Advanced techniques for determining thermal properties on rock samples and cuttings and indirect estimating for atmospheric and formation conditions

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Abstract. A set of advanced methods and instruments was developed to improve essentially quality of experimental data on reservoir thermal properties (thermal conductivity, thermal diffusivity, volumetric heat capacity, coefficient of linear thermal expansion) at atmospheric and reservoir thermobaric conditions. The new thermal core logging technique provides continuous high-resolution profiling rock thermal properties along wells accounting for rock anisotropy and multi-scale heterogeneity. Integration of the technique application results with standard well logging data leads to possibilities of high resolution profiling porosity, rock matrix thermal properties, elastic wave velocities and modulus, rock density, etc. New approaches are described that allow indirect determination of the reservoir thermal properties from standard petrophysical logging data accounting for formation anisotropy. A new laser optical scanning instrument, enhanced theoretical modeling of effective thermal properties and special workflow opened a way to determination of combination for rock thermal properties on rock cuttings at formation temperatures. The new experimental basis improves reliability of data on physical properties of reservoirs, results of specific heat flow determination and reservoir thermal regime modeling within prospecting, exploration and development of geothermal energy fields.

1. Introduction
Serious typical current problems in investigations of rock thermal properties (thermal conductivity, thermal diffusivity, volumetric heat capacity, coefficient of linear thermal expansion) is caused by the fact that most of measuring methods and instruments were designed to study the thermal properties of industrial materials. It is a reason that the traditional experimental basis does not allow to account for essential peculiarities of rocks which include [1]:

- significant thermal anisotropy;
- multi-scale heterogeneity;
- impossibility to polish rock sample surface;
- partial or full destruction of studied rock samples under pressure that should be applied to a measuring probe to reduce contact thermal resistance influence;
- necessity to study number of rock samples within a formation under study to take formation heterogeneity.
All these problems are especially essential for reservoir rocks that are highly porous and often fractured. Neglect of the problems mentioned above causes significant errors in the measurement results that are unacceptable for estimation of reservoir thermal regime, determination of specific heat flow, prospecting, exploration and development of geothermal energy fields [1] as well as for solution of similar problems in oil and gas science and industry.

These circumstances explain a necessity in development of special methods and instruments that provide improvement in quality of experimental data on rock thermal properties at atmospheric and reservoir thermobaric conditions. Methods and instruments, developed by the authors to improve quality of experimental data on reservoir thermal properties, are described in a present paper.

2. Measuring instruments

2.1. Laser versions of optical scanning instrument

Two laser versions of optical instrument (A and B) have been developed (figure 1).

The laser instrument A (figure 1a) was designed [2] to provide profiling thermal conductivity, thermal diffusivity and volumetric heat capacity with spatial resolution on full-size core samples in combination with continuous core sample scratching [3] and gamma-spectrometry on core samples.

The laser instrument B (figure 1b) has been developed for the thermal property measurements on standard core plugs (cylinders 1x1”) and small rock samples (including core cuttings) with a sample length from 10 mm and larger. The laser instrument B allows also to provide the thermal property measurements on pieces of broken core samples, split core samples, full-size core samples, and is being applied for the measurements of effective thermal conductivity and thermal diffusivity on special synthetic samples when a new method for the thermal property measurements on rock cuttings and non-consolidated rocks [4] is used (see below). The laser instrument B can be easily transported to different petrophysical laboratories and core storages to provide numerous measurements. Technical characteristics of the laser instrument B are shown in table 1.

Both laser instruments are based on the optical scanning method [1] and provide accounting for rock thermal anisotropy and the measurements of rock thermal conductivity tensor components on every rock sample under studying.
Table 1. Parameters of the developed laser optical scanning instrument B.

| Parameter                                             | Value                                      |
|-------------------------------------------------------|--------------------------------------------|
| Weight, dimensions                                    | 20 kg, 25×25×90 cm                        |
| Thermal conductivity range                            | 0.2…40 W/(m·K)                            |
| Thermal diffusivity range                             | (0.1…5)×10⁻⁶ m²/s                        |
| Uncertainty in thermal conductivity results (with equal values of accuracy and precision, conf. prob. 0.95) | ±3%                                       |
| Uncertainty in thermal diffusivity measured (with equal values of accuracy and precision, conf. prob. 0.95) | ±4%                                       |
| Spatial resolution of thermal conductivity profiling  | ≥ 0.2 mm*                                 |
| Scanning velocity                                     | Variable, 0.5-10 mm/s                     |
| Effective thickness of rock layer under investigations | 5 – 30 mm*                                |
| Length of rock samples under investigations            | 10 – 400 mm*                              |
| Optical heating source                                 | Laser, wavelength 0.96 μm                 |

*Variable according to variations in measuring regime parameters

2.2. Integration of developed quartz dilatometer and laser optical scanning instrument for measuring coefficient of linear thermal expansion of anisotropic rock samples

The developed quartz dilatometer [2] is applied in combination with the laser optical scanning instrument for the measurements of the coefficient of linear thermal expansion (CLTE) on standard core plugs (1x1”) accounting for rock anisotropy. The overall view of the quartz dilatometer is presented in figure 2.

