The electrical signature of rock samples exposed to hydrostatic and triaxial pressures

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Abstract
The electrical signature of sedimentary (carbonate) and crystalline rock samples was studied in hydrostatic and triaxial pressure experiments up to 300 MPa. The aim was to establish a relation between an electrical signal stimulated by an external pressure acting on the sample and the mechanical stability of the rock. Natural open fractures tend to be closed under hydrostatic pressure conditions, whereas in triaxial pressure experiments new fractures are generated. These contrary processes of either decrease or increase in crack density and geometry, cause a decrease or increase in the inner surface of the sample. Such pressure induced variations in pore geometry were investigated by an interpretation and modelling of the frequency dependence of the complex electrical conductivity. In a series of hydrostatic pressure experiments crack-closure was found in the electrical signature by a decrease of the model capacitor C being related to crack geometry. This capacitor increases in the triaxial experiments where new fractures were formed.

Key words *electrical impedance spectroscopy – fracturing – carbonate – amphibolite – pressure*

1. Introduction

The electrical conductivity of crustal rocks covers more than 10 orders in magnitude. Two principle processes must be separated, electrolytic conduction and electronic conduction. If electrolytic conduction takes place in a salinar pore fluid (ions or water dipols) than pore geometry, permeability, tortuosity and porosity are the most important petrophysical parameters controlling the electrical bulk conductivity. Poor in conductivity are generally low porous and low permeable crystalline rocks like granites, gneisses or amphibolites. They range in conductivity from about 10^{-9} to 10^{-2} S/m depending on the degree of saturation of the pores and the conductivity of the electrolyte. The highest conductivities were detected in ore minerals or in those rocks where an interconnected network of highly conducting phases like graphite or ore minerals can increase the bulk conductivity up to a few S/m (Glover and Vine, 1992; Duba *et al.*, 1994; Nover *et al.*, 1998).

If we focuss on electrolytic conduction in fluid saturated rocks, then the Archie equation relates bulk conductivity and porosity

$$F = 1/\Phi^m$$

with F = formation factor = ρ_0/ρ_ω = bulk resistivity/electrolyte resistivity, Φ = porosity and m the cementation exponent. The pressure dependence of the bulk conductivity was considered in an extended version of the Archie equation by introducing an additional exponent describing the reduction in porosity and the increase of the cementation exponent at elevat-

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ed pressures. This picture relates an electrical parameter to a petrophysical property, the porosity. A much more detailed description of pore geometry variations in pressure experiments can be derived from sophisticated electrical measurements where the frequency dependence of the conductivity is considered. The principle idea is based on the fact, that ions and dipoles in the fluid pore phase are accumulated on the inner surface of the rock due to surface charges of the rock forming minerals. Thus we have «clouds» of charged particles fixed on mineral surfaces. These clouds oscillate (depending on their relaxation time) in the applied electrical field if the frequency is varied in a wide range, *e.g.*, Hz up to MHz. Consequently pressure induced variations in pore geometry change both, the overall electrical (bulk)-conductivity and the frequency dispersion of the complex reponse of the sample. Thus we can correlate changes in pore geometry with electrical properties by measuring the frequency dependence of the complex electrical conductivity.

To prove this idea we have applied hydrostatic pressures to reduce the cross sections of the conducting pathes and thereby increase the rock resistivity (Nover *et al.*, 2000). The reversed experiments were triaxial loading experiments where new fractures were formed, grow and finally form clusters of cracks and microcracks. Thus the «conducting» cross section of the (electrical and hydraulic) conducting paths was increased together with the inner surface of the sample and its mechanical stability was decreased. Such pressure induced variations in pore geometry were monitored by electrical impedance spectroscopy. A modelling of the data provides access to more detailed petrophysical variations like *e.g.*, the correlation with inner surface (BET) data.

In this paper we report on hydrostatic and triaxial pressure experiments performed on carbonate rocks from oil drillings and crystalline rock samples from the KTB. The pressure dependence of the electrical conductivity and permeability were measured and correlated with the petrophysical parameters porosity, inner surface and seismic compressional and shear wave velocities.

2. Experimental

Carbonate rock samples from surface outcrops, oil drillings and garnet-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 on cylindrical plug samples having dimension of 30 mm in diameter and about 30 mm heigh. Porosities were determined using the Archimedian method, mercury porosimetry and the BET method as well.

