Ac conductivity of marble rocks after the application of various stress modes. Correlation with mechanical properties.

C. ANASTASIADIS[1], D. TRIANTIS[1], I. STAVRAKAS[1], F. VALLIANATOS[2]
[1] Department of Electronics
Technological Educational Institution of Athens
Athens, 122 10, Tel/Fax: 0030-210-5316525
GREECE

[2] Department of Natural Resources Engineering,
Technological Educational Institute of Crete,
Chania, Crete, 731 33,
GREECE

Abstract: - Ac conductivity measurements of marble samples have been carried out in the frequency range of 50kHz to 1MHz. Variations of frequency dependent conductivity were studied when the samples had previously suffered uniaxial compressional stress of various magnitudes up to fracture. The conductivity plots that were fitted by using the law $\sigma = A \cdot f^n$ indicated significant dependence of the exponent n on the previously suffered uniaxial compressional stress when the applied stress reached values where the material in terms of mechanical behaviour, shows a deviation from linear elasticity and microcracking occurs. A linear relation between variable ν, which describes the variation of exponent n, and the damage variable D of the samples is evident.

Key-Words: - ac conductivity, marble rocks, damage, rock conductivity, microcracks.

1 Introduction

Electrical properties of rocks are of great interest for the exploration of deep Earth. Ac conductivity is extremely sensitive to physical conditions like temperature and pressure, the chemical composition of the solid and liquid phases, and microstructural factors like porosity[1]. The coupling between conductivity and the mechanical behaviour of upper crust rocks is of great potential importance in very different geological contexts. Mechanical stresses upon rocks create microscopic or macroscopic discontinuities resulting in changes of both mechanical and electrical behaviour of the material[2-4]. Crack propagation and crack nucleation are two major processes that control conductivity.

Dielectric impedance measurements constitute a strong tool to study the evolution of fracture generation[5,6]. Microstructure changes induced by compressive stress cause a loss of connectivity, thus, influence electrical conductivity. Variations of rock conductivity have been studied and correlated to relevant petrophysical properties[7].

Marble is a rock, which from a geological point of view belongs to 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.

The stress S on the material is given as a function of strain ε. For the linear elasticity range it can be stated that:

$$S = Y_o \cdot \varepsilon \tag{1}$$

where Y_o is the Young's modulus of the undamaged material which is constant in the elastic range. When stress takes values that lead further than the elastic range, then microcracks appear. In this range, where the sample is plastically deformed, strain ε is greater than the value given by Eq. (1). Accordingly[4]:

$$S = Y_{eff} \cdot \varepsilon \tag{2}$$

where Y_{eff} is the effective Young's modulus and it is no longer considered to be constant. In the plastic range Young's modulus becomes progressively smaller while stress increases. In this case the damage variable, D, is used [8,9] so that

$$Y_{eff} = Y_o \cdot (1 - D) \quad (3)$$

The damage variable D quantifies the deviation from linearity and the distribution of microcracks in a one-dimensional problem. In general 0<D<1. When D=0 linear elasticity is obtained with Eq. (1) valid, but when D=1 failure occurs. Since damage variable is a function only of the applied stress D(S), for the range that stress-strain relation deviates linearity Eq. (2) may be transformed as:

$$S = Y_o \cdot (1 - D) \cdot \varepsilon \quad (4)$$

Jonscher suggested that the widely observed power law frequency dependence of ac conductivity is a “universal” property of materials. In general, ac conductivity $\sigma(f)$ is found to vary with frequency f as

$$\sigma(f) = \sigma(0) + A \cdot f^n \quad (5)$$

at frequencies well below the lattice vibrational frequency [10]. In Eq. (5), $\sigma(0)$ is the dc limit of conductivity, A is a parameter depending on temperature and pressure and the exponent n takes values between 0 and 1. In the high frequency range ac conductivity follows the power law

$$\sigma(f) = A \cdot f^n \quad (6)$$

once the component $A \cdot f^n >> \sigma(0)$.

In the present work a correlation of the recorded changes of exponent n and the deviation of the Young’s modulus Y_o described by damage variable D, will be demonstrated, by elaborating ac conductivity measurements on various frequencies. The used samples suffered stress capable to cause limited damages in the level of irreversible deformation. In a previous paper a decrease of the exponent n values had been observed for increasing mechanical stress magnitude on marble samples [11].

