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NON DESTRUCTIVE DAMAGE ESTIMATION ON ROCKS WITH LABORATORY MEASUREMENTS OF DIELECTRIC LOSS (tanδ)

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ABSTRACT

Non–destructive testing methods constitute a fast growing field of study that concerns various materials. In addition to the up today bibliography a novel technique is presented here. It deals with the use of Dielectric Spectroscopy (DS) or Impedance Spectroscopy (IS) techniques for the verification of mechanical damages introduced in geomaterial structures. The main merit is that these techniques do not influence the materials structure or properties, thus it can be tested with several other methods. In this work damages are introduced in a geomaterial by uniaxial stress application while temperature is constant. Specifically, the damage caused to marble samples and its influence on the dielectric loss angle were measured and examined with respect to the externally applied uniaxial stress. Results indicate that when the applied stress is capable of leading to microcrack generation then tanδ values increase while for high values of stress, a loss peak is evident in the studied frequency range.

***Keywords**:* Dielectric losses, Geomaterials, Uniaxial stress, Damages

1. Introduction

Marble is a rock, which from a geological point of view belongs to the metamorphic rocks used for constructions of buildings and monuments. 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 electrical behaviour of the material. Mechanical stresses upon rocks create microscopic or macroscopic discontinuities resulting in changes in the mechanical behaviour of the material[1-3]. Microscopic and macroscopic defects can be studied indirectly through the understanding of the electrical properties of the samples. It is common for this purpose to measure the dielectric characteristics of the studied samples.

Dielectric properties relate to the ability of a material to polarise under the influence of an electric field. The polarisability of a material depends on the structure and molecular properties and therefore dielectric measurements can provide information in this respect. The technique for measuring dielectric properties is known as Dielectric Spectroscopy, DS [4-7].

DS can be used to measure the complex relative permittivity of materials over a wide frequency range. For homogeneous and isotropic materials complex relative permittivity ε^* (hereafter,

referred to as complex permittivity for convenience) for the system electrode-solid-electrode is defined as:

$$\varepsilon^*(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega) \tag{1}$$

$$\varepsilon^*(\omega) = \frac{C^*(\omega)}{C_0} \tag{2}$$

where ε' and ε'' are the real and imaginary parts of ε^* respectively, ω is the angular frequency ($\omega = 2\pi f$), $C^*(\omega)$ is the complex capacitance and C_0 is the electrodes capacitance when the material is not present. The complex electric modulus, M^*, is given by:

$$M^*(\omega) = \frac{1}{\varepsilon^*(\omega)} \tag{3}$$

The complex conductivity σ^* is given by:

$$\sigma^*(\omega) = i\omega\varepsilon^*(\omega) \tag{4}$$

Information extracted by Eqs. (1), (2), (3) and (4) for the system electrode-solid-electrode can be classified in two categories:
Information on the solid properties (eg. Dielectric constant, dipole relaxation, charge mobility, conductivity) and
Information of the electrode-solid interface [8].
The loss tangent, tanδ, is the ratio of the energy loss over the stored energy for the system electrode-solid-electrode for each period of the applied electric field, Thus,

$$\tan\delta = -\frac{\mathrm{Im}[C^*]}{\mathrm{Re}[C^*]} = \frac{\varepsilon''}{\varepsilon'} \tag{5}$$

The objective of this work is to investigate microscopic and macroscopic discontinuities and cracks using dielectric spectroscopy and specifically by the study of dielectric loss angle ($\tan\delta$).

2. Mechanical properties of marbles and Experimental details

Marble samples collected from Mt. Penteli, Attica (Dionysos marble) is the kind of marbles that have been typically used since the ancient times for the construction of artifacts and monuments. It is mainly composed of calcite (98%) and other minerals depending on the variety of the marble, such as muscovite, sericite and chlorite. Its content in quartz is very low, about 0.2%. When it is not damaged its characteristics are: density 2.7gr/cm^3 and porosity is approximately 0.4%. This fact suggests that Penteli marble is a geomaterial of low porosity when it is not damaged. Calcite crystals are equisized and polygonic, sometimes exhibiting twinning. Their texture may be characterized as quasi-homoblastic. The rock is white with a few thin parallel ash-green colored veins containing silver-shaded areas due to the existence of chlorite and muscovite.
The experimental setup is shown in Fig. 1. According to the principles of low frequency (f<10MHz) DS that is used for this work the sample of thickness, d, is placed between two metal plates (electrodes) of cross sectional area, A, where ($A>>d^2$). The dielectric measurements were conducted using an LCR meter (Agilent model 4284A), accompanied by a dielectric test fixture

