Precision serial lapping, imaging and three-dimensional reconstruction of minus-cement and post-cementation intergranular pore-systems in the Penrith Sandstone of north-western England

MARK R. COOPER AND ROBERT H. HUNTER

Department of Earth Sciences, University of Liverpool, Brownlow Street, Liverpool, L69 3BX, UK

Abstract

The application of serial lapping, imaging and image processing, and three-dimensional reconstruction techniques are discussed using the quartz-cemented Penrith Sandstone as an example. The study involves the characterization of the three-dimensional pore system microgeometry using combined back-scattered scanning electron and cathodoluminescence microscopy and focuses on the definition of post-compaction and post-cementation intergranular porosity networks. Sample preparation, section spacing and re-orientation and data presentation are described. Aspects of the application of serial section datasets and their limitations are discussed in relation to permeability studies.

KEYWORDS: precision serial lapping, imaging, minus-cement, Penrith sandstone.

Introduction

AN assessment of the factors which influence permeability is an important aspect of the understanding of reservoir quality. In particular, it is important to quantify the factors which influence the evolution of porosity with time, principally, compaction and cementation. Permeability within unfractured porous rocks depends mainly upon the grain-size and porosity and on various factors which describe the pore system microgeometry, i.e. shape, tortuosity and inter-connectivity of the porosity. Grain-size, sorting and porosities can readily be determined and in many rocks, permeability can be measured on cored material. It is possible, therefore, to determine empirically what the shape and tortuosity factors are in any sample in which grain size and shape distributions have been determined, although this gives information only on the present intergranular porosity. Another approach is to define a model pore system and to model numerically the permeability (e.g. Bryant *et al.*, 1993). Such models work well for simple systems (i.e. near constant grain sorting, shape and packing), but are not well suited to more complicated systems and, at present, a general model of permeability as a function of grain characterization is not available. It is because mechanisms, timing and rates of compaction and cementation are influenced by complex physical and chemical interactions during burial, that it is not possible generally to predict how they will affect permeability in all but the most simple systems.

What is needed is a detailed characterization of pore system microgeometries in a variety of natural and synthetic systems and an understanding of the way in which these pore systems develop as a function of compaction and cementation history. To achieve this, it is necessary to be able to distuinguish certain textural elements, in particular the post-compaction–pre-cementation porosity (the 'minus-cement porosity') and the post-cementation intergranular porosity; both reflect important stages of the intergranular porosity evolution during burial. Quartz arenites, in particular aeolian and shallow marine

Mineralogical Magazine, June 1995, Vol. 59, pp. 213–220

sandstones, are mineralogically and texturally the most mature natural systems available. Furthermore, quartz, precipitated as syntaxial overgrowths, is one of the most abundant cements found within such sandstone reservoirs (McBride, 1989) and is therefore an important control on reservoir quality. Understanding the way in which compaction and cementation affect permeability in these relatively simple natural systems will provide the framework for developing more complex models. Even in mature quartz arenites, however, variations in sphericity and roundness of grains may be significant and locally determine the shape and interconnectivity of pores (see Fig. 1*a*). Characterization of the pore system microgeometry is, thus, a complex three-dimensional (3-D) problem and is generally not amenable to standard stereological methods (e.g. De Hoff, 1983).

Three-dimensional geometries of porosity systems can be obtained by impregnation and acid leaching, however, this gives information only on post cementation intergranular porosity. The only way properly to characterize such complex systems is through 3-D reconstruction from serial thin sections or sequentially ground surfaces. The latter requires the ability to characterize features incrementally during repeated lapping. The recent development of image capture hardware and image processing and analysis software has facilitated petrographical data gathering and interpretation. However, serial thin sectioning and serial lapping generally have been regarded as time consuming and labour intensive and, as such, have not been utilized as a general procedure (De Hoff, 1983).