2 Sample characteristics and experimental technique

Marble is a metamorphic rock composed wholly or in large part of calcite or dolomite crystals. The crystalline texture is the result of metamorphism of limestone by heat and pressure. The experiments have been conducted using samples of Pentelicon marble (Dionisos) collected from Penteli Mt., Attica. The basic characteristics of the marble samples used in the experiments are summarized below: they are composed of calcite (98%), and other minerals such as muscovite, sericite and chlorite. The geo-material used can be characterised as quartz-free since its content in quartz is less than 0.2%. Its density is 2.7 Mgr/m^3, its porosity is 0.371% and its absorption coefficient by weight is 0.11% [12].

The used samples were cylindrical with a diameter of 25mm and a height of 8mm. The average fracture limit of the samples was measured to be 15MPa. Special care was taken to maintain constant hydration levels of the order of 0.09% for all used samples. This was achieved by keeping the samples in a hermetically closed chamber during the experiment.

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=6ks before performing the ac conductivity measurements. It should be noted that after removal of the uniaxial compressional stress S, a relaxation of the ac conductivity to a final limiting value is observed[13]. Thus, a certain time elapse -which empirically is not longer than 6ks- after stress removal is required to ensure that the measurements are not influenced by relaxation phenomena. The stressing system comprised a uniaxial hydraulic load machine (Enerpac–RC106) that applied compressional stress to the sample.

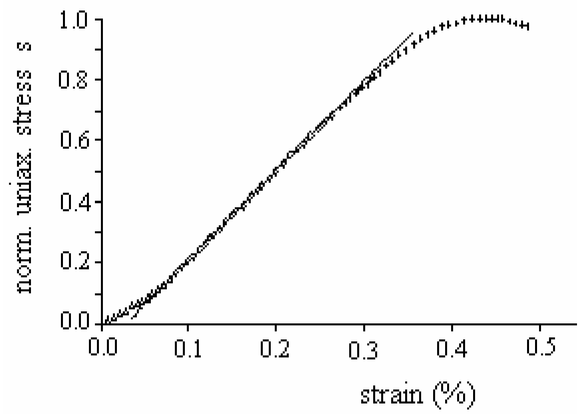

Fig. 1: Experimental stress-strain curve for marble samples

The stress-strain curve of the marble samples is a matter of extensive investigation [14]. Fig. 1 depicts a typical stress-strain curve of the marble samples. The stress axis is normalized to the ultimate compressional stress (i.e. Smax) and it is described by the ratio S/Smax (i.e. normalized uniaxial stress). The linear part of the stress - strain characteristic curve extends for 0.15 < s < 0.70. For s>0.70 the samples exhibit plastic behaviour.

The samples used were classified with respect to the previously applied mechanical stress using the respective code-numbering (see Table 1): Codes with NS prefix refer to samples never subjected to mechanical stress. Codes with SL prefix refer to samples subjected to mechanical stress in the linear (elastic) range (0.15<s<0.70). Finally, codes with SP prefix refer to samples subjected to mechanical stress in the plastic range up to fracture neighbourhood (0.70<s<0.95).

Table 1. Calculated values of exponent n, and variables D and ν for various samples with respect to the previously applied mechanical stress.

Sample code	normalized uniaxial stress s	exponent n	Damage variable D	variable ν
NS01	0.00	0.92	0.00	-
SL02-04	0.2 to 0.6	0.91-0.93	0.00	-
SL05	0.70	0.90	0,01±0,01	0,02±0,01
SP06	0.76	0.80	0,08±0,01	0,13±0,01
SP07	0.80	0.71	0,15±0,01	0,23±0,02
SP08	0.85	0.60	0,23±0,02	0,35±0,02
SP09	0.90	0.46	0,32±0,02	0,50±0,02
SP10	0.92	0.41	0,38±0,02	0,55±0,02

The ac conductivity 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 specimen 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 specific rock is characterised by its low porosity the existing ac conductivity mechanisms depend determinately on the water content of the specimen [15].

