

# Permeability and seismic-frequency elasticity of cracked glass

**Abdulwaheed Ōgúnsàmi \***

Research School of Earth Sciences  
Australian National University  
Canberra, ACT  
Australia  
abdulwaheed.ogunsami@anu.edu.au

**Yang Li**

Research School of Earth Sciences  
Australian National University  
Canberra, ACT  
Australia  
peter.yang.li@gmail.com

**Ian Jackson**

Research School of Earth Sciences  
Australian National University  
Canberra, ACT  
Australia  
ian.jackson@anu.edu.au

*\*presenting author asterisked*

## SUMMARY

With the objective of improved understanding of frequency-dependent properties of cracked and fluid-saturated media, permeability and forced oscillation measurements are being undertaken on dry and fluid-saturated cracked glass media. In particular, we address some of the issues outstanding from a prior intensive study across a wide range of frequency. Our preliminary results demonstrate the potential of improved methods for the measurement of the very low permeabilities of such cracked glass media, the feasibility of torsional forced oscillation measurements at longer oscillation periods and lower differential pressures, and the benefits of improved alignment of our specimen assembly for complementary flexural oscillation tests. Such complementary flexural oscillation tests on glass-rod specimens are expected to provide further insight into the relevant fluid-flow regimes.

**Key words:** Permeability, poroelasticity, dispersion, dissipation, synthetic glass media

## INTRODUCTION

Improved understanding of the effective properties of fluid-saturated crustal reservoirs is of interest in exploration and production geophysics where the role of cracks and pores on elastic properties has increasingly received more attention. This is largely due to increasing economic significance of efficient seismic characterisation and time-lapse monitoring of crustal reservoir rocks for petroleum production, geothermal energy production, carbon dioxide sequestration and efficiency of nuclear waste reposition. For these purposes, more robust interpretation of seismic velocity measurements in terms of rock porosity and fluid content is highly desirable.

In order to constrain seismic data, and to understand the effect of fluid saturation, laboratory ultrasonic measurements of elastic properties are often made. However, such laboratory measurements of elastic velocity, in fluid saturated media often differ significantly from field seismic velocity. The difference in magnitude – called dispersion – is related to the noticeable difference in frequency between the laboratory and the field techniques with the potential to introduce significant uncertainties. Thus, the need to account for this frequency dependent difference between these measurements is key to reconcile laboratory ultrasonic measurements (~MHz range) with field data from borehole (~tens of kHz) and exploration (~10–100 Hz) geophysics.

Dispersion is caused by fluid mobility in the pore spaces during wave propagation and the degree to which the dispersion takes place is largely related to whether the fluid mobility and pressure communication within the pore systems is enhanced completely (i.e. in equilibrium) or inhibited. For example, in the low frequency poroelastic (undrained) regime of Biot- Gassmann's theory, there is sufficient time during the wave propagation for enhanced fluid mobility and pore pressure communication and thus, pores systems are said to be locally in equilibrium. Whereas, in the high frequency (limited time) regime, the mobility of the pore fluid tends to be inhibited as some of the fluids are trapped and isolated in each of the individual pores, limiting the complete pressure communication among the pores/crack and thus leading to pore pressure difference between adjacent cracks/pores - a condition responsible for higher stiffness/moduli often obtained from laboratory measurements at intermediate to high (ultrasonic) frequencies. (Dvorkin et al., 1995)

Despite the extensive theoretical improvement, clear understanding of the mechanism governing the dispersion of elastic properties of crustal rocks remains very topical, particularly as the role of microstructures is yet to be clearly demonstrated by a robust and conclusive laboratory investigation. While it is established that the squirt/microscopic (e.g Mavko and Jizba, 1991) flow models (which are based on the geometries of the cracks and microstructures) can be efficient in modelling the observed dispersion, the often-required input for this model i.e. quantitative information about these microstructures such as aspect ratios, crack density etc., are not easily constrained/obtained from rocks due to the nature of their complex depositional structures and fabrics. In addition, several of the previous laboratory measurements were made either at low or high frequencies, with insufficient frequency bandwidth to observed the expected stress/flow regimes as predicted from the theoretical models (e.g O'Connell and Budiansky, 1977)

