AUTOMATED MINERAL IDENTIFICATION AND ROCK CHARACTERIZATION PROCESS

This invention relates to an automated mineral identification and rock characterization process.

Generally mineralogical information is valuable for many fields of petroleum engineering, for example for controlling reservoir quality, rock/fluid interaction during production operations etc.

Such information on the mineralogical composition of a sample, for instance a sandstone sample, is normally determined by examination of a thin section using an optical microscope. Initially the geologist will only give approximate abundances of the minerals present. If more precise values are required, the sample is usually point-counted. This is a labour intensive technique and involves identification of the minerals present at about two or three hundred points per thin section. In this technique each point is analysed on the basis of its colour and texture. However, such a procedure still gives only semi-quantitative information, especially for minerals of low abundance. Also, as the section is of finite thickness (30 μm), problems are encountered when points fall on grain edges. At such points it is difficult to tell which mineral (or pore) should be counted which further restricts quantitative analysis.

In recent prior art an automatic analysis procedure has resulted in an automatic point-counting method in that an electron beam scans a sample such that a coarse grid of 50 x 50 points is obtained. On each point a full X-ray spectrum is collected and analysed. To identify the mineral composition of each point in the grid X-ray counts in twelve energy regions are compared with normalized values in said regions of current reference materials, wherein the smallest difference between the

unknown spectrum and one of the reference spectra classifies mineralogically the considered point.

Also rock characterization procedures are known in the art, particularly related to analysing reservoir pore complexes. Recent art has disclosed automated scanning electron microscope (SEM) imaging, wherein back-scattered electrons (BSE) form an image of a polished mineral sample by developing analog signals representing in time varying voltages which are proportional to scene brightness. In this procedure prior to analysis the pores are filled with epoxy resin which reacts totally different from the heavier mineral grains.

The resulting images can be processed in several ways. For example the SEM images are scanned by a videoscanner whereafter the resulting analog television signal is converted into a digital form. In such a digital representation the observed field is electronically arranged into an array of grid points or so-called pixels (picture elements) allocated to intervals on a gray level scale. The individual pixels of such a digital image are classified as either rock or pore.

In another example colour filter images are obtained from mineral samples, impregnated with pigmented typically blue epoxy. Such filters also produce distributions of pixel arrays. For each colour image "one" and "zero" values are assigned to distinguish pores from other matter.

In further analysing of the images as obtained above erosion and dilation techniques are used, with which pore complex characteristics can be determined.

Limited mineralogical information can be obtained from BSE analysis. The problem lies in the fact that there may be a grey level overlap for different minerals, also minerals (clays) with a variable composition can cause grey level overlap, as such hampering accurate mineralogical identification.

The above-mentioned investigation techniques are cumbersome in that rather complex processing techniques and indirect imaging procedures are used.

In the above mineral identifying procedure only a few sample points are analyzed. Intermediate sample areas and interstitial spacing are investigated insufficiently. Grain edge and pore wall characteristics cannot be determined at all in that way.

In the above-mentioned rock characterization procedures the required features have been acquired only after performing intermediate procedure steps as scanning of the BSE image and then filtering of the scanned image.

A main object of the present invention is to improve the mineral identification procedures as disclosed above, particularly to provide a process wherein the whole sample will be covered, and more particularly to provide a fully automated process which allows both mineral classification and geometrical rock matrix and pore network characterization in which process no operator interference is required.

A further object of the present invention is to improve the above-mentioned mineral classification procedure.

Yet another object of the present invention is to perform pore network and rock matrix procedures which are directly processing BSE information data.

To arrive at the above-mentioned objects, to overcome the discussed shortcomings and to accomplish said objects the present invention is a computer controlled process for analyzing and characterizing polished mineral samples, comprising the steps of automated compiling, storing, reading, displaying and processing sample data, firstly by using back-scattered electron (BSE) imaging of at least one field of view of a sample through which BSE information data are obtained, further processed by digitizing and directly converted into grey level information data, said data being further subjected to segmentation of grey levels; discriminating individual objects in the field of view, to wit pores from grains by detecting dark and non-dark phases; separating said objects by contour sharpening; reconstructing said objects; determining both rock matrix and pore network; and evaluating said

rock matrix and pore network information data so obtained to arrive at rock matrix and pore network parameters, and

secondly, after having defined in an automatic object defining step objects from said BSE information data, producing X-ray spectrum data of atomic elements in said objects by using inelastic electron scattering X-ray sampling of said field of view to carry out a mineral identification procedure relative to said field by exposing said objects each as a whole to high resolution inelastic electron scattering X-ray object scanning, said data being further subjected to determining elemental ratios of the objects investigated and said elemental ratios being compared with prototype mineral ratios to determine mineral composition of said objects.

