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DIELECTRIC SPECTROSCOPY AS A DIAGNOSTIC TEST METHOD FOR THE DETERMINATION OF MECHANICAL DAMAGE IN MARBLE SAMPLES

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ABSTRACT

Dielectric Spectroscopy (DS) is a well-established technique for the electrical characterization of various materials but may also serve to determine changes in their mechanical properties, since the macroscopic structural changes of a material under stress are strongly related to changes in its electrical conductivity or dielectric properties. In the present work, dry marble samples were subjected to various uniaxial loads up to failure and the complex dielectric permittivity ε was recorded during each load cycle, over a wide frequency range (10^{-2}Hz – 1MHz). The results show a strong dispersion in electrical conductivity spectra, especially at low frequencies. Conductivity values measured at low frequencies (<1Hz) are almost three orders of magnitude larger for samples prior to failure, as compared to the unstressed samples. Dc-conductivity follows an exponential behavior with respect to the relative compressional stress. A frequency dependent relaxation mechanism is observed in the electric modulus representation M* during the application of stress to the sample. The frequency of the observed relaxation peak in the imaginary part of M* and the values of M_∞ recorded in the real part of M*, are strongly affected by the applied stress and may be used to provide reliable information about the stress condition of the investigated material.*

Key words: dielectric spectroscopy, conductivity, electric modulus, rocks, mechanical properties.

1. Introduction

It has been established that the application of mechanical stress on rock or cement-based materials changes its electrical and dielectric properties, as a consequence of microscopic structural changes [1-4]. A macroscopic indication of such kind of changes is the observation of transient weak electrical currents which are produced when the samples are subjected to stress loads up to failure [5-9]. The correlation of the electrical properties of rocks to their mechanical changes has attracted the interest of many researchers not only due to the theoretical significance itself, but also due to its high practical importance. The above correlation may serve as a non-destructive testing method for the verification and quantification of mechanical damages which are introduced to solid samples, in conjunction with other diagnostic tools. In this sense, Broadband Dielectric Spectroscopy (BDS) technique can provide information about dynamical structural changes of materials which are subjected to mechanical stress by means of the changes of their dielectric properties which are recorded over a wide frequency range.

So, in the present work the complex dielectric permittivity ε^* of marble samples was recorded over a wide frequency range, during the application of uniaxial stress loads to the samples, up to failure. Different types of data representation are used for the analysis of experimental data, in order to reveal the correlation of dielectric properties with the applied load.

2. Experimental setup

Dielectric spectroscopy measurements were carried out by means of a high-resolution broadband spectrometer (Novocontrol Alpha-N Analyzer). The frequency range of the applied ac electric field was between 10^{-2} Hz and 10^{6} Hz. The specimen was mounted between two parallel stainless-steel electrodes forming a sample capacitor. The electrodes were electrically isolated from the load platens by thin teflon plates. Good electromagnetic shielding was implemented to the whole sample holder in order to diminish noise problems that are common especially at low frequencies. The stress was recorded continuously with a load cell placed at the bottom side of the capacitor. The experimental apparatus is shown schematically in Fig. 1.

Marble samples (Dionysos), collected from Penteli Mt., Attica, were cut in cylindrical shape (2-3 mm height and 37mm diameter) and dried in an oven at 105^{o}C for one day. Their chemical composition is 98% calcite and 0.2% of quartz and other minerals such as muscovite, sericite and chlorite [10]. Its density is 2.7 Mg/m^3, its porosity is 0.37% and its absorption coefficient by weight is 0.11%. Conductive silver paint was applied at both sides of the sample, in order to achieve good electrical contact with the stainless-steel electrodes.

The marble sample placed between the round plate electrodes of the capacitor can be considered as an equivalent electrical circuit comprised of a capacitance, C(ω), in parallel with a resistance, R(ω). These values which are the output of the analyzer are associated to the complex impedance $Z^*(\omega)$ and dielectric permittivity $\varepsilon^*(\omega)$ through the following relations:

$$\frac{1}{Z^*(\omega)} = \frac{1}{R(\omega)} + i\omega \cdot C(\omega) \tag{1}$$

and

$$\varepsilon^*(\omega) = \varepsilon' - i\varepsilon'' = \frac{C(\omega)}{C_o} - i\frac{1}{\omega \cdot C_0 \cdot R(\omega)} = -\frac{i}{\omega \cdot C_0 \cdot Z^*(\omega)} \tag{2}$$

where $C_0 = \varepsilon_o \cdot \pi \cdot r^2 / d$ is the capacitance of the empty sample cylindrical capacitor, with distance d between the electrodes and r their radius, ω=2πf and ε_o is the permittivity of the vacuum. The specific conductivity σ^* of the sample is related to the dielectric permittivity by the equation:

$$\sigma^* = \sigma' - i\sigma'' = i\omega\varepsilon_o\left(\varepsilon^* - 1\right) = \omega\varepsilon_o\varepsilon'' - i\omega\varepsilon_o\left(\varepsilon' - 1\right) \quad (3)$$

