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Investigation of Microcracks in Marble from Mt. Penteli by Dielectric Spectroscopy

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Abstract: The purpose of this work is to investigate microscopic and macroscopic discontinuities and cracks with the use of dielectric spectroscopy[1]. The technique has been applied to marble samples from Mt. Penteli in Attica, Greece. This material was chosen because it has been a basic constructing material of classical ancient monuments. Measurements of the real and imaginary parts, ε' and ε'', of the complex dielectric permitivity for various frequencies in the range of 150Hz up to 1MHz are presented. Initially, measurements were taken before any application of stress to the samples. Then, the same samples were subjected to mechanical stress by the application of uniaxial pressure S of increasing values from $0.30S_{max}$ to $0.84S_{max}$ where S_{max} denotes the maximum limit of the recorded stress before sample failure. The experimental results clearly demonstrate the change of the dielectric constants, when the mechanical stress on the samples exceeds the linear elasticity region.

Περίληψη: Στόχο της παρούσας εργασίας αποτέλεσε ο εντοπισμός ασυνεχειών και ρωγμών σε μάρμαρα με εφαρμογή της διηλεκτρικής φασματοσκοπίας. Η τεχνική εφαρμόσθηκε σε πεντελικό μάρμαρο λόγω της ευρείας χρήσης του κατά την αρχαιότητα. Τα αποτελέσματα της μελέτης καταφανώς παρουσιάζουν αλλαγές των διηλεκτρικών σταθερών τους όταν η εφαρμοζόμενη μηχανική καταπόνηση στα δείγματα υπερβαίνει την γραμμική ελαστική περιοχή.

Introduction

Marble is a rock, which from a geological point of view belongs to the metamorphic rocks. 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 or macroscopic discontinuities resulting in changes in the mechanical behaviour of the material [2-5]. In this work the marble samples were provided in the form of specimens of thickness t=4mm and cross-section $A=400mm^2$. Initially measurements were conducted without subjection to any stress. The measurements were recorded with an LCR meter (Agilent 4284A), using the dielectric test fixture (Agilent 16451B), which had been further automated using a computer for data recording, storage and analysis. The dielectric test fixture, in which the sample was placed, had already been positioned into a special chamber to ensure constant temperature (298K), inert atmosphere due to continuous inert gas (Ar) flow and constant humidity [6]. The instrument directly provides the values of capacitance C and dielectric loss tanδ. The values of capacitance C and dielectric loss tanδ can be read directly from the instrument. Knowing the values of capacitance C, the real part of the complex dielectric constant (ε′) is calculated by the relation:

$$\varepsilon' = \frac{4tC}{\varepsilon_0 \pi d^2} \qquad (1)$$

where ε_0 is the permittivity of free space. Using the values of ε′ and tanδ the values of the imaginary part of complex relative permittivity (ε′′) were calculated using equation (2).

$$\tan\delta = \varepsilon''/\varepsilon' \qquad (2)$$

Afterwards, the samples were subjected to mechanical uniaxial stress S, using a uniaxial hydraulic load machine (Enerpac–RC106). The stress S had been applied on the sample for t_S=100s and then the sample was allowed to relax for t_r=6ks before the measurements took place. The value of the applied stress S for the strain of the samples ranged from $0.3S_{max}$ to $0.84S_{max}$ where S_{max} stands for the upper limit of the recorded stress before fracture. The stress S in the material is given as a function of strain ∈. For the linear elasticity region we have

$$S = Y_0 \cdot \in \qquad (3)$$

where Y_0 is the Young modulus of the undamaged material, a constant. For the Penteli rock samples this region corresponds up to $S=0.6S_{max}$ approximately. The region for $S>0.6S_{max}$ is a deviation from linear elasticity, thus, microcracking is occurring.

Figure 1 depicts the stress–strain diagram of marble samples for normalized stresses, based on experimental data[7]. The region for $S>0.6S_{max}$ deviates from linear elasticity, thus, microcracking occurs.

Experimental results and discussion

Figure 2 shows in semi-logarithmic plot the real part of dielectric permittivity, ε′(f) of marble samples for four successive values of uniaxial compressional stress S in the range from 0 to $0.84S_{max}$ to which the samples were subjected, under the application of ac field in the frequency range 150Hz–1MHz.

The effect of the applied stress S on the value of ε′ at several frequencies is given in Figure 3. It is obvious that the application of stress on marble samples causes a decrease on the measured values of ε′ in the whole range of frequencies from 150Hz to 1MHz. However, for applied stress $0<S<0.6S_{max}$, no major changes are observed in the values of ε′. A slight distinct change is detected in low frequency ranges, of less than 1kHz. In the range $0.6S_{max}<S<0.84S_{max}$, where the strain leads to the initiation of microcracking, the values of ε′ decrease slightly. Especially when the strain of the samples due to the applied uniaxial stress S reaches the value of $0.84S_{max}$, thus causing significant macrocracks to the sample, the decrease in the values of ε′ at all frequencies from 150Hz to 1MHz ranges between 25 and 30%. For minor strains corresponding to $0.7S_{max}$ the relevant decrease of the value of ε′ ranges from 10 to 15%.

