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Crack Assessment in Cement-based Materials using Ultrasound and T_2 NMR Relaxation Time

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***Abstract*—The elastic properties and durability of concrete based structures, directlydepend on the microstructure of the hydrated cement paste. The microstructurevaries with time due to several chemical reactions and mechanical loads,leading to micro fractures, among other effects. For this reason, it is necessaryto develop techniques to locate and measure fractures. Ultrasound hasproven to be a reliable diagnostic tool, so the primary objective of this paperis to propose a composite nondestructive methodology based on NMR relaxometryand through-transmission ultrasound for crack assessment in cementpaste specimens. While the Hilbert-Huang transform of ultrasonic signals areable to locate the defects, the T_2 transverse relaxation time gives the watercontent in the crack. The results show, that the Hilbert-Huang transformenhanced the ultrasonic echoes coming from the fracture allowing to detectand locate fractures with an error of 15%, and the crack size is given by thedecay parameter of the relaxation time T_2, based by exponential fitted theFID signal.**

***Index Terms*—Detection of cracks; Ultrasound; Hilbert-Huang; NMRrelaxometry.**

I. Introduction

The effects of mechanical loading and environmental effects on concretestructures often lead to the development of microcracks, reducing the overallstrength and stiffness of the structures while increasing their permeabilityto aggressive agents such as chloride ions and Carbon dioxide (CO_2). Water carrying aggressive agents can then travel through these small paths toreach the reinforcing steel and cause corrosion [1]. The demand for reliablenondestructive testing (NDT) techniques to investigate the quality of concretein new or existing structures has drastically increased in recent decades.

Ultrasound [2]-[4] and Magnetic Resonance Imaging [5]-[6], among others arepromising NDT techniques. Information regarding the presence of cracksin cement paste specimens can be obtained using through-transmission ultrasound,in which the pulse amplitude and ultrasonic time of flight areimportant parameters that are used to detect and locate defects in the internalmicrostructures of the specimens. Additional information regardingthe presence of cracks in cement paste specimens can be obtained using protonNMR relaxometry due to its capability of the detection of evaporablewater in cement-based materials and also for the discrimination of water incracks from water hosted in capillary and gel pores based on the relativelylong spin-lattice relaxation (T_1) and spin-spin relaxation (T_2) times of watercracks. The main contribution of this paper is that we present the locationof cracks based on Hilbert-Huang decomposition of ultrasonic signals andexperimental results of measurements of the T_2 relaxation time and its lineardependence on crack size. Both measurements were performed on 18 whitePortland cement paste specimens with a water-to-cement ratio (w/c) of 0.5by weight.

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II. Methodsof Analysis

A. *Throughtransmissionultrasound*

The through-transmission ultrasound technique is widely used for thecharacterization and long-term monitoring of material properties. Elasticwave propagation in composite materials consists of a complex mixture ofmultiple mode conversion and multiple scattering, which results in diffusiveenergy transport. An incident acoustic pulse propagating within the materialis scattered by the materials microstructure. The effect of these inhomogeneitiesmanifests itself in the slowing of the propagation and dispersion ofthe source wave; therefore, the group velocity and the attenuation of incidentacoustic waves are material dependent. The ultrasonic signal also containsinformation regarding flaws or discontinuities in the material.

B. *Analysis of ultrasonic signal*

The innovative part in the context of acoustic signals is the use of theHilbert-Huang transform(HHT) to improve the detection of the signals obtainedfrom the samples, The HHT is based on a decomposition of the analyzedsignal into individual mono-component signals, this transformation iswell studied and used in different nonlinear applications, however its use forsignals from concrete specimens have not been shown in the literature.

C. *Hilbert-HuangTransform*

The Hilbert-Huang transform [7], developed for a non-linear and non-stationarydata analysis, is an adaptive composite method of the Hilbert spectralanalysis and empirical mode decomposition (EMD). The algorithm is basedon applying the Hilbert transform to small number of intrinsic mode functions(IMFs) highlighting the local characteristics of the data. The EMD canbe defined as dyadic filter bank [8] where the acquired signals are decomposedinto data that corresponds of high frequency oscillations (detail), by analyzingtwo consecutive local extrema and finding the maximum value that existbetween them, and its low frequency counterpart. This is an iterative processuntil the slower oscillation in the data has been extracted. Each

residual ordetail waveform is referred as Intrinsic Mode Function (IMF); of zero localmean, finite bandwidth, and its mean frequency is related to the number ofzero-crossings. The set of IMFs represents the energy associated to variousintrinsic time scales and the oscillation mode imbedded in the data, therealso have a well-behaved Hilbert transform. The latter allows to calculatethe instantaneous frequency of each mode resulting as a full time-frequencyof the data.