Figure 2. The quartz dilatometer for the measurements of the coefficient of linear thermal expansion on rock samples: 1 - specimen holding system of vitreous silica; 2 – furnace; 3 – lifting system; 4 – temperature controller and thermal displacement indicator, 5 – computer.
The laser optical scanning instrument is applied to determine directions of principal axes of thermal anisotropy [1] and to study rock sample heterogeneity. After that the CLTE is measured for different orientation of core plugs in the dilatometer according information on principal axes of rock anisotropy found from the optical scanning measurements. Such technique allows us to determine the principal CLTE tensor components and exclude distorting influence of rock heterogeneity. Certified reference standards (fused quartz, silicon single crystal, cuprum, aluminium) with the CLTE values within the representative range of (0.5-24.6)-10^{-6} K^{-1}, that covers in general a range of rocks and rock-forming mineral CLTE values ((2.5-18)-10^{-6} K^{-1}) are used for metrological testing the CLTE instrument developed. A small accuracy plus precision value (±0.1·10^{-6} K^{-1} for temperature step of 20 K) and possibility to determine the CLTE value for every 20 K temperature interval allowed us to establish the CLTE vs temperature dependences within a temperature interval of 20 to 100 K, which was chosen to prevent rock sample destruction, for samples of different rock types.

3. Methods

3.1. Continuous thermal core logging
A novel method is based on continuous high-resolution non-destructive profiling the rock thermal properties on all full-size core samples along wells using the optical scanning instruments [5]. The integrated approach to investigations of the formation is applied. A continuous high-resolution (0.2-1 mm) profiling of rock thermal properties (thermal conductivity, volumetric heat capacity, thermal anisotropy coefficient) on all cores recovered from wells is combined with (1) the thermal property measurements on standard core plugs in three core plug states successively (as-received state, dry, and brine-saturated rock samples), and (2) processing the thermal property logs jointly with the standard petrophysical logging data. The results of high-resolution thermal profiling allows detailing the porosity variations up to scale of 1 mm. Variations of rock mineralogical composition can be defined from inverse problem solution.

Based on the results of continuous thermal core logging with the help of established correlations, it is possible to estimate elastic wave velocities, its anisotropy (γ and ε Tomsen’s parameters) and dynamic elastic moduli [6], density and porosity [7], calculate brittleness index and obtain information about detailed variations of strength properties along the wells [8].

The detailed thermal core logging data provide a well-founded selection of full sized core samples for preparation of core plugs that are studied (1) with a special optical scanning technique at dry state and after their saturation with brine and oil, and (2) with special instruments at elevated temperature and pressure. The additional measurements of the rock thermal properties are performed on small pieces of broken cores with the optical scanning technique if necessary. Variations in high-resolution thermal properties characterize typically rock heterogeneity well, while an understanding of rock heterogeneity is fundamental for adequate core sampling and scaling-up data from laboratory measurements to core- and reservoir-scales. Implementation of express (directly in core storage) continuous thermophysical profiling of full-sized or slabbed cores allows to register detailed variations of basic rocks physical properties and can be used for optimization of the representative core sample collection creation [9].

3.2. Logging of rock matrix thermal properties
The approach was developed to infer the thermal properties (thermal conductivity, thermal diffusivity, and volumetric heat capacity) of rock matrix from integration of thermal core logging data and standard petrophysical well logging data. The approach is based on close correlation between rock thermal properties and porosity (figure 3a) and allows to record continuous variations in rock matrix thermal properties along wells (figure 3b). Information on the rock matrix thermal properties is needed as input data for theoretical modeling geothermal energy reservoirs. Reliable determining the rock matrix thermal properties could be provided earlier only from investigation of correlations between
rock thermal conductivity and rock porosity as well as from correlations between rock volumetric heat capacity and rock porosity [10].

Figure 3. Example for carbonates: correlation of variations in reservoir rock thermal conductivity and porosity (a) and log of rock matrix thermal conductivity (b) inferred from integration of thermal core logging data and well logging data.