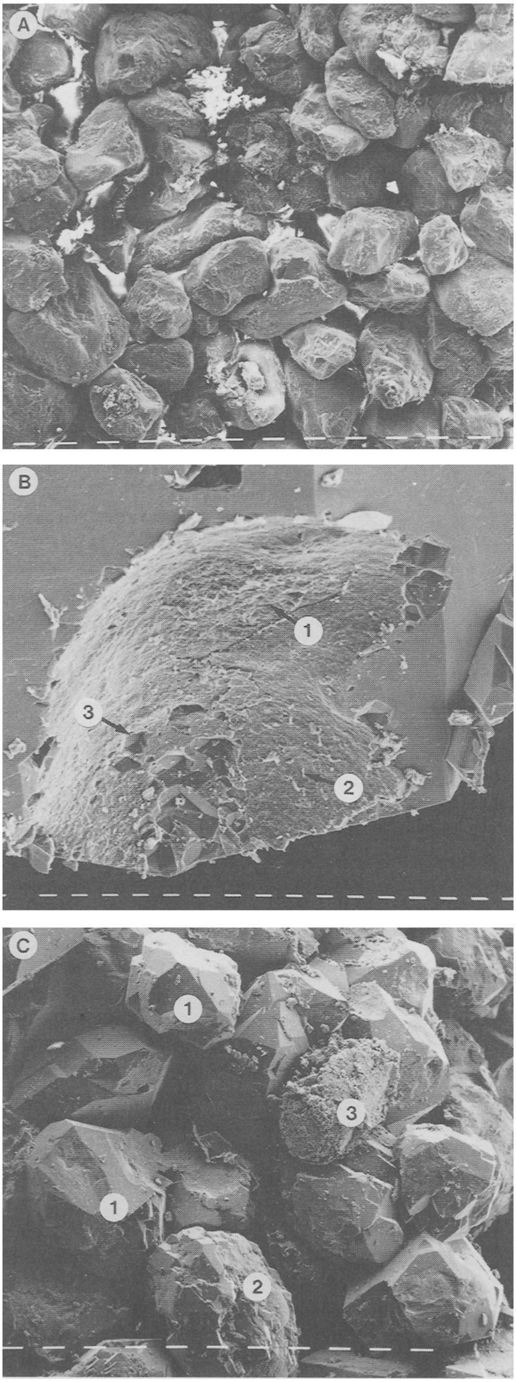


FIG. 1. SEM photomicrographs of selected Penrith Sandstone samples [accelerating voltage = 15 kV]. (A) Example of an uncemented grainflow lamina, which has undergone mechanical compaction and minor pressure solution. The interconnectivity of the intergranular pore-system, relative to cemented examples, is good [Sample location: Bongate Scar; Grid ref. NY 687 200; scale bar = 100 μm]. (B) Photomicrograph of a cemented lamina showing small, shallow pressure solution pits (1) at detrital grain-to-grain contacts Small (<10 μm) moulds of early overgrowths (2) and flakes of grain-coating clay (3) are preserved where overgrowths have grown together or around neighbouring detrital grains [scale bar = 10 μm]. (C) A grainflow lamina within which the volume and interconnectivity of the post compaction intergranular pore-system has been reduced by the precipitation of quartz overgrowths. Structural classes of quartz grains identified include: (1) monocrystalline grains generally showing well-developed single euhedral overgrowths. (2) Polycrystalline grains generally with poorly-developed overgrowths. Grains of a composition other than quartz, e.g. rock fragments (3) lack overgrowths [scale bar = 100 μm: B and C Sample location: Bowscar Quarry; grid ref. NY 520 344].

In this contribution, we describe aspects of the 3-D pore system microgeometry within certain facies of the Penrith Sandstone (Rotliegendes-equivalent) of northwestern England. The method involves serial lapping/polishing and incremental data gathering using scanning electron microscopy, combining back-scatter electron (BSE) and cathodoluminescence (CL) techniques. We present observations on 3-D geometries of minus-cement and post-cementation intergranular pore systems, highlighting different approaches to data collection and presentation and their limitations.

The Penrith Sandstone

The aeolian facies of the Penrith Sandstone is considered to have been deposited as westward-migrating, sinuous-crested to barchanoid transverse dunes (Macchi, 1981; Steele, 1981). The lamination types preserved within individual dunes include grainflow (cf. avalanche or sandflow) and wind-ripple laminae; grainfall is not commonly observed. Details of the depositional processes which form these stratification types are discussed by Goggin *et al.* (1986). A detailed description of the petrography of the Penrith Sandstone is given by Waugh (1970*a,b*).

Detrital texture and mineralogy. For the purposes of this investigation, a sample was selected from a texturally and mineralogically mature grainflow lamina. The grain-size and sorting were determined from a thin section cut perpendicular to the lamina. The grain-size is that of a medium sand (0.42 mm, 1.35 phi) and the lamina is well sorted (s.d. = 0.49 phi). Individual grains show moderate to high sphericity and are rounded to well rounded (Fig. 1*a*). This uniformity of grain size and shape reflect a relatively simple depositional (detrital) texture. Local (grain-scale) variations in the packing control the heterogeneity of the intergranular pore-system microgeometry. The detrital fraction of the lamina comprises ~ 90% quartz grains, the remainder being varied feldspars (5%), rock fragments (5%) and rare heavy minerals; using the mineralogical classification of Dott (1964), the lamina classifies as a subarkose.

