The electrical signature of sedimentary and crystalline rocks in the pre-failure regime in hydrostatic and triaxial pressure tests

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Abstract
The electrical signature (AC-impedance spectroscopy) of sedimentary and crystalline rock samples was studied in hydrostatic (up to 300 MPa) and triaxial pressure experiments (up to 150 MPa axial stress). Pressure-generated variations in pore geometry caused a decrease in conductivity (hydrostatic pressures), while triaxial pressures increased the bulk-conductivity. Model data of the frequency dispersion were fitted to the measured data and thus allowed to derive model parameters (capacitor C) that describe changes in pore geometry. They are based on electrochemical interactions of the fluid pore electrolyte with the inner surface of the sample. When hydrostatic pressures were applied, cracks were closed and thus a decrease of the refined model capacitor C was detected. On the contrary an increase of C of more than two orders in magnitude was found in triaxial experiments where new fractures were formed.

1. Introduction

The Archie equation relates the electrical conductivity of fluid saturated crustal rocks with petrophysical properties (porosity, permeability, degree of pore filling, conductivity of the pore fluid), but it cannot be used to derive a simple relation between electrical rock properties and their variation due to external pressures acting on the sample. Such information can be obtained from frequency dependent electrical conductivity measurements (Glover&Vine, 1992; Duba et al., 1994; Nover et al., 1998). This method detects electrochemical interactions between ions and dipols in the pore electrolyte and the charged mineral surfaces. The electrochemical reactions cause the formation of "clouds" of charged particles (adsorbed on the inner surface of the pore system). These oscillate in the stimulating electrical field applied, depending on their relaxation time. If the frequency is varied, e.g. from Hz up to MHz the relaxation times of these polarisation processes can be measured. Hydrostatic and triaxial pressures induce variations in pore geometry and thereby change both, the overall electrical (bulk)-conductivity by closing and opening of conduction paths, and the frequency dispersion by variations in pore size that cause e.g. the overlapping of such "clouds" on both sides of a fracture thus changing the time constant of the relaxation process (Nover et al., 1999).

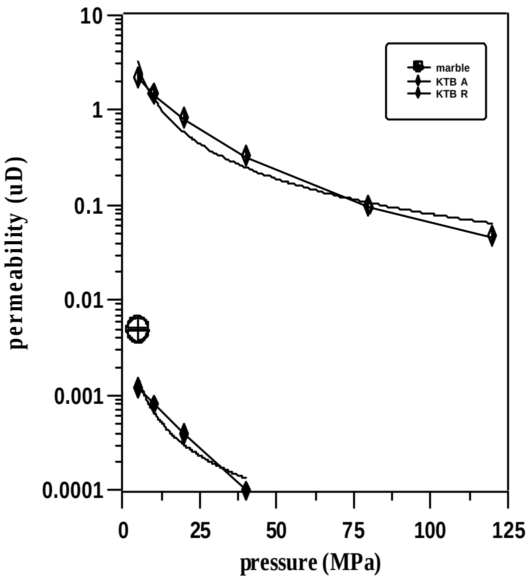
2. Experimental

Carbonate rock samples from surface outcrops and amphibolites from the German Continental Deep Drilling Project (KTB) were chosen for this study. The petrophysical parameters porosity, permeability, inner BET surface and complex electrical conductivity were measured. The pressure dependence of the permeability was determined on cylindrical plugs in an autoclave up to confining gas-pressures of 300 MPa using a pressure transient technique (experimental details can be found in Nover et al., 1995, 1998). The electrical conductivity measurements were performed in an autoclave that allowed the independent adjustment of confining and uniaxial pressure. Confining pressures could be increased up to 350 MPa, the uniaxial load up to 800 MPa. The dispersion of the electrical conductivity was measured on fluid saturated specimens (NaCl solution of 0.1 in molarity).

