High Pressure High Temperature Cell for Electrical Conductivity Measurements of Earth-Type Materials

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Abstract—A high pressure, high temperature cell has been constructed and used to determine the temperature dependence of the electrical conductivity of various geological materials at a frequency of 60 Hz. Test conditions in the cell approximate an *in situ* environment by means of a variable uniaxial confining pressure and a separately variable pore pressure. A wide array of constituents of typical oil bearing formations have been examined at temperatures up to 240°C.

I. INTRODUCTION

Nearly half of the world's heavy petroleum resources are located in northern Alberta, Canada. Most of these heavy bitumens permeate sand layers deep beneath the ground surface forming what are commonly known as the oil sands. At typical formation temperatures, bitumens are very viscous, almost solid, and their separation from sand grains is difficult. The recovery of heavy oils from these subterranean reservoirs can be achieved only when the viscosity of hydrocarbons saturating the formation is reduced to a level such that they are rendered mobile under practically achievable pressure differentials, which can be accomplished by increasing formation temperatures. Several techniques to heat the underground bitumen reservoirs, such as injection of steam and hot water, or fire flooding, have been proposed and are currently under examination [1]. Studies have shown that the efficiencies of these techniques can be improved significantly by employing an electric current to preheat the oil bearing zone through ohmic or dielectric losses [2]–[5]. The general availability and the comparatively low cost of 60 Hz electrical energy make it the prime candidate for use during the first stages of bitumen extraction. Experiments have been performed on scaled physical models and computer simulations have been conducted to determine the best conditions for the electric preheat recovery phase [2]–[5]. The accuracy of both the physical and computer models depends on knowing the behavior of the electrical conductivities of the reservoir materials as a function of temperature. *In situ* techniques [6]–[11] are not well suited for making controlled measurements at elevated temperatures. To obtain conductivity values over the range of temperatures encountered during *in situ* electrical heating, measurements must be performed under laboratory conditions on representative samples.

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Development of techniques suitable for earth-type materials has been of ongoing interest for many years [12]–[16]. The authors have previously reported the development of several measurement cells for determining the electrical conductivity of oil sands at temperatures up to 100°C [12], [13]. As well, the authors have made measurements up to 240°C with a pressurized capacitor-like sample holder [15]. This apparatus relied on a statically applied confining pressure to maintain sample-electrode contact, which in many cases resulted in impedance irregularities which could not be resolved by the two terminal measuring techniques employed [12]. Moreover, the system was not suitable for making measurements on cored samples.

In this paper a four-terminal apparatus [7] is described which permits the determination of the electrical conductivity of reservoir materials at power frequencies, as a function of temperature and under approximately *in situ* conditions, and is especially suited for making measurements on the unconsolidated oil sand and soft shales that characterize much of the hydrocarbon deposits in Alberta in Western Canada. Measurement results for the major geological strata of typical heavy oil bearing formations are then given.

II. APPARATUS CHARACTERIZATION

A. General Concepts

The electrical conductivity can be determined by measuring a voltage drop, V_s , across a sample whose geometrical dimensions are known, and which carries a known current, I_s . This so-called two-electrode method, illustrated in Fig. 1(a), suffers from a systematic error due to the contact resistance at the electrode/sample interface. The sample voltage is therefore overestimated, resulting in an experimentally determined conductivity whose value is lower than the true value. To overcome this problem the modified apparatus of Fig. 1(b) may be used. The two voltage probes P_1 and P_2 of this four-electrode method draw negligible current. Thus, while the contact resistance at these two probes may be quite large, it is now immaterial and does not affect the accurate determination of the true inter-probe voltage, ΔV . The two-electrode conductivity, σ_2 , and the four-electrode conductivity, σ_4 , are computed from the following formulae:

$$\sigma_2 = \frac{L \cdot I_s}{A \cdot V_s} \quad (1)$$

Fig. 1. Experimental concept for measuring the electrical conductivity of cylindrical samples between two electrodes. (a) Two-electrode method. (b) Four-electrode method. (c) Conceptual model of measurement cell.

$$\sigma_4 = \frac{L' \cdot I_s}{A \cdot \Delta V_s} \quad (2)$$

where the meanings of L , L' , and A are indicated in Fig. 1.

