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Innovative Formation Damage Sample Evaluation Techniques0

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تقنية مبتكرة لتقييم تضرر عينة التكوين الصخري

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لقد تم استعمال التقدم في اختبارات الغمد عند الضرورة المكمنية التي تجري على عينات موائع المكمن وأخرى لبية في محاكاة العمليات القاعية مثل الحقن بمياه البحر واستخدام طينة الحفر أو كبس المواد الكيميائية، للتعرف على أي ضرر يحدث للتكوين الصخري (فرانسز وآخرون 1995).

وقد تم في حالات متعددة استعمال طريقة النفاذية الراجعة للتعرف على متى حدوث تضرر التكوين الصخري ولكن هذه الطريقة لا يمكن التعرف بواسطتها على الميكانيكية الفعلية مثل حجم الترسب، مانع المستحلب البوليمر، عزو المواد الصلبة وكبح البوليمير الحيوي / خلية، وإنتاج كبريتيد الهيدروجين. ولكي يتم التعرف على هذه الميكانيكيات والحصول على تفسير مفصل لبيانات النفاذية الراجعة يجب إجراء بعض الإختبارات التحليلية الإضافية التي تشمل التصوير الشعاعي بمساعدة الحاسوب C.A.T، رقائق المقطع الصخري، المسح بواسطة المجهر الالكتروني لعينات جافة وأخرى مائعة عند درجات حرارة منخفضة ودراسة تكاثر الميكروبات.

يمكن إجراء هذه التحاليل قبل وبعد الإختبارات، للمقارنة من أجل التعرف على ميكانيكية الضرر. لقد تم مناقشة تقنيات متعددة وكيفية استعمالها لتفسير بيانات النفاذية الراجعة بالتفعيل في هذه الورقة.

Abstract: *Advanced laboratory reservoir conditions flood tests, performed on live reservoir fluids and core materials, have been used to simulate downhole operations such as seawater injection, drilling mud application and chemical squeezes to identify any associated formation damage mechanisms (Francis et al, 1995).*

In many cases return permeabilities are used to identify when formation damage has occurred, but in itself will not identify the actual mechanism such as scale precipitation, emulsion polymer blocks, solids invasion, bio-polymer/cell blockage and H_2S production. In order to identify these mechanisms and to fully interpret the return permeability data, it is necessary to undertake additional analytical techniques, which include C.A.T. scanning, petrographic thin sections, dry and cryogenic S.E.M. and microbial growth studies.

These analyses can by performed before and after tests for comparison, to identify the damage mechanisms. The various techniques and how they are used to fully interpret the return permeability data are discussed in this paper.

INTRODUCTION

The use of laboratory reservoir conditions flood tests to predict possible downhole formation damage mechanisms, has advanced considerably over the past few years with the development of hastelloy alloy. This alloy does not readily corrode to even aggressive fluids such as HF. As a result, hastelloy

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is used for laboratory flood test rigs and downhole production tubing where corrosion resistance is required, as discussed by Petersen and Bluem (1989) and Asphanhani *et al.* (1991). In addition to the advances in flood test rig design and metullargy, analytical techniques such as dry and cryogenic S.E.M., thin sections, microbial analysis and C.A.T. scanning have allowed the identification of formation damage mechanisms in laboratory core flood tests to fully interpret the return permeability data obtained. Once the formation damage mechanisms are identified using these techniques, it is possible to identify the best preventative/remedial treatments to use downhole to minimize the problems and to pre-screen these treatments in the laboratory using core flood tests, prior to their use in the well. In order to perform effective laboratory formation damage flood tests, to simulate the situation downhole, it is absolutely essential to use carefully selected cores to represent the major injection/production intervals. It is also important to use cores, field muds, crude oil and downhole chemicals from wellsite, so that the laboratory flood tests closely match these materials downhole, with all the chemical additives present. By utilising these materials and by mimicking the relevant temperature, pressure, mud hydrostatic pressure and chemical application temperature and pressure, the laboratory flood tests will closely match any possible formation damage mechanisms which may occur in the well. Through analysing the core before the flood test is performed (untreated samples) and after (treated samples), it is possible by utilising C.A.T. scans (computer aided tomography), thin sections, dry and cryo S.E.M. (scanning electron microscopy) and microbial analysis to determine, the formation damage mechanisms and relate these to the return permeability and fluid loss volumetric data (King and Adegbesan, 1997) and Francis (1997).

COMPUTER AIDED TOMOGRAPHY ANALYSIS

The analytical technique of computer aided tomography has been used for many years to determine, by non-destructive X-ray scans, formation damage mechanisms such as mud solids invasion (Krilov, 1991).

This technique is used primarily to identify from whole cores the best plugging orientation and sites, to ensure that any sedimentary laminations run parallel to the length of the plugs. A simple planar X-ray scan is used to show the position of any circular AXIAL slice (½ cm thick) (Fig. 1a) and is not used for interpretation purposes.

The AXIAL slice (Fig. 1b) is used to show any vertical fractures, burrows and depth of drilling mud solids invasion, so that these can be avoided when taking the plugs, unless they are representative of the entire reservoir section and would thus be included.

