

# Broadband laboratory measurements of dispersion in thermally cracked and fluid-saturated soda-lime-silica glass

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# **SUMMARY**

To better understand the dispersion of seismic velocities arising from stress-induced fluid flow, broadband laboratory measurements have been conducted on a range of synthetic samples. Forced oscillation methods providing access to low frequencies (mHz - Hz) were combined with measurements at MHz frequencies with ultrasonic methods. Either fully dense soda-lime-silica glass or aggregates of sintered glass beads were subject to broadband tests before and after thermal cracking under dry, argon- and water-saturated conditions in sequence. Crack closure effects under pressure are observed on all samples. A systematic increase in shear modulus, attributed to the suppression of 'squirt' flow, has been monitored on the low-porosity (approximately 2%) cracked glass-bead specimen with both argon and water saturation at ultrasonic frequency. The use of samples with different porosities varying from 0 to 6% promises to distinguish the roles of pores and cracks in fluid-flowinduced dispersion.

Key words: rock physics, seismic properties, soda-limesilica glass, poroelasticity, dispersion

# **INTRODUCTION**

The frequency of active-source seismic methods used in the field is within the range of tens to hundreds of hertz. In contrast, the conventional laboratory technique - ultrasonic pulse transmission method - is conducted mainly at megahertz frequencies. The measurements at different frequencies are complicated by the presence of pore fluids, the behaviour of which is highly dependent on the timescale of excitation. A thorough study of performing broadband measurements on samples with different petrophysical properties and saturated with pore fluids with different viscosities is needed in order to better extrapolate laboratory results to in-situ seismic explanations.

Gradients in pore-fluid pressure, either between cracks with different orientations or between cracks and pores, can be created during the passage of a seismic wave through a fluidsaturated medium. But this pore-fluid pressure gradient can be relaxed by local fluid communication ('squirt') between cracks or between cracks and pores. Conditions are described as 'saturated isobaric' if such squirt flow achieves a uniform pore-fluid pressure throughout the whole specimen without fluid exchange between specimen and external reservoirs. At higher frequencies, the pore-pressure gradient cannot be relaxed on the timescale of wave. The suppression of squirt flow in this 'saturated isolated' regime causes stiffening of the fluid-saturated medium (O'Connell and Budiansky, 1977; Mavko and Nur, 1975).

Theoretical research on this fluid-flow-related dispersion is extensive but has not been thoroughly and unambiguously Technically, broadband tested experimentally so far. measurements have been hindered by the difficulties of performing low-frequency tests to supplement the wellestablished ultrasonic techniques. The combined use of innovative forced-oscillation methods at the Australian National University at mHz-Hz frequency and ultrasonic technique at the University of Alberta at MHz frequency makes it possible to access widely-spaced frequencies and fluid-flow regimes. The limited experimental attempts in the past of broadband measurements have also mainly focused on natural geological materials such as Fontainebleau sandstones (David et al., 2013) and carbonates (Adam et al., 2006). The physical mechanism of dispersion is veiled by the mineralogical complexity of natural samples. To this end, a set of synthetic soda-lime-silica glass samples with much simpler composition and microstructure, either fully dense or aggregates of sintered glass beads with porosities ranging from 2% to 6%, was fabricated and measured.

This abstract will report the methods of preparing synthetic samples with required petrophysical properties first, and then broadband techniques with an emphasis on the low-frequency forced-oscillation method. Preliminary results focusing on pressure-dependent crack closure and dispersion will then be presented.

# METHOD AND RESULTS

## **Sample Preparation**

Soda-lime-silica glass is used as the synthetic analogue of sandstone in the sense of a high weight percentage of silica (about 75%). Fully dense soda-lime-silica glass rods (Fig. 1) are used to present non-porous media, whereas samples with prescribed levels of porosity can be achieved by sintering soda-lime-silica glass beads in a controlled way (Fig. 2). The low-porosity (about 2%) samples are made from soda-lime-silica glass beads with diameters ranging from 300 to 350  $\mu$ m (Fig. 2a), which are subsequently sintered at 700 °C for 18 hours.