B. Apparatus

A practical realization of the simple measurement system depicted in Fig. 1(b) for conditions of temperature and pressure approaching those encountered *in situ* requires a rather elaborate measurement cell. Firstly, provisions must be incorporated to recompact the samples to near *in situ* densities, which can be accomplished by applying a uniaxial pressure, P_c , to the ends of the sample as suggested in Fig. 1(c). In the apparatus to be described below, this pressure is provided by allowing one of the end electrodes to move as a piston under the influence of an externally applied gas pressure. With this approach the authors have been able to apply sufficient pressure to recompact the samples to the point where resistivity essentially becomes independent of pressure. An additional advantage of this approach is that as the sample expands under heating, the piston can move to accommodate the increased sample size without damage to the cell.

Secondly, in order to prevent the evaporation of moisture in the samples at elevated temperatures, means must be provided to pressurize the pore space (P_p in Fig. 1(c)). In the case of soft shale samples the confining pressure, P_c , may suffice to prevent evaporation, provided P_c is greater than the saturated steam pressure at the highest temperature at which measurements will be conducted. However, in the case

of rigid samples or unconsolidated oil sand samples, where grain to grain contact has been established by the confining pressure P_c , the pressure in the pore space must be maintained independently above the appropriate saturated steam pressure. In all measurements a net confining pressure ($P_c - P_p$) was maintained in order to ensure positive contact between the sample and the end electrodes. The cell must provide probes for measuring the potential difference ΔV and for monitoring the sample temperatures. The authors have found that the only practical way to insert potential probes was through one of the end electrodes, as suggested in Fig. 1(c). To minimize the number of high temperature-pressure feedthroughs into the pressurized cell, these voltage probes were designed to double as thermocouples, thereby providing cell temperatures at points a and b. With this basic design approach the authors feel that the complex changes that occur during heating in the porosity, density, and saturation levels within the cell will approximate those changes that occur naturally in a heated formation when P_c and P_p are chosen initially to represent the *in situ* overburden and pore pressures prior to heating. Thus, while the cell was not specifically designed to determine the precise nature of these complex changes, the measured electrical conductivity will substantially represent the conductivity of the sample material under conditions of *in situ* heating. The final implementation of the cell shown in Fig. 1(c) is illustrated in cross-section in Fig. 2, and will be referred to as the HPHT cell (High Pressure High Temperature cell).

All steel components are made of type 316 stainless steel, and all O-rings are made of VitonTM. The sample (#8) is radially confined by a TeflonTM liner (#7) and the stainless steel walls of the cell (#9). One end of the sample butts against a copper electrode (#22) which is hard-soldered to the fixed stainless steel plug (#6). This end of the sample is electrically grounded. The opposite end of the sample is energized through another copper electrode (#22) that is hard-soldered to the movable stainless steel plug (#10). These two electrodes (#22) are the current carrying electrodes of the four electrode system shown in Fig. 1(c). The movable plug (#10) is mechanically coupled with the movable stainless steel piston (#12) by means of a brass connecting rod (#20). The rod is electrically isolated from the piston by two VespelTM spacers (#18), one of which is compressed against two O-ring face seals, making the piston able to pneumatically insulate the pore pressure, P_p , from the confining pressure, P_c . The piston unit can move along the axis of the cell as it seals on the TeflonTM liner (#7) and adjacent stainless steel liner (#11). The electrode-to-TeflonTM seals, one on the fixed plug and two on the piston unit, prevent water seepage past the electrodes, precluding the possibility of a shunt between the energized electrode and ground.