(Agilent model 16451B) and supported by a computer for data recording, storage and analysis. The dielectric test fixture that was used to hold the specimen was protected by a chamber providing constant temperature (298K), inert atmosphere by continuous effusion of inert gas and also low humidity. The instrument directly provided the values of capacitance, C , and dielectric loss, $\tan\delta$.

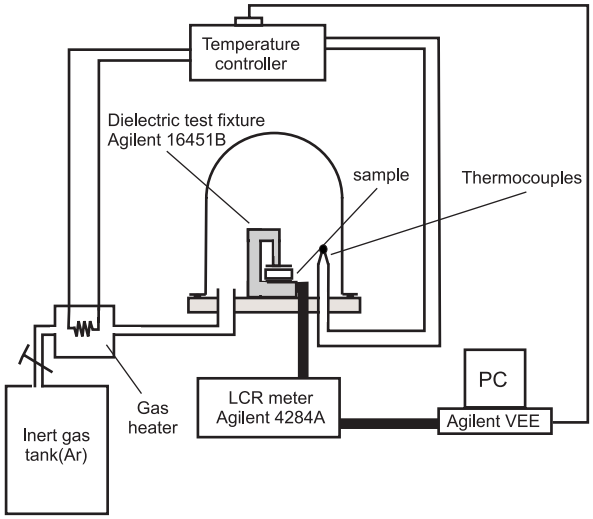

Fig. 1: Apparatus for the measurement of dielectric characteristics. It contains the LCR meter, the LCR meter the holder and the chamber for maintaining constant temperature and humidity conditions during DS measurements, the temperature controller and the recording system for digitizing and storing data.

Damages introduced to a sample can be estimated by its stress-strain curve. Fig. 2 shows the stress-strain curve of the used marble samples. In this work the stress values will always be mentioned as normalised stress which is the applied stress over the maximum stress recorded before fracture ($s=S/S_{max}$).
The stress, s , on the material is given as a function of the strain ε. For the linear range it can be stated that:

$$s = Y_o \varepsilon \tag{6}$$

where Y_o is the Young's modulus of the undamaged material which is constant in the range where stress is linearly related to strain. For the marble samples this region corresponds up to $s = 0.6$ approximately. When the stress takes values that lead further than the linear region (i.e $s > 0.6$) then microcracks occur while stress and strain are no further linearly related. In this range, where the sample is plastically deformed, the strain, *ε, is* greater than the value given by equation (6). Here the strain ε is greater than the value given by Eq (6). Accordingly, we write [3]:

$$s = \mathrm{Y}_{eff} \cdot \varepsilon \tag{7}$$

where $\mathrm{Y}_{\mathrm{eff}}$ is the effective Young's modulus and it is no longer considered as constant. In this range the Young's modulus becomes progressively smaller while stress increases. An advancement in the approach to this process is to introduce a damage variable D so that [9,10]

$$\mathrm{Y}_{\mathrm{eff}} = \mathrm{Y}_0(1 - \mathrm{D}) \tag{8}$$

The damage variable D quantifies the deviation from linear elasticity and the distribution of microcracks. In general 0<D<1. When D=0, linear elasticity is obtained with Eq. (6) valid, but when D=1, failure occurs.
Fig. 3 shows a representative schematic regarding damages nucleation and gradual development with respect to normalised stress. It becomes clear that fracture planes are guided during the last range, that is for normalised stress s>0.98.