Several authors have performed laboratory investigation from low to high frequencies on a few crustal rocks (e.g. Adam et al., 2006, 2009, David et al., 2013, Mikhaltsevich et al., 2014). However, the findings are not without ambiguity, especially on the role of microstructures, as rocks can be complex in microstructures. Li et al. (2017a,b), in a recent extensive laboratory study, investigated broadband frequency dependent elastic properties of synthetic glass media. Synthetic material provides an opportunity for a simplified microstructure, the geometry of which can be carefully controlled during the fabrication process, which provides a chance

of being accurately characterized and thus amenable to systematic interpretation which can be linked with the observed fluid flow related dispersion.

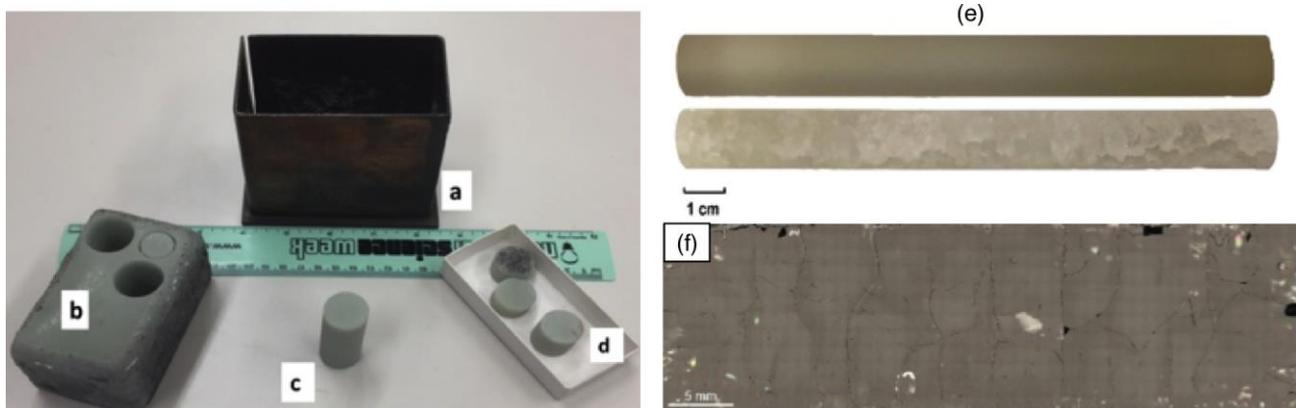
This abstract reports further work related to Li et al (2017ab) with focus on improved methods of preparing synthetic samples as well as additional low-frequency laboratory measurements. With a cracked glass-rod specimen from Li et al. (2017a,b) and work in progress on newly prepared glass-rod and sintered glass-bead specimens, we are addressing the following outstanding issues in order to develop a more robust understanding of the seismic signatures of fluids in saturated cracked media.

- I. Further investigation of the milder-than-exponential variation of argon permeability with differential pressure likely due to the flow of argon between the enclosing annealed copper jacket and the cylindrical surface of the thermally cracked, precision-ground glass specimen;
- II. Exploration of the possibility of observing the transition towards drained conditions at oscillation frequencies below 10 mHz,
- III. Assessment of the feasibility of measurements at differential pressures below the 10 MPa minimum of the Li et al. (2017a) study to better document the pressure-induced closure of cracks of very low aspect ratio
- IV. Acquisition of data complementary to those of torsional oscillation from flexural tests, that were precluded in the study by Li et al. (2017a) by poor alignment of the experimental assembly and finally
- V. Development of a physico-chemical explanation for the apparent impermeability of the specimen to water as a pore fluid

Additional data for the cracked glass-rod material FDSL1 of Li et al. (2017b), from further permeability measurements and forced oscillation tests, along with the results of exploratory flexural mode tests, and progress in the sintering of improved glass-bead materials is presented with preliminary results concerning the pressure dependence of permeability and torsional/flexural compliance.