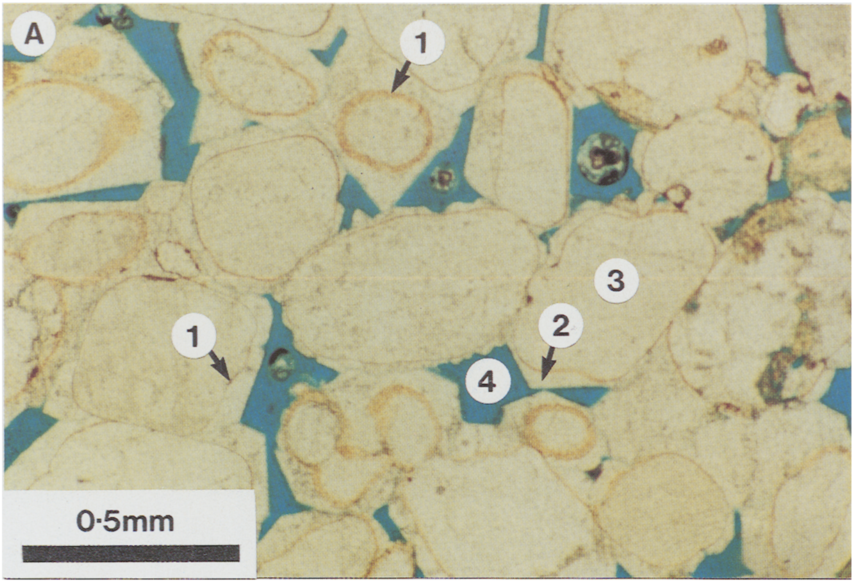
Diagenesis and porosity evolution. Although we have no direct measure of the depositional intergranular porosity, average values measured from modern uncompacted grainflow laminae are close to 50 % of the bulk volume (% bv) (Pryor, 1973; Atkins and McBride, 1992). The depositional porosity has been reduced by the effects of burial-related mechanical compaction, pressure solution and quartz cementation. Mechanical reorganization processes include grain slippage, rotation and fracturing; these occur mainly at relatively shallow burial depths (100s of metres) and can account for significant porosity reduction and bulk volume loss in newly deposited sands. Further, but relatively minor bulk volume loss has resulted from pressure solution at grain-to-grain contacts (Fig. 1*b*; see also Waugh, 1970*b*). Significant pressure solution has been prevented by the precipitation of quartz as syntaxial overgrowths (Fig. 1*c*); evidence supporting this is shown Fig. 1*b*, features such as grain coating clay and moulds of early quartz overgrowths would have been detroyed if further pressure solution had occurred. Local heterogeneity in the degree of cementation relates to the packing, which controls the volume available for cementation (i.e. the minus-cement porosity) and structural/compositional variations of the detrital grains which control nucleation sites and growth rates for quartz overgrowths (captions: Fig. 1*c* and Plate 4*b*).

The minus-cement porosity defines the limit of compaction-related intergranular porosity reduction; 33.6% bv in the Penrith sample determined from point counting. Quartz cementation has further reduced the intergranular porosity to a residual of 11% bv. Thus, the total intergranular porosity reduction associated with compaction and cementation is ~ 80%. These results, although useful in terms of defining the volumetric changes during porosity evolution, give no indication of changes in pore-system microgeometry, i.e. pore-size, pore throat diameter or interconnectivity.

Methods

Approaches and limitations. Transmitted light is used for conventional thin section analysis and scanning electron microscope BSE and CL and reflected light microscopy are commonly utilized methods of sample surface imaging in the geosciences. The absence of identifiable detrital grain boundaries in many quartz cemented sandstones prevents the distinction of detrital grains and syntaxial overgrowths using transmitted light. In the Penrith Sandstone this is not a problem, however, since the presence of well-defined dust lines on detrital grain surfaces does allow this distinction (Plate 4; see colour plate section). Nevertheless, difficulties do arise in determining the exact position of grain or pore boundaries and the nature of grain contacts because such curved or inclined surfaces have an apparent width when viewed in sections of normal thickness (e.g. Plate 4*a*). By imaging the sample surface, errors associated with conventional optical thin section analysis are overcome.