In Figure 4, the dependence of the imaginary part of the complex dielectric constant ε″(f) on frequency f is shown. The marble samples were subjected to uniaxial compressional stress S in the range from 0 to $0.84S_{max}$. It is clear that for an applied stress less than $0.6S_{max}$, no remarkable changes are observed in the measured values of ε″.

The previous mechanical strain of the samples does not seem to have affected the values of ε″, as long as the applied stress had not brought the material into the plastic region ($S<0.6S_{max}$). The changes observed when the applied stress was between $0.18S_{max}$ and $0.45S_{max}$ were approximately 5% and cannot be considered, given that they are in the experimental error margin. When the marble samples were subjected to a stress S equal to $0.56S_{max}$ which corresponds to the limit between elastic and plastic region, then a considerable variation in the values of ε″ is observed, which for the frequency range between 150Hz and 10KHz is from 10% to 15%, whereas for the higher frequency range it does not exceed 10%.

Figure 5 depicts the rapid increase in the values of ε″ caused by the previously applied stress S on the samples when the former was larger than $0.6S_{max}$. The horizontal axis is normalized and expresses the ratio $k=S/S_{max}$. The experimental results show that ε″ follows an exponential law of the following form:

$$\varepsilon''(k)=A\exp(b\cdot k) \quad (4)$$

where A is a pre-exponential factor and b is an exponential factor strongly dependent on frequency. At the low frequency region (f<1KHz), the fitting of the experimental values of ε″ with Eq.4 evaluates the exponent b between 6 and 3 reducing as frequency increases. At higher frequencies (f>1KHz) the experimental values of ε″ follow the practical law of Eq. 4, but the values of the exponential factor b are less than 3 and at 1MHz, b is approximately 0.5.

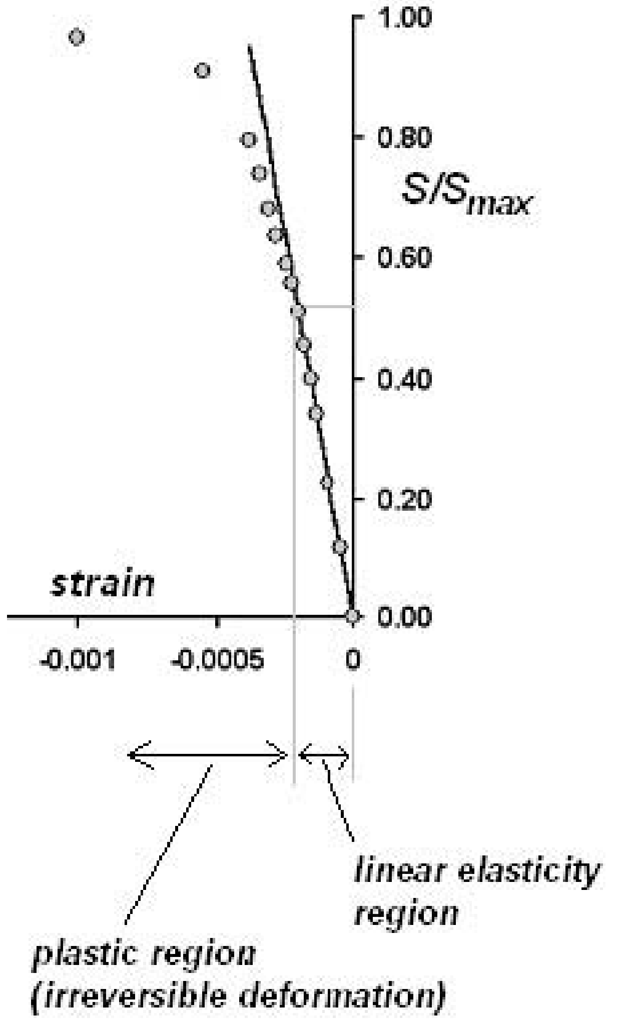

Figure 1: Normalized experimental stress–strain diagram from a Penteli marble sample.