Two thermocouple probes (#23) penetrate the sample to different depths from the fixed-plug end. Each probe consists of an inner core of iron/constantan thermocouple wire housed in a 1.6 mm (1/16-in) diameter brass tube which, in turn, is isolated from the sample by a thin TeflonTM sheath. The left end of each tube has a brass mounting flange soldered to it while, at the right end, a thermocouple junction is hard-soldered to the tip, sealing the tube at that point. Both probes are rigidly installed in the thermocouple flange (#4) which,

Fig. 2. Longitudinal section of HPHT cell. 1-thermocouple wires; 2-large brass nut; 3-stainless steel pore pressure flange; 4-thermocouple flange with probes; 5-bronze retaining screw; 6-fixed stainless steel plug; 7-TeflonTM liner; 8-sample under test; 9-stainless steel main cell; 10-movable stainless steel plug; 11-stainless steel liner; 12-movable stainless steel piston; 13-overburden pressure port; 14-stainless steel spark plug flange; 15-bronze retaining screw; 16-spark plug; 17-beryllium copper sliding contact; 18-VespelTM spacers; 19-fastener for brass connecting rod; 20-brass connecting rod; 21-water absorbing material (fiberglass); 22-copper electrodes; 23-thermocouple probes; 24-VitonTM O-rings.

in turn, is attached to the pore pressure flange (#3). The thermocouple flange (#4) consists of several stainless steel, brass and TeflonTM components, which are not shown in Fig. 2. In addition to sensing sample temperature, these probes act as the voltage probes in the four-electrode method. As such, both probes are electrically insulated from all metallic parts and from each other and are exposed to the sample only over a distance of about 1 mm at the tips.

The thermocouple wires (#1) from the two probes are carried outside the cell through 4.8 mm (3/16-in) diameter stainless steel tubing (not shown in Fig. 2) and terminate at pressure-sealed thermocouple connectors. The thermocouple/voltage probes, the pore pressure flange, and the tubes that house the thermocouple wire form a single unit.

Pressure P_c is applied to the sample by the mechanical force of the piston unit which is activated by high pressure nitrogen gas which enters through a pressure port (#13). This pressure is maintained at a constant level over the entire range of operating temperatures by the action of a pressure regulator and gas leak. Pore pressure, P_p , is applied by surrounding the TeflonTM liner (#7) and sample (#8) with high pressure nitrogen gas which is introduced through another port (not shown in Fig. 2) at the fixed-plug-end of the apparatus. Pore pressure gas enters the sample directly through the thermocouple/voltage probe holes. The nitrogen gas also migrates along milled passages on the outside of the TeflonTM liner into the space behind the movable stainless steel plug to establish a zero pressure differential across it. Unlike the confining pressure circuit, the pore pressure line is fixed in volume so that pore pressure will fluctuate with temperature. Both confining and pore pressure systems are plumbed with stainless steel tubing from a single source of gas.

During an experiment the HPHT cell is placed in a conventional oven where sample temperature can be controlled. The two gas lines are fed through one of the oven walls, as is the electrical connection to the sample. Electric current reaches the sample from outside the cell via a spark plug (#16) which acts as a high pressure, high temperature electrical feedthrough device (and also provides the confining-pressure gas leak). The spark plug electrode engages a sliding contact (#17) on the

piston unit which, in turn, is in contact with one end of the sample. The electric circuit is completed by returning current from the sample to the steel body of the cell.

III. MEASUREMENTS

A. Test Conditions

The cell with an installed specimen was pressurized by confining and pore pressures of 5600 kPa and 2000 kPa, respectively, at least 12 hours before a test to allow the sample structure to accommodate the new stresses. During an experiment, the confining pressure remained at its initial level. The pore pressure was permitted to change with increasing temperature, thus approximating the natural increases in *in situ* pressure that would inevitably occur in a heated formation. The sample temperature was increased from room temperature at 20°C in 12 steps up to 240°C. Conductivity measurements were performed at each of these steps, once the temperature distribution in the sample was approximately uniform.

Electric signals from the cell were monitored by an HP 3825 Automatic Data Acquisition System. An HP 82300B HP BASIC Language Processor controlled the entire experiment by continuously sensing sample temperatures, performing current and voltage measurements at appropriate temperature conditions, computing conductivities and checking for possible loss of integrity of the HPHT cell, printing and displaying intermediate results, and changing oven temperatures. At the end of an experiment, a curve-fitting routine was applied to the collected sample conductivity data to find a third degree polynomial to approximate the measured points. Both the polynomial and the data points were then plotted.