In any imaging technique involving three-dimensional reconstruction, tracing individual pores and their interconnections through consecutive increments relies on the spacing of the sections. Section



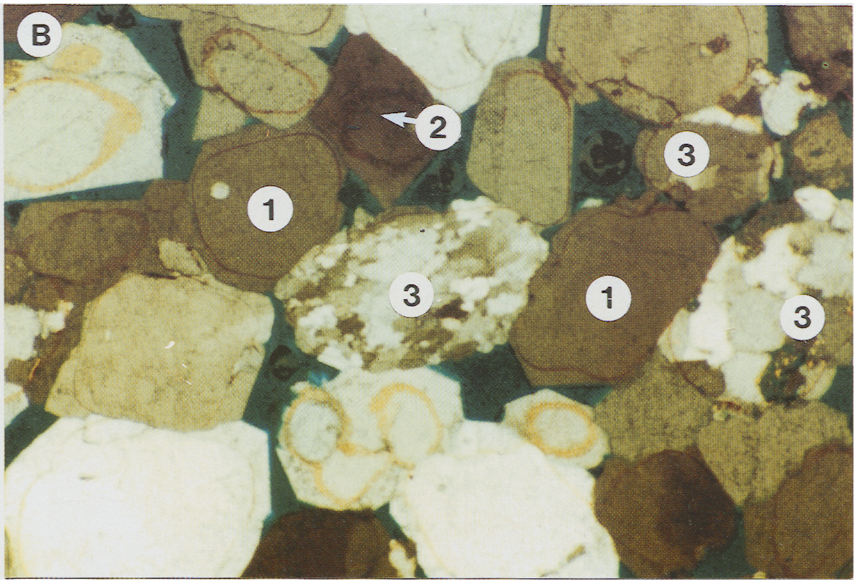


PLATE. 4. (A) Plane polarized light (PPL) photomicrograph of a quartz-cemented grainflow lamina. Outlines of detrital grains are defined by a mixed layer of iron oxide and grain coating clays, the dust line (1); note variations in apparent width. The dust line allows differentiation of the quartz overgrowths (2) from their detrital quartz hosts (3), both of which appear colourless when viewed using PPL. Intergranular porosity (4) has been impregnated with epoxy resin which appears blue under PPL. (B) Partial XPL (cross polarized light) photomicrograph of the same field of view as (A), showing overgrowths in optical (crystallographical) continuity with their detrital hosts. Structural classes of quartz grains identifiable using XPL include unstrained (1) and strained (2) monocrystalline grains, not identifiable using the SEM images. A range of polycrystalline grains (3) are present, for which the overgrowth development is controlled by the structural complexity of the host grain. [Sample location: Bowscar Quarry; Grid ref., NY 520 344].

spacing, using serial thin sectioning, is limited to ~ 300 μm (Bryon *et al.*, this vol.), and thus for grain sizes less than ~ 1 mm, is unsuitable for characterizing individual features such as grains or pores. Also, the hardness of quartz causes severe wear on blades used to cut the serial sections utilizing an annular saw, the normal method of obtaining such sections. Serial lapping allows increments of as little as 1 μm; although our experience suggests that with standard lapping technology, increments of ~ 30 μm are the minimum achievable with any precision. A disadvantage of incremental lapping is the destruction of the sample; any data-collection and imaging of the sample must be undertaken after each grinding increment. The grain-size of the sample utilized in this study is that of a medium sand and precision serial lapping was utilized.

Precision serial lapping. Serial lapping was undertaken utilizing a Logitech PM2A precision lapping and polishing machine and PP5D precision polishing jig. Increments of 100 ± 5 μm of sample were removed. Pore space within the Penrith sample was impregnated with epoxy resin prior to analysis. The resin reduces the risk of grain-plucking during grinding and allows the intergranular porosity to be imaged.

Three-dimensional reconstruction from a series of two-dimensional images requires that individual surfaces are accurately oriented in the x–y plane with respect to successive slices in the z-direction. In order to achieve this, the sample was first cut with mutually perpendicular surfaces using a jig specially adapted for this purpose in conjunction with an annular saw. Orientation of successively ground surfaces in the x–y plane was then achieved using edges and corners, either prior to imaging or using software after image acquisition. The errors introduced in visual orientation/matching of edges are of the order of a few microns, which is small in comparison to the incremental spacing in the z direction and to the features of interest.

