An apparatus to measure elastic dispersion and attenuation using hydrostatic- and axial-stress oscillations under undrained conditions

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ABSTRACT

An experimental apparatus is described for the investigation of frequency dispersion, and related attenuation, of fluid-saturated rocks under confining pressure and undrained boundary conditions. The forced-oscillation method is performed on cylindrical samples. The measurement of stress and strain under hydrostatic oscillations allows the dynamic bulk modulus to be inferred, while axial oscillations give access to dynamic Young's modulus and Poisson's ratio. We present calibration measurements for dispersion and attenuation on standard materials such as glass, plexiglass, and gypsum. Results show that for strain amplitudes below 10^{-5} , robust measurements can be achieved up to 1 kHz and 1.3 Hz, respectively, for axial and hydrostatic oscillations. A new experimental design of the endplatens (sample holders) allows control of drained or undrained boundary conditions using microvalves. The microvalves were tested on a porous Vosgian sandstone. In addition, numerical modeling confirms that the resonances of the apparatus only affect frequencies above 1 kHz, with little sensitivity to the sample's stiffness.

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I. INTRODUCTION

Dispersion (i.e., frequency-dependence) of the elastic properties of subsurface rocks constitutes a major challenge when trying to link seismic (~ 100 Hz) or sonic (~ 10 kHz) field surveys to laboratory standard ultrasonic measurements (~ 1 MHz). Through the causality principle, well expressed with Kramers–Kronig relationships, an elastic dispersion is conjugated with an elastic dissipation, often named “attenuation” (Q^{-1}) by analogy to viscoelastic solids (O’Connell and Budiansky, 1978). Wave induced fluid flows (WIFFs) are thought to be among the principal mechanisms for attenuation in subsurface rocks (Pride *et al.*, 2004). Each of the various mechanisms within WIFFs has a corresponding theoretical or modeling solution (e.g., Winkler and Nur, 1979; Müller *et al.*, 2010; and Sarout, 2012). It is, therefore, essential to experimentally verify these models under controlled and comparable conditions to reliably interpret field measurements.

Under fully-saturated conditions, one major mechanism at play may be the squirt-flow between compliant cracks and stiff pores. Characteristic frequency ($f \sim \xi^3 K_S / \eta$) is dependent on the crack aspect ratio (ξ), the skeleton bulk modulus (K_S), and the fluid's dynamic viscosity (η) (Mavko *et al.*, 2009). Therefore, for reservoir rocks with $\xi \sim 0.001$, $K_S \sim 40$ GPa and $\eta \sim 0.001$ – 0.1 Pa s, the squirt-flow attenuation may affect the frequency range from 400 Hz to 40 kHz.

In order to investigate these dispersive mechanisms, specific devices have been developed to measure the complex elastic moduli over broadband frequencies under the kHz range using the forced-oscillation method. In most apparatuses, if we ignore resonant bar techniques which measure narrow frequency bands in the sonic range (5–20 kHz), Young's modulus or Poisson's ratio is accessible in a large broadband (1 mHz to 1 kHz) through axial oscillations generated by piezoelectric actuators (PZTs) or dynamic shakers [e.g., Spencer (1981), Batzle *et al.* (2006), Mikhaltsevitch *et al.*

(2011), Tisato and Madonna (2012), Szweczyk *et al.* (2016), and Sun *et al.* (2018)]. The apparatus developed by Jackson and Paterson (1993) is able to measure the shear modulus and Young's modulus up to 1 Hz, through torsional and flexural oscillations, respectively (Jackson *et al.*, 2011). The previous apparatus at the Ecole Normale Supérieure de Paris (Pimienta *et al.*, 2015a; 2015b; and 2016b) was able to measure a bulk modulus up to 0.4 Hz with pure hydrostatic oscillations, and Young's modulus/Poisson's ratio up to 100 Hz through axial oscillations.

The new apparatus presented in this paper is able to measure the complex bulk modulus up to 1 Hz, and Young's modulus and Poisson's ratio up to 1 kHz using forced-oscillation methods, under either drained or undrained boundary conditions, and for temperatures between 20 °C and 100 °C. The frequencies of the viscous-driven WIFFs (such as squirt-flow) for a water-saturated rock may be shifted to lower frequencies with the use of more viscous pore fluids, such as glycerin (Batzle *et al.*, 2006). This enables us to extend the "apparent frequency" range of the apparatus (e.g., Borgomano *et al.*, 2017). We have recently reported some experimental results on carbonates or sandstones (Chapman *et al.*, 2018; Borgomano *et al.*, 2019b; and Yin *et al.*, 2019). We wish here to present the apparatus itself and the related calibrations.

II. PRESSURE VESSEL

A standard hydraulic triaxial cell from Top Industrie company (Fig. 1) was modified in order to investigate the

frequency-dependence of the dynamic moduli of cylindrical samples, of 80 mm length and 40 mm diameter. The cell is auto-compensated (i.e., the piston is hydraulically compensated for the confining pressure) and can reach a maximum confining pressure of 100 MPa and a maximum axial stress of 700 MPa, using servo-controlled hydraulic pumps. Initial experiments were conducted at room temperature (controlled at 20 °C). A servo-controlled heating collar located around the confinement cell enables heating and temperature control [through a proportional integral differential (PID) controller] to a maximum of 100 °C (Fig. 1). The temperature feedback is provided by a thermocouple (type K) located inside the confining oil. The sample is jacketed in a neoprene sleeve to separate the confining oil from the pore fluid and is maintained in between a top and a bottom endplaten, respectively, made out of steel and aluminum.

