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● *Contributed Paper*

CONCRETE/MORTAR WATER PHASE TRANSITION STUDIED BY SINGLE-POINT MRI METHODS

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A series of magnetic resonance imaging (MRI) water density and T_2^* profiles in hardened concrete and mortar samples has been obtained during freezing conditions ($-50°C < T < 11°C$). The single-point ramped imaging with T_1 enhancement (SPRITE) sequence is optimal for this study given the characteristic short relaxation times of water in this porous media ($T_2^* < 200$ μs and $T_1 < 3.6$ ms). The frozen and evaporable water distribution was quantified through a position based study of the profile magnitude. Submillimetric resolution of proton-density and T_2^*-relaxation parameters as a function of temperature has been achieved. © 1998 Elsevier Science Inc.

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INTRODUCTION

Understanding the mechanisms of concrete hydration, curing, and freeze/thaw damage requires techniques for the non-destructive assessment of water distribution and dynamics. Several reports have demonstrated the utility of bulk nuclear magnetic resonance (NMR) studies to characterize water in concrete materials. Proton-density and relaxation-time measurements allowed the description of pore structure and population distribution as well as water adsorption and phase transition in cement and concrete materials.[1,2] Quantification of water in reservoir cores and concrete is possible by using stray-field imaging[3] and oscillating gradient-echo[4] techniques.

In the case of cement pastes, the detection of short relaxation components is more challenging at low temperatures, where dipolar broadening becomes dominant over inhomogeneous broadening ($T_2 \sim 100$ μs).[1]

Bogdan et al.[5] investigated the moisture content of White Portland Cement pastes using single-point imaging (SPI).[6] A modified version of the SPI pulse sequence, namely single-point ramped imaging with T_1 enhancement (SPRITE) permits a faster data acquisition and greater experimental flexibility.[7]

In this communication we present some considerations for space-resolved relaxometry of phase transitions by SPRITE. Concrete and mortar imaging is performed in a simultaneous fashion. A comprehensive description of T_1 and T_2 mapping is under investigation.

EXPERIMENTAL

Typical T_1 values for concrete samples are under 5 ms,[1] resulting in a fast SPI acquisition without saturation effects. The small flip angle will allow for repetition times of the order of T_1. Given that the time restriction is then determined by the gradient rise time and amplifier's duty cycle, we invoke the SPRITE sequence, reducing the overall acquisition time and the acoustic vibrations of the gradient set. A 1-scan 64 points one-dimensional profile is collected in under 0.3 s. Acquisition times for more scans are determined by the duty cycle limitations of the gradient amplifiers.

T_2^* mapping can be obtained using SPRITE,[8] varying systematically the encoding time with a constant field of view (Fig. 1). Then, for a given position in the sample, the T_2^*-weighted profiles becomes simply:

$$\rho(x) = e^{-t_p/T_2^*(x)}\rho_0(x) \tag{1}$$

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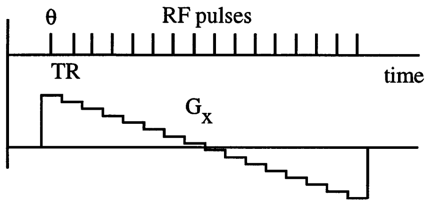

Fig. 1. SPRITE sequence. Varying the encoding time and gradient increment, keeping a constant field of view, permits T_2^* mapping.

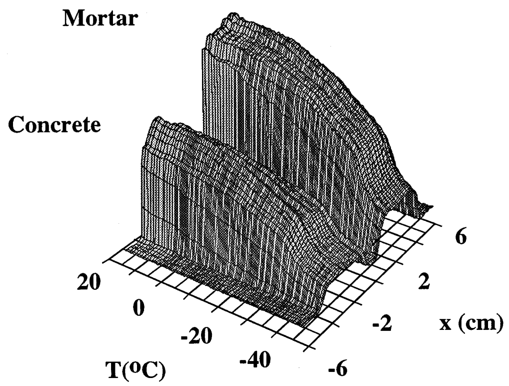

Fig. 2. Temperature dependence of the magnitude profile: 109 one-dimensional 64-point SPRITE profiles.

with $\rho_0(x)$ the spin-density distribution and t_p the encoding time.

The temperature dependence of the non-frozen water density and T_2^* parameters is achieved by introducing a temperature controller system in the magnet bore.

Two cylindrical concrete samples (5-cm diameter, 4-cm length) were prepared using White Portland Cement with a 0.5 water:cement ratio and graded quartz aggregates. The mortar specimen contained aggregates under 0.3 cm and a 2.4 aggregate-to-cement proportion, while graded aggregates of 1.4 cm maximum size were added to the concrete sample. Samples were hydrated at room temperature for 3 weeks.

A sixteen-rod birdcage resonator in a 2.4 Tesla 32-cm horizontal bore superconducting magnet (Nalorac Cryogenics, Inc., Martinez, CA, USA) was used. Experiments were setup by a Tecmag (Houston, TX, USA) Libra S-16 console. Pulse sequence generation and data acquisition were controlled by MacNMR software.

The cooling rate was slower than 2°C/h. At approximately 0.5°C intervals a series of one-dimensional profiles and T_2^* maps were acquired. Thirty-two scans were used for one-dimensional acquisition and four scans for all relaxation time maps. Sixty-four-point profiles with a 14-cm field of view were achieved with an encoding time of 105 μs and a maximum gradient of 5 G/cm.

RESULTS AND DISCUSSION

A series of T_2^* weighted SPRITE profiles were acquired during cooling from 12°C to −50°C. At each temperature a set of ten encoding times were used in the range 55 to 400 μs. A four-scan acquisition was achieved in under 40 s. Fitting the decay for different points along the profile results in a space-resolved T_2^* map. For all temperatures, the profiles decay in a single-exponential manner.

Fig. 2 shows a typical magnitude decay as a function of the temperature. The first noticeable feature is the reduced magnitude in the concrete cylinder region produced by the presence of non-porous large aggregates. The absence of phase transitions at around 0°C indicates that the paste is nonsaturated.

A secondary transition[9] is observed at around −40°C by a pronounced variation in the signal magnitude.

Relaxation times can be determined at all positions in each sample. No significant differences were observed within each sample. T_2^* values for the centre of the concrete and mortar samples corresponding to 12°C are 153 ± 3 and 182 ± 3 μs, respectively, according to a least-squares fitting. The decay for concrete is systematically faster than the decay for mortar as a consequence of the additional inhomogeneity introduced by the coarse aggregates. The spin distribution is obtained applying Eq. (1) and the least-squares output. This results in a reduction to 40% of the intensity at −48°C as compared to room temperature for both specimens, in agreement with reported estimations of non-frozen water for a 0.5 water-to-cement ratio cement paste at −50°C.[10]

This study shows the potential of using the SPRITE method for a space-resolved assessment of the water-phase transition in concrete materials.

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