Image acquisition and processing. Combined BSE and CL were used to image the sample (Fig. 2*a,b*, respectively). Images were collected using a JEOL-840A electron microscope with a LINK BSE detector and an Oxford Instruments CL detector. The BSE and CL images were acquired using a LaB_6 electron gun with accelerating voltage of 15 kV and probe current of 1.9 nA at a working distance of 39 mm. The SEM was linked to a KONTRON IBAS-20 image analysis system, which was used to control the SEM, to collect and store images, and to control a motorized stage (Evans *et al*, 1994). The system allowed predetermined areas of the sample surface to be imaged with minimum effort. The geometry of the CL collection system determined the minimum magnification available (× 100). For the grain size

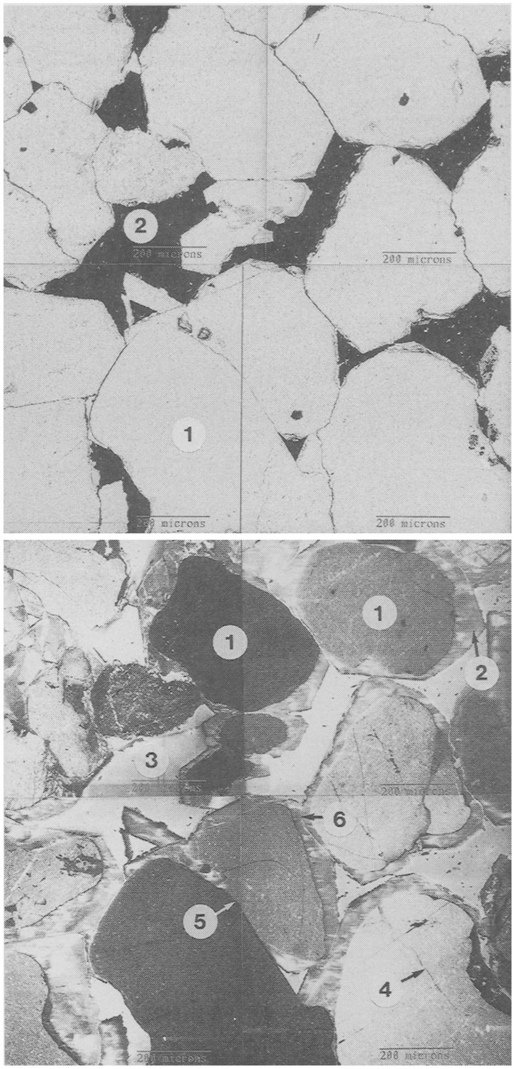


FIG 2. (A) Montage of BSE images of the quartz-cemented grainflow lamina collected between successive lapping increments. Quartz (1) appears light grey, intergranular porosity (2), impregnated with epoxy resin, appears dark grey. (B) CL images corresponding to the BSE images in A, showing detrital grains (1) and their overgrowths (2). The intergranular porosity (3) appears light grey, similar to the grey-level shown by some of the detrital grains and overgrowths. The image also reveals information concerning fractures (4), and pre- and post-cementation grain contacts, (5) and (6) respectively [Scale bar = 200 μm].

of the sample, four overlapping images were required in order to collect an area large enough for analysis;

these were collected at × 120. Pairs of BSE and CL images were collected from each surface between successive lapping increments.

For the BSE images, the atomic number contrast between quartz and epoxy resin allows the post-cementation porosity to be easily and accurately digitized. This contrast also facilitates manual segmentation using a grey-level histogram of the image. Note that the dust lines identified using optical petrography are not visible, it is therefore not possible to differentiate detrital grains from their overgrowths using BSE. For the CL images, the differences in grey-level between the detrital grains and their overgrowths allow them to be differentiated and variations in grey level between individual grains allows them to be traced between consecutive sections with confidence. Under CL, the epoxy used to impregnate the intergranular porosity has a grey level similar to that of some of the detrital grains and overgrowths. With hindsight, impregnation of the intergranular pore-system with a material whose cathodoluminescence is markedly different to the rock components would have eliminated the need for separate BSE images.

The CL images, although containing much useful information, are complex and information regarding detrital grain boundaries, overgrowths and grain contacts cannot be acquired solely on the basis of segmentation using grey-levels but requires manual digitization in order to simplify the image. Although automated image processing packages are available, in general, they cannot distinguish cracks and other superfluous information (noise) from desired image elements. Manual filtering of auto-processed images can produce the desired information, but is dependent on the complexity of the sample and inherent noise. Manual digitization allows the operator to select specific elements relevant to the study and remove completely unwanted information.

The information required to reproduce the 3-D minus-cement porosity and post-cementation porosity was digitized manually from the BSE and CL images and stored as coded polygon network files. Once the cement component had been subtracted from the image, the minus-cement pore-system could be reconstructed.