III. Experimental Methods

A. *Nuclear MagneticResonance*

Nuclear magnetic resonance (NMR) is a nondestructive technique that iscapable to measured water content and proton mobility.The NMR techniquecan detect the evaporable water content in a porous medium through theapplication of radio-frequency pulses (RF) to the sample, this has been usedto study hydrating-cement-based materials [9]-[11]. Protons in water will tendto align themselves along the direction of an external magnetic field B_0,forming a macroscopic nuclear magnetization of the sample. In recent yearsa unilateral magnet is used to provide the external magnetic field, whichthe sensitivity volume is located at the surface of the magnet. When RF pulses are emitted by a superficial RF coil at the resonant frequency, themagnetization is perturbed away from the main magnetic-field alignment.

There will be a gradual dephasing of this magnetization and, consequently, aloss of coherence of the precessing magnetization of the nuclei. The precessingnuclei absorb the energy and subsequently release the absorbed energy andrelax back to the original alignment at a rate that is determined by the $T_1$and T_2 relaxation times. In porous media, T_1 and T_2 depend on the chemicalcharacteristics of a sample and on the size of its pores. T_1 is the spinlatticerelaxation, which involves the loss of excess energy to the lattice orsurroundings as thermal energy; as the spins relax from a high-energy stateback to a low-energy state, RF energy is released back into the surroundinglattice.

The recovery of longitudinal magnetization follows an exponential curve.After time T_1, the longitudinal magnetization has returned to 63% of its finalvalue. T_2 is the spin-spin relaxation time associated with the entropyeffect related to the loss of phase coherence. The dephasing of the transversemagnetization causes a gradual decrease of the signal induced in theRF coil, leading to a decaying NMR signal, which is called the free inductiondecay (FID) and decays with a time constantT_2^*relaxation refers to decayof transverse magnetization caused by a combination of spin-spin relaxationand magnetic field inhomogeneity [12]. Long T_1 and T_2 relaxation times areassociated with large pores, and short relaxation times are associated withsmall ones[13]. The transverse relaxation decay is a sum of the contributionsfrom the spins distributed throughout the entire collection of pores with differentsizes. It is well known that the T_2 decay curves of compartmentedwater are not single exponentials but rather correspond to a distribution ofrelaxation times because various proton spins experience different environments.

In particular, multiexponential decay arises because of the presenceof barriers (e.g., membranes, pores) that lead to water compartmentalizationand determine the rate of water exchange among the compartments.

Therefore, the T_2 decay of bulk water or of water that exhibits a very rapidexchange regime results in a single exponential, represented as:

$$M_{xy}(t) = M_0 e^{-t/T_2} \quad (1)$$

where M_0 is the initial magnetization of the protons

IV. Experimental Setup

Two of the major factors that affect concrete performance are the w/cratio and the curing process[1]. If the w/c ratio is kept below a certainvalue during the mixing process and the curing is properly controlled, thedurability and strength of the resulting concrete at later stages will lie withinacceptable standard values.

For this reason it is very important that the specimen was made correctly.

A. *SpecimenPreparation*

White Portland cement (CPC-30R-B) was used to prepare cement pastespecimens with a w/c ratio of 0.50 by weight. This type of cement waschosen because of its low iron oxide content, which corresponds to a highervalue of the nuclear spin-spin relaxation time T_2[14]. However, the hydrationproducts are of similar nature in both white Portland cement and ordinaryPortland cement[15]; therefore, the mechanical and durability properties ofmaterials prepared using white cement will not be significantly different fromthose of materials prepared using ordinary Portland cement.

The cement was mixed in compliance with the ASTM C305-99 standard.

The specimens were cast in 5 cm x5 cm x5 cm molds. Before the cement pastebegan to dry, thin stainless steel sheets were inserted from the tops of thespecimens to simulate vertical cracks of 12 mm in length and 200, 300, 400,500 and 600 μm in width (figure 1); no stainless steel sheets were inserted intothe control specimens.

Fig 1. Set of 6 out of 18 cement pastes specimens with stainless steel sheets to formartificial cracks

The specimens were placed in a room at a constanttemperature of 23°C. After 24 hours, the stainless steel sheets were removed,and the specimen were further cured for 28 days.