3 Experimental results and discussion

Fig. 2 shows the variation of ac conductivity plotted against frequency for different sample groups classified with respect to the previously applied mechanical stress, which is determined by the normalized uniaxial stress. The relationship between σ_{ac} and f is usually linear in log-log presentation and the slopes of the lines varying with n are related to a conduction mechanism. It becomes clear from Fig. 2 that the results do follow the sample power law relation given by:

$$\sigma_{ac}(f,s) = A(s) \cdot f^{n(s)} \qquad (5)$$

where the parameter A and the exponent n depend on the normalized uniaxial stress, s. The fitting of the experimental data $\sigma_{ac} - f$ whose values are recorded in Table 1 with respect to the normalized uniaxial stress, s, permit the calculation of the exponent n. All calculated exponent values vary between 0 and 1 which indicates the existence of translational hopping motion.
The measurement error of the normalized uniaxial stress, s, for the various samples used is estimated of the order of 2%-4%. The corresponding error of the n factor estimation is not greater than 3%.

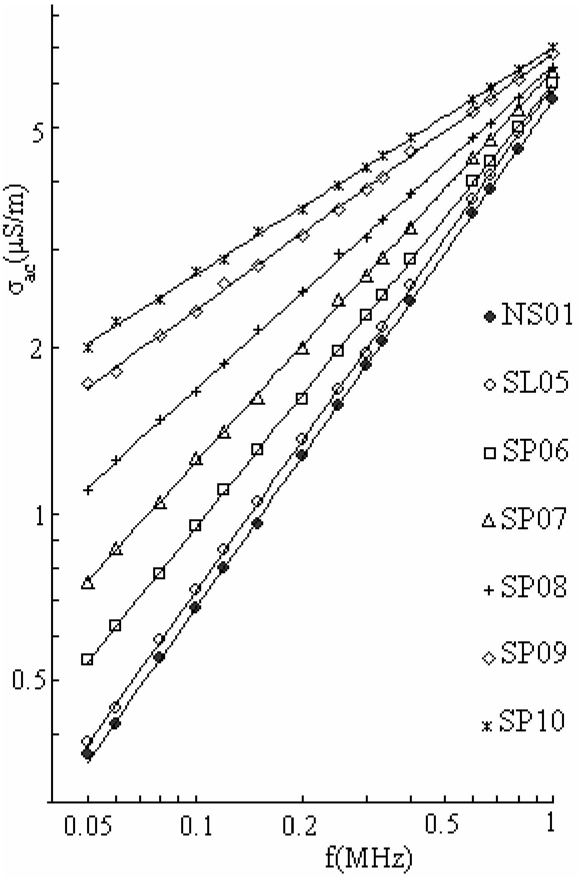

Fig. 2. Frequency dependence of the ac conductivity at different samples of which the previously applied mechanical stress is described by the uniaxial stress, s, (see Table 1). Lines represent the fitted results to the data points according to Eq. 5.

The values of the ac conductivity at different frequencies and the values of the exponent n for the SL sample group that were stressed in the linear range are almost identical with those of the unstressed NS group samples. This is the reason for not demonstrating the experimental data of σ_{ac} in Fig. 2.
The first significant change in the values of ac conductivity (see Fig.2) is detected for the sample

SL-04 (see Table 1), subjected to s=0.7. In this sample the previously applied mechanical stress has driven it to the limit between elastic and plastic ranges (see Fig.1). The exponent value was calculated to be equal to 0.90 which is slightly smaller than the respective values for the NS samples.

As far as the SP samples that were stressed in the plastic range are concerned, it is observed that they exhibit greater ac conductivity values. Samples characterized by an increased s ratio exhibit even greater conductivity values and the resulting exponent values are continuously decreasing as the normalized uniaxial stress increases (see Table 1).

Samples that have previously suffered a mechanical stress very close to failure, give exponent values decreased to half of those corresponding to NS or SL samples.

Fig.3 demonstrates the dependence of the exponent n on the normalized uniaxial stress s. The decrease of the values of the exponent n, for s>0.7 is rapid. This behaviour is straight analogous [16] to the variation of the Young's modulus Y_{eff} as this was defined by Eq. (2).