Image presentation. Simplified, manually digitized images of boundaries of intergranular pores and detrital grains are shown in Fig. 3. Such images, when compared with Plate 4 and Fig. 3, highlight the noise associated with unprocessed 'raw' images. The simplified images are those which are used to reconstruct the 3-D elements of the texture, either by viewing separate incremental images in series, or by viewing stacked images stereoscopically. Figure 4 shows six simplified serial sections showing detrital grains, minus-cement and intergranular porosity;

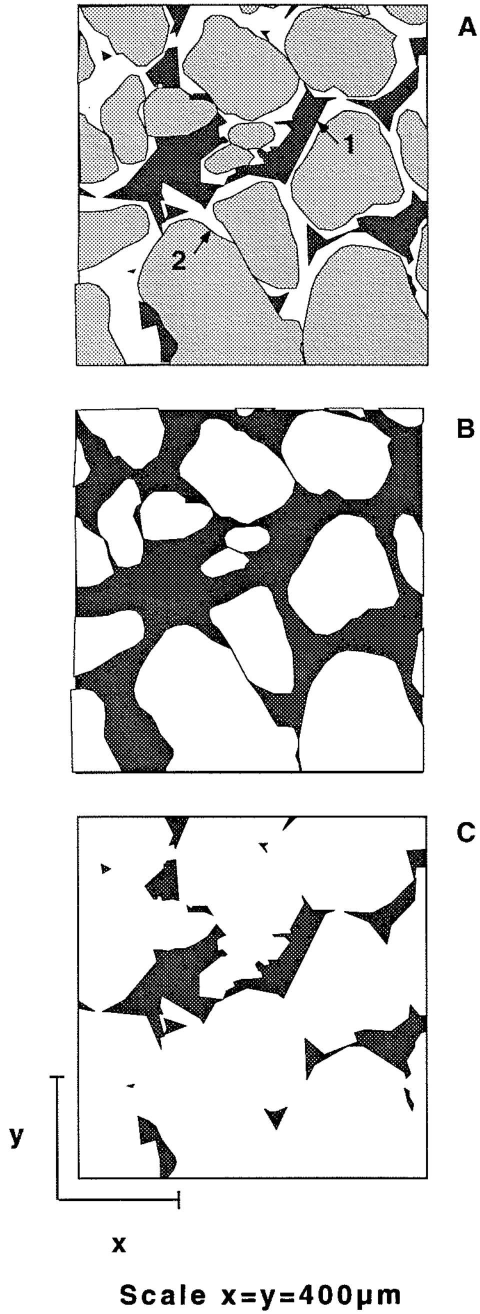


FIG. 3. (A) Boundaries of intergranular pores (1) and detrital grains (2) digitized from the BSE and CL images. (B) Simplified section showing the geometry of detrital grains (white) and minus-cement porosity (shaded). (C) Simplified section showing quartz (white) and intergranular porosity (shaded).

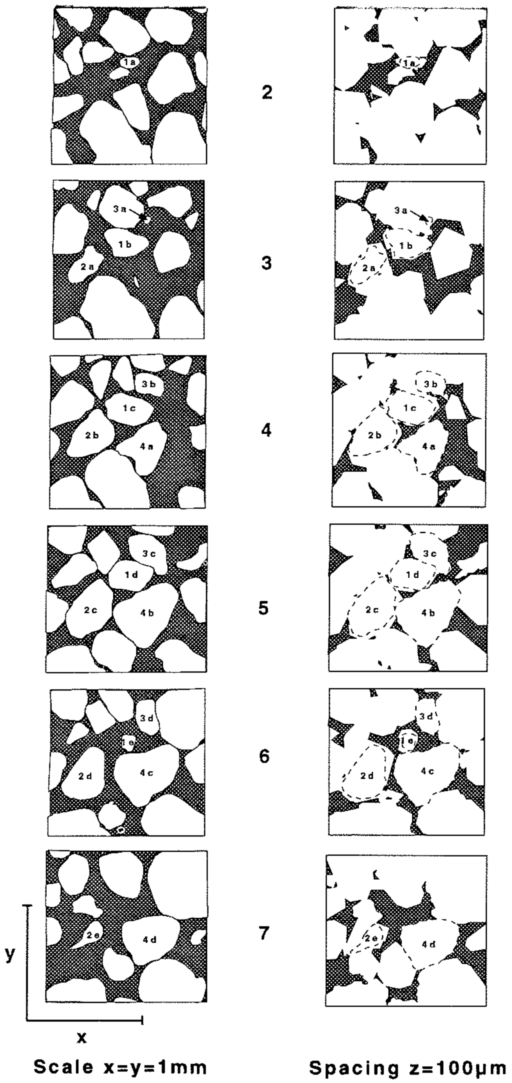


FIG. 4. Six simplified serial sections showing detrital grains and minus-cement porosity in the left hand column; total detrital and authigenic minerals and intergranular porosity are shown in the right hand column. Visually, it is possible for the operator to trace grains through consecutive sections. Grains 1–4 have been labelled as they occur through consecutive sections; their boundaries are shown as dashed on the post-cementation intergranular porosity sections. Grains 1 and 2 have well developed quartz overgrowths which increase the size of the grains and reduce the volume and interconnectivity of the local pore space. Grain 3 is a rock fragment which has undergone ductile deformation during compaction. Grain 4 has not developed a quartz overgrowth, local pore space is unaffected.

details are highlighted in the extended figure caption. The series of images highlight local variations in porosity related to processes operating at the grain-scale during compaction and cementation and demonstrate the complexity of even this apparently simple system.