A further ~~embodiment~~ aspect of the present invention is related to a system for carrying out a computer controlled process for analyzing and characterizing polished mineral samples,

- the system comprising an image processing system (IPS) for compiling, storing, reading, displaying and processing sample data, and
- the IPS being connected to,
- a scanning electron microscope (SEM) for producing a beam of accelerated electrons in order to scan said mineral samples,
- a detector for detecting back-scattered electrons (BSE) in the SEM, its detector response being used in the IPS in a first process step for imaging at least one field of view of a sample through which BSE information data are obtained, ~~(in the IPS)~~ and further processes by digitizing and directly converting into grey level information data, said data being further subjected to segmentation of grey levels; discriminating individual objects in the field of view, to wit pores from grains by detecting dark and non-dark phases; separating said objects by contour sharpening; reconstructing said objects; determining both rock matrix and pore network; and evaluating said rock matrix and pore network information data so obtained to arrive at rock matrix and pore network parameters; and defining in an automatic object defining step objects from said data, and

- an energy dispersive X-ray analyzer (EDX) connected to the IPS and being used in a second process step for producing X-ray spectrum data of atomic elements in said objects by using inelastic electron scattering X-ray sampling of said field of view to carry out a mineral identification procedure relative to said field by exposing said objects each as a whole to high resolution inelastic electron scattering X-ray object scanning, in the IPS said data being further subjected to determining elemental ratios of the objects investigated and said elemental ratios being compared with prototype mineral ratios to determine mineral composition of said objects.

One advantage forms the accurate classifying of the minerals even when slightly changing compositions occur in the sample to be analysed.

However, the most important feature of the process as stated above lies in the combination of the BSE and EDX techniques. With the improved BSE procedure the objects to be analysed are defined

more accurately. With said EDX technique a highly improved mineral classification is achieved, and moreover mapping of said procedures results in an overall characterization of a sample which forms a great advance for its users in the field of petroleum engineering, mining and soil engineering.

Further aspects, features and advantages of the present invention will become clear from the following description and the accompanying drawings in which:

Fig. 1 shows a system with which the process in accordance with the present invention is performed;

Fig. 2 shows schematically the mineral identification procedure of the present invention;

Figs. 3A to 3D are illustrating several processing steps in the mineral identification procedure in accordance with the present invention;

Figs. 4A and 4B present results of classification procedures, respectively known from the prior art (A) and in accordance with the present invention (B);

Fig. 5 shows schematically the pore network and rock matrix characterization procedure in accordance with the present invention; and

Fig. 6 is illustrating a worked-out example of a sample analyzed and characterized in accordance with the present invention.

In the Figs. 2 and 5 the same reference numerals are used for corresponding process steps.

Fig. 1 shows a system with which the process in accordance with the present invention is performed. The system consists of three main parts which are interconnected via several interfaces (not shown; for reason of clarity the interfaces are represented by one single block with electronics for beaming, scanning and imaging): a scanning electron microscope (SEM) 1, an energy dispersive X-ray analyser (EDX) 2 and an image processing system (IPS) 3.

The SEM 1 comprises an SEM column 10 and uses an accelerated electron beam (up to 30 kV) under high vacuum conditions, to scan the sample under investigation. For the work described here, samples are impregnated with resin, in particular an epoxy resin, cut and highly polished to obtain a flat surface. They are then coated with a conductive layer of carbon to avoid build-up of electric charge. The various kinds of electrons and photons generated at the point of incidence of the electron beam on the sample surface are detected separately. As the electron beam scans the sample, the response of any one of these detectors can be used to build up a characteristic image of its surface. The detector normally used in the work described below is a back-scattered electron detector 14.

