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Status Report

RESERVOIR ASSESSMENT AND CHARACTERIZATION IMAGING TECHNIQUES APPLIED TO THE STUDY OF FLUIDS IN POROUS MEDIA
TASK 3
TECHNOLOGY TRANSFER ACTIVITIES

by

Liviu Tomutsa and Daryl Doughty

September 1995

Work Performed Under Contract No.
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Prepared for
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1.1 MULTINUCLEAR NMR MICROSCOPY OF TWO-PHASE FLUID SYSTEMS IN POROUS ROCK

Title: "Multinuclear NMR Microscopy of Two-Phase Fluid Systems in Porous Rock" by Daryl A. Doughty and Liviu Tomutsa

Place: Presented at Third International Meeting on Recent Advances in Nuclear Magnetic Resonance Applications to Porous Media, in Louvain-la-Neuve, Belgium

Date: Sept. 3–6, 1995

1.2 IMAGING TECHNOLOGY DEVELOPMENTS AT NIPER

Title: "Imaging Technology Developments at NIPER" by Liviu Tomutsa

Place: University of Houston, Chemical Engineering Department, Houston, TX

Date: March 3, 1995

1.3 IMAGING TECHNOLOGY APPLIED TO THE STUDY OF FLUIDS IN POROUS MEDIA

Title: "Imaging Technology Applied to the Study of Fluids in Porous Media" by Liviu Tomutsa

Place: DOE Contractors Project Review Meeting, Fountainhead Lodge, Eufaula, OK

Date: June 25–29, 1995

Title: "Imaging Technology Applied to the Study of Fluids in Porous Media" by Liviu Tomutsa

Place: Lawrence Livermore National Laboratory, Lawrence, CA

Date: September 15, 1995

2.0 PUBLICATIONS

2.1 MULTINUCLEAR NMR MICROSCOPY OF TWO-PHASE FLUID SYSTEMS IN POROUS ROCK (PAPER)

Paper by Daryl A. Doughty and Liviu Tomutsa, presented at Third International Meeting on Recent Advances in Nuclear Magnetic Resonance Applications to Porous Media, to be published in *Magnetic Resonance Imaging*. (The complete paper is included at the end of this report.)

2.2 MULTINUCLEAR NMR MICROSCOPY OF TWO-PHASE FLUID SYSTEMS IN POROUS ROCK (REPORT)

Topical Report by Daryl Doughty and Liviu Tomutsa to be submitted to the DOE as a deliverable on September 29, 1995. A diskette with the software developed is available with the report.

3.0 ABSTRACTS SUBMITTED

"Use of Statistical Dispersion Model to Study Polymer Clogging in Sandstone Samples" by E. Kasap, L. Tomutsa, H. Ates, and H. Gao. Submitted to SPE IOR Meeting Tulsa, OK, April 1996.

"Laboratory Validation of Tensorial Behavior of Permeability" by L. Tomutsa, E. Kasap, S. Swan, and H. Ates. Submitted to SPE IOR Meeting Tulsa, April 1996

"Use of X-Ray Scanners in Petroleum Research" by D. Maloney, and L. Tomutsa. Submitted to December 1995 Meeting of AGU, San Francisco, CA.

4.0 TRAINING

Training was provided for two summer AWU engineering students, Harun Asap and Tamara Glover. They were introduced to principles of rock permeability measurements with emphasis on minipermeameter measurements on heterogeneous rock samples and porosity/permeability measurements using the automated petrographic image analysis (PIA) equipment.

Tours of the computed tomography (CT) imaging laboratory and short presentations (10 to 20 minutes) on the applications of imaging technology to improve oil recovery were given on a regular basis to groups of visitors from:

- Department of Energy (Headquarters, Morgantown Energy Technology Center, and Pittsburgh Energy Technology Center)
- Industry (U.S. major oil companies and independents) including independent oil producers from Oklahoma)
- International visitors, including representatives of RIPED from Beijing and Daqing oil field, People's Republic of China, and Hydrocarbon Development Institute of Pakistan.
- Academia members, including National Institute for Petroleum and Energy Research (NIPER) AWU summer students and faculty.
- Middle and high schools students and teachers, including Earth Science teachers participating in the DOE-sponsored exploration program.
- The general public, including visitors to the OK Mozart Festival.

Peer interaction took place through professional interactions at the national Society of Petroleum Engineers (SPE), Society of Core Analysts (SCA), and DOE Contractor Review meetings, SPE Fluid Mechanics and Oil Recovery Processes Technical Committee meetings (of which L. Tomutsa is a member), visits to University of Tulsa, University of Houston, Stanford University, and Lawrence Livermore Laboratory.

