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SCANNING ELECTRON MICROSCOPE EVALUATION OF DRILLING DAMAGE AND ACID TREATMENT USING UNCOATED CORE

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Abstract

A novel SEM technique allows the observation of the same pore on a core face after each step in a series of dynamic flow tests. It requires no conductive coating and facilitates core flow using a one inch (25.4 mm) diameter plug. Three separate studies were undertaken in which the procedure was used to observe the effects of drilling mud invasion, waterflooding, and matrix acidization on individual grains and pores. In the drilling mud study it was found that 2% HF removed most of the siderite weighted mud, but that mud residue and etching of the framework grains resulted in a lowered overall permeability. When seawater replaced formation water during the laboratory waterflood, there was an increase in permeability due to ionic stabilization of clays and the washing out of other loose fines. In the matrix acidization study, 10% HCl created wormholes in a fractured dolomite at elevated temperature and pressure.

KEY WORDS: Formation Damage, Well Damage, Drilling Mud, Scanning Electron Microscopy, Core Flow, Acid Treatment, Waterflood, Uncoated.

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Introduction

Formation damage (or well damage) refers to the reduction in permeability of a reservoir from its native state. It is usually the result of some adverse physical and/or chemical reaction which occurs around the wellbore during the drilling, completion, or production of a well. Some common causes of formation damage are plugging by drilling mud solids, improper treatment of clays in the formation, and scale precipitation.

Petroleum geologists and engineers conduct laboratory flow tests with core plugs in order to minimize formation damage and optimize the stimulation of the formation. A permeability to water (or oil) is measured, then the rock is caked with drilling mud, waterflooded, and/or treated with acid to determine the extent of damage or stimulation. In this way an optimum drilling and stimulation program can be designed. Whether the treatment results in an increase or a decrease in permeability, it is desirable to know the mechanism for that change. In this regard, scanning electron microscope (SEM) analysis is extremely valuable.

At least two approaches have been used for such evaluations. One method involves the removal of a slice from either end of a core plug before the plug is subjected to flow (Boyer and Wu, 1983). The slices are examined in the SEM to determine the pore and grain structure prior to treatment. After the flow test the treated core plug is examined, using the untreated ends as a reference for evaluating changes. Some changes can be clearly viewed in this manner. These include plugging of pores with a foreign material and significant framework dissolution. Since one does not view the same grains or pores after treatment, there are instances where the effects may not be detectable. There may have been a small amount of calcite dissolution or precipitation in a carbonate cemented rock. Also, one could not tell if a pore that is open was previously filled with loose clay or other fines.

Another method of SEM evaluation was used by Thomas et al. (1976). They did relocate the same point in a sample before and after treatment. Their system worked fairly well but had two drawbacks. The samples examined were not used in the flow tests, although parallel core flow tests were run.

Instead, they used cubes of rock which were saturated (rather than flushed) with the treating solutions. As such, the effects of flow on particle movement and filtrate straining could not be evaluated. There is also a potential problem with the acid treatment used to remove the conductive coating. An aluminum coating was applied with a vacuum evaporator for the initial SEM examination. It was removed by immersion of the sample in 28% HCl prior to treatment. The authors demonstrated that this caused no morphological change in kaolinite or illite. But certainly the acid would have an adverse effect on carbonates and chlorite. Also, one cannot be certain that the surface chemical properties of the clays were not changed.

Materials and Methods

The procedure for encasing core plugs in metal sleeves with screens on each end was developed for measuring flow properties of unconsolidated rock (Swanson and Thomas, 1980; Mattax et al. 1975). This "rock" has insufficient cementing material to support itself at surface conditions, a common state for petroleum reservoirs in the Gulf of Mexico. Core is usually recovered from the well with a rubber sleeved core barrel. These full diameter cores are immediately frozen. Smaller cylindrical plugs are then drilled in the laboratory using a liquid nitrogen coolant. The plugs are inserted into a thin metal sleeve with wire screens on both ends. The sleeved core plug is mounted in a standard core holder in which reservoir pressure conditions are reestablished before the rock is allowed to thaw. It was suggested that this might be an ideal configuration for viewing uncoated core plugs in the SEM. The metal would provide electrical conductivity and the screen would serve as a coordinate grid for relocating pores and grains. Freezing is not required for consolidated rocks, such as those used in this study.

