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LABORATORY CHARACTERIZATION OF CARBON-EXPOSED SANDSTONE

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ABSTRACT
To better understand the mechanical behavior of geologic CO_2 reservoir material, the non-linear behavior of dynamics and elastic wave propagation in Berea sandstone samples is investigated. Nonlinear characterization methods, including nonlinear resonance ultrasound spectroscopy (NRUS) and sequential dynamics impact test (SDIT), are applied to pristine and carbon-exposed Berea samples. The results can be used to reveal the characteristics of geologic reservoir materials that are most affected by varying microstructural and environmental conditions. An analysis of the work may lead to bases for test methods that could be deployed in the field in the future to monitor the condition of reservoir formations, and lead to better understanding of CO_2 injection-induced seismic events.

Keywords: GSCO2, nonlinear vibrational methods

NOMENCLATURE
α nonlinear hysteretic parameter

1. INTRODUCTION
With the growing concern about greenhouse gases and global climate change, capture and storage of CO_2 in deep geological formations, such as depleted oil and gas reservoirs, provides a promising solution to mitigate the exponentially increasing release of atmospheric greenhouse gases since industrial revolution [1, 2]. CO_2 injection in geological formations may alter the pore pressure and stress state there, and it is reported that microseimicity rates correlate with periods of elevated CO_2 injection rates [3]. Thus, it is important to investigate the effects of CO_2 injection on the mechanical behavior of reservoir material. Previous researchers reported the influence of CO_2 injection on sandstone samples result in slight variation on mechanical properties, including tri-axial loading strength and ultrasound P-wave velocity [4, 5]. In this study, nonlinear parameters, charactering both fast and slow dynamics behaviors, of the pristine and carbon exposed Berea sandstone samples are investigated using nonlinear vibrational test methods, namely nonlinear resonance ultrasound spectroscopy (NRUS) and sequential dynamics impact test (SDIT).

2. MATERIALS AND METHODS
2.1 Berea sandstone sample and CO_2 treatment
In this study, Berea sandstone was selected for laboratory characterization because it has been used as a standard for reservoir rock formation. Two samples were cut and cored from the same rock block to minimize the variation of material property among test samples, considering the inhomogeneity of natural rocks. The sample geometry and density can be found in Tab. 1.

TABLE 1: SAMPLE GEOMETRY AND DENSITY

	Length (mm)	Diameter (mm)	Density (kg/m^3)
Sample 1	154.0	31.0	2035.9
Sample 2	153.3	30.9	2045.7

For CO_2 treatment, the Berea sandstone sample 1 and 2 were placed into a vacuum chamber for 24 hours to remove any air from the core. While the sample was still under vacuum, deionized water (DI) was forced into the vacuum chamber. The samples then spent 24 hours in DI while under vacuum to ensure saturation. After saturation, the samples were placed in Parr batch reaction vessels (series 4650) with glass liners. DI filled the reactor up to the half way mark on the core. The vessels were then sealed and CO_2 was injected with a Maximator gas booster pump to approximately 100 bar. The vessels were then placed in an oven at 50°C, after equilibrating with the oven temperature, reached pressures of over 170 bar with supercritical CO_2.

After two weeks in the oven, valves on the vessels were opened to allow degasing overnight. This method avoided the formation of dry ice within the reactor and minimized rapid and potentially damaging degassing in the sample. The samples were flipped over and placed back in the vessels, so the end that had

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previously been in direct contact with the supercritical CO_2 was submerged in water and vice versa. The water from the first part of the experiment was reused for the second. Once the samples were back in the vessels, the same procedure was followed for pressurizing as used in the first part of the experiment. After two weeks, the vessels were again degassed and the samples removed.

2.2 Nonlinear resonance ultrasonic spectroscopy

Nonlinear resonance ultrasound spectroscopy, first developed by Van Den Abeele et al. [6], reveals the nonlinear softening of the elastic modulus with increasing drive input energy. The specimen is held with flexible strings with minimum constraint along its axial direction, such that a free-free boundary condition can be assumed. A piezoelectric disk is mounted on one end of the sample, and an accelerometer or Laser Doppler Vibrometer is installed/targeted on the other end to records its dynamic response, as shown in Fig. 1. It is noted that the ultrasound transducers were not used during the NRUS test.

