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Note: Precision viscosity measurement using suspended microchannel resonators

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We report the characterization of a suspended microchannel resonator (SMR) for viscosity measurements in a low viscosity regime (<10 mPa s) using two measurement schemes. First, the quality factor (Q-factor) of the SMR was characterized with glycerol-water mixtures. The measured Q-factor at 20 °C exhibits a bilinear behavior with the sensitivity of 1281 (mPa s)⁻¹ for a lower (1-4 mPa s) and 355 (mPa s)⁻¹ for a higher viscosity range (4-8 mPa s), respectively. The second scheme is the vibration amplitude monitoring of the SMR running in a closed loop feedback. When compared in terms of the measurement time, the amplitude-based measurement takes only 0.1 ~ 1 ms while the Q-factor-based measurement takes ~30 s. However, the viscosity resolution of the Q-factor-based measurement is at least three times better than the amplitude-based measurement. By comparing the Q-factors of heavy water and 9.65 wt.% glycerol-water mixture that have very similar viscosities but different densities, we confirmed that the SMR can measure the dynamic viscosity without the density correction. The obtained results demonstrate that the SMR can measure the fluid viscosity with high precision and even real-time monitoring of the viscosity change is possible with the amplitude-based measurement scheme. © 2012 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4768245>]

Viscosity, a measure of a fluid's resistance to flow, is one of the most fundamental properties of fluids. Precise measurement of the viscosity is important in many industrial and medical applications such as ink printing, painting, food, cosmetics, and health monitoring.¹⁻⁴ In general, viscosity measurements are performed by applying shear motion to a sample fluid and measuring the relevant physical parameters such as time, speed, pressure drop, and damping coefficient. However, conventional viscometers require 1–10 mL per measurement, which may not be suitable for rare or valuable samples such as a variety of bio-fluids and newly developed pharmaceutical materials. To address this challenge, a variety of MEMS-based viscometries have been developed by exploiting resonant damping and capillary effects.⁵⁻⁸ One example of MEMS-based viscometries is the use of micro-fabricated resonators, which measures viscosity by partially⁶ or fully⁷ immersing the microfabricated resonator in liquids. While this approach allows the viscosity measurement for a broad range of the viscosity from 0.22 to 1096 mPa s,⁸ the amount of sample required per single measurement is often comparable to conventional bench-top viscometers, and the sensitivity is limited due to the intrinsically low quality factor (Q-factor) in liquid environments. Though a few studies have explored microfabricated resonators having embedded fluidic channels for viscosity measurements,⁹ detailed characterization was not provided.

In this work, we attempt to measure viscosity by using suspended microchannel resonators (SMRs), which are vacuum-packaged resonators having microfluidic channels embedded.¹⁰ Although SMRs were originally developed

for high-resolution mass sensing applications, they are also useful for measuring other physical properties that are related with energy dissipation such as the viscosity of a fluidic sample. A recent study discovered the nonmonotonic energy dissipation of liquid-filled SMRs as the liquid viscosity increases,¹¹ suggesting that the viscosity can be precisely measured by systematically characterizing the resonance behaviors of the liquid-filled SMR. This note reports two viscosity-measurement schemes using the SMR, i.e., Q-factor-based and amplitude-based methods, for the precision viscosity measurement. In particular, we focus on the low viscosity range below 10 mPa s because bio-fluids such as blood, saliva, and urine are our primary interests while samples with viscosities up to 1000 mPa s can be tested with SMRs.¹¹

The concept of the *Q-factor-based method* is shown in Fig. 1(a), which illustrates how the resonance behavior of the SMR depends on the sample density and viscosity in the range of our interest. Let us consider the SMR initially filled with a heavier and more viscous (high ρ and high μ) sample. When the sample in the SMR is replaced with a lighter and less viscous (low ρ and low μ) sample, the resonance frequency of the SMR increases while both the Q-factor and vibration amplitude decrease. The resonance feature illustrated in Fig. 1(a) is typically measured in the frequency domain – the vibration amplitude of the SMR is monitored under the open-loop frequency sweep, and the resonance frequency and Q-factor are extracted using the lock-in detection scheme.¹² The out performance of the lock-in detection over the ring-down and thermal noise methods has been reported when operating dry SMRs without a liquid sample.¹³

Figure 1(b) shows the resonance frequency and Q-factor of an SMR for glycerol-water mixtures having different weight fractions at 20 °C. The SMR used in the present work

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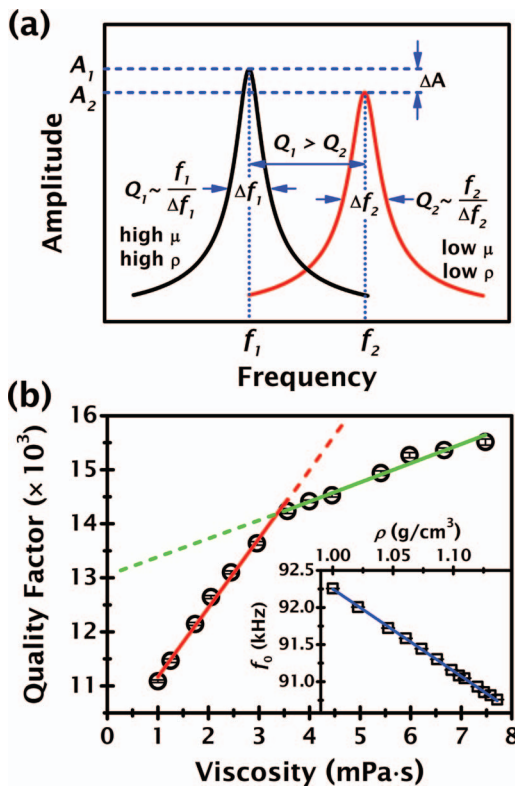