The two longitudinal X-ray slices (Figs. 1c and 1d), taken at 90° to each other, run vertically down the whole core length. These slices are used to identify sedimentary laminations, pebbles and cemented patches so that the plugging tool can be oriented to ensure that any laminations run parallel to the length of the plugs, since fluid flow in the reservoir would be along these laminations. It is also important that permeability barriers to plug fluid flow such as large pebbles, highly cemented regions and clay wisps are avoided as this would not represent the matrix of the reservoir, where fluid flow would occur.

The C.A.T. scan technique is also very useful for determining the best locations to plug unconsolidated cores, as these can be performed with the core still present inside the aluminum core barrel liner, thus avoiding disturbing the unconsolidated core (Figs. 2a, 2b, 2c and 2d).

The section of core in (Fig. 2a) shows that the unconsolidated core is well preserved with no fracturing. The axial slice (Fig. 2b) shows that the annular space between the barrel and core is well preserved (white region) and the drilling mud (black) has not invaded the core to a high degree and thus good plugs can be obtained from this core section.

The section of core immediately below this is highly fractured (Fig. 2c), the annular space is lost and mud solids invades the fractures (Fig. 2d) showing compaction of this region of core has occurred and thus no suitable plugs could be taken from this region of the core.

The C.A.T. scan technique used in this fashion ensures that plugs best represent the reservoir fluid flow paths, contain the relevant sedimentary features, and are <u>not</u> invaded by drilling mud solids prior to the laboratory flood test being performed.

THIN SECTION ANALYSIS

The thin section technique is used to analyse rock samples prior to testing (untreated samples) and again