Alternatively, the reciprocal permittivity or electric modulus M^* representation can be used

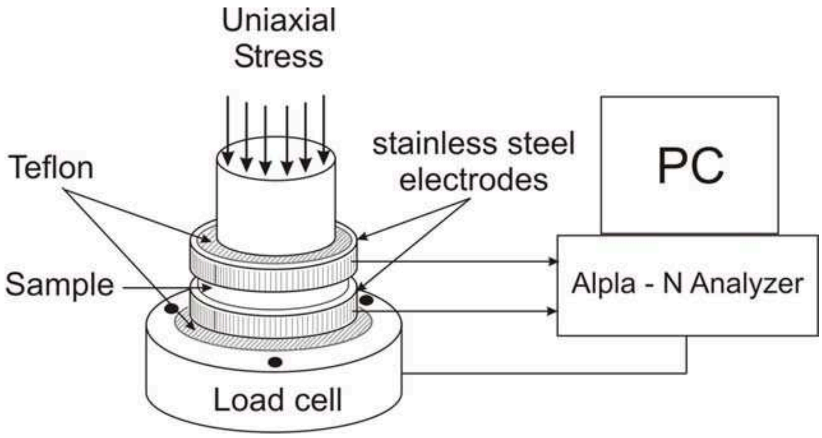

Fig. 1: Schematic representation of the experimental apparatus.

to describe relaxation processes. Electric modulus M^*, which is an electrical analogue to the mechanical shear modulus, is defined as:

$$M^*(\omega) = \frac{1}{\varepsilon^*(\omega)} = M'(\omega) + iM''(\omega) = \frac{\varepsilon'}{\varepsilon'^2 + \varepsilon''^2} + i\frac{\varepsilon''}{\varepsilon'^2 + \varepsilon''^2} \quad (4)$$

The main advantage of the above representation is that the contribution of electrode polarization effects is negligible, allowing the evaluation of relaxation processes at the low frequency range of dielectric spectra [11].
In the present work, conductivity and modulus representations were used to analyze the experimental data and correlate them with the applied stress.

3. Results and discussion

The variation of ac – conductivity as a function of frequency, measured at various stress loads up to failure which detected at 200kN, is shown in Fig. 2. Large dispersion (frequency dependent variation of conductivity) is observed over the whole frequency range, especially at the first stages of load. The conductivity spectra may be roughly divided into three regions. At the high frequency range, a linear increase of conductivity with increasing frequency is observed, namely, polarization conductivity due to the localized hopping motion of charge carriers. At the medium frequency range, a frequency independent region (dc-plateau) is developed at pressures higher than 30kN. Furthermore, at this frequency range, conductivity values prior to failure (200kN) are almost three orders of magnitude higher than that measured at the first stage of load.

At the low frequency region a slight deviation from the dc-plateau should be attributed to electrode polarization phenomena which are common at these frequencies.

The pressure dependence of dc-conductivity recorded at 0.035Hz is shown in Fig. 3, in a linear scale. According to the observed variation, dc-conductivity changes more drastically when the load exceeds about 70% of its maximum value, which corresponds approximately to the elastic limit of the marble sample [5, 7]. The data of fig. 3 are fitted very well with an exponential curve, according to the following relation:

$$\sigma_{dc}(P) = \sigma_0 e^{-P/B} \tag{5}$$

where σ_0, B are fitting parameters, with $\sigma_0 = \sigma_{dc}(P=0)$ and B a constant which should be a function of temperature and volume of mobile species (activation volume) [12].
The large variation of conductivity values recorded at frequencies lower than 10Hz, suggest that low-frequency electrical conductivity is very sensitive even to small changes of