A piezoelectric actuator from PI (PICA Stack P-056-20P) is installed above the top endplaten, and under a cylindrical steel rod that can be put in contact with the axial piston (Fig. 1). A neoprene sleeve isolates the actuator from the confining oil. The axial piston is either not in contact with the top endplaten for pure hydrostatic conditions or in contact to apply a deviatoric stress.

The top and bottom endplatens, which contain the pore-fluid lines, are both equipped with two P- and S-ultrasonic transducers of 10 mm diameter and 1 mm height from PI Ceramics (type PIC255/ref PRYY+0.111), allowing for time travel measurements of P- and S-waves of 1 MHz in the axial direction

FIG. 1. Photo and schematics of the experimental apparatus. The sample is a cylinder of 80 mm length and 40 mm diameter, isolated from the confining oil by a neoprene jacket. A piezoelectric actuator is used to induce axial-stress oscillations (maximum 1 kHz), with the axial piston in contact with a small deviatoric stress of maximum 1.5 MPa. The confining pump is used to perform hydrostatic oscillations while the axial piston is not in contact (maximum 1.3 Hz). P- and S-wave ultrasonic transducers (1 MHz) are mounted on the top and bottom endplatens. Drained or undrained boundary conditions of the sample can be achieved through the two hydraulically piloted microvalves installed in the top and bottom endplatens.

of the sample. They are also equipped with hydraulically piloted microvalves, which enables us to switch between axially drained or undrained conditions for a saturated porous sample (Fig. 1).

The deformations of the sample can be measured with two internal LVDTs, which can measure the global strain in the axial direction with a resolution of 10^{-5} , and with axial and radial strain gauges directly glued on the sample (at mid-height), which can measure local strains with a maximum resolution of 0.3×10^{-7} (Fig. 1). The strain results reported here were acquired with TML 350 Ω foil strain gauges (FCB-6-350-11). Up to 14 strain gauges, connected in the quarter bridge, can be connected to the data acquisition system (DAQ). Four axial strain gauges are reserved for the bottom aluminum endplaten in order to deduce axial stresses (Fig. 1).

III. METHODOLOGY

The technique used to characterize the frequency dispersion and attenuation of the elastic moduli of a sample is the forced-oscillation method (or stress-strain method). This method applies an oscillatory stress to the sample and records the subsequent axial and radial strains (e.g., Spencer, 1981; Paffenholz and Burkhardt, 1989; Jackson and Paterson, 1993; Batzle *et al.*, 2006; Adelinet *et al.*, 2010; Mikhaltsevitch *et al.*, 2011; Takei *et al.*, 2011; David *et al.*, 2013; Madonna and Tisato, 2013; and Pimienta *et al.*, 2015a). All the signals are analyzed using the Fast Fourier Transform method to extract amplitudes and phases. The apparatus enables us to perform

two modes of stress oscillations: (i) hydrostatic, using the confining pump; (ii) axial, using the piezoelectric actuator.

For hydrostatic oscillations [Figs. 2(a) and 2(b)], the confining pressure oscillates with an amplitude (ΔP_c) of about 0.2 MPa around a mean value to induce a strain oscillation amplitude in the order of 10^{-6} . Below 0.1 Hz, the confining pump regulates from the feedback of the confining pressure transducer (Fig. 1). Above 0.1 Hz, the confining pump regulates solely in position. Depending on the bulk modulus of the sample, the pressure oscillation can be adjusted so that the strains meet the previous condition. For an isotropic sample, the volumetric strain (ϵ_{vol}) can be calculated by (i) $\epsilon_{vol} = \epsilon_{ax} + 2\epsilon_{rad}$, where ϵ_{ax} and ϵ_{rad} are the mean values of all the strain gauges glued at mid-height on the sample in the axial and the radial directions, respectively (Fig. 2), or by (ii) $\epsilon_{vol} = 3\epsilon_{mean}$, where ϵ_{mean} are the mean values of all the strain gauges, regardless of direction. In theory, for an isotropic sample, both calculations should give the same result. However, the second method gives a slightly lower uncertainty, since the error on average decreases with increasing number of strain gauges used. Moreover, relation (ii) gives equal importance to the axial and radial directions, whereas the uncertainty in relation (i) is primarily controlled by the radial directions. The bulk modulus K is then calculated by

$$K = -\frac{\Delta P_c}{\epsilon_{vol}}, \quad (1)$$

with the convention that strains are negative in compression [Fig. 2(a)]. For a perfectly homogeneous sample, no significant discrepancy between the different strain gauges is expected. For rocks,

FIG. 2. Strain gauge positions (left) and typical stress-strain recordings of hydrostatic [(a) and (b)] and axial [(c) and (d)] oscillations (around mean values) on a viscoelastic material [modified from Borgomano *et al.* (2017)]. The strains (ϵ_{ax} , ϵ_{rad} , ϵ_{alu}) are each averaged from four strain gauges around the circumference of the sample/endplaten at mid-height. The axial-stress oscillation ($\Delta\sigma_{ax}$) is deduced from the aluminum endplaten axial strain (ϵ_{alu}) and its calibrated Young's modulus of 78 GPa.

however, small discrepancies between the strain gauges may arise from the intrinsic heterogeneity or anisotropy of the sample. An associated uncertainty can, therefore, be calculated when averaging the strain gauges. From Eq. (1), the uncertainty on K (δK) depends on the uncertainty of the confining pressure ($\delta P \approx 0.001$ MPa) and the uncertainty on the average of the strains ($\delta \epsilon$). The relative uncertainty on K is then deduced by $\delta K/K = \delta P/P_c + \delta \epsilon/\epsilon_{vol}$. The error on the strain average can be calculated by $\delta \epsilon = std/\sqrt{n}$, where std is the standard deviation between the strain amplitudes and n is the number of strain gauges considered.