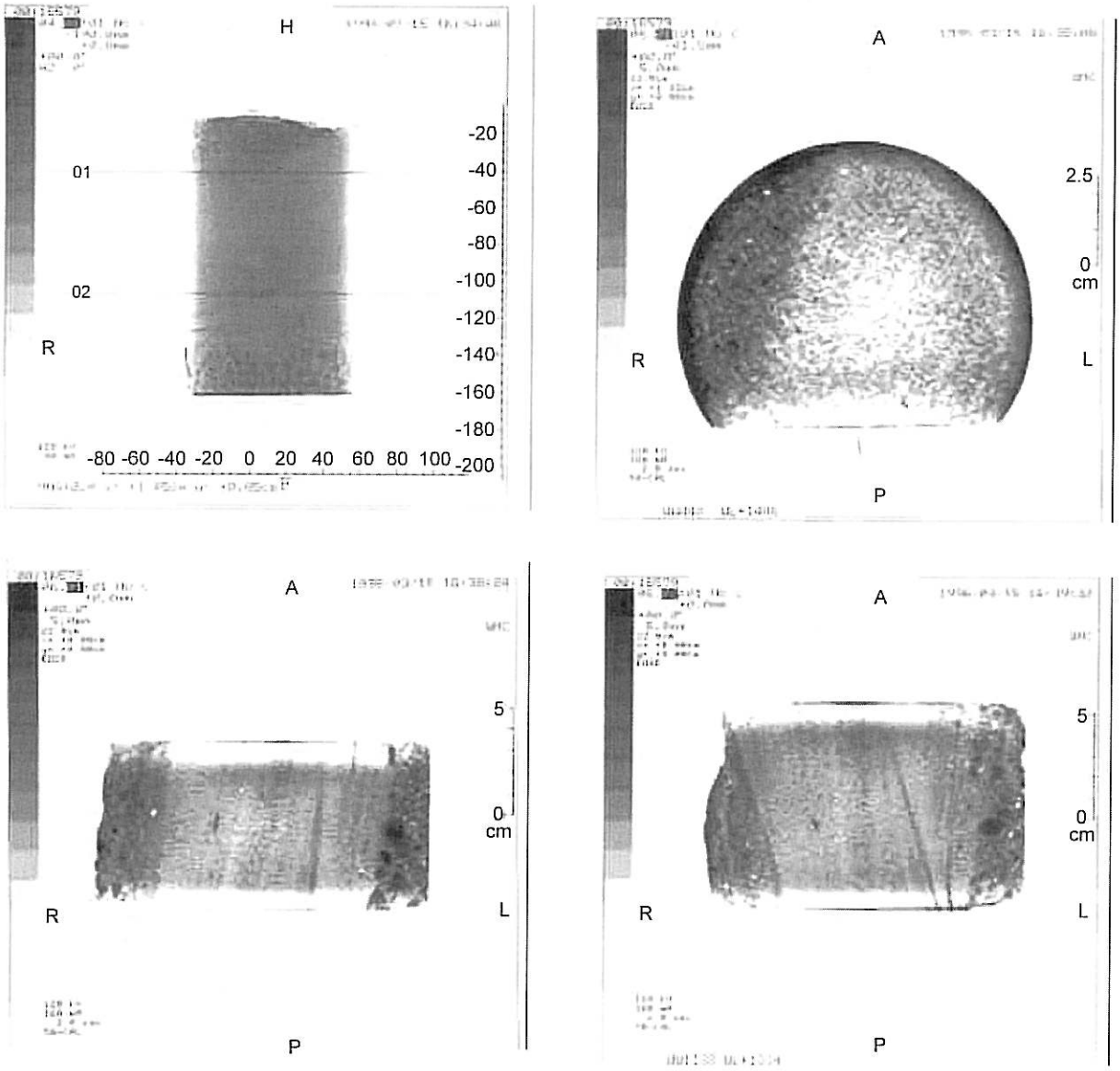


Fig. 1a. Planar X-ray scan shows the position of any axial slices (horizontal lines).
Fig. 1b. This axial slice shows that drilling mud solids have invaded only the outer rim of the core. A bedding surface cross-cuts the axial slices (higher and darker areas).
Fig. 1c. Longitudinal slice showing a conglomerate with rounded pebbles. Central Portion is of a laminated sandstone.
Fig. 1d. Longitudinal slice at 90° to Fig 1c.

after the flood test is performed (treated samples) to determine any solid formation damage mechanism. This technique uses a thin rock slice (30μm thick) which is optically transparent, mounted on a glass slide, to examine the natural distribution of clays and cements in the rock before a flood test and lengthways down a plug at the end of flood test analysis for comparison. The thin sections can identify solid formation damage mechanisms such as drilling mud cake development and degree of invasion (Fig. 3a), acid dissolution of mud during clean-up with associated mud fines migration (Fig. 3b), and acid matrix wormhole dissolution with clay mineral fines migration (Figs. 3c and 3d). These thin sections observe damage mechanisms optically in two dimensions only (30 μm in thickness), whereas dry S.E.M. observes this damage in three dimensions.

DRY S.E.M. ANALYSIS

The dry S.E.M. (scanning electron microscope) analysis is performed on a rock chip prior to flood test analysis (untreated sample) and after flood test