Figure 5 shows the six selected sections, oriented and stacked as stereo pairs to reveal the true 3-D geometry of grains and pores. Experience shows that it is not usually possible to differentiate elements of the texture when more than six sections are used, particularly for complex images. Visually, it is possible to extract information on pore interconnectivity from such images. In order to obtain information on the permeability, more sophisticated processing is required, e.g., using genus and pore map software (see Macdonald *et al.*, 1986*a,b*), although this study did not distinguish between minus-cement and post-cementation intergranular pore systems.

Discussion

This study was directed primarily at optimizing the data collection techniques that are a necessary precursor to further investigations. An important aspect of any approach involving use of spatially-derived information is the determination of area/volume of sample necessary to characterize the parameters of interest, in particular, the heterogeneity of the porosity on the scale of the laminae. Due to limitations imposed by the available SEM/CL technology, the sample volume used in this study was too small to do this. Recent developments in this field will enable larger areas (volumes) to be investigated without the need for montages to be constructed. Clearly the development of more sophisticated image analysis software will enable a more rigorous quantification of the sorts of data presented herein. At present, we cannot attempt to model the evolution of permeability based on this 3-D data set. A discussion of the problems of up-scaling of these microscopic observations to meso- and macroscopic, sandbody dimensions, is beyond the present scope and requires similar information on pore system microgeometries for a variety of associated facies.

Summary

In this contribution, we have discussed approaches and methodology which can be used in the characterization of pore system microgeometries in the study of quartz cemented sandstones which are an important element in the development of general (predictive) models of permeability as a function of compaction and cementation history. The post-compaction–pre-cementation (minus-cement)

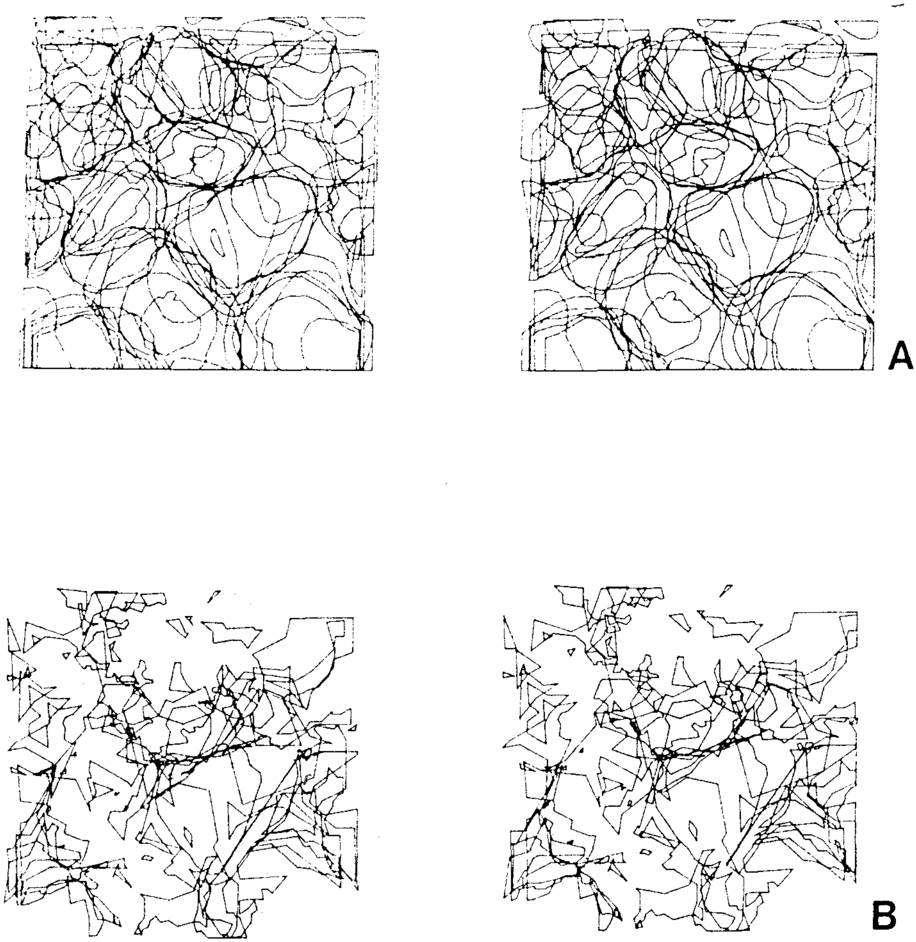


FIG. 5. Stereo pair, of the six sections shown in Fig. 4, showing the 3-D geometry of the detrital grains and minus-cement porosity (A) and post-cementation intergranular porosity (B). The respective pairs (left and right) are rotated by 4°, for each, in the x plane about the vertical. The pairs should be photocopied and cut to facilitate examination with a stereoscopic viewer.

