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Electrical behaviour of rock samples from Puglia 1 borehole (South Italy): from 20 °C to 1000 °C

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Abstract
During 1992, at the Applied Geophysics Laboratories of Florence (I) and Prague (CS) some electrical measurements were made on deep rock samples to understand the source of low resistivity values measured by logs. The studied samples were sandstones (6380 m depth) and dolomites (6070 m depth) coming from the borehole AGIP-PUGLIA 1 (South Italy). The electrical parameters were the real part of the complex resistivity, the loss tangent and the total harmonic distortion. They were measured as functions of temperature ($20 \leq T \leq 1000$ °C), confining and internal pore pressure ($3 \leq p \leq 33$ MPA), and frequency ($0.002 \leq f \leq 1000$ Hz). For the measurements, two different apparatus were used: the Florence apparatus working up to $T = 200$ °C conditioning internal pore fluid, and the Prague apparatus working in temperature (200 ÷ 1000 °C). Both the literature and laboratory research confirm that the electrical results at our experimental physical conditions remain unchanged. The obtained results confirmed that deep rocks can show low resistivity valued at special internal fluid pressures and temperatures, according to their mineralogical composition and petrographic structure.

Key words *high temperature – electrical properties – low frequency*

1. Introduction

It is known that electrical resistivity is a useful parameter to study the physical conditions of the Earth's interior, thanks to its high sensitivity to thermal changes. Geophysical researchers have long been working on the problem of defining the electrical properties of rocks in the laboratory, mainly at high pressures and temperatures (some GPa and up to 1200 °C). These *p-T* value ranges were selected because they simulate the theoretical physical characteristics of the deep crust and of the lithosphere (Ádám, 1976; Dvorak, 1975; Hirsch and Shankland, 1990; Kariya and Shankland, 1981; Keller, 1982; Parkhomenko, 1989). In literature, there is agreement on the strong increase in electrical resistivity owing to oxidrile loss. In fact, a dehydration process appears on minerals at $T \approx 400$ °C, so serpentinization occurs: this temperature is one of the *critical temperatures* found by Duba (Constable and Duba, 1990; Duba *et al.*, 1978) and Olhoeft (Hall and Olhoeft, 1986; Olhoeft, 1979). They spotted five main regions with different values of the activation energy (thermal activation regions) limited by the following temperatures: 100 °C, 450 °C, 700 °C and 1200 °C.

At sub-surface conditions, the rock electrical behaviour is complicated by liquid-vapour equilibrium of the internal fluid and by electrochemical phenomena (Losito, 1989; Llera *et al.*, 1990). In wet rocks, $T \approx 100$ °C is a very important temperature given the transition of the water from the fluid to the gas state, at atmospheric pressure.

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2. Studied samples

AGIP S.p.A. made available some rock blocks from PUGLIA 1 deep borehole and well log (fig. 1). The samples used for the electrical measurements come from 6067-6075 m (core 2 = N2, N6a, N6b) and from 6372-6379 m (core 3 = N3, N4).

Core 2 is a very coarse grained dolomite with interbedded synsedimentary breccias, shales and anhydrite levels (in nodules or large crystals). The porosity is low (0.7 ÷ 1.4%), whereas the permeability can ben high because of microcracks (0.12 ÷ 6.7 md). The age is upper Trias.

Core 3 is a continental sandstone, fine to coarse grained. The grains are quartz, muscovite, biotite, metamorphic and sedimentary fragments; the matrix is argillaceous-sericitic with mica and illite. The fractures are filled by illite, dolomite and calcite more or less iron rich. The porosity is low (0.6 ÷ 1.9%), whereas the permeability can be high (0.06 ÷ 32.8 md). The age is late Permian.

Differential Thermal Analysis (DTA), ThermoGraphy (TG), Differential ThermoGraphy (DTG), RenTgenoGraphy (RTG), and microscopic analysis have been done (fig. 2). The petrophysical analysis identified two groups of samples (see table I):

– N3, N4 (6376.47 m and 6378.28 m) – these samples are sandstones. Their chemical composition is wide: quartz (the richest in SiO_2 is N4) and micas, mixture of carbonates, illites and kaolinites (matrix). A graphitic component is present only inside pelites, quartzite and phyllite fragments.