Typically, the NRUS measurement generates a single extensional vibration mode in samples with bar-like geometry, using the bonded piezoelectric disk. Multi-cycle harmonic excitation signals with sweeping frequencies (within the range of one single resonance) is generated as sinusoidal bursts and amplified as the driving input voltage of the piezoelectric disk; the steady-state measurement is obtained on the other end. With ascending or descending drive input voltage, an amplitude-dependent resonance frequency, and thereby the dynamic elastic modulus, can be observed and correlate with the maximum strain amplitude developed in the sample under a one-dimensional extensional mode assumption. For brittle porous materials (rock, concrete, etc.), the resonance frequencies shift towards lower frequencies with higher strain amplitude (softening). The nonlinear parameter alpha is defined as the relative shift in resonance frequency per unit strain [6]. In this study, NRUS test was conducted on Berea sandstone sample 1 at its pristine and CO_2-treated status, in a controlled test environment (25-26 ℃ and 55-57% relative humidity).

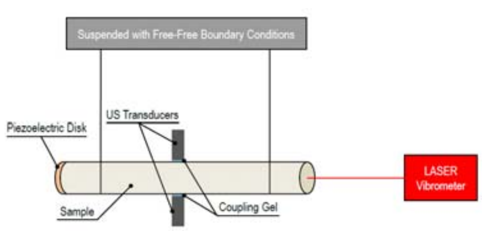

FIGURE 1: SCHEMATIC FOR NRUS

2.3 Sequential dynamics impact test

Sequential dynamics impact test was developed at University of Illinois by the authors, to observe and measure the slow dynamics behavior of materials. Several studies reported that ultrasound P-wave velocity, as index of dynamic elastic modulus, evolves over time with continuous low-frequency high-strain excitation, and it recovers over a period of time after stopping strain disturbance [7]. The SDIT focuses on capturing material conditioning towards non-equilibrium steady-state established by a sequence of impacts.

The setup of Sequential dynamics impact test is illustrated in Fig. 2 (left). For an individual impact, we follow the ASTM C215-14 standard for fundamental transverse resonant frequencies: sample under test is supported at two anti-nodes, impacted at the midpoint of the sample length, and its dynamic response is measured at one end by an accelerometer. Within each impact sequence, an automated impactor carries out 100 sequential impacts on the sample under test with consistent input energy and fixed time interval of 3 seconds, and the accelerometer records all 100 time signals. The control and data acquisition systems were developed based on MATLAB and Arduino platforms. As the sample is being conditioned with sequential impacts, the fundamental transverse resonance shifts towards lower frequencies as shown in Fig. 2 (right), which confirms material softening as conditioning to non-equilibrium status or stiffening as recovering with memory mechanism. In this study, SDIT was conducted on Berea sandstone sample 2 at its pristine and CO_2-treated status, in a controlled test environment (32 ℃ and 58% relative humidity). Multiple impact sequences were conducted to verify its repeatability.

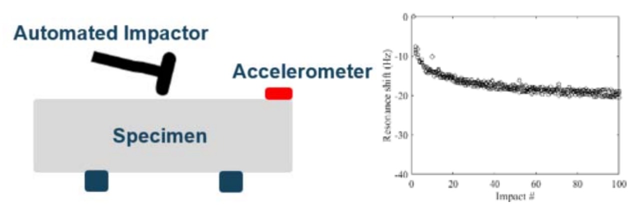

FIGURE 2: SCHEMATIC OF TEST SETUP FOR SDIT AND TYPICAL MEASUREMENT RESUTLS

3. RESULTS AND DISCUSSION

3.1 Density measurement

Table 2 summarizes the change of density from the carbon treatment, which concludes a negligible change in material loss (clay portion dissolves in acidic liquid) resulting from carbon injection.