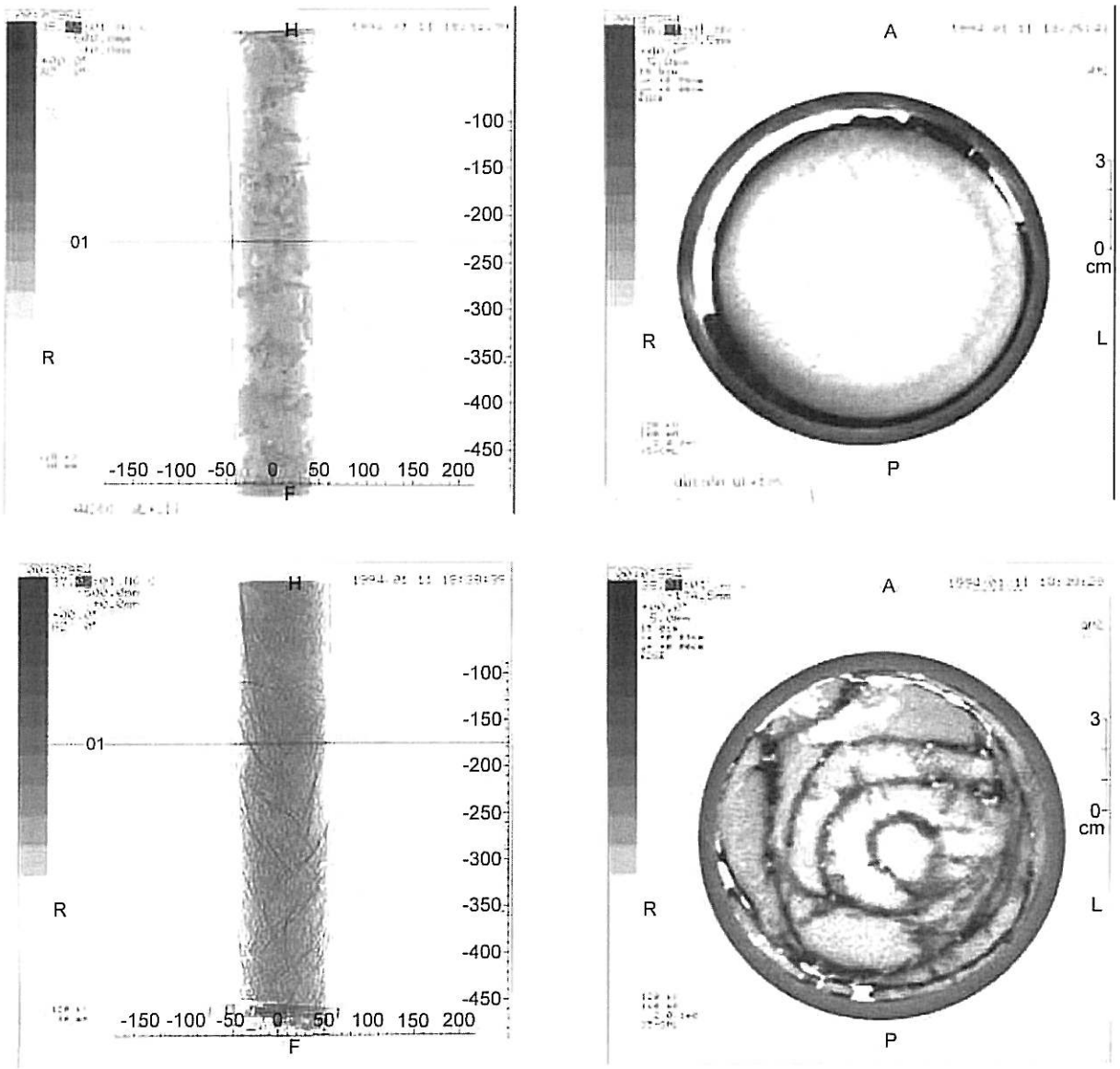


Fig. 2a. Plannar X-ray scan shows the position of the axial slice shown in Fig 2b.
Fig. 2b. The annular space between the barrel and core is well preserved (white region) and the drilling mud (black) has not invaded the core. Good plugs can be obtained from this section of core.
Fig. 2c. Abundant fractures are visible in this section of core.
Fig. 2d. No annular space is visible and mud solids (black) have invaded along fractures. No suitable plugs can be obtained from this section of core.

analysis (treated samples) on selected regions of the plug (injection/production end faces of the plug), to identify solid formation damage mechanisms in three dimensions. Drilling mud cake development and solids invasion (Fig. 4a), sodium formate polymer drilling mud cake and strands blocking and bridging pores (Fig. 4b), and excessive scale inhibitor retention (Fig. 4C) as discussed by Carbone *et al* (1999), can be observed by dry S.E.M. analysis. Incompatibility between injection water and the natural reservoir formation brine can result in scale precipitate formation, such as aragonite (calcium carbonate), which can significantly reduce the permeability of the rock and seriously reduce the injection rate (Fig. 4d). Fluid losses from drilling muds and clean-up chemicals can cause significant collapse of natural pore lining clays, such as platy/hairy illite, causing a stimulation in the near wellbore region if the clay collapses onto the pore walls (Figs. 4e and 4f) or can cause a dramatic reduction in permeability if the clay collapses onto the pore throats.

The dry S.E.M. analysis can therefore be used to identify solid formation damage mechanisms such as scale precipitation, mud solids invasion, clay migration