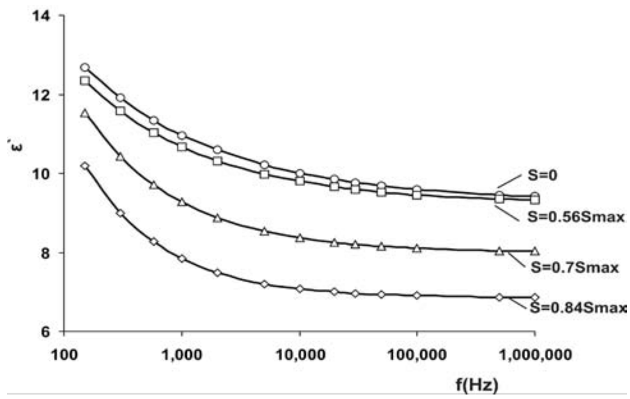

Figure 2: The real part ε′ of the complex dielectric constant versus frequency f for the marble sample, 6ks after the application of various uniaxial pressure S values between 0 and 0.84Smax

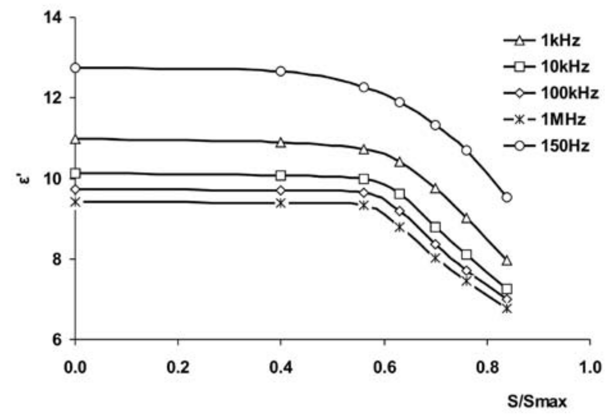

Figure 3: The real part ε' of the complex dielectric constant as a function of the normalized stress S/Smax for various frequencies.

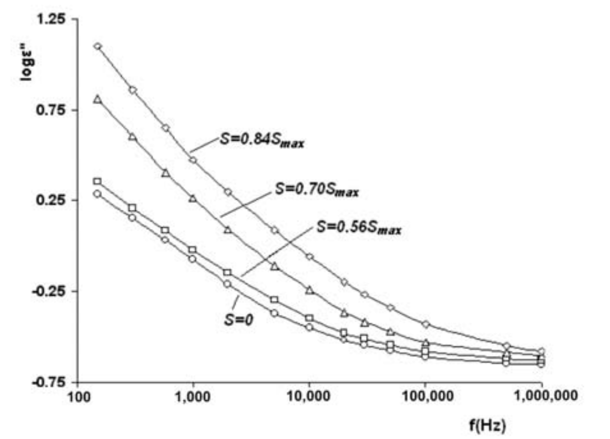

Figure 4: The imaginary part ε" of the complex dielectric constant versus frequency f (log-log plot) for the marble sample, 6 ks after the application of various uniaxial pressure S values between 0 and 0.84Smax

Conclusions

When the samples were subjected to stress S below the plastic region, (as far as the mechanical behaviour of the materials is concerned) no important variations were observed in the values of both real and imaginary part of the complex permittivity .

When the marble samples had been subjected to stresses adequate to lead them to the plastic region, the values of ε′ were clearly smaller than those measured when the samples had been subjected to smaller stresses and had not entered the plastic region. This shows that the appearance of crack-microstructures prevented the reversible displacement of electric charges in the geomaterial.

On the contrary, the values of the imaginary part of the complex permittivity increased rapidly according to an exponential law along with stress S. This happened

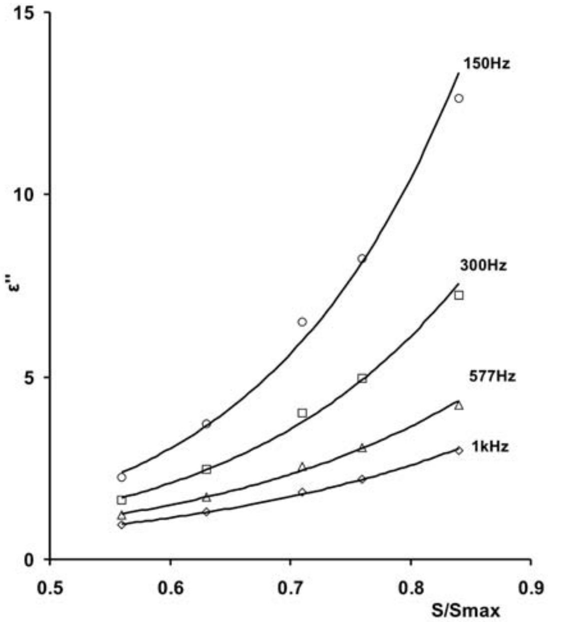

Figure 5: The imaginary part of permittivity ε" plotted against normalized stress values is shown to increase rapidly at low frequencies.

when the samples had been previously subjected to stress S exceeding the plastic region limit ($S>0.6S_{max}$ approximately). In this case, the appearance of crack-microstructures as well as the further appearance of higher values of stress S caused significant increase to the ohmic losses due to the free charge movement in the material.

From the above mentioned results it becomes evident that the non-destructive testing method of dielectric spectroscopy can be easily applied in the investigation of micro-cracks and structural imperfections in marble that have been caused by aging, earthquakes or various other factors to be determined.

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