## METHOD

**Sample preparation.** Sintered aggregates of beads of soda lime silica glass provide a suitable synthetic analogue of a well sorted, matured siliciclastic rocks which has a very huge economic importance as hydrocarbon reservoir rocks (**Figure 1a-d**), while initially fully dense glass rods of soda-lime-silica (**Figure 1e, f**) are used to investigate the effects of crack porosity alone (Li et al., 2017a). The sintered analogue material has residual equant porosities between 2-5%, while the glass rod is fully dense with zero porosity. The specimens were heated to 500°C and then quenched in water at room temperature as a way of introducing compliant cracks of low aspect ratio.

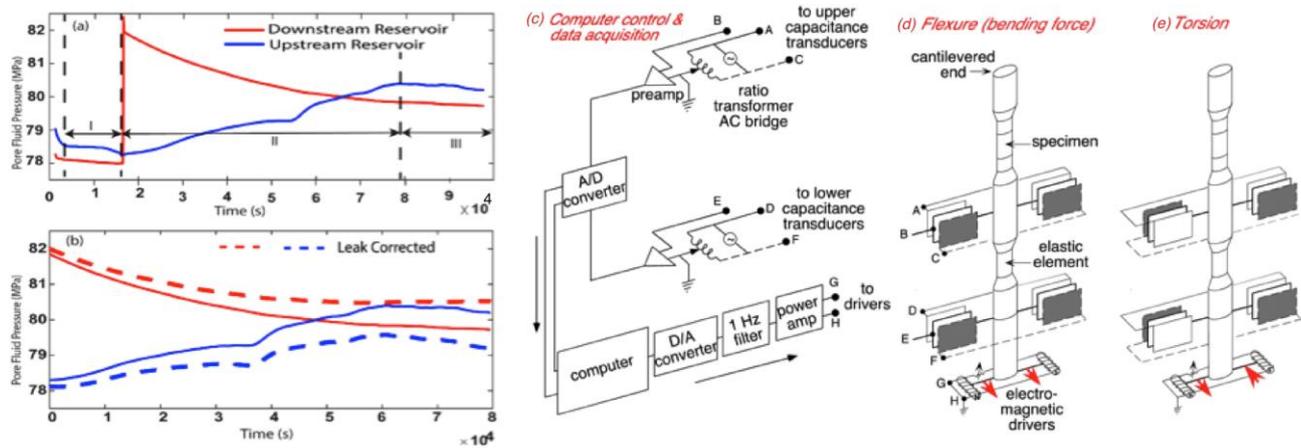


**Figure 1.** (a-d) An improved procedure for preparation of low-porosity samples by sintering soda-lime-silica glass beads: (a) The rectangular mould for the sintering process; (b) glass-bead block sintered at 700 °C for 18 hours; (c) cored specimen; (d) End trims of the specimens for thin section and further analysis. Note the 30cm long ruler as scale. (e) Soda-lime-silica glass rod before and after thermal cracking; (f) A longitudinal cross-section of a glass-rod sample, showing the network of thermal cracks.

To assess the effect of cracking, the specimen dimensions were measured before and after the thermal treatment. About 0.2% increase in sample volume, i.e., crack porosity, was found after thermal cracking (Li et al., 2017a). In this follow-up study, an improved sample fabrication approach is presented. Purer batches of glass spheres of diameter between 100 and 350  $\mu\text{m}$  are used, following the same thermal protocol, but with a rectangular mould from which samples of desired dimension can be cored (**Figure 1a-d**). The specimen is then precision ground into cylindrical shape of  $15.00 \pm 0.01$  mm diameter. This approach promises the possibility of horizontal coring to minimise the impact of any vertical variation in porosity while also guaranteeing specimens of uniformly low aspect ratio of about  $2 \times 10^{-4}$ . (Li et al., 2017b).

**In situ measurement of permeability.** Given that its pressure dependence is an important indicator of crack closure, permeability is an important physical property in the study of fluid saturated cracked media. The pulse decay technique of Brace et al. (1968) is performed on the Jackson-Paterson attenuation apparatus where argon, water, and newly, pentane may be introduced as pore fluids.