3. Results and Discussion

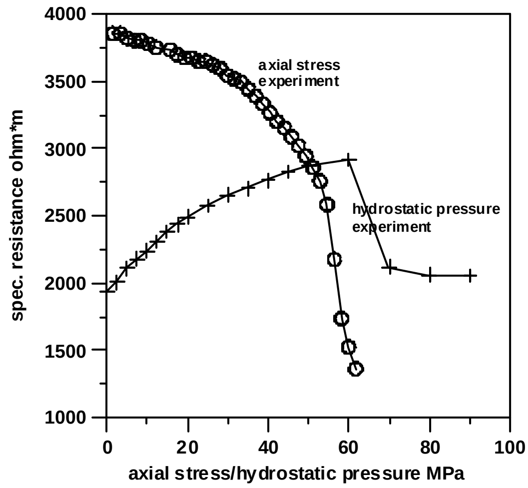
3.1 Petrophysical Properties

The main features of the experiments performed will be discussed on two samples, a marble and an amphibolite fulfilling the criteria: comparable in grain size, permeability and porosity. The



amphibolite (KTB) was fine grained and dense with only minor occurance of open fissures which were mainly caused by pressure and temperature release when the sample was brought up to the surface. Porosities are less than 3 vol % for both samples and permeabilities range from 1 µD to less than 10 nano-Darcy (Fig. 1) depending on the orientation of the sample (amphibolite) thus revaling a significant anisotropy in crack orientation and permeability. Permeability in radial orientation was about three orders in magnitude higher than in direction of the borehole axis.

Fig. 1 Pressure dependence of the permeability of the marble and the KTB rock samples. The permeability was measured using a pressure transient technique with a pressure gradient of 5 MPa across the sample. The transport medium was Argon-gas. The confining (hydrostatic) pressure could be increased up to 300 MPa.



The progressive closure of microcracks results in a pronounced permeability decrease at low confining pressures up to about 80 MPa. At pressures above 100 MPa the pressure/permeability relation (p/k) tends to be more linear reflecting the closure of texture related fractures (Freund & Nover, 1995, Nover et al., 1998). This result correlates with the results obtained from acoustic measurements (Dürrast & Siegesmund, 1999) where a significant increase of the Vp and Vs wave velocities were measured in the low pressure range, while at pressures above 150 MPa intrinsic textural properties dominate the velocity increase.

Fig. 2 Pressure dependence of the volume (bulk) resistivity of samples that were used in a hydrostatic (resistivity increase) and a triaxial pressure experiment (resistivity decrease).

3.2 Complex electrical conductivity in hydrostatic and triaxial pressure experiments

The bulk-conductivity was changed significantly when the samples are exposed to either hydrostatic or triaxial pressure conditions (Fig. 2). When hydrostatic pressures were applied, the bulk resisitivity was increased by the progressive closure of fractures. First fractures with a high aspect ratio are closed, then, at higher pressures, the intrinsic properties control the pore volume reduction (Nover et al., 1998, 1999, 2000). The reverse observation was made in triaxial pressure experiments, where a decrease of the bulk-resistivity, due to the formation of new fractures, was observed. In these experiments the axial stress was increased gradually until failure of the sample (strain: 6 x 10^{-4}, strain-rate: 10-7 s^{-1}). Displacement and axial stress are shown in Fig. 3 for KTB sample VB934C1e. The complex electrical conductivity was measured for each step in pressure, the confining pressure was kept constant at 2 MPa.

