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Mobile NMR for porosity analysis of drill core sections

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Abstract
We apply a novel mobile nuclear magnetic resonance (NMR) scanning system, the NMR-MOUSE®, for measuring porosity of geological drill core sections. The NMR-MOUSE® is used for transverse relaxation measurements on water-saturated core sections using a CPMG sequence with a short echo time. A regularized Laplace-transform analysis by the UPEN program yields the distribution of transverse relaxation times. The signal amplitudes and the distribution integrals correlate directly with the porosity of the cores, in spite of the influence of diffusion in the strong field gradient of the NMR-MOUSE®, which is discussed. The method is particularly attractive because it neither requires a volume calibration nor the samples to be machined to fit the coil, and because the device is mobile and particularly attractive for field use such as on logging platforms and research vessels.

Keywords: NMR, mobile spectrometer, physical properties, porosity, drill cores, ODP/ICDP

1. Introduction

The study of NMR relaxation of fluids in porous media has been motivated by the oil industry’s interest in reservoir rocks (Kleinberg 1996). The apparent NMR relaxation time distribution is influenced in different ways by wall relaxation and internal field gradients in porous media, depending on the translational diffusion of the fluid molecules. Provided the sediment samples are saturated by low-viscosity fluids and the exponential signal decay is dominated by wall relaxation, the decay curve associated with a single pore size will be a simple exponential. The decay constant is proportional to the pore size, i.e. small pores are characterized by shorter relaxation times $T_1$ and $T_2$ than large pores. Thus, the proton NMR signal characteristics of water in porous media are related directly to the porosity and pore size distribution of a sample. This information can be obtained by mobile nuclear magnetic resonance measurements with the NMR-MOUSE® (Eidmann 1996).

Mobile NMR has its origin in inside-out NMR, where NMR spectrometers are lowered into boreholes for analysis of fluids in the surrounding rock. NMR well-logging devices (Coates 1999) are unilateral NMR sensors (Matzkanin 1989). The term unilateral refers to the fact that the study object is not inserted into the magnet for measurement (the common case in NMR) but applied to the NMR sensor from one side. Suitable dimensioning of magnet and coils ensures that the polarizing magnetic field $B_0$ and the radio frequency (RF) magnetic field $B_1$ are only moderately inhomogeneous. Unilateral materials analysis with the NMR-MOUSE® is based on the same principle, however with much larger field gradients (Eidmann 1996).

As the NMR-MOUSE® operates at frequencies higher than that of the well-logging sensors it has less ringing and shorter dead time. This advantage makes the NMR-MOUSE® sensitive to signals with relaxation times $T_1$ and $T_2$ shorter

⁴ NMR-MOUSE (Nuclear Magnetic Resonance Mobile Universal Surface Explorer) is a registered trademark of RWTH Aachen University.
than 0.1 ms and allows one to study rocks with small pores and possibly gas hydrates. Another advantage of the NMR MOUSE® compared to conventional devices is its small size and weight. This makes it particularly attractive for use on ships and drilling platforms (figure 1).

2. Technical construction

The NMR MOUSE® used for these experiments was constructed from a figure-8 coil and U-shaped magnet (Anferova 2002). The value of the magnetic field $B_0$ directly above the gap between the magnets is 0.52 T corresponding to a proton resonance frequency of about 22 MHz. With the device, a signal penetration into the core sample of up to 6 mm can be achieved by reducing the frequency. The magnetic field of the NMR MOUSE® is highly inhomogeneous with a volume-averaged magnetic field gradient of about 12 T m$^{-1}$ at 21.1 MHz. We used the standard ODP core samples, split into two half cylinders along the core axis. The NMR-MOUSE® was then operated on the flat splitting surface.

3. Basic procedures

We studied both sandstone samples of different porosity from the Allermöhre borehole in the North German sedimentary basin and limestone samples from the Caribbean sea drilled during Ocean Drilling Programme (ODP) leg 165. Transverse relaxation was measured with the NMR-MOUSE® using a CPMG sequence with a short echo time of 0.1 ms (Carr 1954) (figure 2). The measurements were performed at a frequency of 21.1 MHz with a penetration depth of 3–4 mm into the sample. Prior to measurement all samples had been saturated with distilled water. A regularized Laplace-transform analysis based on the UPEN program (Borgia 1998) yielded the distribution of transverse relaxation times $T_{2\text{eff}}$ (figure 3).