B. *UltrasonicTesting*

The through-transmission ultrasonic method was used to acquire signalsfrom the specimens. The experimental setup consists of a Panametrics-NDTModel 5058PR High Voltage pulser-receiver, a pair of ultrasonic transducers,one operating as a transmitter and the other as a receiver. The generation ofthe transmitted pulse and the signal acquisition were accomplished using unfocusedtransducer at an operating frequency of 0.5 MHz. A HANDYSCOPEHS3 was used to record the waveforms originating from the tested material.A 300 V negative square electrical pulse at the central frequency of the transducerwas sent to the transmitter with a pulse-repetition time of 13 ms.Considering the frequency of the transducers and according to Shannon'stheorem[16] it is enough to use a sampling frequency of 2 MHz to capturethe signals correctly, however, as we want to observe the signal dispersion,we need to be able to capture the high frequency components, so the signalswere digitized at a sampling rate of 50 MHz, in this way we can captureall the high frequency components generated.To eliminate random noise werecaptured 500 signals and were averaged.

A Teflon support was constructed to allow the correct placement of thetransducers and the alignment of them with the fracture. In this way it wasalso ensured that the measurement was always in the same region, in orderto have no errors due the uniformity of the sample.

The ultrasonic experimental setup is illustrated in figure 2, where thetested specimen is immersed in water and the transmitter and receiver transducersare positioned on opposite sides of it.

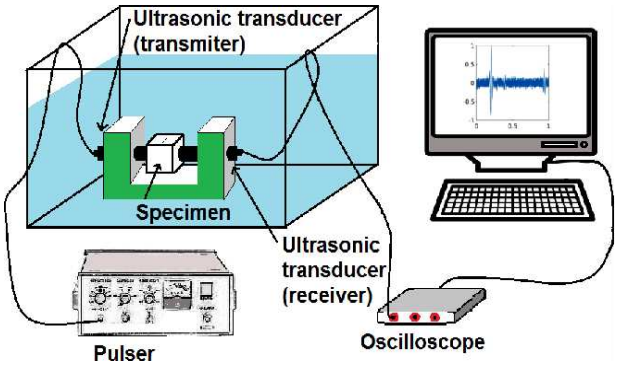

Fig 2. Ultrasonic through transmission testing

C. *NMR Testing*

NMR studies were conducted using the NMR-MOLE (MObile LateralExplorer)[17], which provided a homogeneous field of 75.8 mT centered at 15mm from the array surface and a resonance frequency of $\omega = \gamma B0 =$ 3.226MHz (where γ is the gyromagnetic ratio) that corresponded to the waterresonance frequency. A planar figure-eight surface-coil arrangement, formedby a pair of bundled wires, was used to produce a magnetic field parallel to themain field of the NMR-MOLE. The relaxation times of water molecules in thecement paste were measured using the Carr-Purcell-Meiboom-Gill (CPMG)sequence [18],[19]. The initial exciting $\pi/2$ pulse and the subsequent π pulseshad durations of 30 µs and 60 µs, respectively. The acquisition parameterswere as follows: echo time (TE) =100 µs, repetition time (TR) = 1 s, anda scan number of 64. The chemically bound hydrogen present in calciumsilicate hydrate has a short T_2relaxation time (< 100 µs) and does notcontribute to the NMR signal[20].

The cement probes were located on the NMR-MOLE as shown in figure 3;the RF signal was emitted and received by the RF coil. A portable MagritekKEA2 spectrometer matched the resonant frequency of a particular type ofproton in the sample and recorded the NMR signal at an operating frequencyof 3.226 MHz.

The cement-based specimens were positioned within the sensitive regionof the NMR-MOLE. The specimens with and without cracks were vacuumsaturated with distilled water at 20 mbar to ensure that the specimens werefree of air. The CPMG sequence was used to acquire the NMR signal. Thetransverse magnetization decay was fitted to the exponential function givenby eq. 1. The amplitude of the signal was proportional to the amount ofevaporable water in the specimen [21].

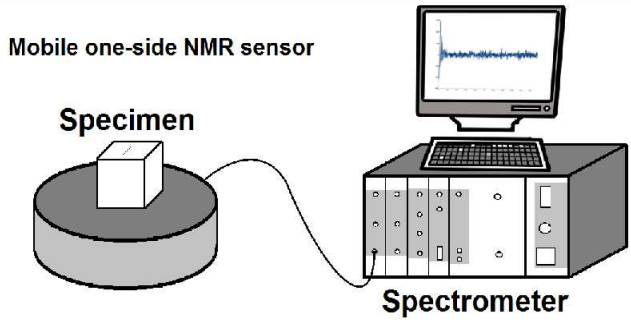

Fig 3. NMR relaxometry probe testing.