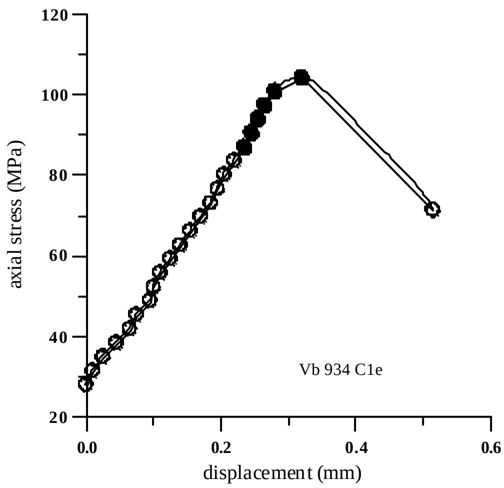


Figure 4 a,b show the results of axial stress experiments performed on a KTB and marble sample. Axial stresses were increased in both experiments until the sample failed. Complex AC-impedance data are presented as Cole-Cole diagrams where the real part of the impedance (Z') is plotted versus the imaginary part (-Z"). The step rate was 3 MPa in the case of the KTB amphibolite (Fig. 4a) and 2 MPa for the marble (Fig. 4b). The KTB sample resists axial stresses up to 91 MPa without significant variations in the shape of the Cole-Cole semicircle.

Fig. 3 Complete load displacement of a KTB amphibolite. The load was increased stepwise, because of measuring the complex electrical response for each step in pressure.

At pressures above 91 MPa a significant variation in the shape of the semicircle was detected, indicating that the time constants of polarisations at the inner surface of the pores have been changed. The point of intersection of the semicircle with the real axis (Z') clearly exhibits a decrease in bulk resistivity. Failure occured at axial stress above 100 MPa. A slightly different result characterises the marble sample. Well-developed Cole-Cole semicircles could be measured, and in contrast to the KTB sample, the shape of these semicircles is varied for each step in pressure (Fig. 4b). Consequently, this sample is deformed even at low axial-stress (1.8 MPa). The final steps of the loading process (above 50 MPa) result in a significant variation of the complex response. Once again bulk-conductivity and time constants of the polarisations were changed. These results show qualitatively that electrical methods are a very sensitive tool for the detection of pore structure variations.

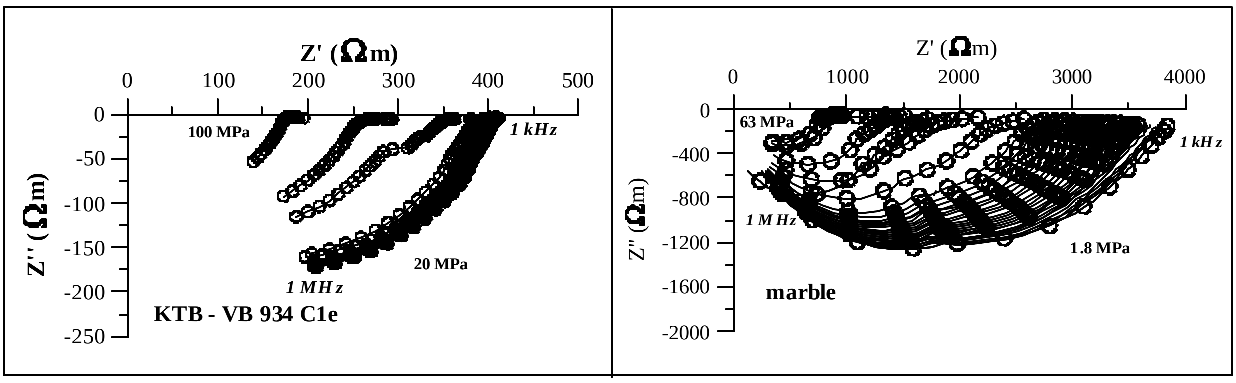


Fig. 4 Complex response of a carbonate rock sample and an amphibilite from the KTB used in triaxial pressure experiments. The data are displayed as Cole-Cole-diagrams where the real part of the impedance is plotted versus the imaginary part. The intersection of the semicircle with the real axis gives the volume or bulk conductivity, which was usually measured at frequencies of about 1 - 5 kHz. Frequency develops from right to left, the left end of the "semicircles" corresponds to the 1 MHz data point.