The pulse decay technique involves a sample bounded by two reservoirs that are initially of equilibrated (equal) pore pressures. A pressure perturbation is suddenly imposed on one of the reservoirs and the consequent pressure evolution on the unperturbed reservoir and/or consequent pressure pulse decay on the perturbed reservoir is monitored with time as the two reservoirs return to pore pressure equilibrium. The transient phase of the exponential pressure pulse decay (versus time) involved in pore-pressure re-equilibration by fluid flow through the specimen is described by linear time dependence of the logarithm of the pressure transient, with a slope proportional to the permeability. The analysis of the slow re-equilibration for cracked glass media of low permeability is being refined to account for the impact of finite leak rates (**Figure 2a, b**).



**Figure 2:** (a,b) The strategy for measurement of permeability: (a) Pore pressure variations with time highlighting the intervals (I) and (III), before and after pore-pressure perturbation and re-equilibration (II), used to estimate the leak rate; (b) Pore pressure variations during the re-equilibration phase (II) before and after leak correction; (c-e) Experimental arrangement for forced-oscillation studies: (c) computer control and data acquisition; (d,e) alternative polarisations of electromagnetic drivers and displacement transducers for (d) flexural (bending force); and (e) torsional modes.

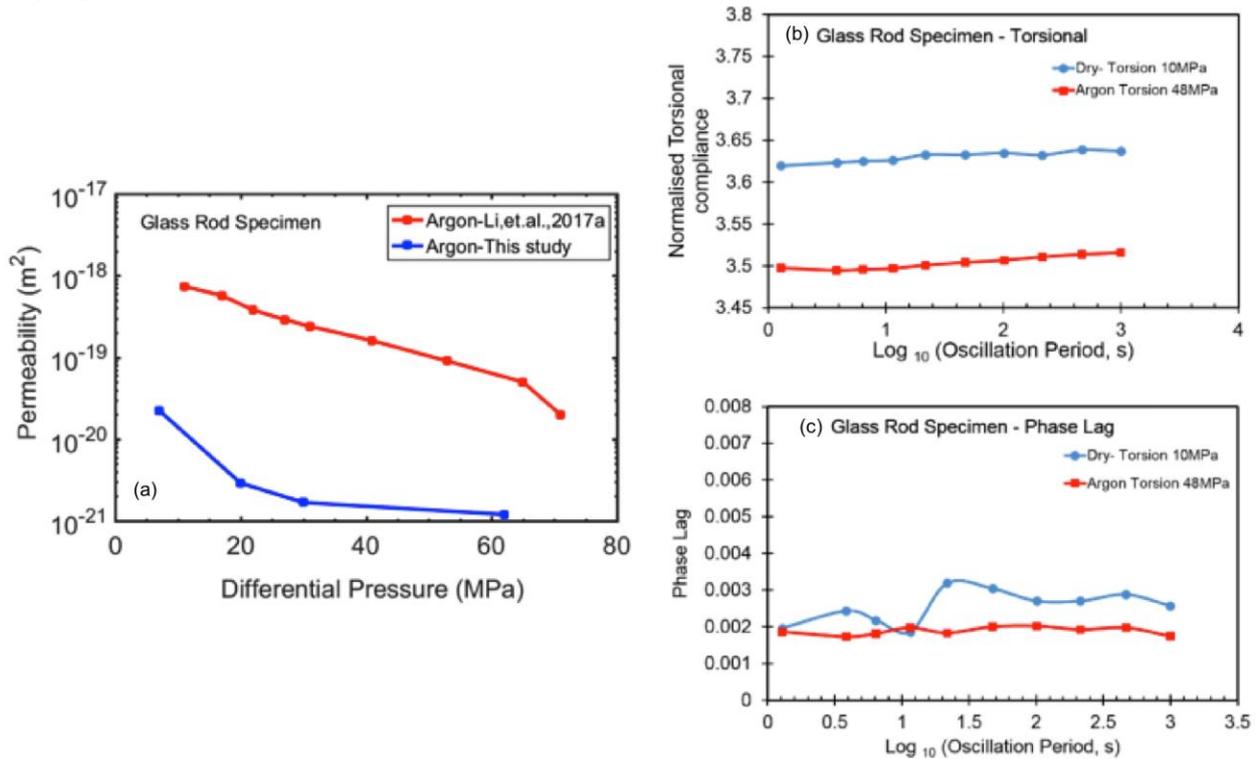
**Forced oscillation method.** For low-frequency forced-oscillation measurements (Jackson and Paterson, 1993), the cylindrical specimen is connected mechanically with a hollow steel elastic standard under pressure to form an integral beam cantilevered at its upper end. A seismic-frequency oscillating torque (torsional mode) or bending force (flexural mode) is applied by a suitably configured parallel pair of electromagnetic drivers near the lower 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 (**Figure 2c-e**). The relative distortions of the specimen assembly, containing the specimen of interest along with steel connecting rods, and the hollow steel elastic element, provide an interim indication (employed in this report) of the compliance and dissipation in the specimen itself, prior to the processing described in the following paragraph. By conducting a parallel experiment with a control specimen with the same geometry and known elastic properties under the same pressure condition, the shear modulus of the studied specimen can be inferred by comparing the behaviours of the two assemblies. 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).