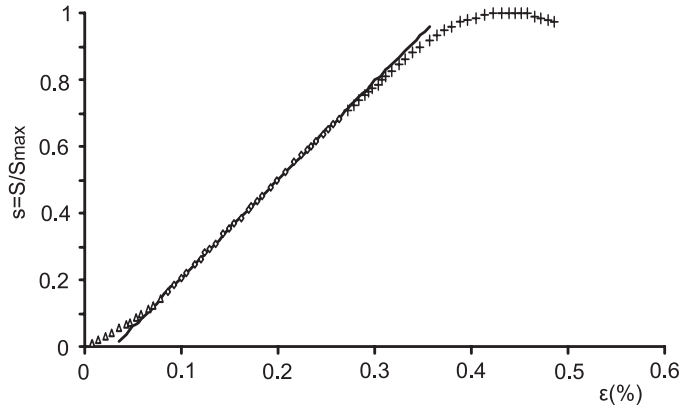

Fig. 2: Representative stress–strain curve for the marble samples used showing all three ranges of mechanical behaviour of marble.

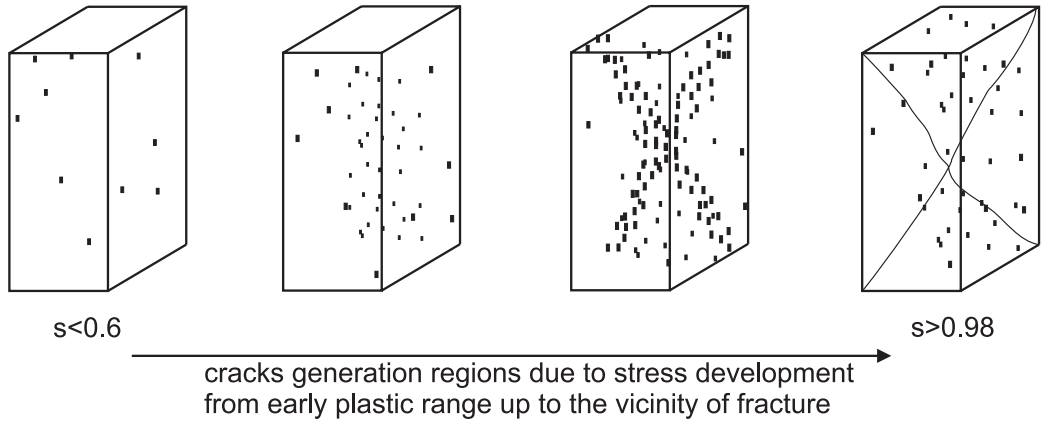

Fig. 3: Schematic diagram for the description of the defect generation and nucleation with respect to the applied stress.

The size of the used samples was 35mmx15mmx7mm approximately and the uniaxial stress was applied parallel to the long edge. The ultimate compressional stress and the elastic-to-plastic transition range were estimated by sequential stress tests. The ultimate stress magnitude was found to be 15MPa and all values were normalized to the maximum value (s=S/Smax). Elastic-to-plastic transition occurs at s=0.6 approximately.
The experimental procedure can be described as follows: Initially dielectric measurements were conducted on unstressed samples. Afterwards, the samples were subjected to mechanical axial stress, *s*, using a uniaxial hydraulic load machine (Enerpac–RC106). The stress was applied on the sample for time $t_s = 300s$ and then the sample was removed and remained unstressed for long time (>3h) during which temperature and humidity were maintained constant before repeating dielectric measurements. The value of the applied stress varied between $s = 0.1$ and $s = 0.97$. Tan was measured for all samples in the frequency range between 100Hz and 1MHz in pre-stress and post-stress conditions.

3. Experimental Results - Discussion

Fig. 4 shows the behavior of tanδ with respect to frequency in a semi-log plot. It can be seen that tanδ begins to vary only when the applied stress exceeds the elastic range. This variation is more intense in the low frequency band. A loss peak becomes clear when stress exceeds s=0.84 and damage factor is D=0.3 approximately, for the frequency of 1kHz. In this range the fracture plane is almost developed and severe damage is observed on the marbles surface. Additionally, more space is now available for the development of solid-fluid systems that affect dielectric behavior of the solid. More specifically, low frequency (i.e. 100Hz) tanδ for unstressed samples (i.e D=0) or for samples stressed in the linear range (i.e. D<0.01 approximately)concerning its mechanical behavior reaches values of the order of 0.15 approximately while for stress that reaches values in the vicinity of ultimate compressional stress it reaches values of the order of 0.4. Another interesting result is that tanδ at high frequencies does not vary, especially for frequencies, of the order of 1MHz where tanδ does not seem to be influenced by the externally applied stress and consequently the damages caused in its structure.