– N2, N6 (6067.20 m and 6072.54 m) – these samples are dolomites from the «Anidriti di Burano» formation. N2 is the richest in CaO. Anhydrite is represented by needles-line aggregates. Spherical elements of ferric hydroxides are present (psammitic and pelitic elements). The permeability is negligible.

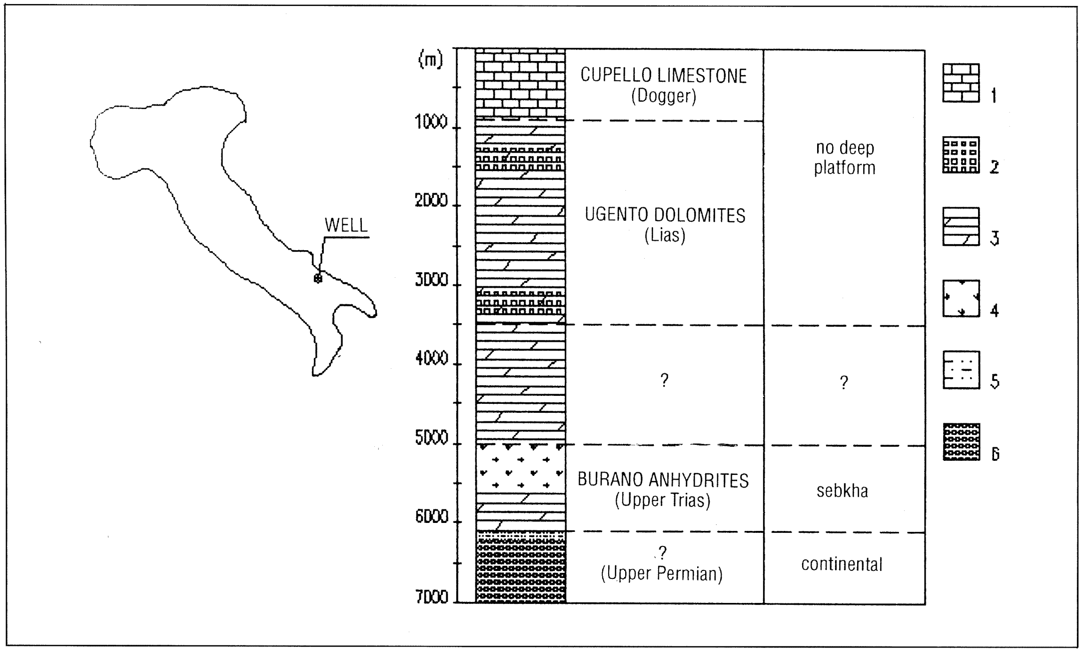

Fig. 1. Location and stratigraphic schema of the well: 1) sandstones with clay and marns interbedded; 2) dolomitic breccia; 3) dolomite; 4) anhydrite; 5) red clay; 6) siliceous-carbonatic sandstones.

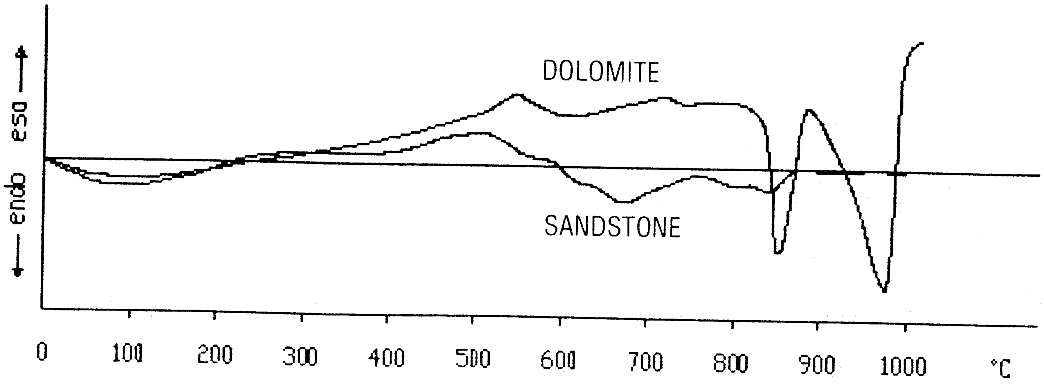

Fig. 2. Differential thermal analysis for sandstone and dolomite samples.