The lead sleeves used are one inch (25.4 mm) in diameter with 150 μm thick walls and contain about 2% antimony. The wire screen is number 40 mesh, with wire thickness of 254 μm and mesh opening size of 381 μm. Stainless steel screen was used at first. After corrosion problems developed, Monel 400 screen was substituted.

One inch (25.4 mm) diameter, one inch (25.4 mm) long, core plugs were inserted into the slightly stretched sleeves. Screens were placed on either end and the tube ends were folded over (Figure 1). For optimum contact between the rock, sleeve, and screens, the assembly was placed in a Hassler sleeve core holder and 300 PSI (2.07 x 10^6 Pascal) of nitrogen gas confining pressure was applied.

Each end of the core was marked at two positions (top and left) with indelible ink and a scribe. Using low magnification SEM micrographs, one can count the number of screen openings down and across to relocate grains and pores of interest.

The screened plugs were examined with an AMRAY 1200C SEM in the secondary electron mode. Instrument conditions were: 5 kV accelerating potential, 25 microamp emission current, 10° tilt, and 15 mm working distance. Low voltage operation was necessary to minimize charging artifacts in the photographs. After the final flow step, some samples were sputter coated with 200 Å of gold to optimize the images.

Three examples of studies which employed this technique are described below. The first involved drilling mud damage and subsequent cleanup using Berea Sandstone. The second illustrates some effects of a laboratory waterflood on a sandstone reservoir. Finally, matrix acidization in a carbonate reservoir was followed.

Results and Discussion

Drilling Mud Cleanup

A one inch (25.4 mm) diameter Berea Sandstone core plug was encapsulated as described. Eight pore/grain sites were photographed with the SEM prior to flow tests. A siderite weighted drilling mud was used to build up a mud cake on the inlet side of the core using a 50 PSI (3.45 x 10^5 Pascal) overbalance. Damage to the inlet face was documented by SEM. The core was then treated with a 6% HCl preflush and a 2% HF acid wash, and examined after each step.

Return permeability was determined before and after mud-up using mineral oil. To remove mineral oil from the sample for SEM examination, Soxhlet extraction with methylene chloride was used. The solvent was then evaporated by placing the sample in a vacuum oven at 50°C. After acid treatment the sample was washed by water flow, then dried in the vacuum oven. For an explanation of return permeability and fines migration, see Gabriel and Inamdar (1983). Soxhlet extraction and other methods of core cleaning have been reviewed by Gant and Anderson (1985).

Siderite (iron carbonate) was used in this evaluation because it is acid soluble unlike barite, the commonly used mud weighting material. Bentonite clay was also present, as it is added to most muds for its sealing properties. Drilling mud has a number of functions including lubrication, balancing reservoir fluid pressure, and transporting rock cuttings.

Initial mineral oil permeability of the Berea Sandstone plug was 325 millidarcies (md). During mud-up the permeability dropped to 85 md before the mud cake caused complete blockage of the core face. After the HF treatment the return permeability was restored to 280 md. This confirms the effectiveness of the cleanup and the presence of some residual damage.

Good quality photomicrographs were obtained of the uncoated rock surface below the screen. Grain and pore features were distinct (Figure 2a). Charging was a problem at a few sites. Relocation of the examination points was facilitated by using the screen as a coordinate grid. After mud-up many of the pore spaces were occluded by drilling mud (Figure 2b). Some grains were dislodged and removed during mud flow. Charging of the sample in the SEM was most severe at this stage.