**Experimental protocols.** In our forced-oscillation measurements, each specimen was encapsulated in an annealed copper jacket to isolate the fluid-saturated specimen from the argon confining pressure medium. Argon, pentane, 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 to a maximum of 100 MPa. Each glass sample was measured both before and after thermal cracking to observe the effects of thermal cracks. Prior pore-pressure re-equilibration tests ensured conditions of uniform pore pressure. Data were collected at ten different oscillation periods (approximately equispaced between 1 and 1000 s) during the forced-oscillation experiments.

## PRELIMINARY RESULTS

**Refinement of procedures for measurement of permeability** Physical properties of porous rocks (permeability, volumetric strain, porosity, etc.) are often found to vary as a function of confining pressure and pore pressure according to an effective pressure law commonly known as Terzaghi's principle. Many studies (e.g. Brace et al., 1968) have demonstrated this theory especially on tight rock samples (such as shales and mudstones) for which permeability follows an exponential (or power law) relationship with pressure. In a similar study on glass material, Ougier-Simonin et al. (2011) measured permeability on a set of glass samples and reported such an exponential relationship between permeability and differential pressure. In contrast, Li et al. (2017a), reported that such exponential variation was limited to pressures  $< 10$  MPa beyond which much milder pressure sensitivity was observed. Such milder-than-exponential variation of argon permeability with differential pressure may reflect flow of argon between the enclosing annealed copper jacket and the cylindrical surface of the thermally cracked, precision-ground glass specimen. To investigate such non-exponential behaviour, further in situ permeability measurements were made on the cracked glass rod specimen FDSL1. The new permeability measurements along with the previous measurement of Li et al. (2017a) are presented in **Figure 3a**. As seen in the figure, the newly measured permeabilities are consistently much lower than those reported by Li et al. (2017a). Since the sample was

last measured, it has been removed from its copper jacket, thoroughly cleaned, re-jacketed and re-measured under various combinations of confining and pore pressure. The possibility of further crack propagation during these procedures cannot be ruled out, but further damage would result in higher permeabilities, yet lower values were obtained. Understanding the differences between these two sets of measurement requires further explanation. The cross-over between the pore fluid pressures in the two reservoirs, during return to pore-pressure equilibrium (Figure 2a), is attributed to the effect of slow leaks from the pore-pressure reservoirs during the lengthy period ( $> 50,000$  s) of pore-pressure re-equilibration. Leak rates estimated from the time-dependence of pore pressures in segments (I) and (III) in Figure 2a, respectively prior and subsequent to the phase of pore-pressure re-equilibration (segment (II)), have been used to adjust the observed pore-pressure versus time trends as shown in Figure 2b. The procedure for such leak correction needs further refinement, and it is possible that inadequacies in the present procedure are responsible for the lower permeability values. However, the non-exponential behaviour from around 20 MPa of differential pressure, remains even in the newly acquired values.