For axial oscillations [Figs. 2(c) and 2(d)], the piezoelectric actuator between the piston and the sample's top endplaten induces axial-stress oscillations. A small deviatoric stress (maximum 1.5 MPa) is constantly applied by the piston to preload the actuator and ensure a good coupling with the sample, while avoiding stress induced anisotropy. In order to limit the displacement of the piston when the actuator is oscillating, the auto-compensation valve is closed. The oscillating axial stress ($\Delta \sigma_{ax}$) (around the mean value) generated by using the actuator is measured from the axial strain gauges (ϵ_{alu}) glued on the lower endplaten (Fig. 2) made out of aluminum by $\Delta \sigma_{ax} = E_{alu} \epsilon_{alu}$, where E_{alu} is the associated Young's modulus of the endplaten that is to be calibrated with standard samples. Young's modulus (E) and Poisson's ratio (ν) of the sample can then be calculated by

$$E = \frac{\sigma_{ax}}{\epsilon_{ax}} \quad \text{and} \quad \nu = -\frac{\epsilon_{rad}}{\epsilon_{ax}}. \quad (2)$$

A total of 4 axial strain gauges are glued on the lower aluminum endplaten, at diametrical opposite locations. Due to the geometry of the endplaten and the position of the strain gauges, we demonstrate later that $E_{alu} = 78$ GPa. In addition, this configuration helps control that the vertical alignment is good and that the axial stress is homogeneous on the sample, as any tilt would result in different strain readings on the aluminum endplaten. Similar to K , the uncertainty on the strain averages ($\delta \epsilon$) can be calculated as previously and propagated to the moduli relative uncertainties by $\delta E/E = \delta \epsilon_{alu}/\epsilon_{alu} + \delta E_{ax}/E_{ax}$ and $\delta \nu/\nu = \delta \epsilon_{rad}/\epsilon_{rad} + \delta \epsilon_{ax}/\epsilon_{ax}$.

The inverse of the quality factor (Q^{-1}) measures the elastic energy dissipation within the sample during the oscillating cycles. In the following text, the term attenuation refers to the factor Q^{-1} . For a purely elastic material, $Q^{-1} = 0$. When dissipation occurs, the rheology of the sample may be considered analogue to a viscoelastic material (O'Connell and Budiansky, 1977). The stress-strain curve presents an elliptic shape that highlights the non-elastic behavior [e.g., Figs. 2(b) and 2(d)]. The slope of the big axis is given by the elastic modulus, while the surface represents the amount of elastic energy dissipated (e.g., Tisato and Madonna, 2012). This elliptic shape is the result of the phase shift between the applied stress (of phase φ_σ) and the induced strain (of phase φ_ϵ). The attenuation is then given by (O'Connell and Budiansky, 1977)

$$Q^{-1} = \tan(\varphi_\sigma - \varphi_\epsilon). \quad (3)$$

The attenuation related to K and E is, therefore, calculated by

$$Q_K^{-1} = \tan(\varphi_{P_c} - \varphi_{\epsilon_{vol}}) \quad \text{and} \quad Q_E^{-1} = \tan(\varphi_{\sigma_{ax}} - \varphi_{\epsilon_{ax}}). \quad (4)$$

Because the lower endplaten is assumed perfectly elastic (as confirmed by the calibration on standard samples), we consider that $\varphi_{\sigma_{ax}} = \varphi_{\epsilon_{alu}}$.

IV. GENERATION OF STRESS OSCILLATIONS AND STRAIN MEASUREMENTS

The controller program of the confining pump has been modified to perform hydrostatic oscillations. Under 0.2 Hz, the program works in controlled pressure mode and imposes a perfect sinusoidal function to the confining pressure. Above 0.2 Hz, and to a maximum of 1.3 Hz, the program works in controlled flow-rate mode, with a constant injection rate between pump-volume bounds. In order to maximize the frequency range, the volume of the confining cell has to be as small as possible (here 4 l), with a high flow-rate confining pump (here 70 ml/min). Due to the limited flow-rate of the pump and the cell volume, there is a compromise between the frequency and the amplitude of the pressure oscillation. Figure 3 represents

FIG. 3. (a) Maximum amplitude of the hydrostatic-stress oscillations achievable with the confining pump as a function of frequency, tested with a gypsum sample of bulk modulus $K \approx 40$ GPa. (b) Corresponding volumetric strain for various theoretical bulk moduli. For a given bulk modulus, the volumetric strain can have values equal or lower than the lines shown.

the maximum amplitude reachable in this apparatus for a given frequency and the corresponding volumetric strain for different bulk moduli. At a low frequency of 10^{-3} Hz and at a maximum flow-rate, the pump can achieve oscillations with an amplitude of 10 MPa [Fig. 3(a)]. The amplitude decreases to about 0.1 MPa at 1 Hz. Due to the intrinsic inertia and joint friction in the pump, any frequency higher than 1.3 Hz cannot be performed.