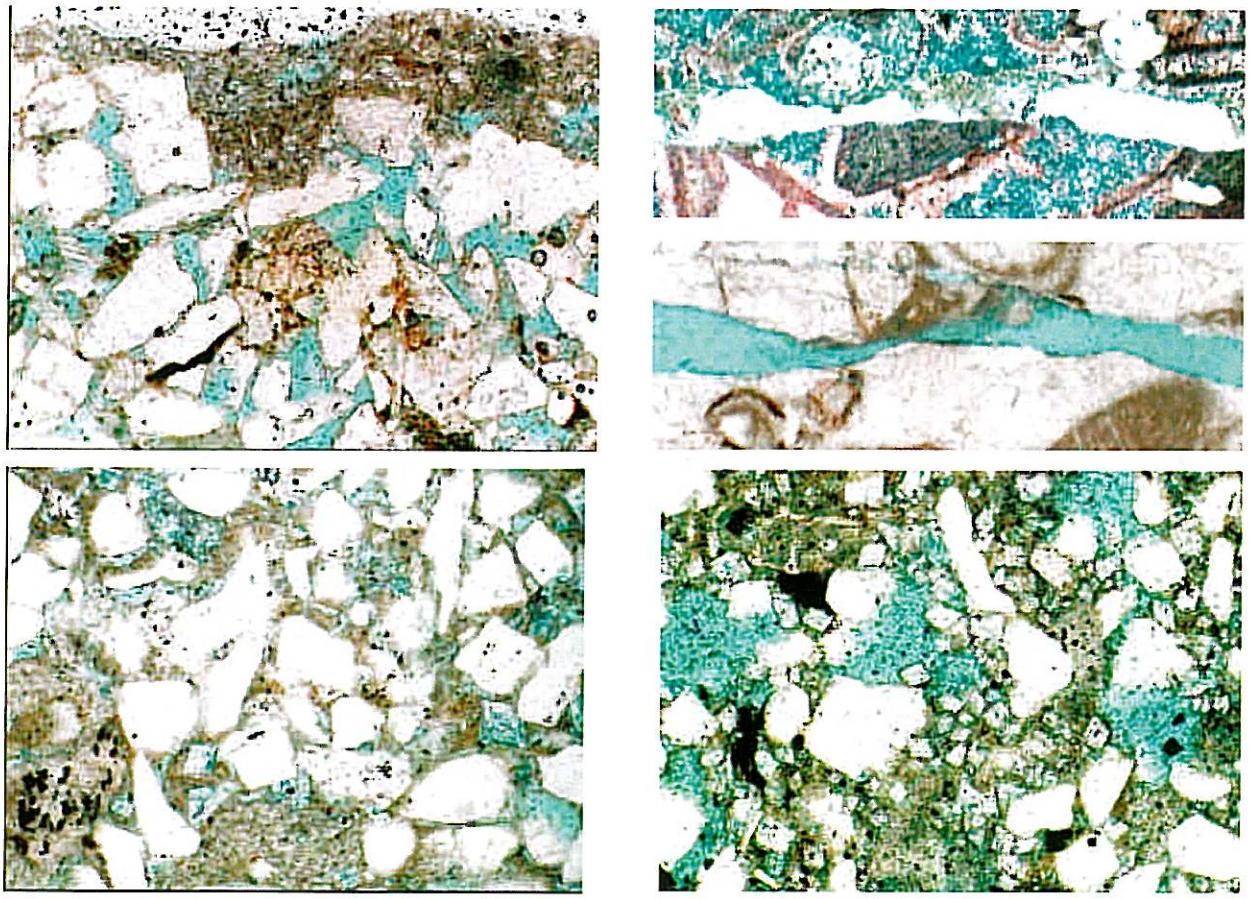


Fig. 3a. Remnants of dolomite weighed mudcake are present on the injection face of the plug. The mud solids fill only those pores exposed at the injection face, with no obvious evidence for mud solids invasion.
Fig. 3b. (Top) Foamed mud has been applied to a naturally fractured sample. Mud solids line and bridge the fracture. (Bottom) Application of acid has caused the removal of the mud, with the solids being smeared along the facture surface.
Fig. 3c. Thin section analysis prior to acid stimulation showed the sample consists of common quartz grains and clay matrix partially replaced by dolomite.
Fig. 3d. After acid stimulation of the sample in Fig 3c dolimite rhombs have been locally dissolved with the formation of wormholes. Acid application has also caused local clay dispersal.

and collapse, and cement dissolution textures in three dimensions.

CRYOGENIC S.E.M. ANALYSIS

The cryogenic S.E.M. (scanning electron microscope) analysis is used to show fluid formation damage mechanisms such as micro-emulsion blocks, water/oil blocks, asphaltene precipitation and polymer blocks, often resulting from fluid losses to the reservoir. These fluid losses can originate from the drilling mud, clean-up/completion chemicals and from chemical squeeze treatments such as scale inhibitors directly into the oil leg of fields. The technique uses fully saturated rock chips taken prior to flood test analysis (untreated samples) and from the saturated plug after completion of the flood test. These saturated rock chips are then cryogenically frozen in liquid nitrogen and placed on a cooled stage (-170°C) in a cryogenic S.E.M. The samples are freshly fractured inside the S.E.M. prior to being analysed. These cryogenic S.E.M. rock chips can be used to show fluid formation damage mechanisms such as drilling mud filtrate retention along channelised flow paths within gas field samples (Figs. 5a and 5b). Water blocks from formate mud fluid losses being retained in a gas field sample (Figs. 5c and 5d), and micro-emulsion formation with KCl brine losses, from a KCl polymer mud system and KCl brine clean-up chemicals (Figs. 5e and 5f) can also be observed.

The cryogenic S.E.M. samples can thus be used to identify any fluid formation damage mechanism providing the liquid phases are immiscible with each other i.e. Oil/water, polymer/water, asphaltene/oil separation.