Following the procedures established in well logging for weakly inhomogeneous fields (Coates 1999) the relaxation curves measured in strongly inhomogeneous fields and the associated relaxation time distributions were then studied for information on rock porosity and the pore size distribution: in addition to the bulk relaxation rates $1/T_{1,\text{b}}$ in porous media there is a contribution $1/T_{1,\text{s}} = \rho_{1,\text{s}} S/V$ from the molecules...
in contact with the pore surface, which determines the overall relaxation rate. Here $\rho_{1,2}$ is the relaxation strength or relaxivity of the surface (which differs for $T_1$ and $T_2$ relaxation) and $S/V$ is the surface-to-volume ratio. The relaxation in most rocks corresponds to the fast diffusion or surface-limited relaxation regime. In this case the rate-limiting step is the relaxation at the surface, not the transport of spins to the surface (for instance by translational diffusion). Therefore, the spins experience a rapid exchange of environments so that the local fields in each region of a pore are averaged to their mean values. As a consequence, a single exponential relaxation decay is observed for a given pore, and the rate of magnetization decay does not depend on the pore shape but only on the surface-to-volume ratio $S/V$ of the pore.

For the longitudinal magnetization in a homogeneous field one obtains (Coats 1999)

$$1/T_1 = 1/T_{1b} + \rho_1 S/V.$$  \hspace{1cm} \hspace{1cm} (1)

For spherical pores $S/V = 6/d$, where $d$ is the pore diameter. For the transverse magnetization, attenuation by diffusion in a magnetic field gradient of strength $G$ needs to be accounted for. The gradient may arise from variations in magnetic susceptibility associated with the pore geometry or from inhomogeneity in the applied $B_0$ field. In inhomogeneous fields one obtains for a CPMG sequence with 90° and 180° flip angles

$$1/T_2 = 1/T_{2b} + \rho_2 S/V + D(\gamma Gt_b)^2/12$$ \hspace{1cm} \hspace{1cm} (2)

where $D$ is the diffusion coefficient, $\gamma$ the gyromagnetic ratio, and $t_b$ the echo time.

In the fast diffusion limit and for a sample with a distribution of $N$ different pore sizes, the transient variations of the transverse magnetization $M_{xy}(t)$ can be expressed as a sum of exponentials:

$$M_{xy}(t) = \sum_{i=1}^{N} M_{xy,i}(0) \exp[-t/T_{2,i}].$$ \hspace{1cm} \hspace{1cm} (3)

For vanishing gradient, i.e. $G = 0$, the spectrum or distribution $P(T_{2,i})$ of relaxation times $T_{2,i}$ in equation (2) is a direct map of the pore size distribution $P(d)$ with $T_2 \propto V/S \propto d$, where the distribution functions $P$ represent probability densities.

The UPEN algorithm yields a regularized inverse Laplace transformation, essentially by fitting a sum of decaying exponentials (each with different decay constants, see figure 3), to the envelope of the echo trains from core samples. All of the decay constants make up the decay time spectrum or the relaxation time distribution.

In unilateral NMR with strongly inhomogeneous polarization and RF fields, the magnetic field gradient $G$ does not vanish. Thus, the decay of the echo envelope is affected by diffusion, and it is characterized by a mixture of $T_1$ and $T_2$ due to a flip angle distribution. The resultant decay time constant is denoted by $T_{2\text{eff}}$ instead of $T_2$.

4. Results

For samples with porosities lower than about 5% the currently achievable signal-to-noise ratio is insufficient for an inverse Laplace transformation based on the UPEN program. Therefore the hardware needs to be improved. However, data from samples with higher porosities were suitable for the Laplace analysis. The shapes of the $T_{2\text{eff}}$ distributions obtained with the NMR-MOUSE® for different core sections from the Allermöhe borehole are similar, but the integral intensities clearly scale with the porosity of the samples (figure 4(b)). On the other hand, the $T_{2\text{eff}}$ distributions of the ODP limestone samples from leg 165 are shifted towards lower $T_{2\text{eff}}$ times (figure 4(a)). This corresponds to smaller pore sizes compared to the Allermöhe samples. Due to the diffusive attenuation of the signal measured in the presence of the stray field gradient of the NMR-MOUSE® ($G_{\text{eff}} \approx 12 \text{ T m}^{-1}$) the distributions of $T_{2\text{eff}}$ are compressed for long relaxation times and cannot be related directly to pore size distributions. Nevertheless, they are still fingerprints of the pore size distributions.