The piezoelectric actuator (PI PICA Stack P-056-20P) is controlled through a programmable function generator (TTi TG1010A) and an electric amplifier (PI PICA E-482). The amplifier can deliver a maximum voltage of 1150 V with a voltage gain of 100 V/V. The actuator can tolerate stresses up to 30 MPa (blocking stress), over which there is a risk of depolarization and permanent damage. Figure 4 shows the amplitudes of axial-stress oscillations obtained with a glass sample for constant voltage inputs of 200 V, 400 V, and 600 V, and on a plexiglass (PMMA) sample for a voltage of 600 V. The axial stress (σ_{ax}) is deduced from the aluminum endplates average strain (ϵ_{alu}) and considering a Young modulus of $E_{alu} = 78$ GPa. The axial-stress amplitudes are stable over the frequency range up to 10^3 Hz, although there does seem to be a little decrease in amplitude above 200 Hz when the voltage input is 600 V. The results on the plexiglass and on the glass, of very contrasting Young's moduli of around 5 GPa and 80 GPa, respectively, are fairly similar with an input voltage of 600 V. This shows that the stress produced by the piezoactuator is solely proportional to the input voltage, with little effect of the sample present between the actuator and the endplates. The voltage can then be adjusted to Young's modulus of the sample in order to obtain the desired strain amplitude.

The current data acquisition system (DAQ) used both for the pressure transducers and the strain gauges (1/4 bridge) can record with a sampling frequency up to 4 kHz. It is composed of four measurement modules provided by Gantner instruments. The pressure transducers are connected to two Q.bloxx A107 universal modules, while the strain gauges are connected in a 1/4 bridge configuration to two Q.bloxx A166 multichannel modules. The maximum recordable frequency without aliasing being under the Nyquist frequency

FIG. 4. Amplitude of stress during axial-stress oscillations using the piezoelectric oscillator at oscillating voltages of 200 V, 400 V, and 600 V on a glass sample, and 600 V on a plexiglass (PMMA) sample. The axial oscillations were performed on the samples at a confining pressure of 5 MPa and a deviatoric stress of 1 MPa. The axial stress (σ_{ax}) is deduced from the aluminum endplates average strain (ϵ_{alu}) and considering a Young modulus of $E_{alu} = 78$ GPa.

of 2 kHz, we limit our measurements to a maximum of 1 kHz. In order to manage the size of the output files, the sampling frequency is adjusted depending on the frequency of the measurement. The sampling frequencies are always chosen to be at least 20 times greater than the frequency of the stress oscillation. This rule is of course violated for frequencies between 200 Hz and 1 kHz, but the uncertainty on the Fourier transform is then lowered by acquiring a greater number of periods. For both hydrostatic and axial-stress oscillations, we record at least 50 periods of the signal to perform the FFT. For frequencies above 1 Hz, the number of recorded periods can be reasonably increased without substantially penalizing the total duration of the experiment. For frequencies between 2 Hz and 20 Hz, we measure at least 400 periods, and above 20 Hz at least 1200 periods.

V. PROTOCOL

Measurements can be done at various effective pressures on homogeneous materials (mainly for calibration), and principally on natural cored porous rocks. The top and bottom surfaces of the plug are necessarily rectified to be perfectly parallel (tolerance of $1 \mu\text{m}$). Once the sample is installed in the cell, it runs through several hydrostatic seasoning cycles, in order to minimize non-linear cycling effects caused by microcracks (Hart and Wang, 1995). The confining pressure varies several times between 0.2 MPa and 30 MPa, which is the maximum pressure tolerable of the piezoelectric actuator. The strain gauges and the internal axial LVDTs (Fig. 1) are monitored until the final cycles are repeatable. The static bulk modulus, during loading and unloading (tangent modulus for a given confining pressure), may be deduced from the last cycle.

For a given effective pressure, either for rocks or non-porous materials, the hydrostatic and axial oscillations are performed in sequence, followed by ultrasonic measurements of P- and S-wave travel times between the top and bottom endplates (Fig. 1), and eventually permeability measurements using the Darcy flow method. The effective pressure is then changed, generally by changing the confining pressure, allowing enough time to equilibrate the pore pressure through the sample.

After measurements are performed on an oven-dry porous rock, the sample can be fully-saturated in the cell following a specific procedure. A vacuum is generated throughout the whole sample from a vacuum pump connected on the top pore line. The pore fluid is then injected from the bottom of the sample with one of the pore-fluid pumps, with a pressure of at least 2 MPa. The vacuum pump is connected to the top pore line via a transparent tube which enables us to see the fluid that has crossed the sample. Moreover, the volume injected by the fluid pumps can be monitored. A volume of fluid of at least 2–3 times the pore volume, at high pressure, is drained through the sample to ensure the absence of air bubbles. The sample's unconfined porosity is measured prior to installation in the cell, via the triple weight method. Porosity variations with effective pressure may eventually be deduced from the volume of the fluid ejected from the sample, which can be measured with the fluid pumps.