The next step was a preflush with 6% HCl. Several pore volumes were displaced followed by a short shut-in. Due to the presence of bentonite gel

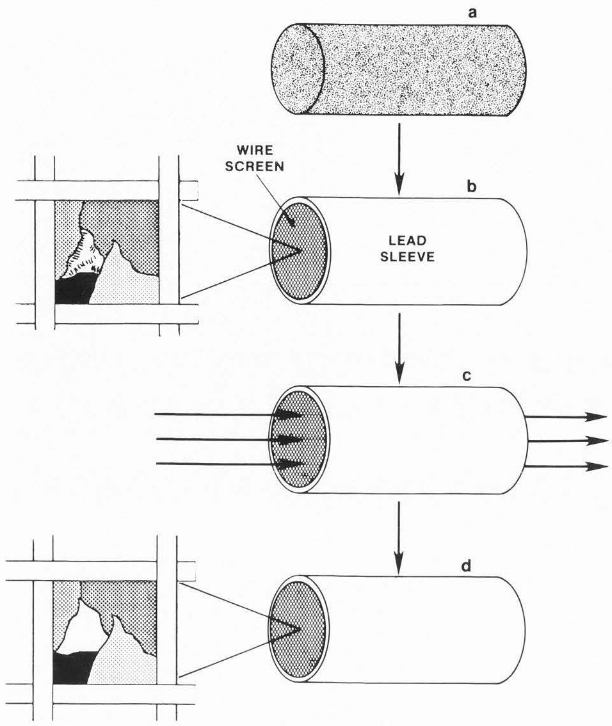

Figure 1. Schematic diagram of encapsulation and examination process: a. One inch (25.4 mm) diameter core plug drilled from larger rock sample; b. Plug inserted into lead tube with wire screens on ends. Sites on core face are located and photographed; c. Rock is subjected to a single treatment step (e.g. mud-up, acid flow); d. Sites are relocated and the effects of treatment evaluated.

in the drilling mud, the HCl was not effective in mud removal (Figure 2c).

The sample was examined again after the final treatment, flow displacement with 2% HF. This was very effective in removing drilling mud (Figure 2d). The remaining mud residue will, however, restrict flow by bridging pore throats, adding to mobile fines, and increasing grain surface roughness. The HF treatment also caused pitting in framework grains of the rock, especially feldspars.

Sandstone Reservoir Waterflood

In the second study a flow test was conducted with a petroleum reservoir sandstone to evaluate the effects of displacing formation water (22 gm/l total dissolved solids) with seawater (33 gm/l TDS), as would occur in a field waterflooded with seawater. This was simply a water-water and water-rock compatibility test and, as such, did not include oil.

The petroleum reservoir sandstone had an initial permeability to formation water of 72 md. A dilute seawater (18 gm/l TDS) was used to displace the formation water. The permeability increased to 76 md but dropped to 67 md after a two day shut-in. The increase was probably due to the washing out of loose material, and the subsequent drop may have been caused by clay sensitivity at the lower salt concentration. Displacement by full strength (33 gm/l TDS) seawater raised the permeability to 82 md, perhaps due to ionic stabilization of the clays. Finally, the core plug was displaced with fresh water, causing a permeability drop to 72 md.

The SEM photomicrographs show no more than a washing effect on the core plug. Loosely held clays and fine chert particles were removed from grain surfaces and pores on the inflow side of the plug (Figures 3a and 3b). On the outflow end of the plug, the number of those fine particles increased. In the field these fines may cause pore or perforation blockage of production wells. Interestingly, kaolin clay booklets which were tightly packed into pores showed little tendency to move or degrade (Figures 3c and 3d). Natural siderite in pores was also unaffected by the seawater flood.

Carbonate Reservoir Acidization

The final example is a study of the effects of an HCl "matrix" stimulation on a naturally fractured dolomite reservoir. In matrix acidization of carbonates, wormholes are created which extend past the drilling damaged zone to increase reservoir drainage.

A lead sleeved core plug was saturated with 10% HCl plus a corrosion inhibitor and an iron chelating agent. The acid solution was then pumped to a pressure of 1500 PSI (1.04×10^7 Pascal) at 180°F (82°C). After a 1h shut-in it was flushed with 2% potassium chloride. The salt residue was removed by Soxhlet extraction prior to SEM examination.