Figure 1. Fully dense soda-lime-silica glass rod before and after thermal cracking.

A different batch of glass beads of small diameter between 180 and 210  $\mu$ m is used, following the same thermal protocol, to achieve a higher residual porosity of about 5% after sintering (Fig. 3, right).



Figure 2. The process of fabricating low-porosity (about 2%) glass-bead samples. (a) soda-lime-silica glass beads with diameters between 300 and 350  $\mu$ m; (b) glass-bead specimen sintered at 700 °C for 18 hours; (c) precision ground specimen with 15 mm diameter and 50 mm length; (d) cracked specimen by quenching from 500 °C into liquid water at room temperature.

All sintered aggregates of glass beads are precision ground into cylindrical shape with length of 50 mm and diameter of 15 mm. Thermal cracks are later introduced to facilitate fluid saturation by quenching samples from 500 °C into liquid water at room temperature. About 0.2% increase in sample volume, i.e., crack porosity, was found after thermal cracking. All cracks have uniformly low aspect ratio of about  $7 \times 10^{-4}$ .



Figure 3. Comparison in microstructure between lowporosity (about 2%, left) and high-porosity (5 - 6%, right) samples.

#### Forced-oscillation and Ultrasonic Methods

Ultrasonic method is a standard laboratory technique which involves the measurement of the traveltime of an ultrasonic pulse generated by a piezoelectric transducer at 1 MHz, travelling through the studied sample and finally received by another piezoelectric transducer at the other end of the sample. The forced-oscillation technique (Jackson and Paterson, 1993) may be less familiar and will be briefly described below.



Figure 4. Experimental arrangement for (a) computer control and data acquisition for forced-oscillation studies in (b) flexure (bending force) (c) torsion modes with alternative polarisations of electromagnetic drivers and displacement transducers.

The cylindrical specimen is connected mechanically with a hollow steel elastic standard under pressure to form an integral beam cantilevered at its top. A seismic-frequency oscillating torque (torsional mode) or bending force (flexural mode) is applied by a suitably configured parallel pair of electromagnetic drivers near the end of the beam. Displacements associated with either the twist or flexure of the beam are measured by two pairs of three-plate capacitance transducers with a precision of measured strain amplitude down to  $10^{-8}$  (Fig. 4).

By conducting a parallel experiment with a purely elastic control specimen with the same geometry and known properties under the same pressure condition, the shear modulus of the studied specimen can be inferred by comparing the behaviours of both. In addition, the phase difference between the studied specimen and the mechanically coupled elastic standard subject to the same oscillating torque can provide information on dissipation (1/Q). A filament elongation model is used to extract the optimal Young's modulus of specimen to match the observed displacements of the cantilevered beam subject to a bending force at the bottom end (Jackson et al., 2011).

# **Experiment Setup**

Specimens were encapsulated in either an annealed copper jacket (for forced-oscillation measurements) or rubber tubing (for ultrasonic measurements) to separate pore fluids from the confining pressure medium. The range of confining pressure was within 150 MPa and 100 MPa, respectively, for forcedoscillation and ultrasonic measurements. Argon and water were introduced into the crack network of sample as pore fluids, combined with confining pressure to create various differential pressures (confining pressure minus pore pressure) spaced at intervals of  $\sim 10$  MPa.

All glass samples were measured both before and after thermal cracking to observe the effects of thermal cracks. Data were collected at seven different oscillation frequencies (10, 21, 46, 87, 156, 260, and 781 mHz) for forced-oscillation experiments and 1 MHz for ultrasonic method.