V. RESULTS

Ultrasonic and NMR signals were collected for 18 cement paste cubes preparedin accordance with the ASTM C305-99 standard. Three were flawlesscontrol specimens, and the remainder consisted of five sets of three specimenseach that contained artificial cracks with widths of 200, 300, 400, 500 and600 µm.

A. *UltrasonicSignals*

The longitudinal ultrasonic speed of the cement paste specimens was calculatedby measuring the time required for the wave to travel the full width(50 mm) of the flawless specimens. To confirm the repeatability of the experimentalspeed value, several through-transmission measurements were conductedusing three control specimens. The speed value was 2403 ± 360 m/sat 500 KHz. The location of a fracture in the test specimen could be determinedbased on the speed of sound in the medium and the time requiredfor the wave to travel the total path. The set of raw signals acquired using 0.5 MHz ultrasonic transducers is depicted in figure 4, where the control signalwas used as a reference pattern. Based on a simple comparison betweenthe flawless-specimen signals and the remainder of the acquired data, thepresence of a defect was suspected but not conclusive. Further analysis wasnecessary to enhance the ultrasonic echoes that represented the flaws withinthe tested material. For that purpose, the Hilbert-Huang transform was chosenbecause of its ability to decompose the signals and denoise, this allowto select the information that we want to detect from the decomposition.

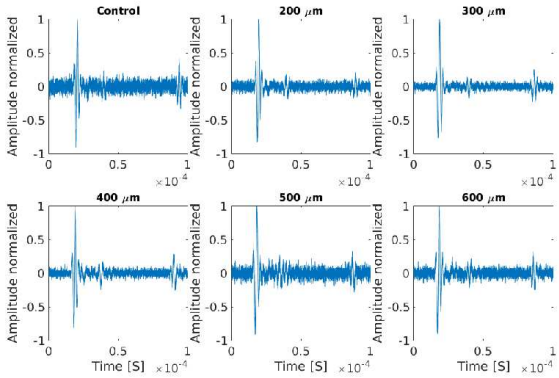

Fig 4. Raw ultrasonic signals acquired using a 0.5 MHz transducer.

The filtered signals from one set of samples are plotted in figure 5; thesesignals correspond to the maximum amplitudes observed in the decomposition.The resulting signals preserve an isolated peak at time t, in the 29 to47 μs range, that corresponded to the fracture position. The Hilbert-Huangtransform was applied to all acquired signals, allowing the computation ofthe average flaw distances for each set of samples under the same conditions.

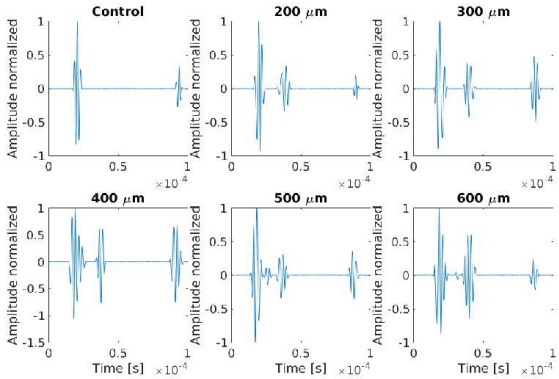

Fig 5. Signals filtered with the transform of Hilbert-Huang.

The complete results are summarized in Table 1.

TableI: Location and detection of fractures. The ultrasound speed was 2403±360 m/s.

Thickness of the fracture [mm]	Position of the fracture [mm]
0.2	25.6±3.9
0.3	24.6±3.8
0.4	24.7±3.9
0.5	24.4±4.0
0.6	24.5±3.7

Observing the frequency spectra, we can conclude from the figure 6 that,as the size of the fracture increases, there is less energy in the main lobe sothe amplitude of this one decreases as the size of the fracture increases. Thisallow us to make a qualitative analysis, however, if we want to quantify thesize of the fracture it is necessary the use of other tools.

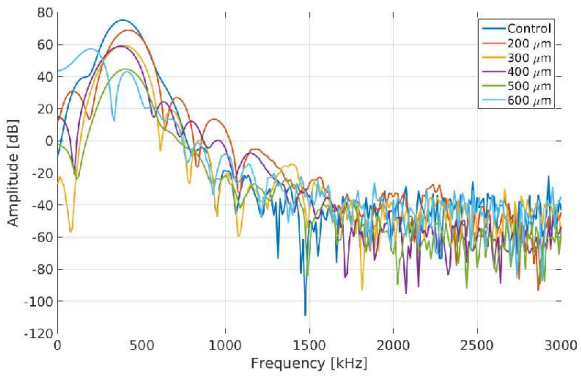

Fig 6. Frequency spectrum of the filtered signals.