Only four-electrode results were used to prepare the final data shown in this paper. However, since the four-electrode voltage was measured across only a section of the sample, the natural inhomogeneity of tested materials could produce erroneous results, particularly when the voltage electrodes were placed in a region of locally higher conductivity. In order to detect this condition, four-electrode conductivity measurements were always compared to two-electrode conductivity measurements. An exceptionally high discrepancy

TABLE I
SUMMARY OF SAMPLE CHARACTERISTICS

Material	Density [g·cm ⁻³]	Water Content [weight %]	Oil Content [weight %]	Sample Description
Amoco Shale	2.03 to 2.24	10.3 to 14.0	—	soft, non homogeneous, greyish, stratified structure
Amoco Shale & Oil Sand	2.04 to 2.17	8.5 to 10.6	2.3 to 3.6	soft, non homogeneous mixture of oil sand and shale
Syncrude Shale	2.09 to 2.31	9.2 to 17.5	—	relatively soft and uniform cores, dark grey-blackish in color
UTF Shale	2.08 to 2.22	13.8 to 18.1	—	grey, non uniform structures of hard material interspersed with softer clay-like lenses
Syncrude Limestone	2.57 to 2.63	1.1 to 2.3	—	very hard, whitish, uniform cores
Mined Lean Oil Sand	2.09 to 2.21	7.2 to 8.8	6.6 to 8.0	uniform blackish mixture of sand and oil
Mined Rich Oil Sand	1.93 to 2.03	1.5 to 2.6	11.7 to 13.6	uniform blackish mixture of sand and oil
UTF Oil Sand	1.89 to 2.01	1.9 to 3.8	12.1 to 14.4	frozen cores with uniform sand grain matrix
Silica Sand & Brine	1.97 to 2.11	11.1 to 14.1	—	cle in silica sand saturated with 5000 mg/L NaCl brine

between the two values of conductivities thus obtained could be an indication that the sample was most likely highly inhomogeneous and that the test results should be discounted.

B. Sample Preparation

All geological samples described in this paper originate from the Athabasca Oil Sands deposit, located near Fort McMurray in northeastern Alberta, Canada. They were supplied by Syncrude Canada Ltd., Amoco/AOSTRA¹/GLISP² and the AOSTRA UTF³ project. After extraction from the borehole, all cored samples were immediately sealed in PVC pipes. Unconsolidated oil sands provided by the Alberta Research Council's sample bank were obtained from mined oil sand in sealed pails. Both the cored and the unconsolidated samples were stored in temperature and humidity-controlled conditions at about 8°C until conductivity tests could be performed. The UTF oil sand samples were frozen at temperatures of about -20°C after their extraction and were stored in this condition prior to conductivity tests. Some typical geophysical properties of the samples are listed in Table I.

Before installation in the HPHT cell, all samples were shaped into cylinders 38 mm in diameter and between 92 and 108 mm long. The Syncrude shales, as a relatively soft material, were formed and sized to the required dimensions in a simple trimming device and then inserted into the TeflonTM liner. The unconsolidated oil sands, as well as the Amoco oil sands and shales, were directly compacted in the TeflonTM liner. The frozen UTF oil sands were machined to the required dimensions and, still frozen, installed in the TeflonTM liner. This procedure was used to minimize damage to the internal structure of the sample. The UTF shales and the Syncrude limestones were very hard and fragile and required careful machining or coring before their installation in the sample holder. Two 4.8-mm (3/16-in) holes were subsequently drilled into the ground-end of the various samples to accommodate the thermocouple/voltage probes. The TeflonTM liner with the sample was then installed in the HPHT cell which was sealed and pressurized.