Throughout the experimental surveys on saturated porous media, the pore fluid is maintained at a minimum pressure of 2 MPa, also limiting the presence of air bubbles. A second pair of pore-fluid pumps containing another fluid can be connected to the pore lines

(Fig. 1). Up to now, two pore fluids have been used in rocks: glycerin and water, due to their viscosity contrast (ratio 1000:1). Since glycerin dissolves in water, the sample and the tubings will first be saturated by the glycerin. For low permeability rocks, the saturation with glycerin can be accelerated by heating the cell, which decreases the glycerin's viscosity. Prior to the installation in the cell and the first "dry" measurements, all the samples are generally oven dried at 80 °C for at least 1 day to remove potential moisture effects.

VI. CALIBRATION OF DISPERSION AND ATTENUATION

Calibration measurements of the forced-oscillation method have been performed on three standards at room temperature (20 °C): gypsum, glass, and plexiglass (PMMA). The gypsum is the same sample used by Pimienta *et al.* (2015a; 2015b) for the calibration of another apparatus. The glass sample is a borosilicate glass that was used in the studies of Mallet *et al.* (2013; 2015). It was synthesized by the French Atomic Energy Commission (CEA) laboratory by a reproducible method, under conditions of slow cooling that prevent any crack formation. The plexiglass was provided by abaqueplast company (France).

The hydrostatic and axial forced-oscillation methods were applied at low confining pressures of 5 MPa and of 30 MPa, the maximum pressure allowed by the piezoactuator. Consistently with uncracked media, the three calibrating samples exhibited no dependence on confining pressure (Fig. 5). The bulk moduli dispersion

and attenuation inferred from hydrostatic oscillations are presented in Figs. 5(a) and 5(b), respectively. For the pure elastic materials, there was consistently no dispersion of the bulk moduli, with constant values around 58 GPa and 44 GPa for glass and gypsum, respectively, in the frequency range 0.004–1.3 Hz. In this frequency range, the bulk attenuation (Q_K^{-1}) for glass and gypsum can be considered negligible, although the results seem to scatter between 0 and 0.02 [Fig. 5(b)]. For the plexiglass, although dispersion is difficult to observe on the bulk modulus [Fig. 5(a)], which is about 5 GPa, an attenuation around 0.04 was observed between 0.004 Hz and 1.3 Hz [Fig. 5(b)] with a maximum around 1 Hz.

Young's modulus dispersion and attenuation inferred from the axial-stress oscillations are presented in Figs. 5(c) and 5(d). For glass and gypsum, again, no dispersion of E is visible in the frequency range 0.01 Hz–1 kHz, with constant values of 80 GPa and 42 GPa, respectively [Fig. 5(c)]. Consistently, the related attenuation (Q_E^{-1}) is negligible, with scattered points between -0.01 and 0.01 [Fig. 5(d)]. However, for the plexiglass, a noticeable attenuation peak ($Q_E^{-1} = 0.08$) centered between 1 Hz and 2 Hz was observed [Fig. 5(d)], with a very low Young's modulus increasing from 2.5 GPa to 5 GPa between 0.01 Hz and 1 kHz [Fig. 5(c)]. This behavior is consistent with the results obtained on the bulk modulus and with previous experimental observations at room temperature on plexiglass (PMMA) (Batzle *et al.*, 2006; Tisato and Madonna, 2012; Madonna and Tisato, 2013; and Pimienta *et al.*, 2015b).

Poisson's ratio dispersion, deduced from the axial oscillations, is presented in Fig. 5(e). For the non-dispersive glass and

FIG. 5. Calibration results of plexiglass (PMMA), glass, and gypsum, for confining pressures of 5 MPa and 30 MPa at room temperature (20 °C). Bulk modulus dispersion (a) and attenuation (b) are inferred from hydrostatic oscillations. Young's modulus dispersion (c) and attenuation (d), and Poisson's ratio dispersion (e) are inferred from axial oscillations. Relative uncertainties for K , E , and ν are, respectively, 0.3%, 2%, and 3% for PMMA, 2%, 5%, and 6% for gypsum, and 1%, 4%, and 5% for glass.

gypsum samples, ν seems to be constant around 0.25 and 0.34, respectively, up to 100 Hz. At higher frequencies, Poisson's ratio seems to abruptly diverge either up or down [Fig. 5(e)]. This is an error issued from the Fourier transform on the low amplitude radial strains, which decrease above 100 Hz as axial stress decreases (as seen in Fig. 4), reaching the strain gauge's uncertainty limit. Over the frequency range of 0.01–100 Hz for the plexiglass, a slight decrease with increasing frequency is observed [Fig. 5(e)], as was previously observed in the study of Pimienta *et al.*, 2016b.

Overall, these calibration results ensure that the apparatus is capable of reliably measuring elastic or viscoelastic dispersion and attenuation using forced-oscillation methods up to 1.3 Hz for the bulk modulus, up to 1 kHz for Young's modulus, and up to 100 Hz for Poisson's ratio.