In spite of the field gradient, porosity can still be accurately determined in either of two ways: (1) the amplitudes measured in CPMG experiments are normalized by the amplitude measured on pure water, which corresponds to 100% porosity (figure 5); for improved accuracy the echo amplitudes should be extrapolated to zero echo time; (2) the integrals $S_p = \int P \log(T_{2\text{eff}})$ of the distribution curves are normalized by the integral of the pure water signal. Instead of normalizing the amplitudes or integrals of the distribution curves, both quantities can also be calibrated by independently determined porosities. Both, the amplitude and the integral methods yield good results for the Allermöhe samples (figures 5(a) and (b)).

The amplitudes of the signals and the values of the corresponding distribution integrals $S_p = \int P \log(T_{2\text{eff}})$ are directly related to the number of spins in the sensitive volume probed by the NMR-MOUSE®, even though the relaxation time distribution at large relaxation times is compressed with respect to distributions measured in homogeneous fields. In water-saturated samples, the number of spins in the fluid normalized by the sensitive volume of the NMR-MOUSE®
Figure 5. (a) Dependence of the amplitudes of CPMG experiments at an echo time $t_E = 0.1$ ms on the porosity $\Phi$ of the Allermöhe samples saturated in water 24 h. (b) Variation of the integrals of the distribution curves $S_P = \int P \, d \log(T_{2\text{eff}})$ with porosity $\Phi$: Allermöhe samples ($\bullet$) and ODP samples (leg 165, site 999, hole B) saturated for 24 h ($\circ$) and 48 h ($\times$). Black and grey lines are corresponding linear regressions. The numbers at symbols are hydraulic permeabilities in mD ($1 \text{ mD} = 10^{-8} \text{ ms}^{-1}$).

is proportional to the sample porosity. Because the sensitive volume of the NMR-MOUSE® is well defined, absolute values of porosity can therefore readily be determined from unilateral NMR-MOUSE® measurements.

Figure 5(b) shows that measurements on ODP samples (leg 165, site 999, hole B) immersed in water for 24 h yield results which differ from those obtained on Allermöhe samples. Doubling the saturation time to 48 h moves the data closer to the porosity curve of the Allermöhe samples. This means that the samples had not been completely water saturated within 24 h. This is attributed to the finer grain texture of the limestone of the ODP samples which results in the shift of $T_{2\text{eff}}$ compared to those of the Allermöhe samples (figure 4). Measured hydraulic conductivity is higher for the Allermöhe samples than for the ODP plugs (figure 5(b)). Thus, more water molecules can pass through the Allermöhe samples during a given saturation period compared to the ODP plugs. This is reflected by the different slopes of the corresponding regression lines. Therefore, and in order to avoid incomplete saturation as well as changes in the sample due to repeated drying and re-saturation, porosity measurements with the NMR-MOUSE® should be performed preferably directly on fully saturated specimens sampled immediately after drilling.

5. Discussion and conclusion

We find that the current MOUSE® technology is well suited to determining porosity for sediment samples with porosities in excess of 5%. To ensure complete water saturation and avoid sample changes by drying and subsequent re-saturation NMR measurements should be conducted on fully water-saturated samples directly after drilling. Compared to porosity measurements by NMR in homogeneous fields and by other methods, the use of the NMR-MOUSE® has three advantages: (1) the samples need not be machined to fit the coil; (2) a volume calibration is not required; (3) the device is mobile and therefore particularly attractive for field use such as on logging platforms and research vessels.

Despite the strong field gradient and associated diffusive attenuation and although $T_{2\text{eff}}$ is a combination of $T_1$ and $T_2$, the apparent relaxation time distributions vary along a core so that porosity profiles can be mapped along the core using the NMR-MOUSE®.

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