Table I. Chemical and mineralogical composition of PUGLIA 1 samples studied at the Geophysical Laboratory in Florence. The values are in percent wt. Matrix = illite-kaolinite-carbonate mixture; sedimentary fragments = pelites, graphitic siltites; metamorphic fragments = quartz-graphite-muscovite schists.

Chemical composition

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	CO_2
PUGLIA 1										
N1	0.2		0.6	0.1	36.6	11.2	0.1			49.5
N2	1.4		0.7	0.1	21.5	29.3	0.1	0.2		46.8
N3	67.3	0.8	13.6	3.9	2.5	1.1	0.3	2.5	0.1	7.6
N4	75.0	0.5	8.7	3.8	2.6	1.2	0.2	1.3	0.1	6.2

Mineralogical composition

Mineral	N3	N4	N6
PUGLIA 1			
Quartz	34.5	56.6	
Quartzite	2.5		
Light mica	9.2	3.6	
Clay	6.5		
Dolomite			85.0
Anhydrite			14.2
Sedimentary fragments		15.3	0.8
Metamorphic fragments	2.9	11.0	
More	0.5		
Matrix	43.9	13.5	

3. Superficial environment (up 3000 m)

At the Applied Geophysics Laboratory electrical rock properties *versus* temperature T (°C), confining hydrostatic pressure pe (MPa) and internal pore fluid pressure pi (MPa), and energization signal (frequency f (Hz), waveform, amplitude) are studied (Bernabini *et al.*, 1988; Losito, 1989; Losito *et al.*, 1991). The apparatus, designed by Losito and Trova to simulate the thermo-baric conditions of both geothermal areas and high crust in the laboratory, is well described in Losito (1989) and in Losito *et al.* (1991).

The samples are cut into cylindrical form (diameter = 36 mm and lenght $= 20 \div 120$ mm) before the electrode setting. The current electrodes are stainless steel porous electrodes, to permit the fluid to fill and pressurise the sample internal pores.

The fluid used is distilled water so as not to alter the sample chemism (only dilution appears), introducing stranger polarisation phenomena. This choice follows from personal experience (Losito *et al.*, 1991) and from the works of Glover and Vine (1995), Knight and Dvorkin (1992), Llera *et al.* (1990) and Losito (1989).

The measurements are made in the frequency range $2 \cdot 10^{-3} \div 2 \cdot 10^{2}$ Hz: the signals are continuously monitored and the parameters are calculated for every decade of frequency.

3.1. *Results at low temperatures*

The samples studied at low depth simulated conditions are two sandstones (N3 and N4), and one dolomite (N6). For each one the real part of the complex resistivity $\rho\,(f)$, the dielectric loss tangent tg δ, and the Total Harmonic Distortion in % of the current signal, were measured as functions both of the temperature T and of the differential pressure Δp $(= pe - pi)$.

In detail, their electrical behaviours are:

– N3 (fig. 3) = the system is regular for $T \leq 120$ °C. The increase is $\rho\,(f)$ for high temperatures ($T \geq 150$ °C) and extreme range frequencies is probably linked to the internal fluid physical state (vapour) and to the phase shift due to the mineralogical composition (mainly clay components, see table I).