VII. CONTROLLING THE UNDRAINED BOUNDARY CONDITIONS

In forced-oscillation experiments on saturated rocks, controlling the pore fluid's boundary conditions in the sample may prove to be essential to control the wave induced fluid flow (WIFF) into and out of the sample. If the boundary conditions are drained (opened), then a dispersive transition between a drained and an undrained fluid-flow regime will appear. This mechanism has been pointed out first by Dunn (1987) for a radial flow out of unjacketed samples under axial-stress oscillations (no confinement) and named the “Biot–Gardner effect.” In our apparatus, the samples are jacketed radially, but the mechanism may still occur axially through the end-platens and pore-fluid lines (Fig. 1). This transition was regularly defined as the drained/undrained transition in previous studies and is a significant contributor to elastic dispersion and attenuation in broadband studies (e.g., Pimienta *et al.*, 2015a; 2016a; Borgomano *et al.*, 2017; and Chapman *et al.*, 2018). It bears a characteristic frequency (f_c), equal to (Cleary, 1978)

$$f_c = \frac{4kK_d}{\eta L^2}, \quad (5)$$

where k is the permeability, K_d is the “drained bulk modulus,” η is the dynamic viscosity of the pore fluid, and L is the length of the sample.

Closing the valves on the top and bottom pore-fluid lines (necessarily located outside the confining cell) does not achieve purely undrained conditions, as small dead volumes of about 3 ml remain (purple in Fig. 1). The storage capacities of these dead volumes are large enough to still generate a drained/undrained dispersive transition (Pimienta *et al.*, 2016a and Borgomano *et al.*, 2017), which may be misinterpreted as a local WIFF mechanism like squirt-flow between cracks and pores.

In the past, few studies proposed a poroelastic model to take into account these dead volumes. Pimienta *et al.* (2016a) proposed a 1D poroelastic model, accounting for a vertical global flow, which solves the pressure diffusion equation accounting for different boundary conditions with the presence of dead volumes. The boundary conditions were then defined as “experimentally undrained” with the dead volume as an input parameter. One can deduce from the previous model that near drained conditions can be achieved when the cumulated dead volumes (top and bottom) are at least 10 times larger than the pore volume of the sample (Pimienta *et al.*,

2016a), which can easily be achieved by adding the internal volumes of the fluid pumps (Borgomano *et al.*, 2017). The model can further be extended to 3D using finite element methods to verify the possible effect of the radial flow, especially near the pore lines in the end-platens, which has the main effect to shift the transition to lower frequencies (e.g., Borgomano, 2018 and Sun *et al.*, 2019).

Although the drained/undrained transition may be of interest to verify the applicability of Biot–Gassmann's equations (e.g., Borgomano *et al.*, 2017 and Yin *et al.*, 2019), it may be problematic when investigating other dispersive mechanisms (e.g., squirt-flow), especially when the characteristic frequencies of each mechanism overlap (e.g., Pimienta *et al.*, 2015a). In order to remove the drained/undrained effect, we developed piloted microvalves settled in the end-platens (Fig. 1) that enable us to impose either open or closed boundary conditions. With microvalves being very close to the sample, we limit the dead volume to a maximum of 20 μl for each side. By default, the microvalves are opened, but can be closed by applying pressure through a pilot line (Fig. 1).

The microvalves were first tested on a Bleurswiller sandstone, a 25% porosity Vosgian sandstone, which is composed by ~60% quartz, ~30% feldspars, and ~10% clays (Fortin *et al.*, 2006; 2007; and Baud *et al.*, 2015). The permeability was measured to be around 200 mD ($2 \times 10^{-13} \text{ m}^2$) using the Darcy flow at an effective

FIG. 6. Results of bulk modulus dispersion (a) and attenuation (b) under dry and glycerin-saturated conditions from hydrostatic oscillations on a Bleurswiller sandstone at $P_{\text{eff}} = 5 \text{ MPa}$. Relative uncertainty on K is around 3%. Under glycerin-saturated conditions, the end-platens' microvalves were either open to achieve axially drained boundary conditions or closed to test undrained boundary conditions. The theoretical drained/undrained characteristic frequency ($f_c = 0.75 \text{ Hz}$) was calculated using Eq. (5). The horizontal dashed lines correspond to Biot–Gassmann's equations.

pressure of $P_{eff} = 5$ MPa. Hydrostatic oscillations between 0.01 Hz and 1.3 Hz were performed under oven-dry and fully-saturated conditions with pure glycerin (Fig. 6). In the saturated case, the microvalves were kept either open or closed during the whole oscillation sequence, with very large dead volumes connected to them (>200 ml). One experiment was performed without any microvalve installed to compare with the open configuration.

From the bulk modulus dispersion and attenuation results [Figs. 6(a) and 6(b), respectively], we can clearly observe that there is no dispersion/attenuation for both the dry and the closed microvalve cases, whereas a strong dispersion is observed for the open configuration [Fig. 6(a)]. In the open configuration, we clearly observe that the bulk modulus at 0.01 Hz is consistent with the “dry” condition, then progressively increases to the “closed” configuration values at 1 Hz [Fig. 6(a)]. From Eq. (5), the theoretical characteristic frequency of the drained/undrained transition was evaluated to be $f_c = 0.75$ Hz, taking $K_d = 6$ GPa, $k = 2 \times 10^{-13}$ m², $\eta = 1$ Pa s, and $L = 8 \times 10^{-2}$ m. This frequency is consistent with the observed transition and the center of the attenuation peak [Fig. 6(b)], supporting the evidence of the drained/undrained transition. Moreover, the bulk moduli for both drained (6 GPa) and undrained (16 GPa) configurations are consistent with Biot-Gassmann’s equations [horizontal dashed lines in Fig. 6(a)], when using a skeleton modulus (solid without pores) of 39 GPa (from a Voigt-Reuss-Hill average of the mineral composition, Mavko *et al.*, 2009), and a glycerin bulk modulus of $K_f = 4.36$ GPa.