Permeabilities (and their pressure dependence) were measured in an autoclave that allowed an increase of a confining gas-pressures of up to 300 MPa. A pressure transient technique was used to calculate the permeability. The pressure gradient across the sample was fixed at 5 MPa. The time required for pressure compensation was used to calculate the permeability k which is defined as

$$q = -kA/\eta dP/dx$$

where q = flow rate, volume of gas per unit in time, k = permeability, A = area, η = viscosity, dP/dx = pressure gradient (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 using a NaCl solution of 0.1 in molarity. Ohm's law relates conductivity σ of a material, current density J and stimulating electric field E

$$J = \sigma E.$$

The electrical charge transport in fluid saturated rock samples comprises both, ohmic conductivity and diffusion controlled processes. Consequently the conductivity becomes a complex quantity

$$\sigma^* = \sigma' + \sigma''.$$

Measurements of the real σ' and imaginary part σ'' of the conductivity σ were performed in the frequency range 1 kHz up to MHz. Thus pressure induced variations in pore geometry could be interpreted in terms of changes in dielectric properties. Experimental details can be found in Nover *et al.* (1995, 1998, 2000).

3. Results and discussion

3.1. *Petrophysical properties*

Though a huge number of samples were measured, the results of only two samples will be presented in more detail in this study. The samples selected (marble, amphibolite) fitted to the following 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 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). In general KTB samples exhibit a significant anisotropy in crack orientation and thus in the permeability. A power fit approximately fits the expression $\log(k) = -1.5 \log(p) + 5.6$ for the pressure dependence of permeabilities in radial direction, while $\log(k) = -2.6 \log(p) + 4.7$ fits KTB data in direction of the borehole axis. Permeabilities in radial (in regard to the borehole axis) orientation generally are two orders in magnitude higher than in direction of the borehole axis. The progressive closure of microcracks results in a pronounced permeability decrease at low confining pressures (up to about 80 MPa), while at pressures above 100 MPa the pressure/permeability relation (*p/k*) tends to be more linear reflecting the closure of texture related fractures (Freund and Nover, 1995; Nover *et al.*, 1998). This closure of fractures as detected by the permeability measurements correlates with the results obtained from acoustic measurements

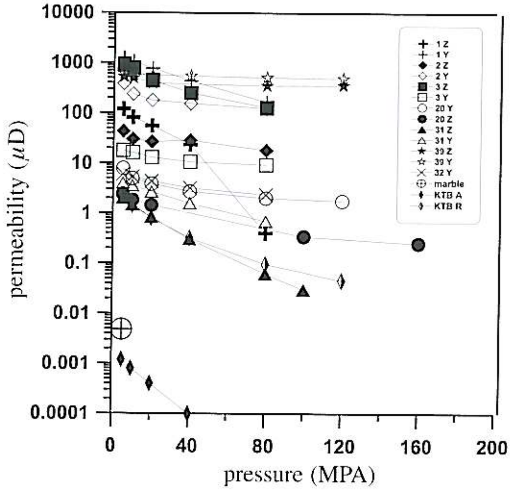

Fig. 1. Pressure dependence of the permeability of carbonate and crystalline rock samples. The permeability was measured using a pressure transient technique with a pressure gradient of 5 MPa across the sample. The transport medium was Argon-gas. The confining (hydrostatic) pressure could be increased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

(Dürrast and Siegesmund, 1999) where a significant increase in the V_p and V_s wave velocities were measured in the low pressure range. At pressures above 150 MPa intrinsic textural properties dominate the velocity increase.

3.2. *Complex electrical conductivity*

Two kinds of pressure experiments were performed, *hydrostatic* and *triaxial* pressure tests.

3.2.1. Hydrostatic pressure experiment

Results of hydrostatic pressure experiments are already published (Nover *et al.*, 2000). Here we will present only a brief summary of important features as required for the understanding of the triaxial pressure experiments. The hydrostatic experiments exhibit the typical increase of the bulk resisitivity as a function of pressure which is caused by the progressive closure of

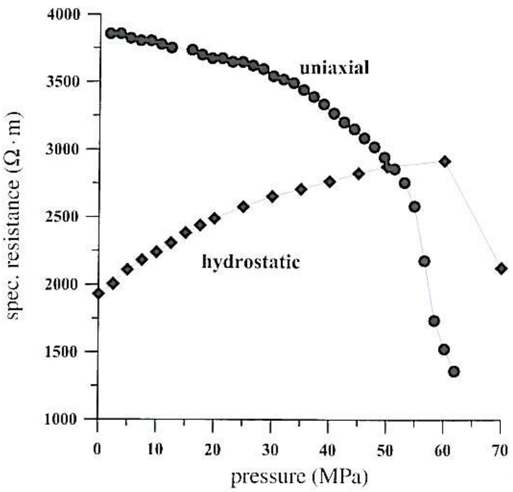

Fig. 2. Pressure dependence of the volume (bulk) resistivity of samples that were used in a hydrostatic (resistivity increase) and a triaxial pressure experiment (resistivity decrease).