In the Fig. 1 several beam conditioning functions such as magnification, spot size, scan mode, detector, and focus are represented by a single line 12, and X- and Y-stearing by a line 13. Also beam stabilization means are comprised in the represented SEM column (not shown for reason of clarity).

On a stage 15 a sample 16 is arranged, or more samples can be arranged in that the sample(s) can be repositioned and substituted as required. For each sample at least one field of view, i.e. a beam spotted area, will be scanned. Also for one sample more fields of view can be scanned, for example eight fields. In such a way a more representative sampling is obtained, which results in a more accurate, mineral identification and/or characterization of the sample. Generally the samples under investigation are mineral samples such as rock samples.

It is noted that the above functions are controlled by electronical circuitry indicated in a general way in the drawing by a block 17 with electronics for beaming, scanning and imaging.

The energy dispersive X-ray (EDX) detector and analyser consists of a detector and analyzer means 20 also connected to said block 17. The analyser detects the X-rays generated by the interaction of the electron beam and the sample with a detector

as the X-ray energy is measured to obtain qualitative and quantitative information about the chemical composition of said sample. This is possible since the energy distribution of emitted X-rays is characteristic for each atomic element in the sample.

The image processing system (IPS) 3 comprises a central processing unit (CPU) 31, based on a very fast array processor and allows image acquisition, enhancement, reconstruction and analysis at high speed. Images to be processed are obtained from the SEM 1, while the X-ray data, needed for mineral identification are obtained from the EDX. The SEM, EDX and the IPS are all interfaced. Under fully automated operation the CPU of the IPS the controls all pertinent functions of the microscope and the X-ray analyzer. As mentioned above, magnification, beam spot size, scan mode, detector selection, automatic focussing and coil supply of the SEM can be controlled and even so for the EDX system, X-ray data collection and processing.

The IPS 3 comprises also an operating and displaying unit 32 used by operators to start and set the required functions and operations.

It will be clear that with an automated system as described operating time will be reduced substantially.

In Fig. 2 the mineral identification procedure in accordance with the present invention is shown schematically.

Two main techniques are employed in the determination of mineralogy using an SEM/EDX system namely back-scattered electron (BSE) imaging and X-ray analysis. First an outline of the principles of these techniques is given below. Then a detailed description relative to the different procedure steps will be given referring to the Figs. 2 and 3A-D.

As a result of the primary electron beam scanning the sample, back-scattered electrons are generated. The detector for these electrons (14 in Fig. 1) is mounted in the SEM just above the sample. The signals provided by the back-scattered electron detector as BSE information data reflect the average atomic density at the spot of impact on the sample and so provide

information about the average composition at that spot. As a rule the resolution of BSE images for mineral samples such as rock samples is approximately 1 micron at an accelerating voltage of 20 kV. BSE imaging in SEM studies is mainly used to obtain information about the spatial distribution of minerals, grains and pores.

Figure 3A shows a BSE image of a sandstone sample where the grey level is related to the average atomic number of the mineral. Hence, the heavier the mineral is the brighter it appears in the BSE image. The pores are dark as they are filled with a low density epoxy resin. From such an image the image analyser can recognise particular groups of minerals by detecting only those areas whose grey levels fall within a specified range. This procedure is called segmentation. However, as different minerals can have very similar average atomic numbers (e.g. quartz, dolomite and sodium feldspar) the segmentation allows only partial discrimination. In order to distinguish such minerals X-ray analysis must be used.

The X-ray analysis system measures the X-ray energy spectrum, i.e. the intensity and the energy of the X-ray radiation. In an SEM, X-rays are generated during inelastic scattering of the primary (beam) electrons. These X-rays are formed by two distinctly different processes: (1) the deceleration of the beam electrons in the Coulomb field of the atomic nucleus gives a continuous background spectrum ("Bremsstrahlung") and (2) the interaction of a beam electron with inner shell electrons results in characteristic lines in the spectrum. The continuous (background) part of the spectrum is not used in the mineral identification procedure. Detection and classification of the characteristic X-ray lines allow identification of the atoms (elements) in the excited sample volume. Also, the number of X-rays emitted by each element is related to the concentration of that element.

The minimum element concentration detectable in X-ray micro-analysis is 0.1% in the best cases and typically less than

1%. For most rock samples the spatial resolution is approximately one micron.