The core plug was taken from a brecciated zone and had an initial air permeability of 56 md, much higher than average for this reservoir. After acid treatment the permeability rose to 6200 md due to the formation of wormholes. Areas of open fractures were often the sites of wormhole development.

The effect of the acid on grain surfaces and pores is shown in Figures 4a and 4b. Pores are enlarged by etching back the dolomite grain surfaces. Quartz and other less soluble minerals are exposed and perhaps liberated from the matrix.

Conclusions

At various stages in a core flow test one can observe the microscopic features which are responsible for the macroscopic flow properties. This can be done without altering the core by the application of a conductive coating. The method described has general applicability in core flow studies for reservoir damage evaluation and should be used in conjunction with measurements of porosity, permeability, and pore throat size. The SEM used was an inexpensive model, available for about $50,000. The results show that this technique does not require a highly sophisticated instrument. Research grade microscopes can produce better results with uncoated core due to higher gun and chamber vacuum, and better beam control.

Acknowledgements

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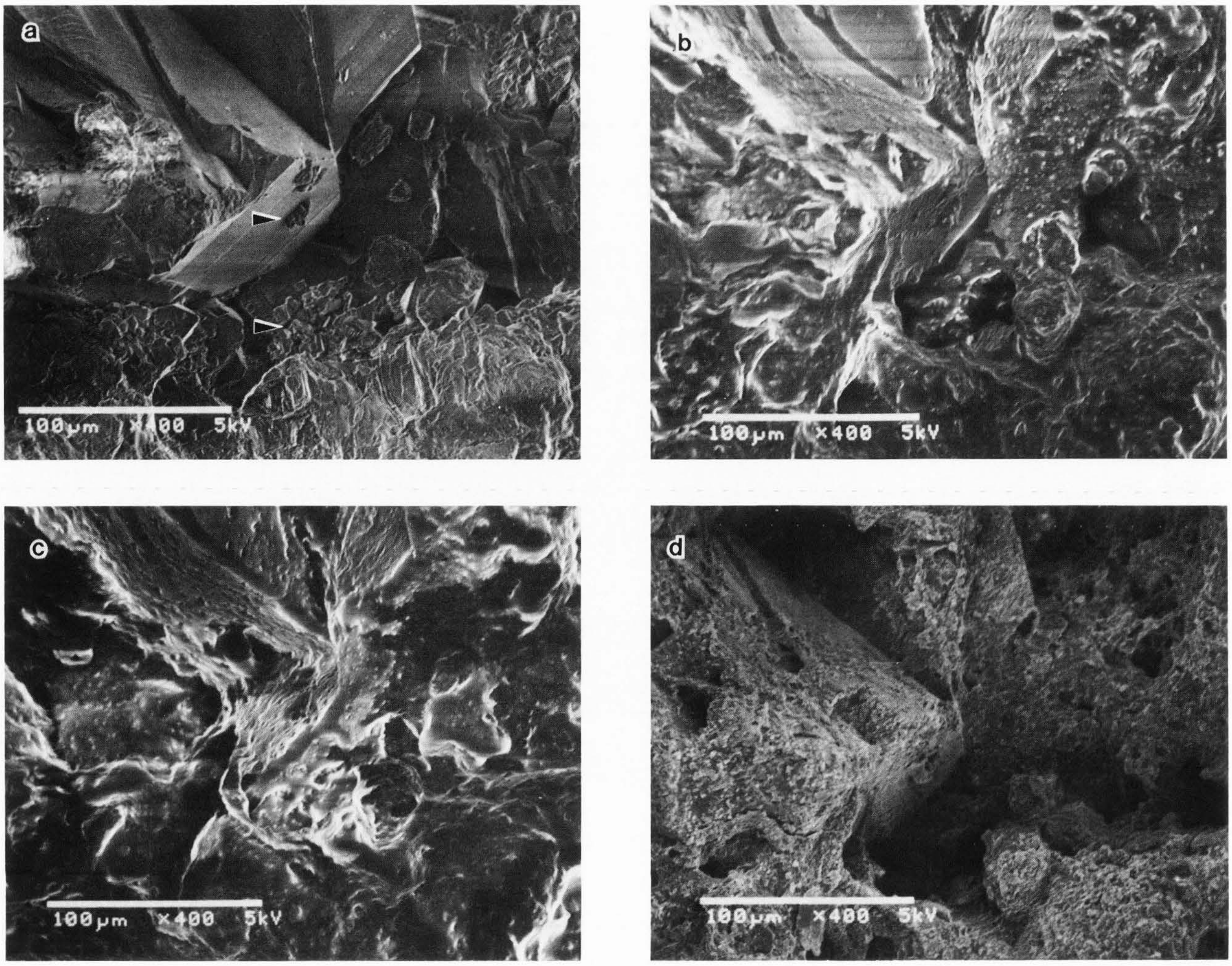