fractures (fig. 2). First fractures having a high aspect ratio are closed, then at higher pressures the intrinsic pore volume is reduced. Consequently the inner surface, that acts in an electrical sense as a capacitor, reduces its value and thus causes a variation of the complex electrical response of the sample. The refined model parameters and especially the capacity that is directly related to variations of the inner surface are decreased by up to two orders of magnitude. A qualitative correlation with BET data of the inner surface improved this finding. For comparison we have included in fig. 2 the results of a triaxial pressure experiment which was performed on an identical marble sample. In this kind of experiment the typical decrease in bulk-resistiviy was measured.

3.2.2. Triaxial pressure experiments

Figure 3 displays the results of triaxial pressure tests that were performed until failure of the sample. Data are presented as Cole-Cole diagrams where the real part of the impedance (Z') is plotted *versus* the imaginary part ($-Z''$). Frequencies develop from low (1 kHz) to high (1 MHz) from the right end to the left end of each «semicircle». The uniaxial load was increased stepwise until failure of the sample. The step rate was 3 MPa in the case of the KTB amphibolite sample and 2 MPa for the marble. It took about 10 min to measure the complex response for one step in pressure with 15 frequencies in a log scaling for one decade in frequency.

The complex responses of both samples show typical common features. In the low pressure range a quarter of a semicircle is well developed. The increase in pressure causes only minor changes in the shape of the semicircle, the indicating that the electrical properties experience only minor changes. But when the uniaxial load was increased to certain threshold values close to the uniaxial compressive strength, the significant variations in the complex response of the samples were detected. Though the step-size was still 3 resp. 2 MPa these final semicircles exhibit dramatic changes in volume resistivity and frequency dispersion. Sample failure occurred after increasing the load up to 100 resp. 63 MPa.

Using a non-linear least-squares program, the measured frequency dispersion data were fitted to model data describing the electrical charge transport in rock samples. The equivalent circuit model used considered pure electrolytic conduction and surface related conduction and polarization processes employing two parallel RC elements in series. The electrochemical basis of this interpretation is due to the fact that polarisations of ions or water dipols take place in the interface layer rock-matrix/fluid-phase. This layer is known in electrochemistry as the electrochemical Double Layer (DL). The relaxation times τ of surface adsorbed cations and hydrated anions and cations within this DL differ in time constants depending on size, ion-concentration and interaction in narrow cracks. The chemistry of the pore electrolyte was fixed during the experiments, variations in relaxation times therefore must be due to geometric changes of the cracks or pores. From the non-linear least squares refinement four model parameters were derived (R1, C1, R2, C2). These are related to the above described effects. The product

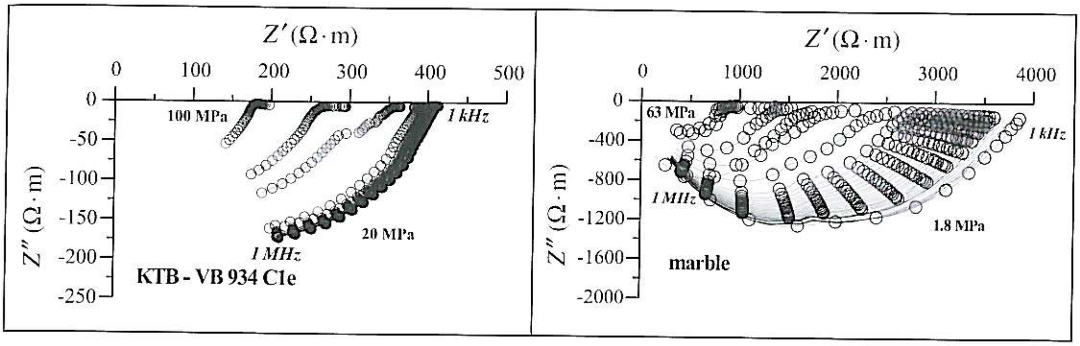

Fig. 3. Complex response of a carbonate rock sample and an amphibilite from the KTB used in triaxial pressure experiments. The data are displayed as Cole-Cole-diagrams where the real part of the impedance is plotted *versus* the imaginary part. The intersection of the semicircle with the real axis gives 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.