The automated mineral identification procedure basically involves the recognition of individual objects (i.e. grains, diagenetic minerals, clays) in the sample. Each object is subjected to X-ray analysis in order to obtain X-ray spectrum data of elements present in the sample which can be classified in terms of mineralogy. Advantageously X-ray spectra of nine elements are acquired. The complete procedure 40 consists of a number of steps 41-50 which are described in detail below and is represented in Fig. 2.

To obtain an image from the SEM the IPS takes control over several functions of the microscope. First a field of view on a sample, i.e. an area to be beam-spotted by a scanning spot, is chosen, represented by a block 41. Then in a BSE-imaging step 42 the appropriate detector and magnification is selected and scanning of the electron beam on the sample is controlled. While the SEM is scanning the sample, the IPS digitizes the incoming BSE information data in a grey level discrimination step 43 and stores it in a video memory. For example a stored image has a standard format of 512 x 512 points (or pixels) in which each point can have a value from 0 to 255 presented as grey level information data. Advantageously for mineral samples the grey values range from 0, corresponding with black (pores), via grey (quartz) to 255 corresponding with white (heavy minerals). It is noted that with advanced data processing equipment more detailed image information can be obtained. Increased test facilities for example up to 10 bit x 10 bit pixels per image with 4 byte grey values for each pixel may be enabled.

Once the data have been stored in a video memory by the IPS, a multitude of image processing functions is available to enhance the image. These functions can filter out noise, improve the contrast, sharpen up contours, discriminate, separate and identify certain objects in the image as indicated by a block 44 in Fig. 2.

In BSE images of sandstone samples the grain edges and grain-to-grain contacts often have a darker appearance than the grain interiors, (Fig. 3A). If this darker rim would be present around the complete grain, then each grain could be distinguished from every other grain by a simple grey level segmentation procedure. However, the grain-to-grain contacts are sometimes not visible along their complete length and so a more sophisticated grain detection procedure is needed. This again involves segmenting the BSE image to give a binary image in which for instance the grains are white and the pore space is black. In this image there will still remain groups of two or more grains which have not been "separated" but which are joined at constrictions as illustrated in Fig. 3B. These constrictions are removed using various morphological operations, resulting in "separated" individual objects. This is illustrated in Fig. 3C.

Once the objects (i.e. separated grains) in the image have been recognised and identified, the coordinates of all the points which belong to an object are known. The IPS then takes control of the electron beam in the microscope and the beam is scanned over the area of each object in turn. During this procedure, X-rays are generated. A problem may occur when two objects of different elemental composition are in contact. For instance, X-ray counts generated from an object other than the one under investigation may be detected. To prevent this all the objects are mathematically eroded, i.e. pixels from the object boundaries are deleted, thus reducing the actual scan area from which the X-ray counts are detected as illustrated in Fig. 3D. Such a procedure indicated in Fig. 2 in block 45 will result in the removal of small objects from the image. Therefore only those objects which will not disappear in this procedure will be scanned. In the present procedure advantageously an X-ray spectrum of each object is obtained in an object scanning step 46 by using 9 predefined energy windows, each window representing an element. The X-ray counts are normalised and sent to the IPS as a

vector of 9 elements. In the IPS the contribution of the background is removed prior to the mineral classification.

Once the X-ray data have been collected and sent to the image analyser, classification of each object or grain can be attempted in a classification step 47. This is done by comparing the vector of X-ray counts obtained from an object with vectors previously obtained for known minerals or "prototypes". This procedure can be considered as an "absolute method" as a result of using absolute values in said classification. If the vector from the unknown mineral is similar enough to one of the prototype vectors the unknown mineral can be classified.

A procedure as known from the prior art is shown in Fig. 4A using the concentrations of Si and Al only (roughly equivalent to the X-ray counts at appropriate energies) to classify some silicate minerals. In said classification procedure a vector of 9 elements (Na, Mg, Al, Si, P/Zr (combined), K, Ca, Ti/Ba (combined), Fe) is composed to enable discrimination between a greater number of minerals. The elements P/Zr and Ti/Ba are combined because of peak overlap in the X-ray spectrum. This procedure works successfully for many minerals but problems are encountered when classifying minerals of which the composition varies slightly. For example, dolomite can contain minor amounts of iron. If the X-ray counts from such a grain are compared with those of a "pure" dolomite, the vectors may differ too much to allow proper mineral classification. To try to define a prototype composition for every slightly different mineral would be an extremely difficult if not impossible task. Another problem with this absolute method is the amount of analysis time required to reduce statistical fluctuations in the data which can result in misclassifications of some minerals.