CT imaging and minipermeameter permeability data for the Bartlesville sandstone sample have been used by an oil company to study the feasibility of setting up a CRADA research with NIPER for further research of the effect of heterogeneity on oil recovery in fluvial type deposits.

A contact has been made with an independent oil producer/consultant to gain insight regarding areas of research important to independents. During his visit to NIPER he viewed the CT, the PIA, and Relative Permeability laboratories. The research results were discussed along with the need for further contacts between independents and NIPER. He was appraised of new improvements in BOAST simulator made at NIPER and obtained a copies of the programs from DOE. He expressed interest in improved log and well test interpretation techniques for low cost evaluation of rock and rock/fluid parameters for reservoir simulators and in NIPER providing courses for independents in the use of simulators.

5.0 WORK FOR OTHERS

Technology transfer took place on a continuous basis during the work on a research project funded by a major oil company.

MULTINUCLEAR NMR MICROSCOPY OF TWO-PHASE FLUID SYSTEMS IN POROUS ROCK

Paper by Daryl A. Doughty and Liviu Tomutsa presented at Third International Meeting on Recent Advances in Nuclear Magnetic Resonance Applications to Porous Media, to be published in *Magnetic Resonance Imaging*.

Abstract

The high field magnetic resonance (MR) characteristics of fluids in porous reservoir rock are more like those of solids than liquids, exhibiting short T_2 relaxation times and broad natural line widths. These typical characteristics severely restrict the MR imaging (MRI) methodology which can be used to obtain high-resolution, porescale images of fluids in porous rock. An MR microscopy protocol based on 3D back-projection using very strong imaging gradients was developed to overcome many of these constraints. To improve the image quality of the two fluid phases, multinuclear MRI, a new technique using proton MR to image the brine phase and 19F MR of a perfluorinated hydrocarbon to image the oil phase, was used. With this protocol, multinuclear images of two-phase fluid systems in rock samples have been obtained using up to 65536 projections and 256 complex points per projection in spherical coordinate space. Resolution as high as 25 microns per pixel was obtained for fluid/rock systems in Bentheim, Fontainebleau, and Bartlesville sandstones. Image processing software, developed to extract the pore size distribution from the binarized 3D image of the fluid filled porosity, will be described. The pore size distributions obtained from 3D images have been broader than those obtained

from petrographic image analysis of thin sections because some pores extend a considerable distance into adjacent layers. End point, two-phase fluid distributions (residual brine or residual oil) and their dependence on pore size and connectivity for several two-phase fluid/porous rock environments are presented. The multinuclear images demonstrated the distribution of residual oil in ganglia within the pore space.

Introduction

In petroleum reservoir assessment and characterization, MRI is a valuable nondestructive imaging technology used to image fluids within cores, providing information about porosity distribution and how it is affected by rock heterogeneity. MR microscopy provides information about the pore network connectivity which is directly related to fluid flow characteristics within the rock matrix, pore sizes, and fluid distributions in pores.[1] The high resolution achievable allows visualization of the effect of rock/fluid interactions on oil and water distributions within the pore spaces of reservoir rocks. Such a capability aids in understanding oil displacement processes taking place at the pore level which are essential in understanding the mechanisms of various oil recovery processes. High resolution MR microscopy was recently expanded to include multinuclear imaging for direct imaging of separate fluid phases using fluids which are tagged by an MR-active marker such as fluorine-19.

Experimental

The most efficient 3D imaging process for fluids in porous rock is the 3D projection reconstruction sequence.[2] The imaging gradient is of fixed amplitude and its direction in space is controlled by the three X-, Y-, and Z-components. Because the gradient is switched on prior to the RF pulses in the spin echo and remains on until the signal is acquired, rapid switching of the gradient is not required and the time to echo (TE) can be shorter, maximizing signal strength for fluid/rock systems which have a short T_2 relaxation time. However, achieving high resolutions using this protocol requires strong gradients and high RF bandwidths. A commercial high field MR spectrometer was modified for imaging using a high field gradient coil assembly to provide the strong gradients as high as 150 gauss/cm. A home-built imaging probe is used which has a horizontal solenoid RF coil capable of operating at both proton and fluorine-19 frequencies. A high power RF amplifier is used to provide the short RF pulses and a high speed 2-channel AD converter interfaced with a separate computer is used to acquire the imaging data.