B. NMR relaxometry measurements

^{1}H proton relaxometry measurements provide information regarding thewater content in a porous sample. The transverse magnetization decay wasmeasured and fitted to an exponential decay function (figure 7(a)). As canbe seen in figure 7(b) the exponential decreases more slowly as the fracturesize increases, since the amount of water inside the specimen increases, in thetable 2 we can observe the decay factors for The different fractures. Figure 8displays the linear relation between the inverse of the decay constant α andthe fracture size, so we can make a linear adjustment to predict the size ofthe fracture knowing the decay of the signal. From the equation 1. We knowthat this gives us the relaxation time T^2.

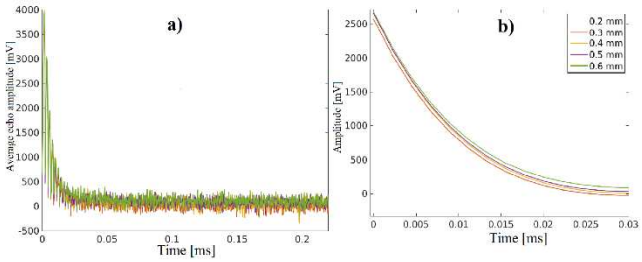

Fig 7. a)T_2 relaxation time distributions determined from NMR decay. b)Exponentialfitted to relaxation time T_2

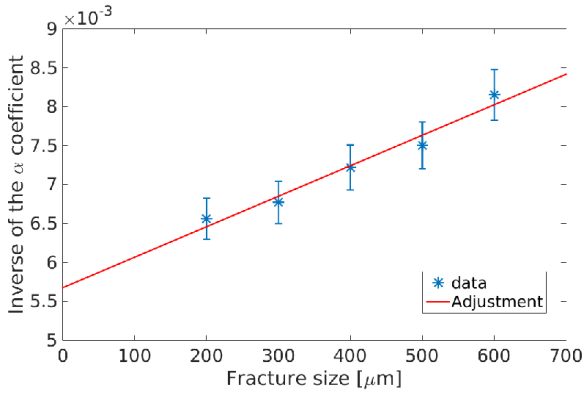

Fig 8. Linear relationship between the crack size and the decay constant with y =3.92x10^{-6}X + 0.00568.

Table 2 Decay constants for each specimen.

Thickness of the fracture [mm]	A [mV]	α	T_2 [ms]
0.2	2673	152.4	7.55
0.3	2671	147.7	7.78
0.4	2754	138.8	8.10
0.5	2740	133.3	8.36
0.6	2743	122.7	8.89

VI. Conclusion

The primary objective of this paper is to propose a composite nondestructivemethodology based on NMR relaxometry and through-transmissionultrasound for crack assessment in cement paste specimens. Single-crackspecimens were investigated in a preliminary study of the capabilities of thecomposite Ultrasound-NMR technique. Through-transmission ultrasound isa well-established nondestructive technique for the assessment of heterogeneousmaterials, especially for the detection and location of inhomogeneitiesor cracks within the tested media. Because a cement paste mixture is ahighly dispersive medium, the acquired ultrasonic signals are very complex,and the crack information is embedded within the echoes originating fromthe microstructure (see figure 4); therefore, some signal-processing transformationmust be applied to enhance the defect echoes. For that purpose, theHilbert-Huang transform was chosen based on its ability to analyzing nonlinearand nonstationary data. In this study, the methodology was provedto be capable of locating a single fracture, but it is expected to be applicableto ultrasonic B-scan signals for the multiple-fracture case.

On the other hand, NMR relaxometry, a more recently developed nondestructivemethodology, provides information regarding the microstructureand crack size based on the water content of the material and the mobilityof water in the pores. Two relaxation times are generally measured in suchNMR experiments: T_1 is related to the hydrogen adsorbed onto surfaces,which has almost no mobility, and therefore, T_1 is typically long (4100 ms);T_2 corresponds to the hydrogen present in water confined on the fracture(tipically T_2< 10 ms). Thus, the size of the fractures within cement pastespecimens with a w/c ratio of 0.5 could be obtained by determining the T_2relaxation time. The T_2 value was found to be between 7 ms and 9 ms, that exhibited a linear dependence on the crack size(figure 8), with a larger crackcorresponding to a longer relaxation time. The filtered ultrasonic signals permitthe detection and location of fractures within the tested specimen whenthe microstructure of the medium is defined as grain noise, and the fittedNMR distribution allows the dimensions of the fractures to be determined.

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