porosity and post-cementation intergranular porosity are important textural elements which relate to burial and compaction history. In general, serial sectioning or incremental lapping are the only way to properly characterize three dimensional geometries of both. Although such procedures may appear laborious, this study has highlighted the potential for generating significant databases which will form bases for more sophisticated models of fluid flow in porous media. We have noted that orientation and spacing of serial increments are critical to the three-dimensional reconstruction of features of interest. In particular, the grain-size is important; grain orientation can be significant although this was not a factor in the Penrith Sandstone sample used in this study. In view of the amount of 'noise' present in the sample, manual digitization of features of interest was an important element of the imaging procedure and, in our opinion, is likely to continue to be so in related studies for the forseeable future.

Acknowledgements

This research was funded by B.P. Exploration. Precision lapping and image acquisition were undertaken at the B.P. Research Centre, Sunbury-on-Thames; Mark Hopkins provided training and technical assistance. Aspects of the serial lapping and imaging methodology were developed in conjunction with D.P. McKenzie and M. Spiegelman at Cambridge, and funded by the N.E.R.C. Dr David Bryon provided valuable input at different stages of this investigation.

References

Atkins, J. E. and McBride, E. F. (1992) Porosity and Packing of Holocene River, Dune, and Beach Sands. *Bull. Amer. Assoc. Petroleum Geol.*, **76**, No. 3, 339–55.

Bryant, S., Cade, C. and Mellor D. (1993) Permeability Prediction from Geologic Models. *Bull. Amer. Assoc. Petroleum Geol.*, **77**, No. 8, 1338–50.

De Hoff, R. T. (1983) Quantitative serial sectioning analysis: preview. *J. Microscopy*, **131**, 259–63.

Dott, R. L., Jr. (1964) Wacke, graywacke and matrix — What approach to immature sandstone classification. *J. Sed. Petrol.*, **34**, 625–32.

Evans, J. and Hogg, A. J. C., Hopkins, M. S. and Howarth, R. J. (1994). Quantification of Quartz Cements using Combined SEM,CL and Image Analysis. *J. Sed. Res.*, **A64**, 334–8.

Goggin, D. J., Chandler, M. A., Kocurek, G. A. and Lake, L. W. (1986) Patterns of Permeability in Eolian Deposits. Conference Proceedings, *5th Symposium on enhanced Oil Recovery of the Society of Petroleum Engineers and the Department of Energy, Tulsa, OK.*

Macchi, L. (1981) *Sedimentology of the Penrith Sandstone and Brockrams (Permo-Triassic) of Cumbria, North-West England.* Unpublished PhD Thesis, The University of Hull.

McBride, E. F. (1989) Quartz Cement in Sandstones: A Review. *Earth Sci. Rev.*, **26**, 69–112.

Macdonald, I. F., Kaufmann, P. and Dullien, F. A. L. (1986*a*) Quantitative image analysis of finite porous media: I Development of genus and pore map software. *J. Microscopy*, **144**, 277–96.

Macdonald, I. F., Kaufmann, P. and Dullien, F. A. L. (1986*b*) Quantitative image analysis of finite porous media: II Specific genus of cubic lattice models and Berea sandstone. *J. Microscopy,* 144, 297–316.

Pryor, W. A. (1973) Permeability-porosity patterns and variations some Holocene sand bodies. *Bull. Amer. Assoc. Petroleum Geol.*, **57**, 162–89.

Steele, R. P. (1981) *Aeolian Dune Sands.* Unpublished PhD Thesis, University of Durham.

Waugh, B. (1970*a*) Petrology, provenance and silica diagenesis of the penrith sandstone (Lower Permian) of Northwest England. *J. Petrol.*, **40**, 1226–40.

Waugh, B. (1970*b*) Formation of quartz overgrowths in the Penrith Sandstone (Lower Permian) of Northwest England as revealed by scanning electron microscopy. *Sedimentology*, **14**, 309–20.

[*Revised manuscript received 12 September 1994*]