The small core plugs of reservoir rock used for MR microscopy are typically about 5 mm in diameter and 7–10 mm long. They are mounted inside a core holder made from two Teflon end caps and two layers of Teflon shrink tubing. The inner layer is an FEP type Teflon which shrinks at 206°C and the outer layer is PTFE Teflon which shrinks at 320°C. This higher temperature softens the inner layer, bonding the end caps and core plug into a leakproof assembly. Small diameter Teflon tubing is clamped into the end caps and permits the exchange of fluid components in the core plug while mounted in the spectrometer. Typical imaging parameters are: 256 x 128 gradient orientations, 256 complex points per projection, 4–8 summed accumulations per projection, 1.28 msec TE, and 0.4 second PD for proton imaging of brine. A longer PD is used for imaging the oil phase. The brine used is 0.5% NaCl by weight with 0.023% $MnCl_2$ added as a T_1 relaxation agent. The oil phase is usually Soltrol or a fluorinated compound such as hexafluorobenzene (HFB) or 3,5-bis (trifluoromethyl) bromobenzene (TFMBB).

The algorithm used to process the projection reconstruction image data is a two-stage implementation of the convolution method for parallel beam reconstruction tomography from the Donner Laboratory algorithms.[3] In adapting this algorithm for MR imaging, the MR projection is treated like a parallel beam emission projection of source intensity across the dimension of the object defined by the gradient direction. The 3D projection reconstruction occurs in spherical coordinate space defined by the angles, θ and ϕ, for both imaging and reconstruction. In Stage 1, separate 2D reconstructions are done for each plane of projections defined by the angle ϕ. Within each plane the orientation of the projections, $P_{\theta\phi}$, is defined by the angle θ. The result of Stage 1 is an ordered set of 2D square Cartesian projections indexed by the angle ϕ. In Stage 2, the columns of each resulting 2D projection represent a series of 1D projections defined by ϕ. Selecting a specified column from each of the 2D projections leads to a 2D reconstruction in the orthogonal plane defined by the column index. Doing this reconstruction for each set of columns in sequence results in a 3D Cartesian image of the fluid-filled porosity of the sample.

Results

Two-phase brine/Soltrol fluid-filled porosity was determined for a core plug of Fontainebleau sandstone for brine and Soltrol/residual brine conditions. Image resolution was about 30 microns per voxel edge. The initial brine-saturated total porosity was imaged using a TE of 1.28 milliseconds. The corresponding oil occupied porosity was obtained using a longer TE to suppress the brine signal. The corresponding residual brine occupied porosity was obtained by subtracting the oil image from the initial brine image. Even though at least 10 pore volumes of oil flowed through the core plug before the second experiment was run, the relative volume of the total porosity occupied by Soltrol is decreasing. Also, the volume occupied by brine is increasing as the distance from the inlet port increases, which is a typical end effect noticed in fluid flow in core plugs.

Using a longer TE to suppress the more quickly relaxing brine phase is a common approach to separately imaging two-phase fluid systems. However, effects on the projections at certain gradient orientations were noticed in these experiments compared to the shorter TE experiments. In an experiment on a small spherical, water-filled phantom at a TE of 10.24 milliseconds for varying gradients strengths, off-axis gradients involving combinations of the Z-gradient with either X- or Y-gradients were discovered to cause a suppression of projection signal strength, increasing with TE and gradient strength. The average signal area for all gradient orientations is strongly affected by gradient strength with the stronger gradient causing a greater suppression of the signal at a given TE. Also the influence of gradient orientation on the signal is much less pronounced at the weakest gradient of 7.45 gauss/cm. The average suppression of the MR signal in the presence of a gradient is caused by the diffusion of fluid molecules from one location in the sample to a region of different magnetic field during the TE. This causes the contribution from that molecule to disappear from the echo signal. An explanation for why this effect should be influenced by gradient orientation is not yet available. Using multinuclear MRI with separate frequencies to image the two phases with similar short TEs was chosen to eliminate the impact of this effect.

Another image effect in a high resolution MR image of a fluid-filled porous medium is the variation in voxel brightness across the different pores or from pore to pore in the images. One such interpretation is that the dimmer voxels represent places within the rock/fluid system where only a part of the voxel is occupied by fluid-filled porosity contributing to the signal. A wedge-shaped phantom sample formed by placing a 0.250 millimeter thick spacer between two small glass plates was saturated with brine and imaged. At the spot where the wedge was one voxel wide, the image spread over three or four voxels causing a reduced intensity. The intensity falls off even more as the thickness of the wedge falls below one voxel. This variation in intensity could be used to make a quantitative estimate of fractional voxel filling in different regions of MR images and effectively increase resolution.