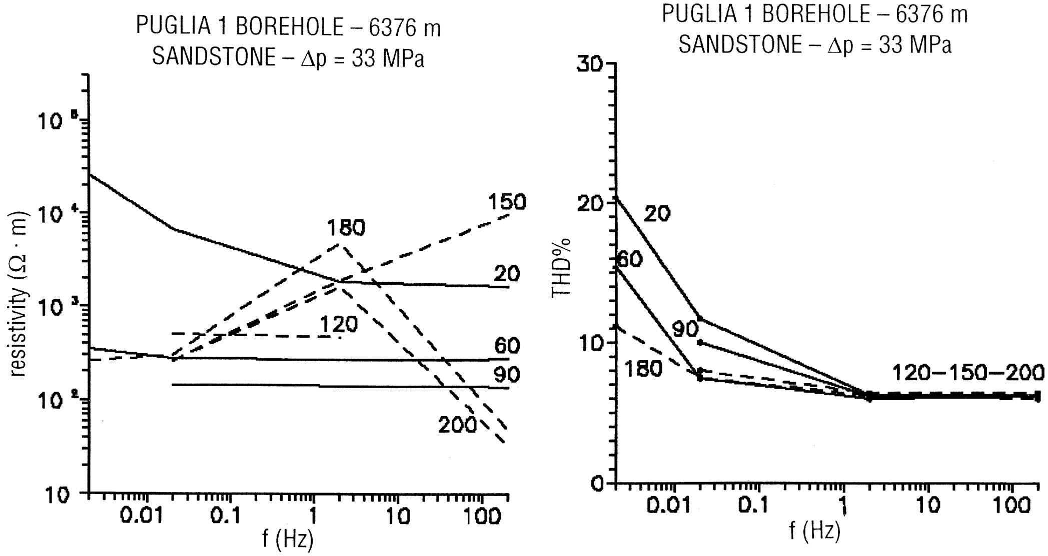

Fig. 3. Typical electrical behaviour of sandstone samples: resistivity and signal distortion.

Distortion effects are present only for $f < 2$ Hz. The temperature dependence is quite regular and related to the internal fluid physical state: at 90 °C the values are already comparable with the signal noise.

– N4 = like the foregoing sample, the system is regular for $T < 120$ °C, then $\rho(f)$ increases with T.

Also the TDH% and tg δ parameter trends are similar to N3 ones.

– N6 (fig. 4) = this sample has an electrical hebaviour very different from N3 and N4, given the very different composition (see table I) and the poor porosity which prevented its hydration.

At first, the resistivity is higher ($2 \div 3$ orders), and the distortion is about twice as great.

Moreover, the pressure effect is valuable: there is a linear decrease of the distortion with the temperature at small Δp (6 MPa); at Δp maximum (33 MPa), the distortion increases with the temperature up to $T = 150$ °C, then it decreases rapidly. Probably, this is related to the chemical-physical composition of the sample which becomes more uniform.

The distortion effects are present only for $f \leq 2$ Hz.

In general, the resistivity range for the studied sandstones is $50 \div 10^5$ $\Omega \cdot$ m, and for the dolomites it is $5 \cdot 10^4 \div 5 \cdot 10^6$ $\Omega \cdot$ m (see table II), reflecting their mineralogy and petrographic structure (Muschietti, 1993, 1995). In addition, the laboratory results evidence $\rho(p) \approx$ constant for the sandstone samples, meaning that, at these T-p conditions, these samples are structurally homogeneous (Losito, 1989; Rauen and Soffel, 1992).

The high resistivity values at low temperature ($T = 20$ °C) are probably due to chemical phenomena dependent on the restoration of the fluid inside the sample (partial hydration and dissolution). It is interesting to note how the resistivity of these samples is low at physical conditions near the natural ones (the *in situ* temperature is about 110 °C).

Also the effect of the frequency is important, because it singles out the presence of rock clay components showing distortion phenomena low frequencies ($f \leq 0.2$ Hz), (Losito, 1989): in fact the mineralogical analysis confirms a content of clay minerals and illite group up to 50% in pounds for the sandstones.