Given the above problems in accordance with the present invention the elemental ratios of the unknown grain are compared with those of known prototype minerals. Fig. 4B shows this procedure, to be considered as a "ratio method", for the same silicate minerals as in Fig. 4A. In this way statistical

of fields of view or samples can be carried out in one procedure without operator interaction (e.g. overnight).

Besides the analysis of mineral abundancies it is also of interest to determine various grain textural parameters (e.g. size, shape, etc.) of the samples. This can be done for the complete detrital assemblage or only for certain minerals (e.g. quartz). Various size and shape parameters are available to the user (e.g. area, maximum or minimum diameter, elliptic shape factor (i.e. ratio of minor:major ellipse axes)) and it is possible to define others if required. Before these analyses can be carried out it is necessary to remove those grains from the image which touch the edge of the field of view, because their geometrical parameters cannot be accurately determined.

Another procedure which can easily be performed after mineral identification is the determination of the pore wall mineralogy. The determination of grain textural parameters and the pore wall mineralogy add very little time to the total analysis.

In Fig. 5 the pore network and rock characterization procedure in accordance with the present invention, indicated by 60, is shown schematically. Said procedure also uses a BSE image of an impregnated and polished rock sample, collected and transferred as said BSE information data from the SEM to the CPU. In order to obtain sufficient statistical accuracy in the analysis, a number of fields of view is collected for each sample at an appropriate magnification. Particularly, the magnification chosen should be low enough to avoid statistical scatter, but high enough to resolve small pores and small mineral particles. As discussed above with respect to the mineral identification procedure, particularly with respect to the steps 41, 42, 43, a BSE image gives a certain amount of mineralogical information with heavier (high atomic density) minerals appearing brighter. The pores are filled with a low density epoxy resin and appear by contrast very dark. The BSE image is improved using grey level processing techniques such as local noise filtering.

fluctuations have less influence on the classification procedure resulting in a more accurate classification. The analysis time per grain is approximately 1-2 seconds. It is also possible to account for slight variations in composition without the necessity of defining a large number of prototype compositions for each mineral. The results of classifying the grains detected from the BSE image of Fig. 3A can be represented in coloured pictures, the different colours representing the different mineral classes considered as indicated in Figs. 4A, B. Further processing of such an image is required before quantification of mineral percentages can be attempted.

Because of the initial erosion of the grains, applied to prevent edge effects in the object scan procedure, the original object boundaries are lost. Before measuring the mineral percentages in the image, the objects have to be restored in a reconstruction step 48 to their original form. The restoration procedure uses a technique similar to that used for the object separation. For a homogeneous sample, measurement of the area percentage of a mineral on 2-D sections is a good estimate of its volume percentage in the bulk rock. The greater the total section area considered, the better the estimate. Measurement of the area percentages of the pores and various minerals in an evaluation step 49 is a simple and fast procedure using image analysis. Finally the mineralogy of said sample can be obtained in a mineralogy step 50, said mineralogy being greatly improved over the prior art by using the above "ratio method".

The above procedures are all linked and can be applied for the analysis of one field of view. However, in order to allow analysis of more fields of view, movement of the sample is necessary. This is carried out by a motor driven scanning stage (as shown in Fig. 1) mounted on the SEM which can be remotely controlled from the IPS. Before starting the procedure the coordinates of the required analysis positions are stored in the memory of the IPS. This means that automatic analysis of a number

The pores are then discriminated from the mineral components in the image by detecting only the dark phases. Mathematical morphology techniques represented by a sequence of steps 61, 62 63 allow definition of individual pores where the pore necks are defined as being constrictions in the 2-D pore network (pores touching the edges of the image are excluded from the analysis as their geometrical properties cannot be accurately determined). A similar procedure represented by a sequence of steps 64, 65, 66 is applied to extract the rock matrix from the BSE image wherein the grains are discriminated from the pores by detecting only non-dark phases. Pore area and pore neck length distributions are measured, as well as the two dimensional coordination number (i.e. the average number of pore necks per individual pore). From the rock matrix image, grain diameter (grain size), grain smoothness and aspect ratio (grain shape) are measured.