¹ Alberta Oil Sands Technology and Research Authority

² Gregoire Lake In Situ Steam Pilot

³ Underground Test Facility

TABLE II
SUMMARY OF MEASURED ELECTRICAL
PARAMETERS OF TESTED GEOLOGICAL SAMPLES

material	number of samples that contributed data	σ_{24} [S·m ⁻¹]	b [K ⁻¹]	c [K ⁻²]	d [K ⁻³]
Amoco Shale	4	260·10 ⁻³	2.49·10 ⁻²	1.07·10 ⁻⁴	-6.10·10 ⁻⁷
Amoco Shale & Oil Sand	3	223·10 ⁻³	2.30·10 ⁻²	6.43·10 ⁻⁵	-4.60·10 ⁻⁷
Syncrude Shale	6	N/A	2.99·10 ⁻²	6.94·10 ⁻⁵	-6.01·10 ⁻⁷
UTF Shale	4	277·10 ⁻³	3.65·10 ⁻²	-1.16·10 ⁻⁶	-3.43·10 ⁻⁷
Syncrude Limestone	4	37·10 ⁻³	2.21·10 ⁻²	1.11·10 ⁻⁴	-4.42·10 ⁻⁷
Mined Lean Oil Sand	11	73·10 ⁻³	2.77·10 ⁻²	6.85·10 ⁻⁶	-2.72·10 ⁻⁷
Mined Rich Oil Sand	8	1.7·10 ⁻³	2.75·10 ⁻²	1.45·10 ⁻⁴	-8.84·10 ⁻⁷
UTF Oil Sand	5	5.4·10 ⁻³	1.35·10 ⁻²	2.32·10 ⁻⁴	-1.13·10 ⁻⁶
Silica Sand and Brine*	5	179·10 ⁻³	2.50·10 ⁻²	1.13·10 ⁻⁵	-1.68·10 ⁻⁷

* Sample prepared in laboratory: clean silica sand saturated with 5000 mg/L NaCl brine.

Fig. 3. Normalized conductivity vs. temperature functions obtained for tested geological materials. 1a-Syncrude limestone; 1b-7000 mg/L NaCl brine; 2-UTF Shale; 3-Amoco Shale; 4-Mined Rich Oil Sand; 5-Syncrude Shale; 6-Mined Lean Oil Sand; 7-Amoco Shale and Oil Sand; 8-UTF Oil Sand.

C. Results

During each of the HPHT tests, 12 conductivity measurements were taken at temperatures from about 20°C (room temperature) to 240°C. The 12 data points were approximated by a third degree polynomial, using the least squares technique:

$$\frac{\sigma(t)}{\sigma_{24}} = 1 + b \cdot (t - 24) + c \cdot (t - 24)^2 + d \cdot (t - 24)^3 \quad (3)$$

where σ_{24} is the sample conductivity at 24°C, b , c , and d are the normalized polynomial coefficients, and $\sigma(t)$ is the conductivity at temperature t . Note, that the above equation represents the electrical conductivity normalized with respect to σ_{24} . Table II summarizes electrical parameters that have been obtained for different materials during the current measurement program and Fig. 3 displays the corresponding conductivity functions. Each curve displayed is based on an average of multiple sample measurements.

D. Discussion of Results

Fig. 3 indicates that the majority of the conductivity functions can be characterized by a substantially linear increase with temperature at lower temperatures and by a decline at higher temperatures. The data suggests that the following two factors have the dominant influence on the the shape of the

Fig. 4. The measured conductivity of a 100% brine (0.086 mol) saturated clean sand sample and the computed conductivity of a 0.086 mol brine solution.

polynomials:

- ion concentrations in the connate water of the samples,
- content and type of clays present in the samples.

For example, consider the change in the electrical conductivity with temperature of the connate water itself, which contains mainly dissolved sodium chloride at concentrations ranging from 1000 - 10 000 mg/L for typical formation materials [17]. The behavior of the electrical conductivity of such geothermal brines has been studied extensively [18], [19]. Initially, as the temperature is increased the viscosity of the brine decreases, resulting in an increase in the mobility of the ions in solution and a nearly linear increase in the conductivity. As the temperature continues to increase the density in the pore water is reduced, resulting in a lowering of the dielectric constant. In turn, this leads to an increased association between ion pairs and a tendency to decrease the conductivity of the brine, thereby at least partially offsetting the increase in conductivity related to the decrease in brine viscosity.