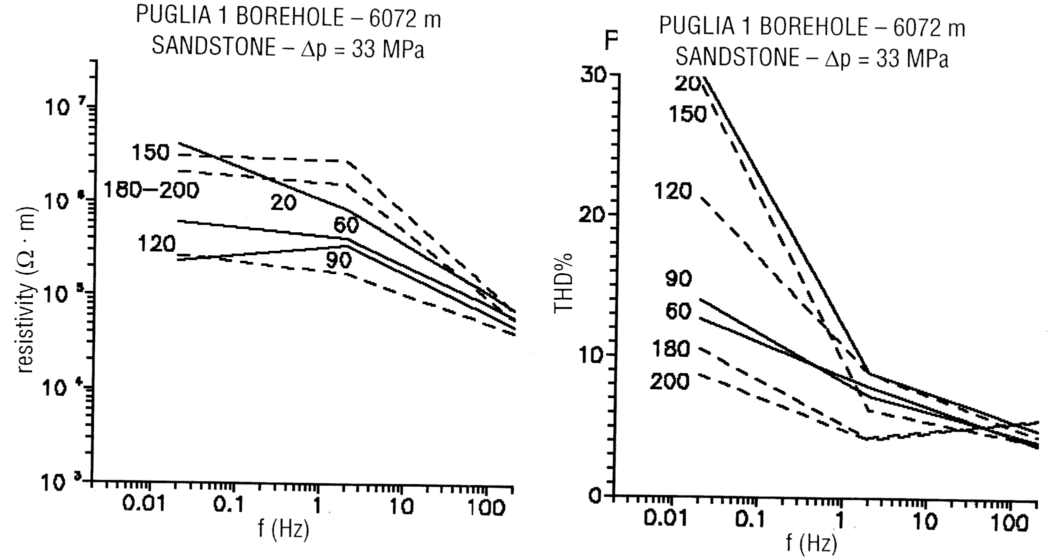

Fig. 4. Typical electrical behaviour of dolomite sample: resistivity and signal distortion.

Table II. ρ logarithms of PUGLIA 1 samples studied under simulated physical conditions at the Geophysical Laboratory in Florence ($T \leq 180$ °C, saturated samples) and in Prague ($T = 1000$ °C, dry samples). MPC = measurement physical conditions, to simulate natural conditions; sample = sample code; PSP = parameters of physical simulation; f = frequency; pi = internal pressure; pe = confining hydrostatic pressure; log ρ = resistivity logarithm at stated experimental temperatures; D = sample depth; surf = surface sample; δ = sample density.

			ρ Logaritms of rock samples							
Area rock type MPC	Sample	PSP	log ρ 20 °C	log ρ 80 °C	log ρ 110 °C	log ρ 150 °C	log ρ 180 °C	log ρ 1000 °C	δ g/cm^3	D (m)
PUGLIA 1	N2		6	6	5	5	4	2		6067
Sandstone	N3	f	4	2	2	2	2		2.71	6376
N3, N4		pi	3	2	2		2			
Dolomite N6		pe	3	2	2		2			
Mix N2			4	4	4	4	5	1		
	N4	f	3	2	2	1	3			6378
$f = 2 \cdot 10^{-3}$		pi	3	2	2	1	3			
$\div 2 \cdot 10^{1}$Hz		pe	3	2	2	1	3			
$pe \leq 39$ MPa			5	5	4	4	5	2		
$pi \leq 36$ MPa	N6	f	6	5	5	6	6			6072
		pi	6	5	5		6			
		pe	6	5	5		6			
			7	6	6	6	6	2		

4. Deep environment (up to 30 km)

The measurements made at simulated natural deep conditions are important both for ρ values used in modelling and as *historical rock thermometers*. In fact, they can disclose the maximum temperature reached during the geological time (sharp change in the resistivity curve slope means $T_{\text{lab}} > T_{\text{natural}}$).

This research was developed at the Czech Academy of Sciences of Prague, where an apparatus to measure the physical parameters of rocks (electrical or magnetic ones), *versus* temperature was operating. This apparatus, described in Lastovicková (1991), is a thermal cell filled with argon (it does not allow oxidation phenomena) and an energization-measure circuit to select the temperature cycle ($20 \leq T \leq 1000$ °C) and the input frequency ($f = 10^3$ Hz or DC).

Inside, the sample is cut in a cylindrical form (fig. 5) (diameter = 8 mm and lenght = 10-15 mm), then the 2-platinum electrodes are inserted. The fixed frequency agrees with superficial measurement results, as follows from Lastovicková and Bochnicek (1992).

4.1. *Results at high temperatures*

The electrical resistivities up to 1000 °C were measured for samples N2, N3, N4, N6a, N6b.