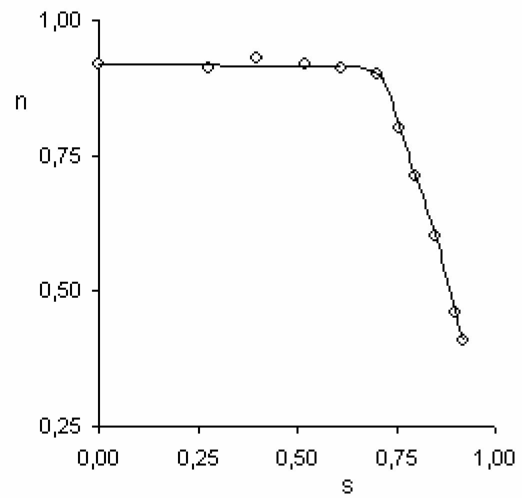

Fig. 3. Variation of the exponent n with respect to the normalized uniaxial stress, s.

In an effort to correlate the decrease behaviours of both the exponent and the Young's modulus, a relation analogous to Eq. (3) is used for the exponent n as follows:

$$n = n_o \cdot (1-\nu) \qquad (6)$$

where the exponent n_o refers to values of n for unstressed samples ($n_o \approx 0.92$). Variable ν is characteristic of the variation span of the exponent n_o due to previously applied mechanical stress. It is obvious that $\nu = \nu(s)$

Based on the experimental data of the stress - strain curve (Fig.1), the damage variable was determined as it was described by Eq. (4) with respect to the values of s. The values of variable ν, were calculated respectively after Eq. 6. Table 1 contains the values of the variables D and ν, for various states of mechanical behaviour of the samples with respect to the previously applied mechanical stress, including the corresponding calculation error.

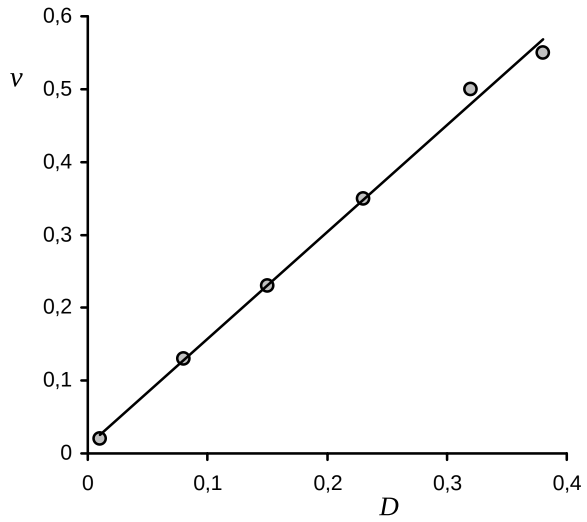

Fig. 4. Graphical representation of variables D and ν. A linear relation is evident with slope to be 1.5 approximately.

Fig. 4 shows the dependence of variable ν, to the damage variable D. It is evident that there exists a linear relation between the two variables which is verified by the experimental result of the present work.

4 Concluding remarks

Since cracks are the origin of structural imperfections which result in numerous local polarizations and in the generation of instantaneous electric fields it may certainly be stated that the main conductive paths follow cracks. Since conductivity differs from grain to grain of the material one may conclude that its mechanical properties may also follow the scaling of the structural inhomogeneities which differ slightly from grain to grain and thus n can in principle characterize the degree of structural heterogeneity of the material. The values of ac conductivity at high frequencies corresponding to fractional power laws with exponents 0.7<n<0.9, are characteristic of slowly moving hopping charge carriers[17]. This corresponds to cases that the previously applied mechanical stress upon the samples has not caused serious structural imperfections in the material. In the cases of mechanical stress upon samples in which the normalized uniaxial stress becomes larger than 0.8 the increased ac conductivity values observed, manifest a greater freedom of charge motion at longer distances between the electrodes which may be favoured by the extended creation of crack micro-structures. The decreasing exponent values that become smaller than 0.5 when the samples are in states fairly close to fracture manifest even faster hopping charge carriers. Future work focusing on the values of the dielectric constant, conductivity and relevant parameters associated with dielectric dispersion will certainly enlighten the factors governing the microscopic or macroscopic discontinuities related to the variations of the mechanical behaviour of the geomaterial.

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