Figure 2. SEM micrographs of Berea Sandstone. Scale Bars = 100 µm: a. Pre-treatment. Angular faces are quartz overgrowths. Arrows indicate kaolin clay; b. After mud-up. Drilling mud coats surfaces and bridges pores; c. After 6% HCl treatment. Damage appears to have increased as mud moves into pore at center; d. After 2% HF treatment. Pores have been partially re-opened and some of the native clay was removed. Surface roughness and pore bridging will cause flow restriction.

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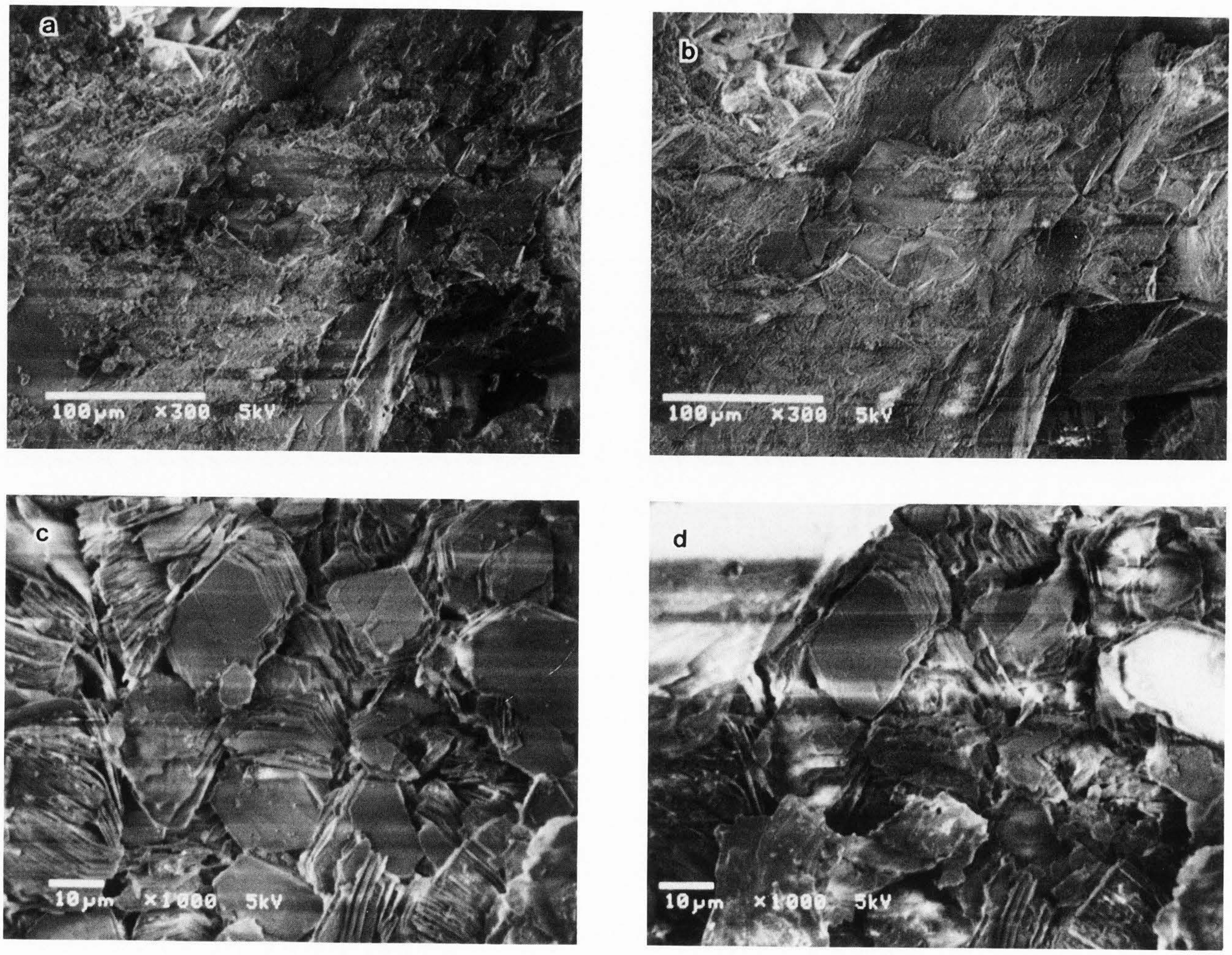