In detail, their electrical behaviours are:

– N3 and N4 = these samples (sandstones) show at $T = 100 \div 200$ °C a non-linear trend, with a strong resistivity increase ($10^4 \rightarrow 10^6$ $\Omega \cdot$ m). Then the curve decreases uniformly up to 50 $\Omega \cdot$ m. At high temperatures, the resistivity trend of rocks that are composed mainly of

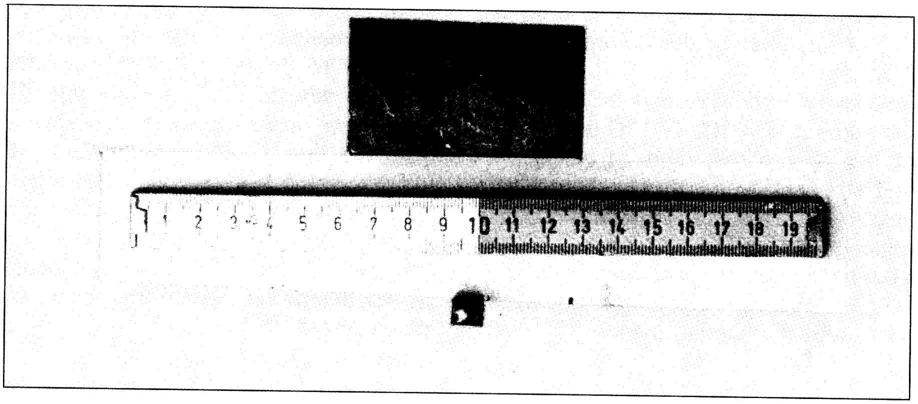

Fig. 5. Different dimensions of samples.

quartz and muscovite (sandstones) is typically monotonous. The quite different type of electrical resistivity behaviour at high temperatures of graywackes agrees with their different mineral and chemical composition, TG, and DTA results. So, the sharp change in the trend of resistivity is because of a small dolomite quantity. The undulations on the curves are due to the release of OH groups from clay minerals, and of CO_2 from dolomite (Lastovicková, 1991). The influence of the main quartzitic component is suppressed by the effect of more conductive assemblage of clay minerals and graphite.

– N2 and N6a,b = these samples (dolomites) exhibit non-linear and rather complicated resistivity curves. The first anomaly is at $T = 100 \div 200$ °C ($10^4 \div 10^6$ $\Omega \cdot$ m), the second anomaly at $T = 450$ °C, (10^4 $\Omega \cdot$ m), and only for N2, belongs to the activation of ions from the crystal lattice before the first release of CO_2; a second activation phenomenon appears at $T = 800$ °C, and the resistivity fall to 10^2 $\Omega \cdot$ m. Both activations took place before the individual steps of CO_2 release, manifested in TG and DTA (strong double endoreaction) records (Lastovicková, 1991; personal note of M. Chlupacova). The resistivity decrease depends on the sample, in fact N2 has a complicated trend with four steps of different slopes while N6 have monotonous increase: this is because samples N6 have more CaO than N2.

The results show two different trends according to the rock type: in fact, N3 and N4 have typical sandstone trends, while N2 and N6 have a dolomite trend (fig. 6). In general,

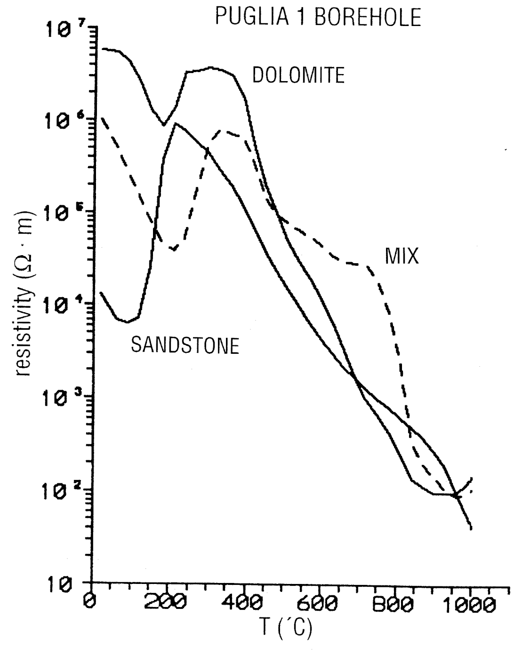

Fig. 6. Resistivity trends of the samples at high temperatures: sandstones, mixed sandstone-dolomite and dolomites.

the resistivity range for the studied sandstones is $50 \div 10^6\ \Omega \cdot$ m, and for the dolomites it is $10^2 \div 10^7\ \Omega \cdot$ m.

