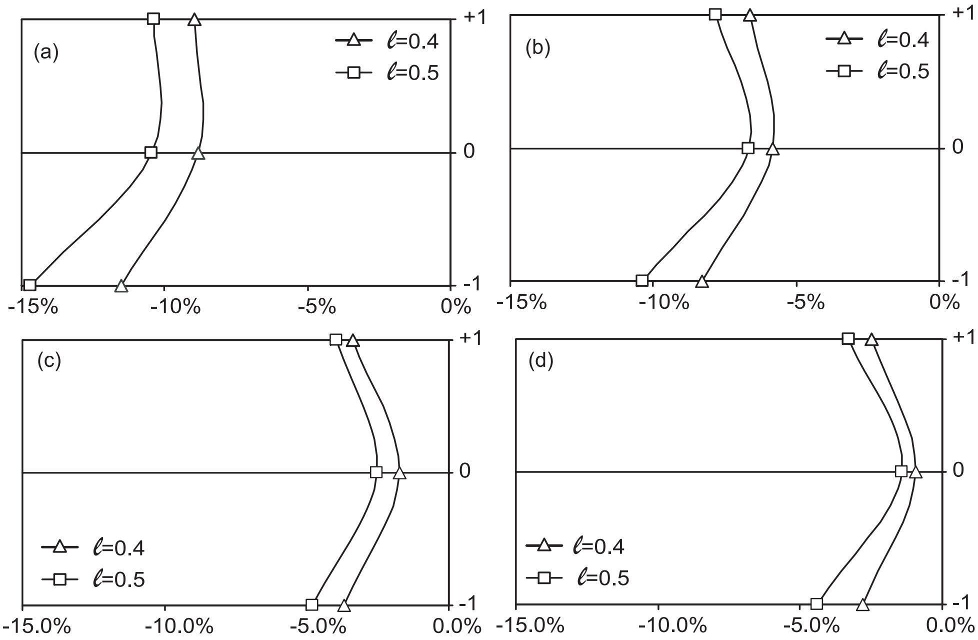

Figure 4: The average % reduction of the real part of the dielectric permittivity with respect to the three selected measurement areas (+1, 0, -1) at the frequencies of 10kHz (curve a) and 100kHz (curve b) for the saturated samples after consequent bending stress (ℓ =0.4 and ℓ =0.5). The curves (c) and (d) correspond to the dried samples.

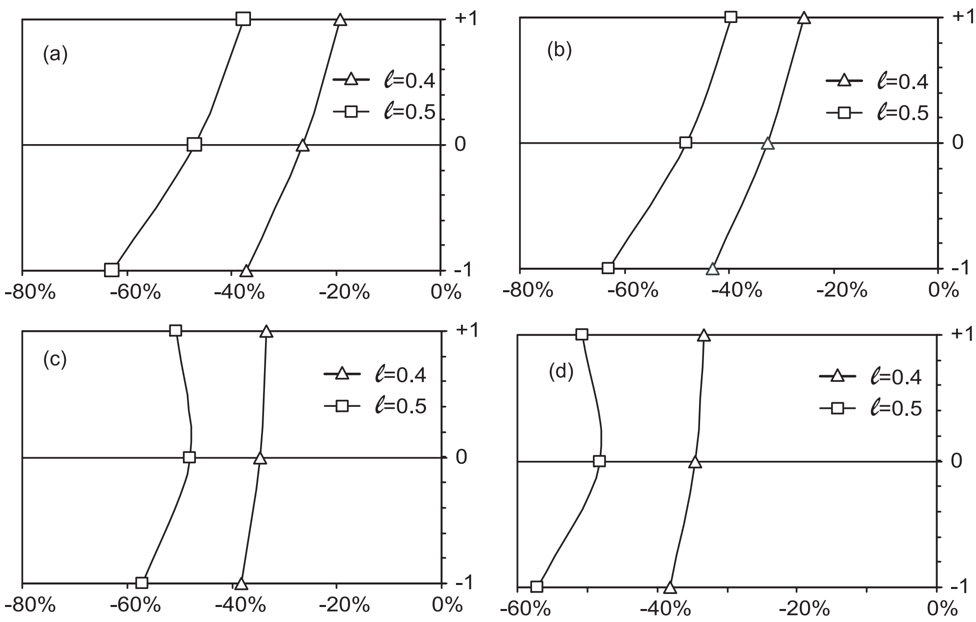

Figure 5: The average % reduction of the imaginary part of the dielectric permittivity with respect to the three selected measurement areas (+1, 0, -1) at the frequencies of 10kHz (curve a) and 100kHz (curve b) for the saturated samples after consequent bending stress (ℓ =0.4 and ℓ =0.5). The curves (c) and (d) correspond to the dried samples.

The changes of the dielectric properties due to bending are summarised in Figures 4 and 5. More specifically, Fig. 4 depicts the behaviour of the real part of the dielectric permittivity with respect to the bending load. A detailed study of the experimental results presented in Figures 4 and 5 can lead to the following comments:

Along the bending region (areas +1, 0 and -1) the applied load causes a reduction of the values of both real and imaginary parts of the complex permittivity of the material. The reduction of the imaginary component is more intense thus its values can be used as an index of the damage processes caused by the bending procedure. It is indicative that the most significant changes are detected at the tensile area (-1) which is in accordance to the fact that the tensile strength of the geomaterials is much lower than the corresponding compressional strength [12-14]. The corresponding changes for the dry samples, as it is expected, are much smaller diminishing the deviations between the three areas +1, 0 and -1 of the bending region.

3. Conclusions

The presented experimental results manifest that mechanical loading, uniaxial compressional or 3PB, causes significant variations to dielectric properties of rocks and specifically marble.

The applied uniaxial stress when leading to microcrack generation in the bulk of the samples makes conductivity to increase due to hydration within the microcracks.

For the 3PB case the real part of the complex permittivity shows a small decrease while for the imaginary part the decrease is much larger for a bending load of the order of 50% of the total strength. It is interesting to further study such variations for larger load values. The results extracted for saturated samples manifest systematic variations of the dielectric characteristics along the bending area.

Acknowledgements

This work is supported from the project ARCHIMEDES II: “Support of Research Teams of Technological Educational Institute of Athens”, sub-project entitled “The electric behavior of geo-materials” in the framework of the Operational Programme for Education and Initial Vocational Training.

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