Figure 3. SEM micrographs of a petroleum reservoir sandstone. Pairs of photographs show the same location before and after formation water and seawater flow: a-b. Loose clays and fine chert particles were removed from quartz grains by flow at this inlet end site. Scale Bar = 100 µm; c-d. Tightly packed kaolin clay booklets are essentially unaltered by fluid flow in the laboratory. Notice the increase in loose debris at this outflow end site. Scale Bar = 10 µm.

Discussion with Reviewers

C. W. Keighin: In the section on waterflooding in sandstones, you mention effects of seawater (with higher TDS) displacing formation water. What are the constituents which contribute to higher TDS in seawater? What are the effects of higher TDS? Where are these effects documented?

Authors: Although components of the formation water and seawater differ somewhat, the higher TDS of seawater is mainly due to higher concentrations of sodium, chlorine, and magnesium ions. The effect of displacing with a higher TDS is often a chemical stabilization of the clays, as indicated by the higher permeability. Also, chemical differences between the formation water and seawater resulted in a small amount of scale precipitation in flow tests run at higher temperatures than used here (21°C). Gabriel and Inamdar (1983) have demonstrated some effects of salinity change on fines migration.

C. A. Callender: Were the solids in the effluent analyzed by SEM/EDS techniques to determine the nature of the fines migration problems in the waterflood tests?

Authors: On other tests in that series the effluent was passed through a 0.20 µm Nucleopore filter. SEM/EDS analysis of the filter papers revealed fine chert particles, and occasional kaolin flakes and illite/smectite clays. Some metallic corrosion products were also found. But pore blockage can occur with no fines leaving the core plug because particle diameters are often larger than pore throats.

A. H. Johnson: Do you feel that the repeated pressure changes encountered by the core plugs (reservoir pressure, 1 atmosphere room pressure, SEM vacuum) have a significant effect on the results of your analysis?