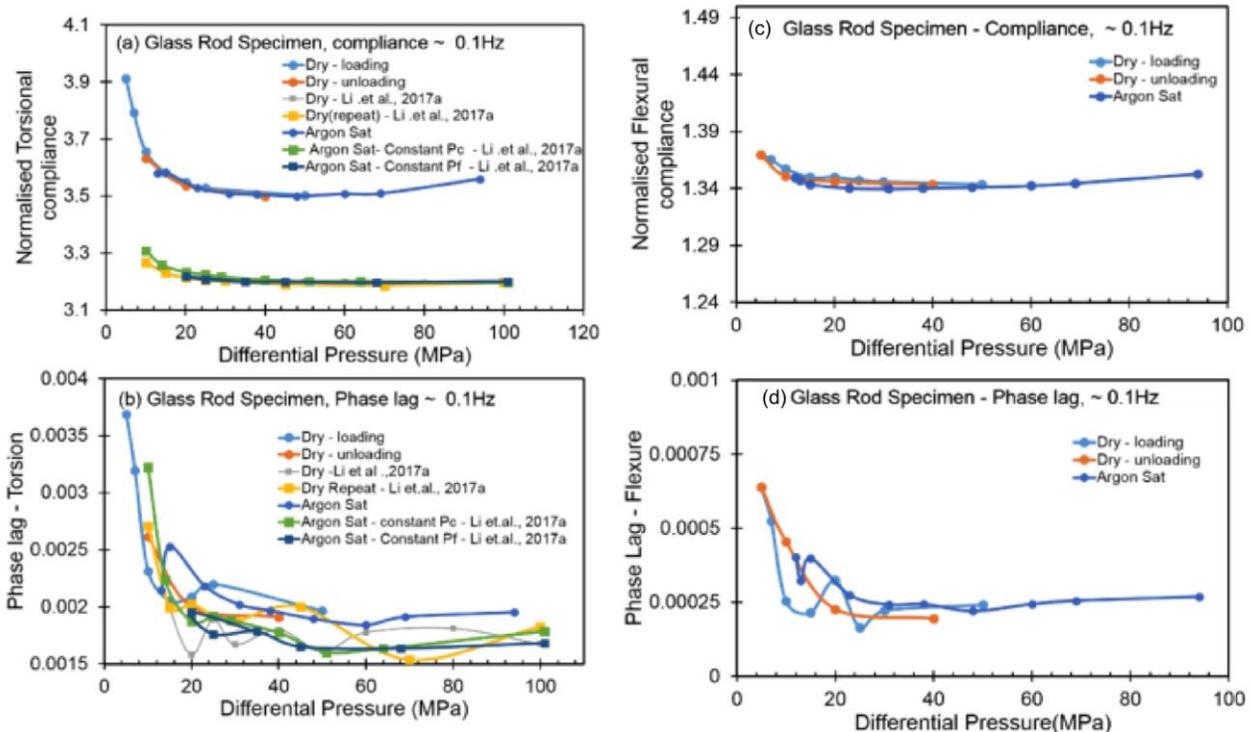


**Figure 3.** (a) Argon permeability measured as a function of differential pressure by the transient decay method on the thermally cracked glass-rod specimen FDSL1. Values from Li et al. (2017a) are plotted for comparison. (b) Normalised compliance, and (c) associated phase lag measured in torsional forced oscillation on the cracked glass-rod specimen FDSL1, versus oscillation period for representative dry and argon-saturated conditions over an extended range of oscillation periods.

**Draining transition at frequencies  $< 10$  mHz?** Stress-induced fluid flow in cracked fluid-saturated media is usually interpreted within a theoretical framework such as that of O’Connell and Budiansky (1977) with its conceptually distinct regimes of stress-induced fluid-flow. These fluid-flow regimes (from drained, through saturated isobaric, to saturated isolated) are separated by a set of transitions (with characteristic frequencies) involving both markedly significant dispersion of the elastic properties, and associated dissipation – reflecting the causality relationship between dispersion and dissipation. In the literature, there has been only limited explicit evidence of fluid drainage on suitable timescales (e.g. Pimienta et al., 2015a, b). Such conditions only occur (and can be sufficiently observed) at low frequencies, where the moduli are expected to be insensitive to fluid saturation. In this regard, the broadband (mHz - MHz) results for cracked glass-rod specimens (Li et al., 2017a) are somewhat surprising. For example, a marked increase in shear modulus is observed on water saturation even at sub-Hz frequencies. Such an increase in shear modulus should indicate saturated isolated conditions typically expected at kHz frequencies, which implies that that the drained condition for this set of specimens may possibly require much lower frequencies.