The resistivity trend presents a first low *anomalous* value at T = 100-200 °C due to the ionic mobility, then a maximum of resistivity is observed for $T < 400$ °C, due to the release of the adsorbed atmospheric water, and the lowest value is nearly the same.

The cooling curves are different from the heating curves because irreversible changes took place during the heating cycle.

5. Conclusions

The experimental results agree with the literature data on the low resistivity values of rocks at very deep environment. The literature contains many hypotheses on the deep rock electrical conductivity (Duba *et al.*, 1994; Hall and Olhoeft, 1986; Llera *et al.*, 1990; Losito *et al.*, 1991; Rauen *et al.*, 1994), which are based on ions and/or electrons mobility, increased by the temperature following the natural physical and petrographic conditions of rocks: melting, carbonaceous components, fluids, conductive impurities, and clays.

In this context, the low resistivity values measured on the PUGLIA 1 samples, varying the frequency at not very high temperatures, could be connected to the presence of carbonaceous components inside the rocks, which can constitute a conductive net (Glover and Vine, 1995). But in this case the sample's petrophysical analysis shows graphitic elements only in traces, so the conductivity cause must be another one.

In fact, the measurements show rock electrical behaviour where the hydrated silica minerals prevail over carbonaceous-sulphide minerals. In fact, strong distortion phenomena of the electrical signals appear at low frequencies (THD% at $f < 0.2$ Hz).

Also the resistivity values, obtained at p-T values comparable with the low deep environment (few kilometres), are anomalously low. This behaviour can be explained with the good permeability (22 md) of samples, hold during the sedimentation of the old complex, which admitted to preserve fluids inside the rocks down to several kilometres' depth. The fluid circulation in the depth in this case also explain the low temperature gradient (10 °C/km for the first 5 km). Besides, the internal pore fluid pressure, contrasting with the lithospheric pressure, lets the hydrated clay minerals survive the diagenetic mineralogical changes, and/or phyllosilicates like illite grow from the alteration of feldspars (low grade metamorphism).

These results can also be useful to understand a possible source of signal dispersion in some electric and electromagnetic soundings (Lastovicková *et al.*, 1993; Rauen and Lastovicková, 1995). In fact, the analysed samples are representative of the area's lithology, as follows from the mineralogical-petrographic data and from personal conversation with AGIP researchers. Further confirmation on the possibility of extrapolating the laboratory data to the field situation follows from the correspondence between the resistivity log data (around 50 $\Omega \cdot$ m) of several AGIP's boreholes and the hydrated sample laboratory data (see table II).

Other information on the rock electrical behaviour follow from the high temperature measurements on dry samples (Lastovicková, 1991; Lastovicková and Bochnicek, 1992). In fact, these measurements provide both the rock thermal history of the past ($T \approx 200$ °C is the maximum temperature reached from the rock during its geological time) and its possible thermal electrical resistivity evolution ($10^{6 \div 7} \rightarrow 10^{1 \div 2}\ \Omega \cdot$ m).

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REFERENCES

Ádám, A. (1976): Geoelectric and geothermal studies, *KAPG Geophysical Monograph*, Akademiai Kiado, 1-755.

Bernabini, M., G.B. Borelli, G. Finzi-Contini, G. Losito, M. Muschietti and A. Trova (1988): Impiego di una apparecchiatura pilota di laboratorio per lo studio di proprietà elettriche di litotipi dell'area di Bolsena in condizioni termo-bariche di interesse geotermico, in *Proceedings II Sem. In. At. Ric.*, CNR-PFE, SP Energia Geotermica, 385-396.

Constable, S.C. and A.G. Duba (1990): The electrical conductivity of olivine, a dunite and the mantle, *J. Geophys. Res.*, **95**, 6967-6978.

Duba, A.G., A.J. Piwinskii, M. Santor and H.C. Weed (1978): The electric conductivity of sandstone, limestone and granite, *Geophys. J. R. Astron. Soc.*, **53**, 583-597.