TABLE 2: DENSITY OF SAMPLES BEFORE AND AFTER CARBON TREATMENT

	Density-Pristine (kg/m^3)	Density-Carbon treatment (kg/m^3)
Sample 1	2035.9	2034.5
Sample 2	2045.7	2044.4

3.1 NRUS results of sample 1

The NRUS results of sample 1 at its pristine and carbon treated status are shown in Fig. 3, at its third extensional mode about 19 kHz. In Fig. 3 (left), the measurement results on pristine sample are demonstrated with black curves and the resonance peaks are identified with a black circle; the results on carbon treated sample are shown with red curves and the resonance peaks are identified with a red circle. It is obvious that sample 1 after carbon treatment has a higher third extensional resonant frequency compared to its pristine condition, in contrast to the case of thermal damaged or defective samples, which shall come with lower resonant frequency. Secondly, with similar levels of input energy, the resonance shifts to lower frequencies at a higher rate, compared to the carbon-treated condition, indicating less softening in the carbon treated sample. This phenomenon can be further identified in Fig. 3 (right), in which the relative frequency shift is plotted against the maximum strain level developed in the Berea sandstone samples: the negative slope of the pristine sample (α = 0.0044) is significantly lower than the carbon-treated case (α = 0.002). The carbon exposure on Berea sandstone sample results in reduction of nonlinearity.

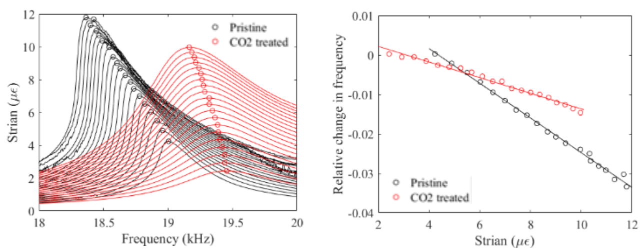

FIGURE 3: NRUS RESULTS FOR PRISTINE AND CARBON EXPOSED BEREA SANDSTONE SAMPLE

3.4 SDIT results

Multiple impact sequence data with SDIT on sample 2 at its pristine and carbon treated states are shown in Fig. 4, where the y-axis represents relative resonance shift in Hz and the x-axis represents time in seconds in logarithm scale. The large drop between the first and second impacts can result from different contact condition between impactor and sample surface. With repeated impulse excitations, the fundamental transverse resonance shifts towards lower frequencies, as shown in Fig. 2 (right) (softening with conditioning on brittle materials).

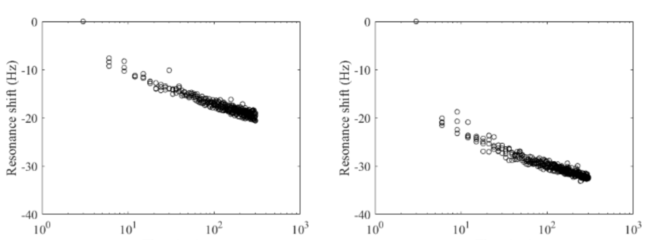

FIGURE 4: RESONANCE SHIFT VS LOG(TIME) WITH THREE REPEATITIONS ON PRISTINE AND CARBON TREATED BEREA SANDSTONE SAMPLE 2

For reliable slow dynamics behavior characterization, the slope of the relative resonance shift vs log(time) of the last 90 impacts out of 100 within each impact sequence is estimated to quantify the rate of conditioning with the same impact configuration. Three impact sequences were conducted on sample 2 at both the pristine and carbon treated status, which provide consistent measure for each case as shown in Fig. 4. The averaged slope at each status characterizes the slow dynamics behavior with the rate of converging towards non-equilibrium state under impulse conditioning: the averaged slope of resonance shift vs log(time) on pristine sample 2 in Fig. 4 (left) is -6.37; after the carbon treatment, the averaged slope in Fig. 4 (right) is -5.39, which reflects the influence of carbon injection on the slow dynamics behavior of Berea sandstone material. Again, the carbon exposure results in reduction of nonlinearity.

4. CONCLUSION

In this study, the influence of carbon injection in Berea sandstone has been investigated with nonlinear vibrational methods. Amplitude-dependent dynamics elastic modulus (fast dynamics) and material conditioning to non-equilibrium status (slow dynamics) are both observed, and affected by the designed carbon treatment. While metrics, such as density, are unable to indicate significant difference, the appreciable sensitivity offered by the nonlinear vibrational methods with both the fast and slow dynamics behavior confirms its effectiveness for geomaterial characterization. The carbon treatment on Berea sandstone samples reduced the nonlinearity of the material in both the fast and slow dynamics behavior.

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