3.3. Rock thermal property measurements on rock cuttings and non-consolidated rocks

3.3.1. The technique of synthetic solid samples preparation was developed. Totally more than 200 synthetic samples (SS), consisting of core cuttings or non-consolidated rocks or industrial/natural materials mixed with material-fillers (wax, water, air), were produced and studied during the research (figure 4). Materials with well-known thermal properties (blocks of several types of rocks with studied thermal conductivity and samples of technical glasses with officially certified thermal conductivity) were used for synthetic samples fabrication. Effect of the following factors on technology stability and properties of a synthetic sample was studied experimentally: (1) thermal properties of material-filler, (2) dimensions of particles provided by vibrations frequency of mill machine and duration of treatment during preparation of rock cuttings, (3) pressure in the press machine during SS fabricating, (4) time of pressing during SS fabricating, (5) temperature of mixture of rock cuttings and wax to provide the best thermal contacts between components and remove air from SS, (6) volume fraction of wax in SS.

A following workflow for synthetic sample fabricating was developed: (1) choosing of material-filler with known thermal properties, (2) preparation of rock cuttings and material cuttings with satisfactory dimensions of particles using a ball mill, (3) mixing particles of rock cuttings and material-filler and (4) pressing the mixture using press machine with the fabricating parameters established.

Using of a particular material-filler has both its advantages and disadvantages, which must be taken into account when fabricating a synthetic sample. Three approaches to synthetic sample fabrication were proposed: (1) pressing a heated mixture of rock cuttings with a wax, (2) pressing a mixture of rock cuttings with water, and (3) pressing rock cuttings without any material-fillers (that is with air). The proportion of air in the sample has a significant effect on the effective thermal conductivity of SS. The smaller the volume fraction of air, the higher the effective thermal conductivity of SS. And the greater the volume fraction of rock cuttings in SS, the closer the effective thermal conductivity to thermal conductivity of rock.
Figure 4. Workflow and features of synthetic sample fabrication.

Pressing a mixture of wax with particles of various materials in most cases allows creating stable SS, but the volume fraction of rock cuttings in such sample is about 60% while air volume is about 5-10%. The fabrication of SS with water as a filler have some limitation because of samples stability. But volume fraction of the rock cuttings in such sample is about 70-85% while air volume is about 1-8%.

Preparing SS in different ways, performing high-precision measurements and solution of the inverse problem allowed to determine optimal parameters of the synthetic sample fabricating. More details are described in [4] and [11].

3.3.2. Metrological testing of the technology on the samples with known properties was successfully performed. Different natural and industrial materials - technical optical glasses (TF-1, K-8, LK-5, KV), two types of marble, granite and sedimentary rocks - have been used for synthetic samples fabrication. Thermal conductivity of used materials is within 0.7-4.5 W/(m·K) that covers whole possible range.

Theoretical model to estimate thermal conductivity of rock cuttings was developed. Different theoretical approaches have been considered during the research to solve this inverse homogenization problem. Among considered theoretical formulas (Lichteneker, Lichteneker-Rother, Wiener, etc.) only modified Lichtenecker’s formula [12] allows to determine rock matter thermal conductivity with an average error of less than 10%.

$$\lambda_{SS} = \left( \sum_i S_i \lambda_i^{-1} \right)^{-1} \left( \sum_i S_i \lambda_i \right)^{\beta}$$

Here \( \lambda_i \) is the thermal conductivity of \( i \)-th constituent element, \( S_i \) is the volumetric fraction of \( i \)-th component, \( \beta \) is the structural-sensitive parameter determined by established empirical formula. The
The desired value of thermal conductivity of rock particles (grains) is found by minimization of misfit between the calculated and measured values of SS thermal conductivity [11]. This approach works well for both filling materials – wax and water.

3.4. Adaptation of DTC-300 instrument for measuring rock thermal conductivity at elevated temperatures

Necessary adaptation of the DTC-300 instrument, which was designed mostly for investigations of industrial materials, to peculiarities of rocks was carried out. The adaptation includes:

- provision of the possibility of thermal conductivity measurements on samples with different diameters with corresponding corrections as DTC-300 instrument in its standard application is applicable only for cylindrical sample diameter of 50 mm;
- correction of thermal conductivity values for non-parallel sided core samples as required parallelism cannot be reached technically for fractured and highly porous samples of reservoir rocks;
- improvement of metrological quality of thermal conductivity measurements using integration of DTC-300 instrument with laser optical canning instrument B (see figure 1b).

3.5. Technique for measurements of rock thermal properties on rock cuttings at elevated temperatures

Thermal conductivity measurements on rock cuttings is the only possibility to estimate thermal properties of rocks at high temperatures when consolidated core samples cannot be recovered. The divided-bar technique [1], which is the most popular method for measurements of thermal conductivity on core samples at high temperature, cannot be applied with its traditional technique.

A special measuring technique, based on divided bar instrument DTC-300 application, was developed for estimation of thermal conductivity on rock cuttings at elevated temperatures (figure 5).