The increase in the electrical conductivity with temperature of actual geological samples must in part be due to the effects described above. Consider a hypothetical sample consisting of clean silica sand compacted to achieve grain to grain contact and 100% saturated with a brine solution. In this case (provided the confining pressure is enough to ensure that porosity changes with temperature are negligible) Archie's law [20] suggests that the variation in electrical conductivity with temperature for such a sample will be identical to that of the brine itself.

Such a sample was prepared, using clean silica sand 100% saturated with a 5000 mg/L (0.086 mol) NaCl aqueous solution. Measurements of the electrical conductivity were made over the range 24°C to 240°C, and the results are shown in Fig. 4. Also shown in Fig. 4 is the temperature response of the brine solution itself as calculated from the data provided by Ucock *et al.* [19]. Although the two responses are drawn to different vertical scales, their congruence indicates that the increase in conductivity of the brine-sand sample depends solely on the ion concentration in the brine. Further, this agreement verifies that the measurement cell is capable of accurate measurements of the temperature response of geological samples over the temperature and pressure range of interest.

Of all the reservoir materials that have been tested in the HPHT cell, only the Syncrude limestone (curve #1a in Fig. 3) exhibited a conductivity whose variation with temperature could be explained by only considering ion concentrations. Indeed, using the data of Ucock *et al.* [19], it has been shown that the calculated conductivity of 7000 mg/L (0.12 mol) aqueous solution of sodium chloride (curve #1b) almost exactly matches the limestone characteristic. For the limestone samples, then, it is inferred that the brine ionic conduction is the predominant factor determining their electrical conductivity. Indeed, examination of Fig. 3 for the response of the electrical conductivity below about 180°C indicates that the dominant conduction mechanism in all samples is governed by the ions in solution.

Since the variation of the conductivity with temperature of the remaining samples tested in the HPHT cell did not follow the brine-sand response, it may be concluded that their conductivities cannot be explained solely in terms of the ionic conductivity of the pore water. A common feature of these materials is the presence of clays in their structures. Clay particles have an excess of positive ions (so called counterions) adhered to their surfaces. In an aqueous environment, the counterions acquire some mobility and increase the overall conductivity of a mineral. In general, this conductivity increases faster with temperature than that of clay-free samples [21], [22]. At high enough temperatures, complex chemical reactions may transform the molecular structure of clays and the electrical conductivity of clay-electrolyte compounds may be reduced. These temperatures can be as low as or below 200°C [23].

Detailed compositional analysis on the samples was not undertaken and it was therefore not possible to quantitatively discuss the relative influence of the clays on the increase in electrical conductivity with temperature. However, such a quantification was not the objective of the measurement program. Rather, the intent was to provide a functional relationship between conductivity and temperature that would be useful in the computer simulation of electrothermal processes.

IV. CONCLUSIONS

A high pressure, high temperature cell used for measurements of the 60-Hz electrical conductivity of earth-type materials as a function of temperature and under approximately *in situ* conditions has been described. The performance of the apparatus was verified by testing many samples consisting of pure silica sand fully saturated with brine. Additional calibrating tests and their evaluation, as well as the metrological analysis of the measuring system showed that the results obtained with the HPHT cell were repeatable and accurate to within $\pm 10\%$.

The variation of electrical conductivity with temperature for the main geological components of an oil bearing formation have been extensively studied. The electrical conductivity of all tested materials increased linearly with temperature, up to about 150–200°C. The ionic conductance of interstitial brines and the counterion conductance of clays are believed to be the main factors contributing to the conductivity growth in this region. Beyond the linear part, the conductivity rise

was slower, and all but one sample (limestone) exhibited a conductivity drop at temperatures above 200°C. Insufficient data has been collected at these elevated temperatures to permit the development of a conductivity model at these elevated temperatures and future measurement programs should address this issue.

The family of normalized conductivity characteristics shown in Fig. 3 and described in Table II can be used in predicting the *in situ* temperature behavior of electrically heated oil sands and the adjacent strata. These functions are being applied by the authors in numerical simulators to model heating patterns for different electromagnetic oil recovery systems [24].

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