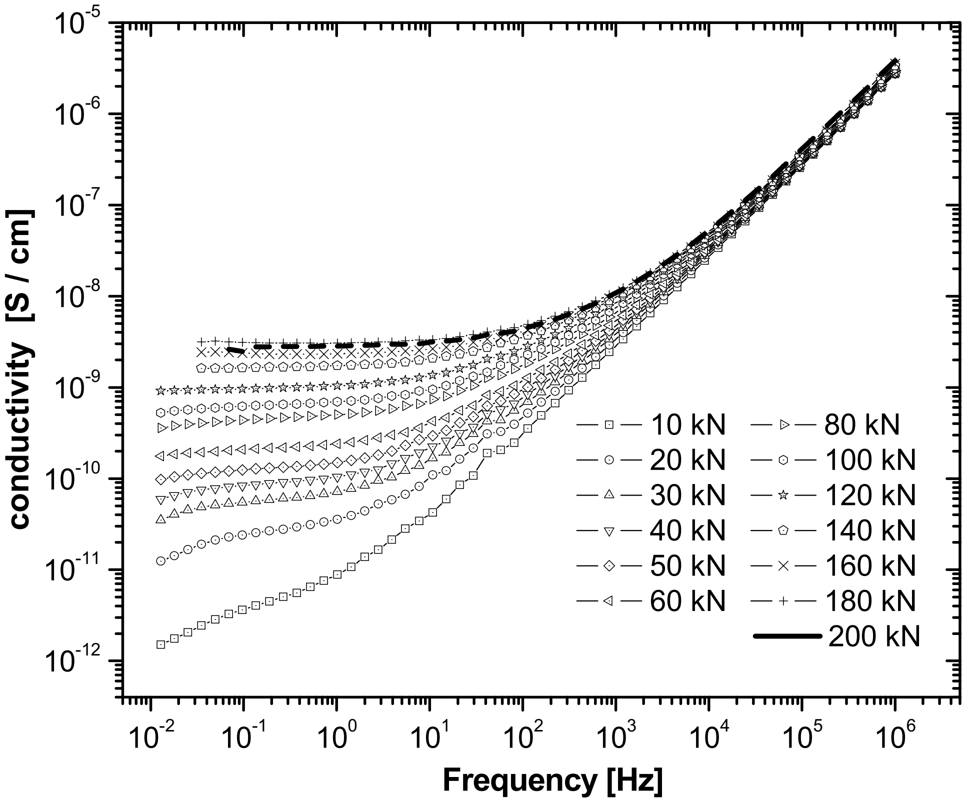

Fig. 2: Ac – conductivity spectra at various stress loads. The dashed line represents the recorded spectrum after failure of the sample.

stress load and may be used to detect damage within the bulk of geomaterials due to externally applied mechanical loading.

Conductivity data were transformed through equations 3, 4 to the electric modulus representation M*, since at this representation ionic conductivity appears as a single relaxation peak, allowing a different approach to data analysis [11].

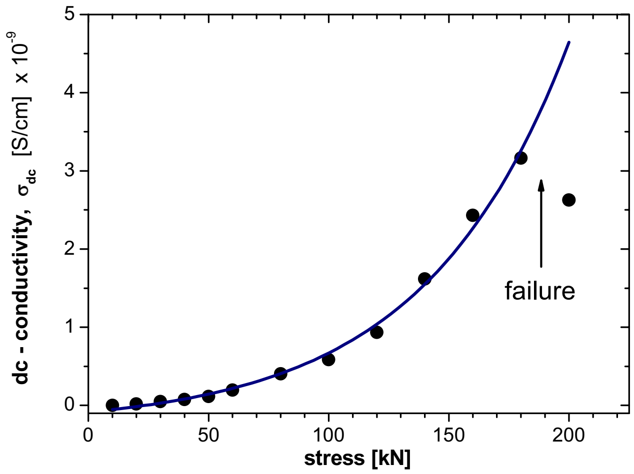

Fig. 3: Dc – conductivity values as a function of the stress load. Solid line represents the fitting curve of an exponential function. The dc-conductivity value at 200kN has been excluded from fitting, since it has been measured after failure of the sample.

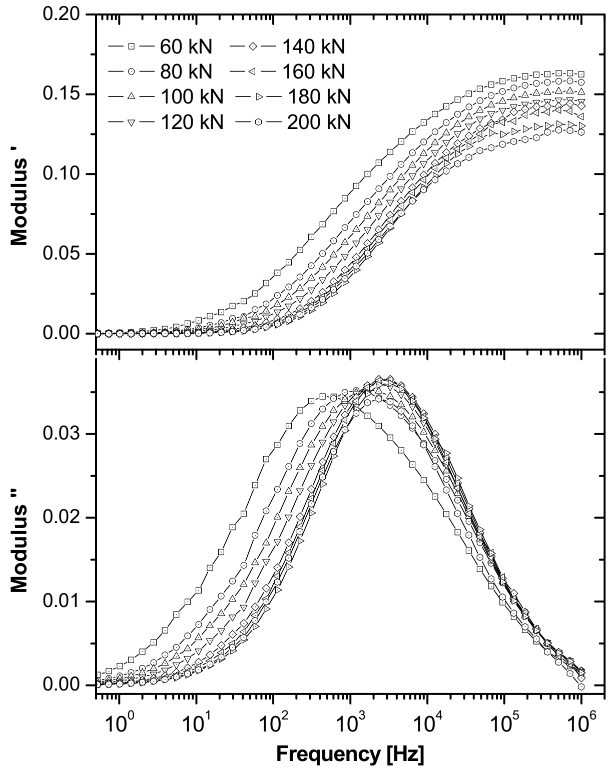

Fig. 4: Real (M´) and imaginary (M´´) part of electric modulus M*, as a function of frequency, at various loads of the marble sample.