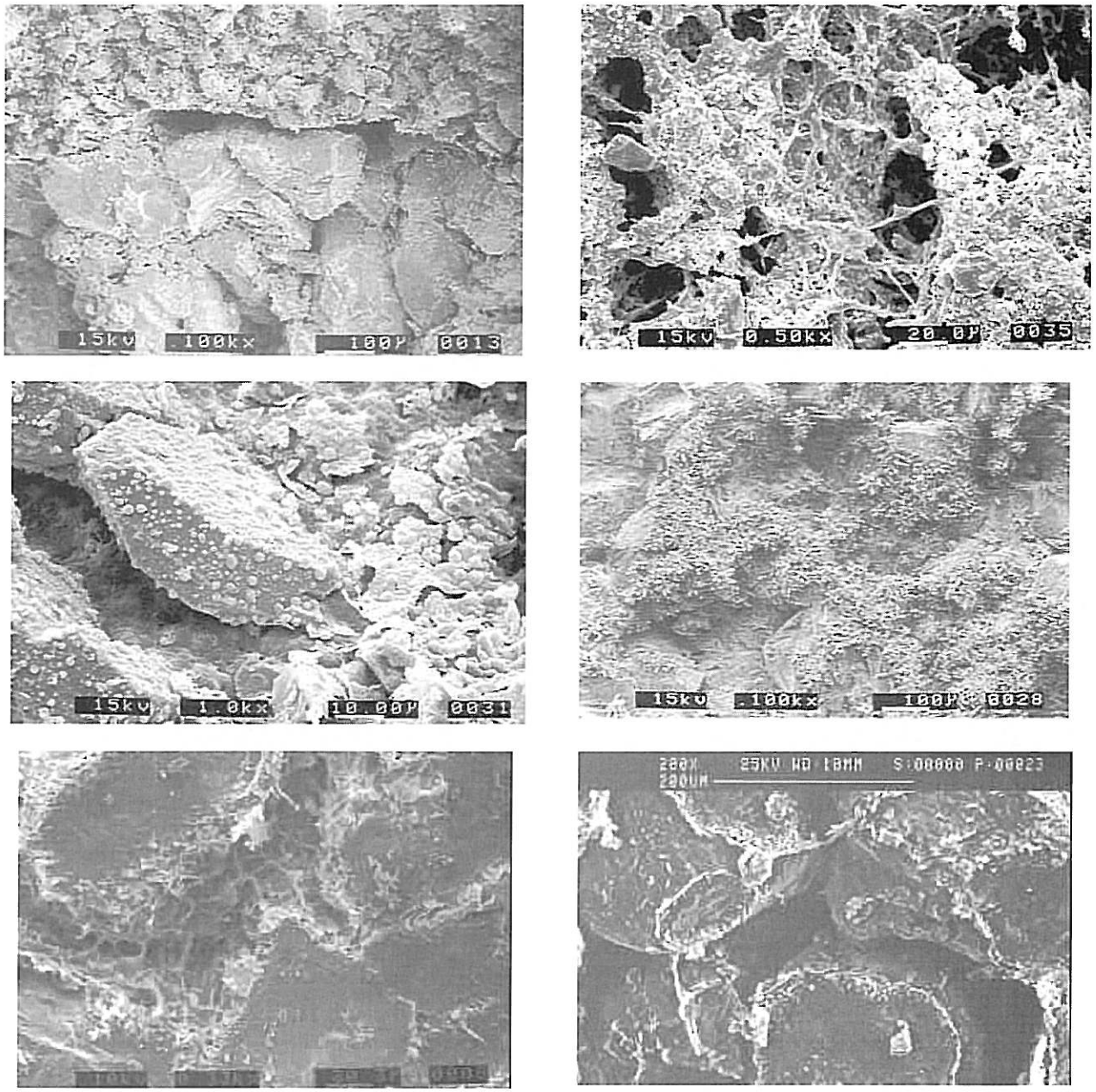
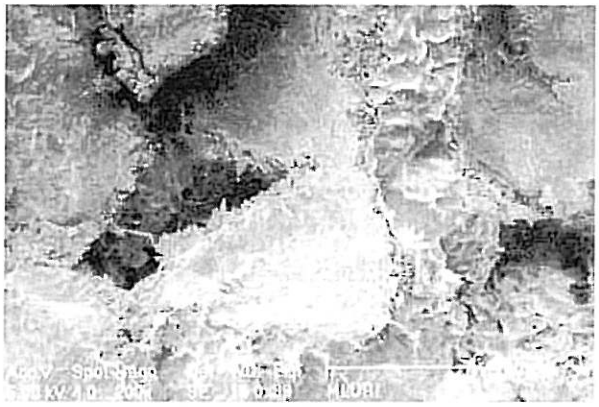
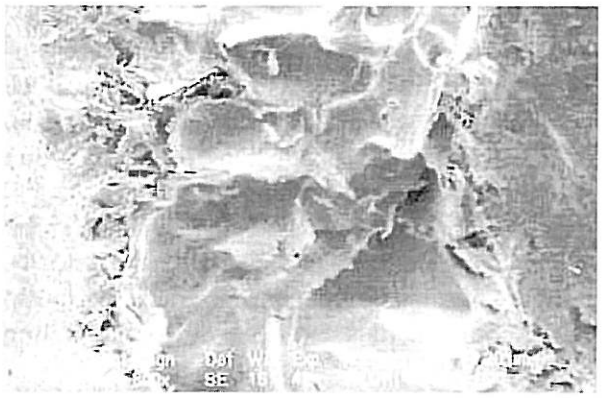


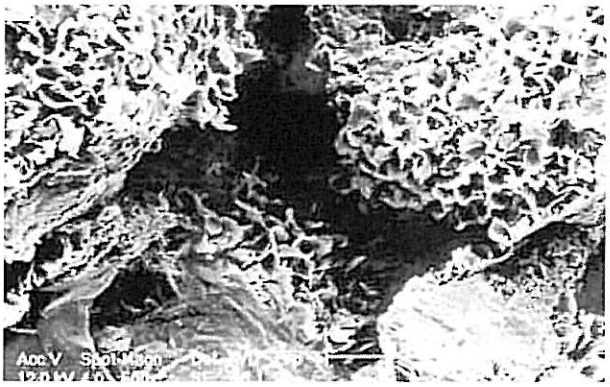
Fig. 4a. A well developed calcite mudcake in excess of 300µm has formed on the injection face. Evidence for mud solids penetration to approximately 400µm.
Fig. 4b. A view looking down on to the injection plug face. Strands of solium formate polymer based drilling mud coat grains and locally block pores.
Fig. 4c. Scale inhibitor formed as a thin film (1µm thick), now partially desiccated and globules on quartz grains, lowering rock permeability.
Fig. 4d. An aragonite scale has formed locally on quartz grains and partially blocks the pores.
Fig. 4e. Pore lining and pore bridging, illitic clay is present (x200).
Fig. 4f. After testing, mud and clean-up chemical filtrate losses have caused the collapse of pore lining, illite clay significantly increasing the permeability (x200).