#### **Results and Discussion: Pressure-induced Crack Closure**

A spheroidal crack with an initial aspect ratio  $\alpha$  is expected to be closed at differential pressure  $P_d \sim E\alpha$ , where E is Young's modulus of the solid. For the glass-bead specimen, with E  $\approx$  70 GPa, and  $\alpha \approx 0.0007$ , crack closure is thus expected at differential pressure of about 50 MPa - consistent with the observations (Fig. 5). The thermally cracked glassbead rod has minimum pressure sensitivity for shear modulus beyond ~50 MPa. This also indicates that few cracks within this glass-bead sample has aspect ratio higher than 0.0007.



Figure 5. Pressure dependence of shear modulus of sintered glass-bead rod after thermal cracking. All cracks are firmly closed beyond 50 MPa.

The slightly decreasing trend of shear modulus with increasing confining pressure may result from the negative pressure derivative of shear modulus for silica-rich material.

#### **Results and Discussion: Dispersion Related to Fluid Flow**

Systematic stiffening was observed for argon and especially water saturation at low differential pressures for ultrasonicfrequency measurements (Fig. 6). From the Gassmann equation (1951), relevant to the saturated isobaric regime, the shear modulus of fluid-saturated medium should be equal to that of dry medium. The observed increase in shear modulus for both argon and water saturation may therefore indicate the transition from saturated isobaric to saturated isolated regime with the local "squirt" flow suppressed.

In work in progress, this notion is being checked by theoretical prediction. The characteristic frequency that separates saturated isobaric and saturated isolated regimes is  $K_0 \alpha^3 / \eta$  given in O'Connell and Budiansky (1977), where  $K_0$  is the solid's bulk modulus,  $\alpha$  is aspect ratio of crack, and  $\eta$  is viscosity of pore fluid. Taking  $K_0 = 40$  GPa for uncracked soda-lime-silica glass,  $\alpha = 0.0007$  for cracks within this set of samples, and  $\eta = 30$  µPa·s for argon viscosity at 10 MPa, the

characteristic squirt-flow frequency is about 0.5 MHz, which indicates the fluid flow at 1 MHz is marginally within saturated-isolated regime. Considering the viscosity of water is significantly higher than that of argon by two orders of magnitude, the behaviour of water flow within the sample is certainly in the saturated isolated regime.



Figure 6. Shear modulus of the cracked glass-bead specimen of about 2% porosity measured under dry, argon-saturated, and water-saturated conditions at ultrasonic frequency. A systematic stiffening effect caused by pore-fluid saturation can be observed at this frequency.  $P_c$ ,  $P_f$ , and  $P_d$  denote confining pressure, pore pressure, and differential pressure, respectively. The labels "up" and "down" correspond, respectively, to data obtained during increasing and decreasing  $P_c$  or  $P_d$ .

David and Zimmerman (2012) proposed a micromechanical model based on Eshelby's results and using differential effective medium scheme to extract crack-density parameter and the aspect-ratio distribution from the pressure dependence of dry elastic moduli. The model assumes that closable and nonclosable pores can be presented as spheroids (a spheroid is an ellipsoid with two equal axes) that have an aspect ratio  $\alpha$ . This model is being applied to the soda-lime-silica glass sample to predict the effect of fluid saturation with observations under dry conditions as input.

The crack-density parameter  $\Gamma$  is first inverted at each pressure from the modulus deficit relative to that for the uncracked glass under dry conditions. Use of another crack-closure equation allows the distribution of crack aspect ratio to be extracted from the pressure dependence of crack density. The differential effective medium scheme is then used in the forward-modelling sense, with the previously inverted crackaspect-ratio distribution, to predict the consequence of argon or water saturation for comparison with the experimental data.

#### CONCLUSIONS

The fluid-flow related dispersion has been clearly documented on the synthetic soda-lime-silica glass samples. The transition from saturated-isobaric to saturated-isolated regime clearly occurs between the seismic and ultrasonic frequency for both argon and water saturation. The implication from this finding is clear: the use of ultrasonic results will overestimate values relevant to seismic frequency with the presence of pore fluids, and allowance must be made for the squirt-flow-related dispersion.

Future work of this project will involve resonant bar technique to provide access to the intermediate "sonic" frequency to better resolve the fluid-flow-regime transition.

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