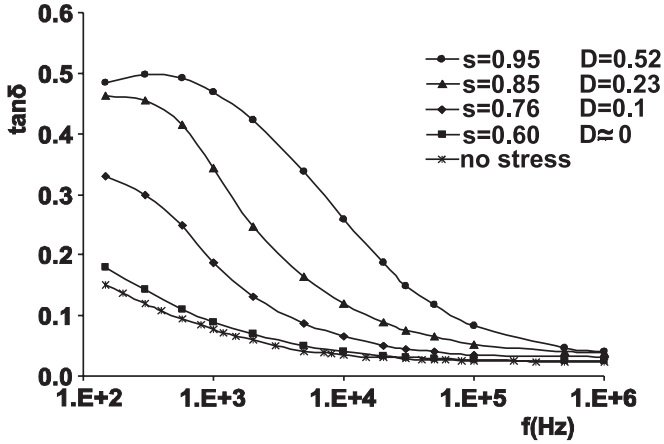

Fig. 4: The behaviour of tanδ when samples were subjected to various stress levels adequate to cause microcrack generation and corresponding damages. Curves of tanδ for stress values lower than s=0.6 and D<<0.01 are not included since they are coincident with those of unstressed samples.

Fig. 5 shows the behaviour of tanδ for specific frequencies and for various stress levels. From this figure it becomes evident that low frequencies (i.e. 150Hz or 1kHz) are much more affected by stress rather than high frequencies where no significant change of tan*δ* is observed even for high stress values.

According to Fig. 4 and Fig. 5 a dielectric loss mechanism seems to migrate from lower frequencies as the mechanical stress applied on the marble samples increases. In parallel to the increasing stress values, dielectric loss reaches higher values demonstrating the relation of the observed mechanism to the increase of space charges due to the movement of free charges on the samples bulk leading to higher conductivity.

Interfacial polarization mechanisms are developed in the samples bulk and specifically between damaged and undamaged regions.

The relatively high value of the dielectric loss implies that such mechanisms cannot be attributed to dipoles but they are rather associated to charge motion in the bulk of the material. Taking into consideration the inhomogeneous microstructure of the sample, especially when microcracks occur, the above mechanisms must be attributed to interfacial polarization Maxwell-Wagner-Sillars (MWS). Interfacial polarization occurs in samples that become inhomogeneously polarized when an external electric field is applied. The free carriers (i.e. space charge) move to arrive at the phase edges, thus, they get orientated increasing this way the polarization of the

sample. The charge carriers are trapped in regions of different polarization creating new MWS edges and forcing more and more grains to behave as large dipoles.

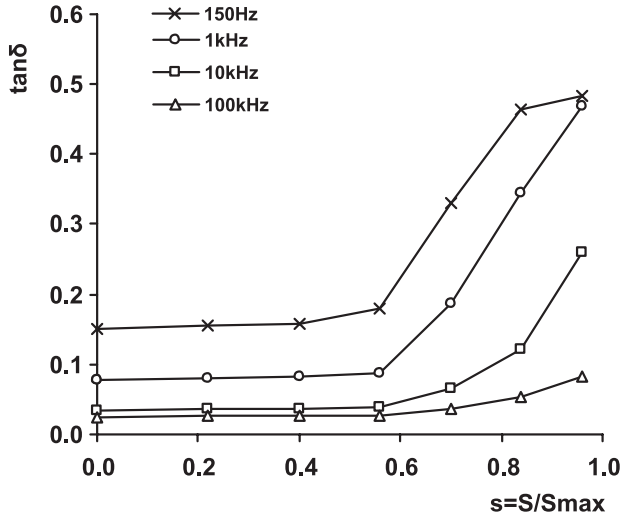

Fig. 5: Tanδ behaviour for four selected frequencies with respect to the applied stress.

4. Conclusions

In this work the influence of damages introduced to marble samples by externally applied uniaxial stress on tanδ was examined. It becomes evident that the value of tanδ is a valuable tool capable of providing information regarding damage of marble samples due to stress. For the studied frequency range, which was 100Hz – 1MHz, it also became evident that it is easier to detect damages caused by stress application in the frequency range of. 100Hz-1kHz approximately while it is more difficult to detect such damages in the frequency of 1MHz.

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