A multinuclear MR microscopy experiment was performed on a core plug of Bentheim sandstone using brine and TFMBB. The same imaging gradient strengths were used for both the proton and fluorine-19 images. Four experiments were performed: (A) the brine saturated core plug; (B) the oil phase using fluorine-19 MRI; (C) the brine imaged at residual oil; and (D) the residual oil, again imaged using fluorine-19 MR. In the images the voxel intensity is displayed using a 0–255 level gray scale. The fluorine-19 images had to be scaled by the ratio of the 1H/19F MR frequencies to obtain the same size and resolution. If the images from (C) and (D) were added, which would represent the total fluid-filled porosity in the core plug, good agreement with the initial brine image would be obtained. This lends confidence to using multinuclear MR imaging to separately image the two phases.

One goal of our MR microscopy was to use the 3D images to investigate the connectivity of the pore space at pore scale. We developed software at NIPER to enable us to track pores through 3D space and develop information on pore size and connectivity using an erosion/dilation process similar to that developed by Ehrlich for petrographic thin section analysis.[4] Erosion/dilation works on binarized images where voxels at or above a selected intensity are set to an intensity value of 255 and those below the reference intensity are set to zero. The kernel is a 3 x 3 x 3 cross with seven voxels and is scanned progressively through the image volume. As the kernel encounters filled voxels in the image plane, in the erosion process the value of the central voxel is replaced by the minimum value of the kernel voxels. For a binarized image this is equivalent to being either on (255) or off (0). Erosion is used to break narrow connections between larger pore spaces. In the dilation process the value of the central voxel is replaced by the maximum value of the kernel voxels. Dilation is used to replace the previously eroded outer layer of voxels for the larger pore spaces. The software will permit up to six successive stages of erosion/dilation.

The pore size determination is used on the binarized image data following any erosion/dilation. As the layers of the image are scanned, a pore index is established for each separate activated voxel encountered during the scan. If the newly encountered voxel is determined to be a nondiagonal neighbor to a previously counted voxel considering all three dimensions, the voxel count is incremented for that pore index. If a bridge is later discovered connecting two previously isolated pores, the voxel count for the highest pore index is added to the lower pore index, and the higher pore index zeroed and freed for later use. The end result of the pore size determination is a histogram of pore size for the image volume and a table showing the ranges in X, Y, and Z for each pore.

The binarized image of a core plug of Bentheim sandstone of 24% porosity was compared to a similar image of a core plug of Fontainebleau sandstone of 12% porosity. The images show a considerable variation in appearance. The Fontainebleau image shows much greater variation in voxel intensity and pore size with isolated large pores separated by low porosity areas of much smaller pores. The Bentheim image shows a higher image uniformity with more connectivity between neighboring pores.

Pore size/connectivity analysis was performed on the two systems using different stages of erosion/dilation. Comparing the largest pore in the pore size histogram for each rock expressed as a fraction of the total porosity remaining at each stage, the binarized image of the Bentheim sandstone showed that even after one stage of erosion/dilation most of the porosity is contained in one large pore throughout the image pore space. The largest pore size then falls off sharply after an additional erosion/dilation and then starts to increase. In contrast to the Bentheim system, the results for the Fontainebleau system shows an opposite trend with the largest pore size starting off small and then increasing modestly after several erosion/dilation stages. After two stages of erosion/dilation, the Fontainebleau system behaves similarly to the Bentheim result. These statistical results confirm the visual differences noted above and indicate the high connectivity in the Bentheim core plug compared to the Fontainebleau sample.

Conclusions

A technique of MR microscopy has been developed at NIPER using the projection reconstruction pulse sequence on small core plugs with strong imaging gradients which has successfully imaged fluids at pore scale in sandstones. Voxel resolutions as high as 25 microns have been achieved.

Multinuclear MR imaging of two-phase fluid systems using fluorine-19 substituted hydrocarbons permits obtaining unambiguous images of the separate water and oil phases leading to better understanding of fluid distributions in porous rock.

Software has been developed at NIPER which can perform a true 3D erosion/dilation process on the binarized, pore scale, MR images and then obtain pore size measurements and track pore connectivity.

Results from pore size measurements show significant differences in the nature of the pore network in Fontainebleau and Bentheim sandstones.

Acknowledgments

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