To further explore transition towards saturated isobaric, and ultimately, drained conditions, forced-oscillation work in progress involves systematic exploration of an expanded range of oscillation periods: 1-1000 s (frequencies of  $\sim 1$  mHz – 1 Hz). Figure 3(b,c) shows representative results across three decades of period. Normalised torsional compliance and phase lag obtained on glass-rod specimen FDSL1 tested dry at 10 MPa and argon saturated at 48 MPa differential pressure are presented. Each data set reveals mildly increasing compliance (implying decreasing shear modulus) with increasing oscillation period. For the corresponding phase lag data, there is essentially no significant change with increasing period. Such systematic exploration of the wider range of oscillation period has potential for the investigation of transitions between saturated isolated, saturated isobaric, and drained conditions, in our ongoing experimental program.

**The effects of crack closure at low differential pressure.** Experimental measurement of the low-frequency elastic properties (compliance and phase lags) of the glass-rod specimen FDSL1 made in this study are presented and compared with those of Li et al. (2017a) in **Figure 4 (a,b)**. The plotted compliances and phase lag are average values for periods of ~ 4 – 100 s. There are three key observations in this figure. Firstly, the newly measured compliances are significantly higher than those of Li et al. (2017a). Secondly, the new dataset extends to lower differential pressure (5 MPa) - important for study of the behaviour of the most compliant cracks. Despite these differences, however, a third observation is that the two datasets reveal broadly similar compliance versus pressure trends - consistent with our expectations concerning pressure-induced crack closure. In the previous study of Li et al. (2017a), the lowest differential pressure was 10 MPa. To better understand and document the pressure-related behaviour of low aspect ratio thermal cracks, the feasibility of measuring to somewhat lower differential pressures is being explored. The higher and more strongly pressure-dependent compliance and phase lag below 10 MPa in the newly acquired dataset in **Figure 4(a,b)** demonstrates the potential value of such measurements at lower differential pressure. Although the torsional compliances measured by Li et al. (2017a) and in the present study are markedly different in absolute value, the observed pressure dependencies of, both compliance and phase lag are broadly similar.



**Figure 4.** (a,b) Torsional forced oscillation measurements of the cracked glass-rod specimen FDSL1, versus differential pressure for dry and argon-saturated conditions. Plotted values of (a) compliance and (b) phase lag are averaged for periods of ~4 to 100 s; (c,d) Flexural forced oscillation measurements on the same cracked glass-rod specimen, versus differential pressure for dry and argon saturated conditions.

In **Figure 4(c,d)**, the flexural forced oscillation data across a range of differential pressure on the dry and argon-saturated glass-rod specimen FDSL1 are presented. The normalised flexural modulus varies systematically with confining/differential pressure within a narrow range (1.37 – 1.34) corresponding to Young’s moduli of 40-65 GPa approximately (Li et al., 2017a). A somewhat milder decrease in compliance, corresponding to an increase in Young modulus, can be seen when a cracked rod is saturated with argon.

**Apparent impermeability to water as pore fluid?** Work in progress with pentane and water, as condensed pore fluids of similar compressibility and viscosity but contrasting chemical properties, is designed to address the issue of the apparent impermeability of both glass-rod and glass-bead materials to water reported by Li et al. (2017a).

## CONCLUSIONS AND PROSPECTS

Substantial progress is thus being made addressing issues remaining from the previous study of Li et al. (2017a,b). Improved procedures for correction of pore-pressure re-equilibration records for the influence of pore-fluid leaks are showing promise in refining the measurement of the very low permeabilities of the cracked glass materials. Torsional oscillation measurements have been extended to longer periods and lower differential pressures with encouraging results. Complementary flexural oscillation tests on glass-rod specimen FDSL1 are expected to provide further insight into the relevant fluid-flow regimes. Finally, work in progress is expected to clarify the apparent impermeability to water of the cracked glass-rod specimen FDSL1. Additional specimens of glass-rod and sintered glass bead materials are being prepared for further broad-band inter-laboratory studies.

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