As mentioned before, the electrical charge transport in fluid saturated rock samples consists of both, ohmic conductivity and diffusion-controlled electrochemical processes with the consequence that the conductivity is a complex quantity: $\rho^* = \rho` + i\rho``$. Measurements of the real $\rho`$ and imaginary part ρ``of the conductivity therefore include informations on pressure induced variations in pore geometry. These were interpreted by fitting model data to the measured data (experimental details in Nover et al., 1995, 1998 and 1999). Two parallel RC elements in series were used to consider pure electrolytic conduction and surface-related conduction. The time constants of the polarisations are given by their relaxation times τ =R*C. The non-linear least squares refinement resulted in four model parameters (R1,C1,R2,C2), which described the electrical charge transport (Table 1)

Table 1: Refined model parameters of the equivalent circuit model consisting of two parallel RC elements in series.

Sample	R1 ohm	C1 µF	R2 ohm	C2 µF	Pressure MPa
Marble	1570	2.6e-3	2013	2.8e-3	1.8
	387	1.12e-2	1251	3.e-3	57
	213	2.13e-2	988	3.2e-3	61
	86	5.66e-2	604	4.00e-3	63
KTB VB 934	327	1.80e+1	54	4.3e-1	21
	301	6.70e+1	39	5.5e-1	90
	204	3.30e+1	64	7.5e-1	94
	172	4.50e+2	29	9.e-1	97
	110	1.37e+3	12	1.1e-1	100

Figure 5 displays the variations of only two of the model parameters, C1 and C2. Both capacitors are related to polarisations where C2 considers bulk and C1 surface-related polarisations. C2 does not change significantly as a function of pressure, while C1 increases continously and most remarkable gives a sharp increase before the sample fails (see last 5 data points in Fig. 3). Thus we can clue that impedance spectroscopy data provide a significant precursor signal before failure.

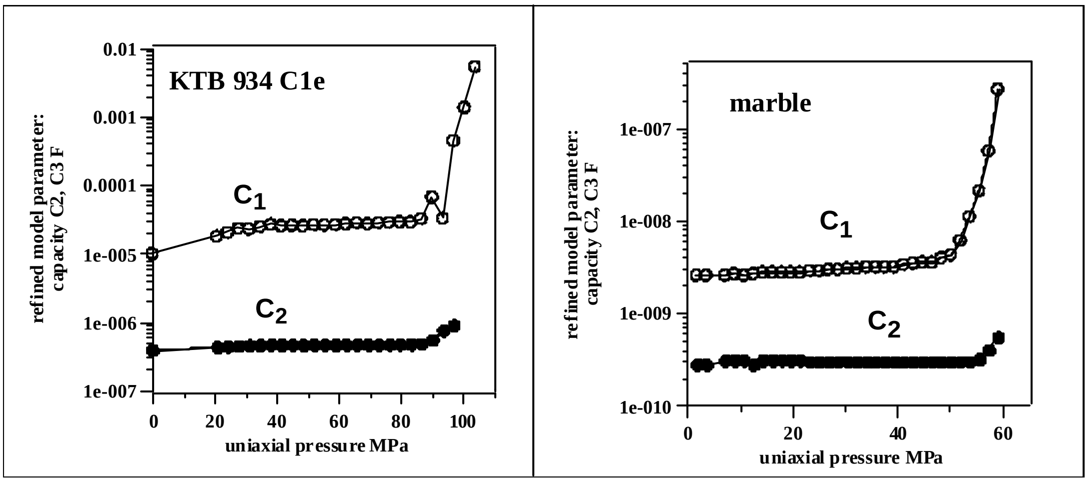


Fig. 5 Variation of the refined model parameters C1 and C2 as derived from the equivalent circuit model. Capacity C1 is the electrical parameter that correlates with the inner surface of the sample.

The voltage decay curve of IP measurements is related to the Cole-Cole parameters by the proportionality $Z(\zeta)=1 / (1+(i\zeta\tau)c)$, where   is the relaxation time of the relaxation process (τ=RC) and $\zeta=2οf$ (f=frequency) and c a parameter describing diffusion controlled processes (Pape et al., 1999). Consequently this "laboratory precursor signal", as derived from AC-impedance data (1 kHz up to 1 MHz) can be compared with field measurements of SP or IP signals by a Fourier-transformation from frequency into time domain. This work is still in progress.

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