In the scheme as shown in Fig. 5 the two way approach of the characterization procedure is indicated clearly. When pore network and rock matrix information data are obtained then pictures will be produced wherein rock matrix data and pore network data have to be imaged separately. Then a "def." image-step 67 is executed to define these images respectively.

The last two steps in this procedure comprise evaluating the image data in an evaluation step 68 and measuring pore network and rock matrix parameters in a step 69 as indicated in Fig. 5.

As an example the results of identification and characterization of the sample as mentioned above, i.e. as results accumulated over eight fields of view, are shown in Fig. 6, exemplifying the great advantage of combining the above BSE imaging and X-ray sampling procedures.

It will be clear for those skilled in the art that the example worked out above, and the used procedures can be extended respectively to other parameter measurements and further procedures which are not used at this moment such as neutron scattering methods and 3D-analyzing.

It is needless to say that the presented process and system can be modified and changed without exceeding the scope of the present invention.

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C L A I M S

1. A computer controlled process for analyzing and characterizing polished mineral samples, comprising the steps of automated compiling, storing, reading, displaying and processing sample data, firstly by using back-scattered electron (BSE) imaging of at least one field of view of a sample through which BSE information data are obtained, further processed by digitizing and directly converting into grey level information data, said data being further subjected to segmentation of grey levels; discriminating individual objects in the field of view, to wit pores from grains by detecting dark and non-dark phases; separating said objects by contour sharpening; reconstructing said objects; determining both rock matrix and pore network; and evaluating said rock matrix and pore network information data so obtained to arrive at rock matrix and pore network parameters, and
secondly, after having defined in an automatic object defining step objects from said BSE information data, producing X-ray spectrum data of atomic elements in said objects by using inelastic electron scattering X-ray sampling of said field of view to carry out a mineral identification procedure relative to said field by exposing said objects each as a whole to high resolution inelastic electron scattering X-ray object scanning, said data being further subjected to determining elemental ratios of the objects investigated and said elemental ratios being compared with prototype mineral ratios to determine mineral composition of said objects.

2. The process as claimed in claim 1 wherein said rock matrix and pore network parameters comprise pore area, pore neck length, distribution of said pore area and pore neck length, and coordination number.

3. The process as claimed in claim 2, further comprising the calculation of grain diameter, grain smoothness and aspect ratio from said parameters.

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4. A process as hereinbefore described with reference to the drawings of the figures 1, 2, 3, 4B, 5 and 6.

5. A system for carrying out a computer controlled process for analyzing and characterizing polished mineral samples, the system comprising an image processing system (IPS) for compiling, storing, reading, displaying and processing sample data, the IPS being connected to a scanning electron microscope (SEM) for producing a beam of accelerated electrons in order to scan said mineral samples; in the SEM, a detector for detecting back-scattered electrons (BSE) its detector response being used in the IPS in a first process step for imaging at least one field of view of a sample through which BSE information data are obtained, ~~in the IPS~~ and further processed by digitizing and directly converting into grey level information data, said data being further subjected to segmentation of grey levels, discrimination of individual objects in the field of view, to wit pores from grains by detecting dark and non-dark phases, separation of said objects by contour sharpening, reconstruction of said objects, determination of both rock matrix and pore network, and evaluation of said rock matrix and pore network information data so obtained to arrive at rock matrix and pore network parameters, the IPS defining in an automatic object defining step objects from said data; and an energy dispersive X-ray analyzer (EDX) connected to the IPS and being used in a second process step for producing X-ray spectrum data of atomic elements in said objects by using inelastic electron scattering X-ray sampling of said field of view to carry out a mineral identification procedure relative to said field by exposing said objects each as a whole to high resolution inelastic electron scattering X-ray object scanning, in the IPS said data being further subjected to determining elemental ratios of the objects investigated and said elemental ratios being compared with prototype mineral ratios to determine the mineral composition of said objects.

6. A system as hereinbefore described with reference to the drawings of the figures 1, 2 and 5.