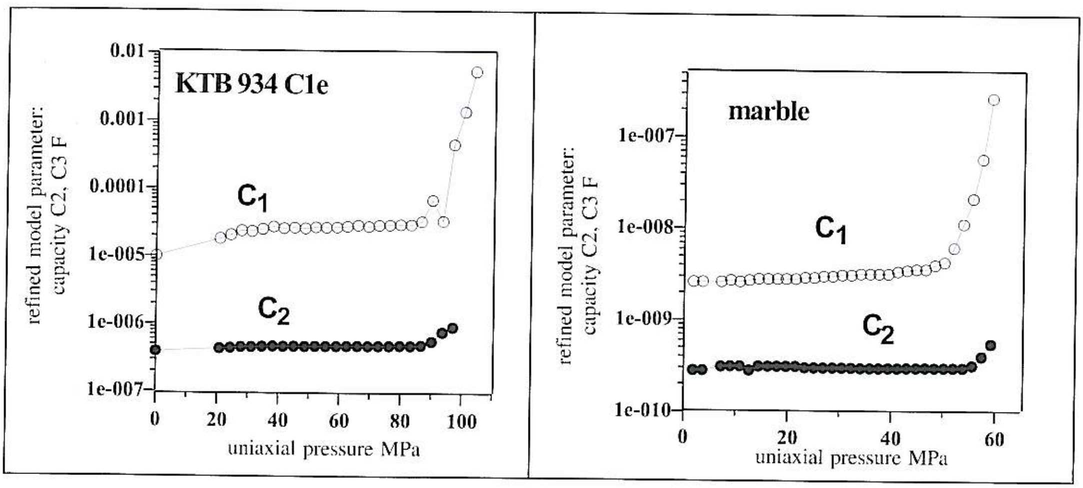

Fig. 4. Variation of the refined model parameter C2 as derived from the equivalent circuit model. Capacity C2 is the electrical parameter that correlates with the inner surface of the sample. Pressure induced forming of microcracks increases the inner surface and the model capacity C2.

RC = τ defines the relaxation time. Figure 4 displays the variations of only two of the model parameters, C1 and C2. Both capacitors are related to polarisations where C1 considers bulk and C2 surface related polarisations. As expected, C2 does not change significantly as a function of pressure, while C1 increases continously as a function of pressure and most remarkably gives a sharp increase right before failure of the sample (table I). Thus we can conclude that impedance spectroscopy provides a significant precursor signal before failure.

This «laboratory precursor signal» cannot be directly applied in field geophysics. But the Fourier transformation of the frequency dispersion data results in voltage decay curves in

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

Sample	R1 Ω	C1 μF	R2 Ω	C2 μF	Pressure MPa
Marble	1570	2.60E − 03	2013	2.80E − 03	1.8
	387	1.12E − 02	1251	3.00E − 03	57
	213	2.13E − 02	988	3.20E − 03	61
	86	5.66E − 02	604	4.00E − 03	63
KTB	327	1.80E + 01	54	4.30E − 01	21
	301	6.70E + 01	39	5.50E − 01	90
	204	3.30E + 01	64	7.50E − 01	94
	172	4.50E + 02	29	9.00E − 01	97
	110	1.37E + 03	12	1.10E − 01	100

the time domain (Vallianatos and Tzanis, 1998, 1999; Colangelo *et al.*, 2000). Thus laboratory AC-impedance data can be compared with field SP or IP signals. The voltage decay curve of IP measurements can be described in terms of Cole-Cole parameters with the proportionality $Z(\omega) \propto 1/(1 + (i\omega\tau)^c)$, where τ is the relaxation time of the relaxation process (τ = RC) and $\omega = 2\pi f$ (f = frequency) and c a parameter describing diffusion controlled processes (Pape *et al.*, 1999). To check this assumption we are currently performing laboratory SP and AC-impedance spectroscopy measurements to prove the validity of the Fourier transformed dispersion data.

4. Conclusions

Hydrostatic and triaxial loading experiments on carbonate and crystalline rock samples indicate that a relation between an electrical parameter (complex conductivity) and a petrophysical parameter (inner surface) exists. In this sense increasing inner surface means an increase in crack density, a parameter being linked to the mechanical stability. This variation in crack density correlates with a variation of the complex electrical response. Consequently AC-impedance spectroscopy data can be used as a sensor for detecting the prefailure stability in rocks.

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