Duba, A., S. Heikamp, W. Meurer, G. Nover and G. Will (1994): Evidence from borehole samples for the role of accessory minerals in lower-crustal conductivity, *Nature*, **367**, 59-61.

Dvorak, Z. (1975): Electrical conductivity models of the crust, *Can. J. Earth Sci.*, **12**, 962-970.

Glover, P. and F.J. Vine (1995): Beyond KTB-electrical conductivity of the deep continental crust, *Surveys in Geophysics*, **16**, 5-36.

Hall, S.H. and G.R. Olhoeft (1986): Non-linear complex resistivity of some nickel sulfides from Western Australia, *Geophys. Prospect.*, **34**, 1255-1276.

Hirsch, L.M. and T.J. Shankland (1990): Electrical conduction: from laboratory to mantle conditions, in *Abstracts of 10th Workshop on Electromagnetic Induction in the Earth*, Ensenada Mexico.

Kariya, K.A. and T.J. Shankland (1981): Electrical conductivity in lower crustal rocks, *Eos, Trans. Am. Geophys. Un.*, **62**, 267.

Keller, G.V. (1982): Electrical properties of rocks and minerals, in *Handbook of Physical Properties of Rocks*, edited by R.S. Carmichael (CRC Press), vol. I, chap. 2, 217-293.

Knight, R. and J. Dvorkin (1992): Seismic and electrical properties of sandstones at low saturations, *J. Geophys. Res.*, **97** (B12), 17425-17432.

Lastovicková, M. (1991): A review of laboratory measurements of the electrical conductivity of rocks and minerals, *Phys. Earth Planet. Inter.*, **66**, 1-11.

Lastovicková, M. and J. Bochnicek (1992): A contribution to the measurements of frequency dependence of electrical conductivity of rocks, *Studia Geophys. Geod.*, **36**, 94-99.

Lastovicková, M., G. Losito and A. Trova (1993): Anisotropy on electrical conductivity of dry and saturated KTB samples, *Phys. Earth Planet. Inter.*, **81**, 315-324.

Llera, F.J., M. Stato, K. Nakatsuka and H. Yokoyama (1990): Temperature dependence of the electrical resistivity of water-saturated rocks, *Geophysics*, **55**, 576-585.

Losito, G. (1989): A new rock physics laboratory apparatus with programmed thermobaric cycles and controlled pore fluid internal pressure (theory, hardware-software, electrical measurements), CNR-PFE, SP Energia Geotermica, LB-21.

Losito, G., M. Muschietti and A. Trova (1991): Laboratory electrical resistivities of rock samples under geothermal temperature-confining hydrostatic pressure conditions, *Geothermics*, **20**, 165-178.

Muschietti, M. (1993): Procedure elettriche integrate per l'individuazione di strutture geologiche profonde: misure di magnetotellurica e di laboratorio, *Tesi di Dottorato*.

Muschietti, M. (1995): First results in the electrical behaviour comparison between graphitic and no graphitic deep rock samples, in *Proceedings of XX EGS Gen. Ass., Hamburg (D)*, Part I.2, SE16, C119.

Olhoeft, G.R. (1979): Non-linear electrical properties, in *Non-Linear Behaviour of Molecules, Atoms and Ions in Electric, Magnetic and Electromagnetic Fields* (Elsevier, Amsterdam), 395-409.

Parkhomenko, E.I. (1989): *Geoelectrical Properties of Minerals and Rocks under High Pressures and Temperature* (Nauka, Moskow), 1-314.

Rauen, A. and M. Lastovicková (1995): Investigation of electrical anisotropy in the deep borehole KTB, *Survey in Geophysics*, **16**, 37-46.

Rauen, A. and H.C. Soffel (1992): Electrical resistivity and anisotropy measurements in the KTB field laboratory, in *Abstracts of I International Geophysics Workshop on Rock Physical Properties, Firenze*.

Rauen, A., J. Duysre, A. Heikamp, A. Kontny, G. Nover and Th. Röckel (1994): Electrical conductivity of a KTB core from 7000 m – effects of cracks and ore minerals, *Sci. Drilling*, **4**, 197-206.

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