Therefore, these microvalves enable us to suppress the drained/undrained transition and achieve pure undrained conditions, regardless of the permeability of the sample. The good

agreement between the open configuration results and the results without microvalves [Fig. 6(a)] implies that they do not affect the connectivity of the pore lines, which is essential in order to not affect permeability measurements using the steady state flow-rate method (Darcy flow).

VIII. NUMERICAL INVESTIGATION OF RESONANT FREQUENCIES

One major concern when developing dynamic oscillatory setups is the possibility of resonant frequencies disrupting the results. A simple method to verify the frequency ranges affected by the resonances of the machine can be computed through numerical solutions. Here, we used Comsol Multiphysics to model the relevant parts of the apparatus [Fig. 7(a)] and simulate its dynamic response. Since we expect resonant frequencies to be in the kilohertz range according to the size of the apparatus, we only focused on the axial-stress oscillations performed by the piezoelectric actuator that may reach these frequencies.

The main body and piston of the triaxial cell are made out of a quenched and tempered (+QT900) 1.4418 stainless steel ($E = 200$ GPa, $\nu = 0.28$, and $\rho = 7700$ kg m⁻³). In the axial column, the piezoelectric actuator (PZT) is made out of PIC151 piezoceramic ($E_{11} = 55$ GPa, $\nu_{12} = 0.38$, in polarized state, Fett *et al.*, 2002). The top endplaten is again made out of stainless steel and the lower endplaten of aluminum 2017A ($E = 72.5$ GPa, $\nu = 0.33$, and $\rho = 2790$ kg m⁻³). The piston load chamber at the top of the cell was modeled with the properties of Therminol SP mineral oil ($K = 1.9$ GPa, $G = 0$ GPa, and $\rho = 898$ kg m⁻³). Finally,

FIG. 7. Numerical model of the experimental setup, with the definitions of materials (a) and the free tetrahedral mesh (b). The materials are all purely elastic and isotropic. The system is solved in the frequency domain for axial oscillations of amplitude 0.2 MPa, and the maximum vertical strains are represented here for 0.1 Hz (c) and 10 kHz (d) (illustrated here for the sample with $E_s = 36$ GPa).

we tested three virtual samples with varying Young's modulus ($E_s = 93\,690\text{ GPa}$) for the same Poisson's ratio ($\nu_s = 0.2$) and density ($\rho_s = 2500\text{ kg m}^{-3}$). The materials are all considered purely elastic in the model.

The stack of pieces in the vertical column can freely expand radially, with continuous boundaries between each other [Fig. 7(a)]. Only the bottom of the lower endplaten is fixed. The top of the piston is radially fixed on an axially mobile surface that can compress the oil (no radial displacement). The piston slides without friction in the upper part of the cell's frame. Since the piston is auto-compensated for the confining pressure, we did not simulate the contribution of the latter, but simply added a deviatoric pre-stress in the vertical column (1.5 MPa). For the finite element calculation, a free tetrahedral mesh was applied to the whole geometry [Fig. 7(b)]. An oscillatory boundary load of $\pm 1\text{ MPa}$ was simulated on the top and bottom surfaces of the piezoactuator, and the displacements of all the nodes were calculated in the frequency domain. Analogous to the real experimental conditions, the sample's axial and radial strains were deduced from the nodes' displacements at mid-height on the surface of the sample, averaged on a grid surface equivalent to the experimental strain gauges ($6 \times 2\text{ mm}^2$). Similarly, the aluminum axial strains (deducing the axial stresses) are inferred from a surface grid at mid-height on the bottom endplaten.

The frequency range of the simulations goes from 10^{-5} Hz to 10^4 Hz and was applied to the three samples of varying Young's modulus ($E_s = 93\,690\text{ GPa}$). The strain amplitude results, inferred from the virtual strain gauges, are presented in Fig. 8(a). Negative values represent compressive strains. The strain amplitudes are not affected by resonances for frequencies up to 600 Hz. It seems that the axial strains of the sample start to diverge at slightly lower frequencies than the radial or aluminum strains [Fig. 8(a)]. Moreover, the stiffening of the sample would seem to slightly increase the frequency upon which the phenomenon appears.

When converted to Young's modulus dispersion [$E = (\epsilon_{alu}/\epsilon_{ax})E_{alu}$] and attenuation Q_E^{-1} [using Kramers–Kronig relationships (O'Donnell *et al.*, 1981)], and Poisson's ratio ($\nu = -\epsilon_{rad}/\epsilon_{ax}$) as would be measured experimentally in the cell (Fig. 8), it seems that E becomes significantly affected only above 2 kHz, while ν may start to diverge at 1 kHz, but insignificantly in terms of absolute value. The moduli values at low frequency match the materials elastic properties used for the model [Figs. 8(b) and 8(d)]. Although the materials used in the model are purely elastic, the resonances above 3 kHz seem to result in an apparent attenuation on E , with potential increasing or decreasing dispersion of E , which may appear non-physical for purely elastic materials. This is related to our experimental