MICROBIAL FORMATION DAMAGE

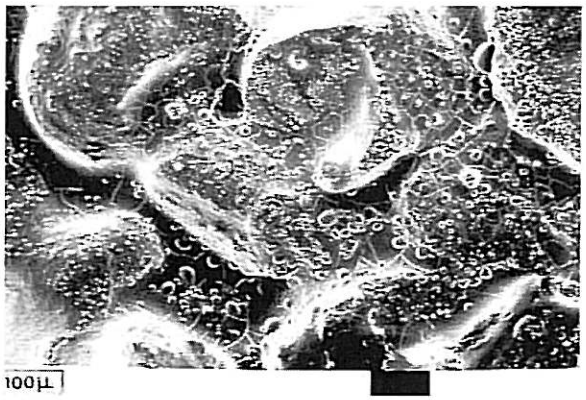
The effect of microbes on oilfield reservoirs in terms of indigenous bacteria (present in field prior to any drilling) and introduced bacteria from drilling muds (Ezzat, 1997) and other well operation chemicals has been very actively researched in recent years. The major formation damage mechanisms associated with microbial activity within wells and reservoir rocks are biopolymer pore plugging, microbe cell pore blockage, H_2S gas production and insoluble metal sulphide precipitation. The major controls on microbe











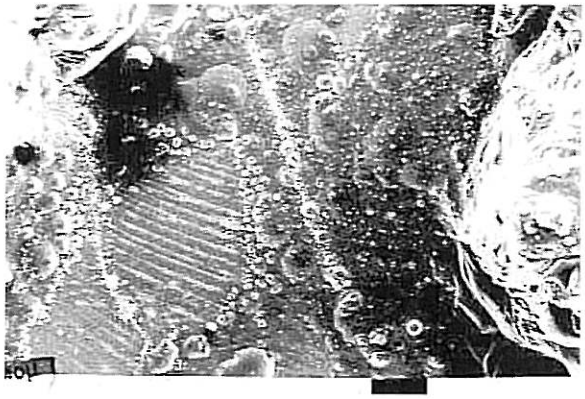


Fig. 5a. Mud filtrate losses pass along a distinct channel within a gas well. Pores adjacent to this channel within a gas well. Pores adjacent to this channel are filled with gas only.
Fig. 5b. Higher magnification view of plate 5a showing that the liquid within the channel fills the large, intergranular pores, but not the micropores between the illitic clay fibres.
Fig. 5c. (Before testing). Plug sample from a gas well in which no liquid was visible within the larger, intergranular pores or the micropores of the pore lining clay.
Fig. 5d. (After testing of sample shown in Fig 5c). Excessive filtrate losses from a mud have been retaine within the sample forming water blocks.
Fig. 5e. (Before testing) Plug sample from an oil well in which oil is the main liquid filling pores, pore lining droplet of formation brine are commonly present.
Fig. 5f. (After testing of sample shown in Fig 5e). Filtrate losses after KCl mud and KCl clean up application resulted in the formation of a microemulsion. Locally coalescence of droplet has formed larger KCl brine droplets.

growth activity are nutrient availability (often fractions of the crude oil e.g. Acetate, butyrate, propionate, hexadecane), a source of phosphate, nitrate, sulphate, sulphur or hydrogen, and probably of most importance the temperature. The SRB (sulphate reducing bacteria) are mainly responsible for the production of H_2S gas and insoluble metal sulphide precipitates in reservoirs. By undertaking microbial analysis of fresh cores (must be well preserved) and drilling muds, and by inoculation into suitable growth media at various temperatures up to the field temperature, it is possible to determine whether H_2S production is

likely to be a problem from either the indigenous and/or introduced bacteria (Wood and Spark, 1999). Once the thermal gradients of the field have been assessed to the water injection profiles, it is possible to model the bacterial populations to temperature and thus predict where H_2S production, polymer plugging, insoluble metal sulphide precipitation and microbial cell plugging are likely to occur within the field. The presence of sulphate reducing bacteria in live drilling muds, core materials and reservoir formation brine from new oil fields is discussed in detail by McGovern-Traa, C. *et al* (1997). Dry and cryogenic S.E.M. techniques when applied to microbial samples can be used to determine bio-polymer/cell plugging (Fig. 6a and 6b), water based drilling mud microbial degradation with the precipitation of iron sulphide (black colour), complete breakdown of mud properties and liberation of H_2S gas (Fig. 6c), and the size, distribution and morphologies of the different cell types (Figs. 6d, 6e and 6f).

