ESRF

Experiment title:

Use of energy dispersive diffraction to study composition and fabric of rocks.

Experiment number:

CH-64

Beamline:

ID9-BL3

Date of Experiment:

from: 26.09.95 to: 02.10.95

Date of Report:

24.07.96

Shifts:

Local contact(s):

Dr.D.Häusermann

Received at ESRF:

30 JUL 1996

Names and affiliations of applicants (*indicates experimentalists):

Barnes*, P.
Cockcroft*, J.K.
Jacques*, S.D.M.
Jupe*, A.C.
Turrillas*, X.

Industrial Materials Group, Dept. of Crystallography, Birbeck College (University of London), U.K.

Hall*,C.

Schlumberger Cambridge Research Ltd., U.K.

Hanfland*, M.

E. S.R.F., France.

Häusermann*, D.

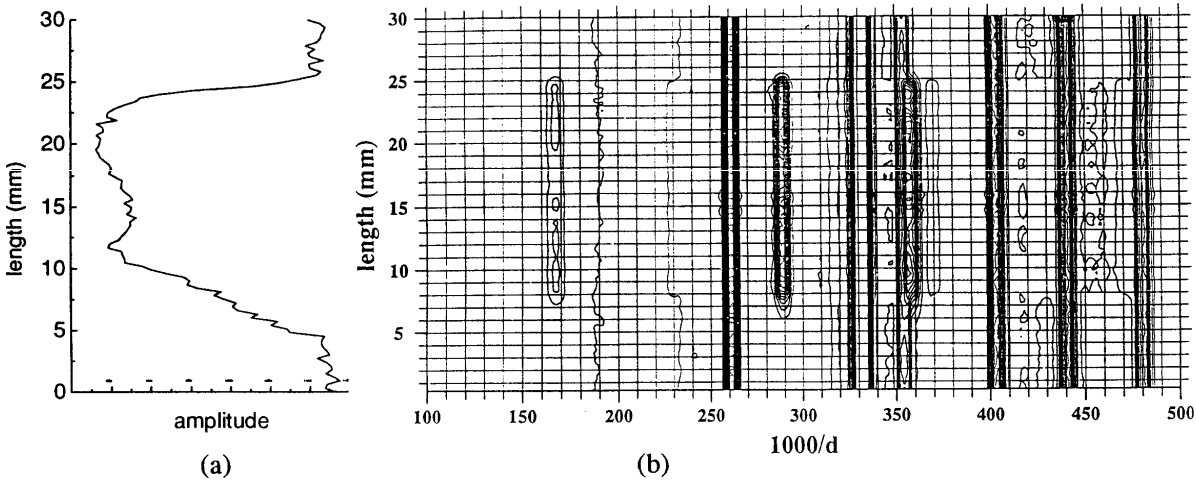
Report:

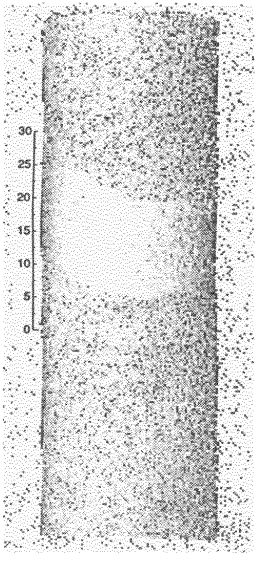
In experiment CH-64, energy-dispersive diffractometer (EDD) facilities at station ID-9 on beamline 3 were used to study the composition and fabric of intact rocks. Initially, sample penetrability, counting statistics, reproducibility, signal averaging, and ideal spacing range were investigated to determine the viability of the technique for studying bulk rock samples. A suite of homogeneous sedimentary rock cores was studied. Patterns for a set of artificial rocks, composed of mineral standards, of known densities were recorded using the EDD apparatus. Next, patterns for actual oilfield reservoir rocks were recorded. Finally, a set of 1-dimensional EDD traverses were carried out to study compositional and texture changes in various interesting rock samples.

From the initial experiments it was shown that the beam readily penetrated 50 mm diameter rock cores to give sharp identifiable diffraction patterns with data acquisition times of a few seconds. Fixed 20 angles of 1.5°,3°, and 7° gave a suitable combined d-spacing range for the identification of sedimentary rock components. Studies of homogeneous rock cores demonstrated that signal averaging was required to increase the population of crystallite orientations in the diffraction lozenge. The oil reservoir rock cores, which have complex mineralogy, produced complicated EDD patterns, but all mineral phases could be identified.

A l-dimensional traverse of a geode, shown to be composed almost entirely of quartz and calcite, demonstrated how the EDD system at the ESRF could be used to study texture: ratios of diffraction peaks were used to monitor changes in preferred orientation along the traverse.

Figure 1. Diode plot (a) and contour plot (b) of diffraction patterns in a l-dimensional traverse of





The 1-dimensional traverses produced some very interesting results: an

was shown to be composed of calcite (see Fig. 1). A phase, identified as γ–anhydrite, appears in the traverse between 7 and 25 mm, this corresponding to the visible vein; this second phase is distributed in the gaps between the oolytes. The accompanying diode output, which is a measure of the total transmission of X-rays, shows a decrease in flux through the sample in the vein region, which in this case is an indirect measure of permeability.

The work breifly outlined above (comprising of *ca.* 2500 data sets) has formed the foundations for further composition and texture mapping studies embracing 2 and 3-dimensional traverses, as well as time-resolved work, and a preliminary report has been published in (1). Further studies are scheduled for November 1996.

(1) Hall, C., Barnes, P., Cockcroft J. K., Jacques, S. D. M., Jupe, A. C., Turrillas, X., Hanfland, M., and Häusermann, D. *Analysis and Texture Mapping of Rocks by X-ray Powder Diffraction.* Analytical Communications,