The real (M´) and imaginary (M´´) part of electric modulus M*, as a function of frequency are shown in Fig. 4. A steady decrease of M´ is observed over the whole frequency range, leading to zero values at low frequencies. Furthermore, the values of M´ measured at 10^6Hz (M_∞) decrease with increasing stress load. A broad relaxation peak is developed at the imaginary part of M*, which

is shifted to higher frequencies with increasing stress load, while its spectral shape becomes narrower. The latter suggests a different distribution of relaxation times in the sample, as the pressure increases.
The frequency values which correspond to the peak maxima and the values of M_∞ as a function of stress load, are presented in Fig. 5. A drastic increase of relaxation frequency appears at the first stage of load, covering about 50% of the elastic region. When the sample exits its elastic region around 140kN [5], the position of peak seems to stabilize. However, M_∞ variation is continuous with pressure up to sample failure. The variation of M_∞ with stress may be divided into two linear regions with a change in slope at the point where relaxation frequency starts to vary slowly.
The above findings, in combination with the variation of electric conductivity, show a strong correlation of electric – dielectric quantities with the applied load in rock samples. The underlying physical mechanisms of charge transfer or charge polarization due to mechanical stress have to be investigated further, in detail.

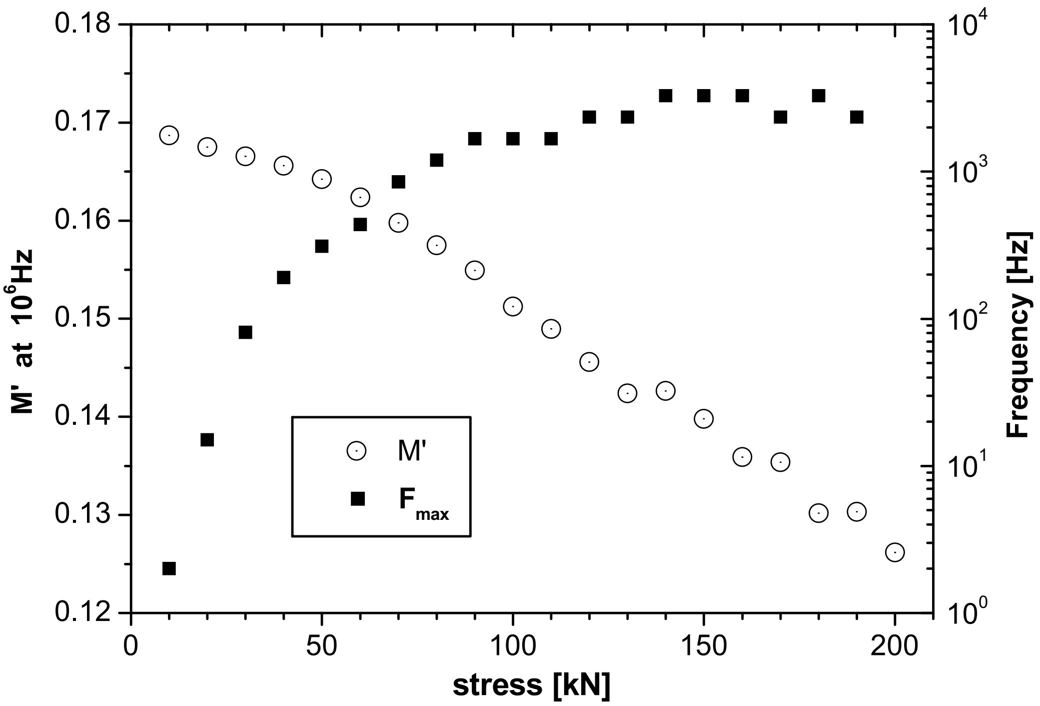

Fig. 5: Relaxation frequency in M′′ spectra and M_∞ variation as a function of stress load. Note that the scale of frequency is logarithmic.

4. Conclusions

In the present work, broadband dielectric spectroscopy measurements were carried out in marble samples, while they were subjected in mechanical stress up to failure. Ac- conductivity and electric modulus formalisms were use for data representation and analysis. Ac-conductivity shows a strong dispersion all over the measured frequency range, while σ_{dc} exhibits an exponential variation with the applied stress. The electric modulus representation M′′ of experimental data, exhibits a relaxation peak in the Hz region which is shifted to higher frequencies, as the stress increases. Prior to failure this relaxation peak is stabilized at 2.3 kHz. Conclusively, BDS is proved to be a powerful Non-Destructive Testing tool that can be used to detect damage within the bulk of geomaterials due to externally applied mechanical loading.

5. References

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