FIG. 8. (a) Results of the numerical simulation for axial, radial, and aluminum endplaten strains (positions shown in Fig. 2); the induced (b) Young's modulus, (c) Young's modulus attenuation Q_E^{-1} , and (d) Poisson's ratio as a function of frequency, as would be interpreted from the strain gauges. Simulations were performed here for three samples of Young's moduli $E_s = 93\,690\text{ GPa}$, Poisson's ratio $\nu_s = 0.2$, and length $L = 80\text{ mm}$.

configuration and our method to calculate the different moduli. The non-homogenous stress field in the vertical column at a resonance frequency [Fig. 7(d)] induces an apparent phase shift between the strains on the sample and the strains on the aluminum endplates (used as the axial-stress reference), resulting in an apparent dispersion/attenuation of the sample's Young's modulus. Therefore, it is highly probable that changing the vertical distance between the sample's and the aluminum's strain gauges will affect the apparent dispersion/attenuation results of E [Figs. 8(b) and 8(c)]. This also explains why Poisson's ratio [Fig. 8(d)] is much less affected (in absolute value) since the radial and axial strain gauges nearly coincide.

Although Poisson's ratio of the glass standard [Fig. 5(e)] seemed to diverge and decrease in a similar manner to the model [Fig. 8(c)], we do not believe it to be related to a resonance problem but to the lower-amplitude limit of the radial strain gauges, since for stiff materials, the resonances seem to affect higher frequencies [2–3 kHz, Fig. 8(c)] and with negligible amplitude for ν . Nevertheless, the numerical model suggests that the apparatus should not be affected by resonance frequencies below 1 kHz, allowing use of the forced-oscillation method up to that frequency. However, such tri-axial cell designs will inevitably be affected by resonant frequencies above 1 kHz.

IX. APPLICATIONS

The apparatus was first employed on fluid-saturated low-permeability limestones (pure calcitic), prior to the installation of the microvalves (Borgomano *et al.*, 2019b). The drained/undrained transitions were at a relatively low frequency allowing the study of potentially higher frequency squirt-flow effects. Similar to the results on a dual-porosity oolitic limestone (Borgomano *et al.*, 2017), little or no squirt-flow dispersion was observed for the Indiana limestone (bioclastic and cemented) or a Rustrel limestone (rudist grainstone and cemented). However, thermal cracking on the Indiana sample generated sufficiently low aspect ratio cracks, mainly located in the intergranular cement, to generate squirt-flow dispersion affecting seismic frequencies (~ 100 Hz) (Borgomano *et al.*, 2019a). In addition, a coquina sample (detrital), cored from a deep offshore well, seemed to present some squirt-flow dispersion at sonic frequencies (~ 10 kHz). The main interpretation proposed for this sample was that, similar to sandstones, the squirt-flow originated from the uncemented grain-to-grain contacts, as opposed to the cracked Indiana sample (Borgomano *et al.*, 2019b). Consistently with the “crack” closure, the squirt-flow phenomenon disappeared with an increasing effective pressure above 15–20 MPa for both latter samples. For all samples, drained to undrained transitions were well in accordance with Biot-Gassmann's equations.

Forced-oscillation measurements were then performed on a glycerin-saturated Berea sandstone (Chapman *et al.*, 2018), again to investigate the squirt-flow mechanism and compare with the dispersion/attenuation results on a similar Berea sandstone in another apparatus (Mikhaltsevitch *et al.*, 2016). This comparison under similar pressure conditions confirmed the very high sensitivity of the squirt-flow mechanism to variations in the characteristic aspect ratios of the cracks, as the dispersive frequency ranges appeared quite different in both these studies (Mikhaltsevitch *et al.*, 2016 and Chapman *et al.*, 2018).

More recently, using the benefit of the drained/undrained transition, fluid substitution and shear weakening at seismic frequencies were investigated in a clay-bearing sandstone (Yin *et al.*, 2019). It was shown that the shear-weakening under water-saturated conditions, due to the reduction in surface free energy in the clays, affected mainly the shear modulus at seismic frequencies (< 100 Hz), while seemingly not affecting the ultrasonic results (1 MHz). The observation at a high frequency (1 MHz) was interpreted as a combination of shear weakening due to clays that decrease G over the full frequency range, and squirt-flow in cracks that re-increases G at a high frequency (Yin *et al.*, 2019).

X. CONCLUSION

A new apparatus was developed to perform both hydrostatic and axial-stress oscillations on saturated rock samples, enabling the characterization of the dispersion and attenuation of the elastic moduli over a large frequency range (0.004 Hz to 1 kHz). The cylindrical samples can have selectively drained or undrained boundary conditions at the top and the bottom with the use of microvalves, while the lateral surface is jacketed. Various conditions were tested on a porous sandstone, demonstrating the effect of the microvalves on the drained/undrained fluid-flow regimes. The apparatus has been successfully tested on known elastic and viscoelastic materials, such as glass, gypsum, and plexiglass (PMMA). The complex Young's modulus can safely be characterized up to 1 kHz, while Poisson's ratio measurements seem to be reliable up to 100 Hz, using axial-stress oscillations. The complex bulk modulus, inferred from hydrostatic oscillations, can reliably be measured up to 1.3 Hz. The dynamic limits of the apparatus were investigated through a numerical model, showing that frequencies above 1 kHz will undoubtedly be affected by resonance.

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