![Figure 5. The technique of estimation of thermal conductivity (TC) of rock cuttings at different temperatures using jointly TCS and DTC-300 instruments (see details in the text).](image)

The developed technique of the rock thermal conductivity measurements at elevated temperatures on rock cuttings includes following principal stages:

- measurement of thermal conductivity of solid filling material (cylindrical sample by 40 mm in diameter prepared in press machine) (1) at elevated temperatures (30-300 °C) with DTC-300 instrument;
determination of thermal conductivity versus temperature characteristics for solid filling material including correction for thermal conductivity obtained with DTC-300 at room temperature using thermal conductivity of filling material \( (\lambda_{01}) \) obtained with thermal conductivity scanner (TCS) as non-contact optical scanning method provides more reliable data than contact method (DTC-300);

- measurement of thermal conductivity of solid synthetic sample (cylindrical sample by 40 mm in diameter prepared with press machine by mixing of filling material and studied rock cuttings) (2) at different temperatures with DTC-300 instrument;

- determination of thermal conductivity versus temperature characteristics for solid synthetic sample including correction for thermal conductivity obtained with DTC-300 at room temperature using thermal conductivity of solid synthetic sample \( (\lambda_{02}) \) obtained with TCS as non-contact optical scanning method, that provides more reliable data than contact method (DTC-300);

- determination of thermal conductivity of rock cuttings \( (\lambda) \) at different temperatures using a function describing thermal conductivity of a synthetic sample.

3.6. Determining formation thermal conductivity from standard petrophysical well logging data

Absence of instruments for in-situ thermal property measurements and lack of wells with coring are reasons for many long-term attempts to develop indirect methods for thermal properties determination from standard petrophysical well-logging data. Two principal ways were suggested to determine thermal conductivity of rocks within non-coring intervals using the well-logging data: (1) correlation analysis based approach [13], [14], and (2) theoretical modeling of effective thermal conductivity [15], [16]. The correlation analysis based approach implies the determination of thermal conductivity from regression equations established preliminary between rock thermal conductivity measured on rock samples within depth interval with coring and well logging data. Theoretical modeling of effective thermal conductivity requires a reliable corresponding theoretical model, a volumetric lithological model inferred from the well logging data, and reliable data on thermal conductivity of volumetric lithological model components to determine thermal conductivity of rocks within non-coring intervals.

A poor database of thermal conductivity of rock-forming minerals and a limited amount of core samples involved in correlation analysis usually were reasons for restricted application of both approaches mentioned. Furthermore, rock anisotropy, which is essential often, has never been accounted for earlier.

Our enhancement of both approaches allowed to exclude the disadvantages mentioned above due to following improvements:

- application on the continuous high-resolution thermal core logging to measure rock thermal properties on all core samples within depth intervals where drilling was performed with core recovery;
- establishing regression equations between thermal conductivity tensor components (parallel and perpendicular to bedding plane) and well logging data;
- establishing regression equations between thermal anisotropy coefficient and Thomsen’s anisotropy parameters determined from acoustic well-logging;
- development of representative database on the thermal conductivity of rock forming minerals from numerous measurements on single crystals of minerals from mineralogical museums due to application of the non-destructive measuring optical scanning technique [1];
- development and application of advanced theoretical model of effective thermal conductivity.

Testing and application of the enhanced approaches demonstrated that discrepancies between predicted and measured thermal conductivity in the target wells does not exceed 10%. An example of application of an enhanced approach based on theoretical modeling rock thermal conductivity is shown in figure 6. The volumetric lithological model was derived from well-logging data. In a reference well (figure 6, left panel) thermal conductivities of volumetric lithological model components were determined during minimization of a misfit between calculated and measured
thermal conductivities of rocks. To process results of core measurements and logging data jointly, the results of measurements were preliminary averaged with 0.5 m moving window. Knowing thermal conductivities of each model components and having a volumetric lithological model in a target well (figure 6, right panel) from logging data interpretation allowed predicting vertical variations of thermal conductivity in a target well.

Figure 6. Volumetric lithological models, results of thermal conductivity measurements upscaled to the well-logging data (red lines) and results of thermal conductivity calculation from the well-logging data (black line) within the organic reach shale interval.

4. Summary
A set of advanced methods and instruments was developed to improve essentially quality of experimental data on reservoir thermal properties (thermal conductivity, thermal diffusivity, volumetric heat capacity, coefficient of linear thermal expansion) at atmospheric and reservoir thermobaric conditions. The new thermal core logging technique provides continuous high-resolution profiling rock thermal properties along wells accounting for rock anisotropy and multi-scale heterogeneity. Integration of the technique application results with standard well logging data leads to possibilities of high resolution profiling porosity, rock matrix thermal properties, elastic wave velocities and modulus, rock density, etc. New approaches are described that allow indirect determination of the reservoir thermal properties from standard petrophysical logging data accounting for formation anisotropy. A new laser optical scanning instrument, enhanced theoretical modeling of effective thermal properties and special workflow opened a way to determination of combination for
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