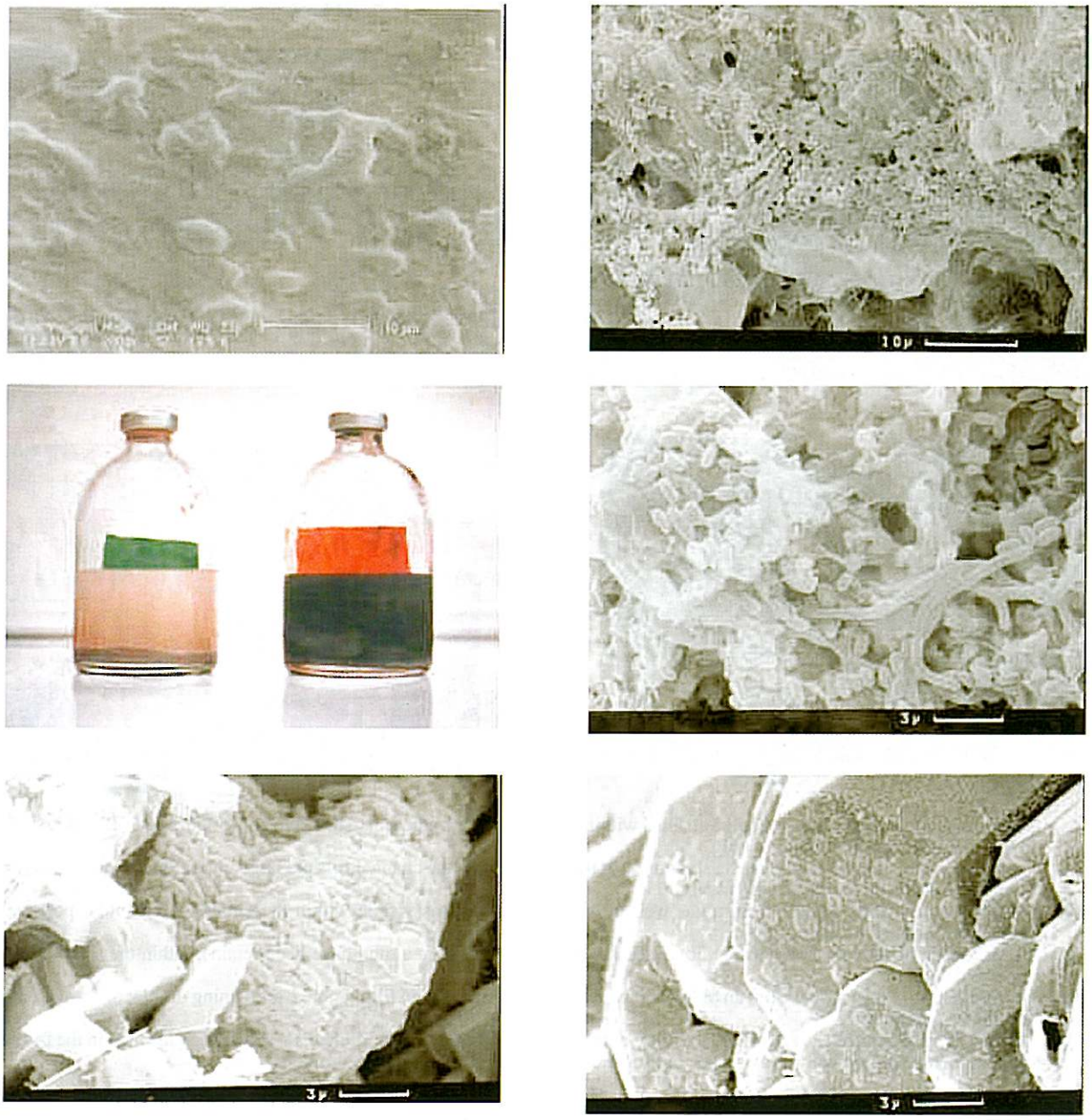


Fig. 6a. Cryogenic S.E.M. showing the occurrence of biopolymer in formation brine.
Fig. 6b. Dry S.E.M. showing the occurrence of biopolymer coating framework grains and partially to completely blocking pores.
Fig. 6c. (Left) Live WBW inoculated into growth media at 30C (0 time). (Right) Same mud after 48 hours incubation shows FeS precipitation and H_2S gas production at 400ppm. The mud has been completely broken down due to microbes (SRB).
Fig. 6d. Elongate, rod shaped and ovoid bacteria within a chalk sample.
Fig. 6e. Ovoid cells forming locally in pores as aggregated beehive colonies.
Fig. 6f. Circular aggregates of tiny coccoid (nanobacteria?) individually up to 0.025µm in size.

CONCLUSIONS

By careful utilisation of CAT scanning, thin section petrography, dry and cryogenic S.E.M. analysis and microbial testing it is possible by combining all these techniques, to fully determine the formation damage mechanisms, which can then explain return permeability and filtrate loss data gained from reservoir conditions flood tests.

It is essential to ensure that all materials used in the reservoir conditions flood tests and the pressure/ temperature conditions are relevant to the field and wells in question, if accurate determination of the formation damage mechanisms is to be made using the analytical techniques described above.

After the formation damage mechanisms have been identified, it is then possible to suggest the most suitable remedial and/or preventative measures to be taken to minimise any formation damage in the reservoir.

It is then possible to pre-screen chemicals in the laboratory using reservoir conditions flood tests such as drilling muds, clean-up/completion chemicals, squeeze chemicals and injection water prior to their use in the well to avoid anything which may cause significant formation damage.

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