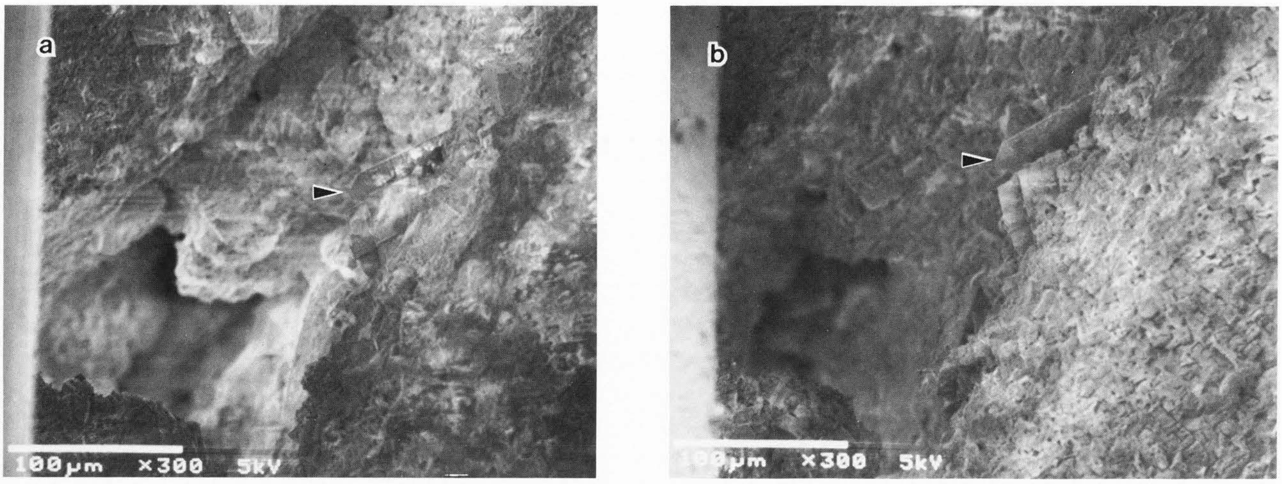

Figure 4. SEM micrographs of a carbonate reservoir core plug. Pair of photographs show the same location before and after acid treatment. Scale Bar = 100 μm: a. Note the elongate quartz crystal (arrow), partly exposed dolomite rhombs, and generally smooth surface of the rock; b. After acidization the dolomite surface has been etched back and the quartz crystal (insoluble in HCl) is unaffected.

Authors: Let us assume that the flow tests were run under conditions which duplicated the petroleum reservoir, and only consider whether the microscopic observations accurately reflect the results of the flow tests. The slight grain volume difference induced by the pressure change should not affect the SEM analysis of those tests. The high vacuum environment will have the same drying effect on hydrated minerals which occurs in normal SEM operation.

R. Klimentidis: After the 2% HF in Figure 2d the sample shows surface roughness. What is it due to? Mostly quartz dissolution or migrated fine particles coating?
Authors: The rough surface texture is a combination of acid etching and mud coating. Grain removal by dissolution is apparent on the right side of Figure 2d. Fine particles liberated from the rock and the drilling mud also contribute to the surface roughness.

A. H. Johnson: In the Berea Sandstone study, you state that 2% HF was very effective in removing drilling mud. Where is it removed to? In the subsurface, could the removal of mud near the well bore create permeability problems at some distance from the well bore?
Authors: Several steps can be taken in the field to prevent precipitation from spent acids. They include the addition of chelating agents, minimizing acid volumes, and recovering as much acid as possible after treatment.

C. A. Callender: The described method appears to be valid for observation of changes on the face of the core, but shouldn't X-ray diffraction mineralogy be performed at some point in the analysis to determine the composition and amount of clay minerals, etc. to more accurately evaluate the effects of the acid or waterflood tests?
Authors: X-ray diffraction was used on other core plugs in both tests, but it did not provide microscopic information on changes within pores and on grain surfaces at relocatable sites. It is a destructive analysis and would require multiple slices to determine if any movement has occurred. Also, a 2% change in mineral content may not be detectable by X-ray diffraction.

C. A. Callender: Have the authors considered the use of a backscattered electron detector to image uncoated, non-conductive samples? BSE detectors have fairly high resolution and are generally not as sensitive as secondary electron detectors to the magnetic field created by a charging surface.
Authors: Backscattered electron imaging is inherently lower in resolution than secondary electron imaging due to the greater depth of generation. Our solid state backscattered detector has limited topographic sensitivity and requires higher accelerating voltages than is practical with uncoated rocks. A scintillator-type backscattered electron detector might be useful in this application.