FIG. 1. (a) Q-factor-based viscosity measurement in the frequency domain. The Q-factor increases with increasing viscosity for a specified viscosity range. (b) Q-factor as a function of sample viscosity at 20 °C which shows a bilinear behavior around the viscosity of ~ 4 mPa s. Inset shows resonance frequency as a function of sample density at 20 °C which linearly decreases with increasing density.

is 406 μm long, 28.5 μm wide, and has an embedded microchannel whose cross sectional area is 7.9 $\mu\text{m} \times 8 \mu\text{m}$. The Q-factor shows a bilinear behavior as the viscosity increases, characterized by sensitivities of 1281 (mPa s) $^{-1}$ for a lower (1–4 mPa s) and 355 (mPa s) $^{-1}$ for a higher viscosity range (4–8 mPa s), respectively. On the other hand, the resonance frequency is inversely proportional to the density with the sensitivity of $-11,035 \text{ Hz}/(\text{g}/\text{cm}^3)$, as shown in the inset. While the inverse proportionality of the resonance frequency to the density is a well-known feature, the increase of the Q-factor with increasing viscosity is counterintuitive and a unique feature of the SMR.¹¹ Since the highest noise levels in measuring the Q-factor are 45.2 for the lower and 55.4 for the higher viscosity range, the resolution for viscosity measurement is estimated as 0.035 mPa s for the lower viscosity range and 0.156 mPa s for the higher viscosity range. With better laser alignment and environmental control, the viscosity resolution of ~ 0.01 mPa s could be achieved.

The second method for viscosity sensing is based on the vibration amplitude. When compared to the Q-factor-based method that needs the open-loop frequency sweep in the frequency domain, the *amplitude-based method* monitors the oscillation of the SMR in the time domain with the closed-loop control mode. While the feedback control keeps track of the SMR resonance, its frequency and vibration amplitude are simultaneously measured at a sampling rate higher than 1 kHz. Figure 2(a) shows the measurement using the amplitude-

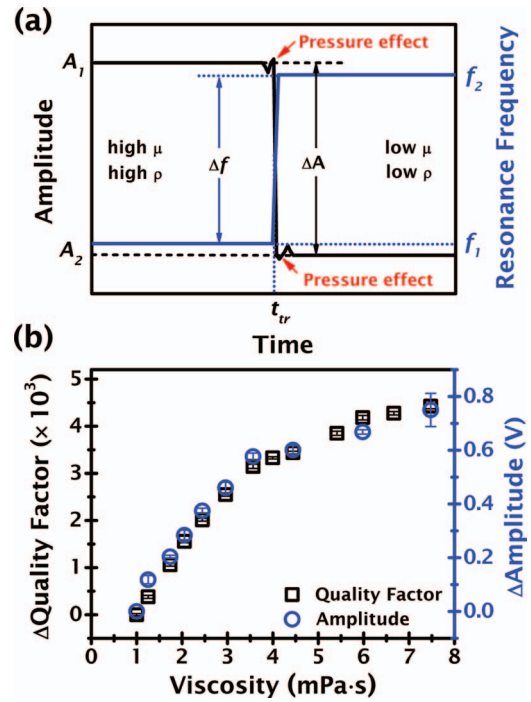


FIG. 2. (a) Amplitude-based viscosity measurement in the time domain using an SMR device. (b) Δ Quality factor (left y axis) and Δ amplitude (right y axis) as a function of viscosity. All measurements are performed at 20 °C.

based method, where the resonance frequency increases step-wise upon the decrease of the density, and the vibration amplitude decreases upon the decrease of the viscosity. It should be noted that the small dips in the amplitude before and after the sample exchange are due to the pressure effect, i.e., pressure fluctuation during the sample exchange in the microfluidic channel. This pressure effect is typically seen in both amplitude and resonance frequency signals, but more pronounced in amplitude.

Figure 2(b) compares the Q-factor and amplitude-based methods by plotting the relative changes of the Q-factor and vibration amplitude for glycerol-water mixture samples. The experimental setup previously reported¹³ is modified with additional beam splitter and photodetector to enable the amplitude-based method. Pure water is taken as a reference to depict the differential values. With glycerol-water mixtures, changes in Q-factor and vibration amplitude of the SMR show good agreement, suggesting that the amplitude-based method can provide the real-time monitoring of the dynamic viscosity. The amplitude-based measurement takes only 0.1 ~ 1 ms which are orders of magnitude faster than the Q-factor-based measurement. However, the viscosity resolutions of the amplitude-based measurement are 0.096 and 1.375 mPa s for the lower and higher viscosity ranges, which is around three times worse than the Q-factor-based measurement. The better viscosity resolution of the Q-factor-based method mainly comes from the use of the lock-in scheme and the consequent reduction of the noise level.

As shown in Fig. 1, the resonance frequency is inversely proportional to the sample mass density. Since the Q-factor is roughly expressed as $Q \sim f/\Delta f$ where f is the resonance frequency and Δf is the full-width of the resonance peak

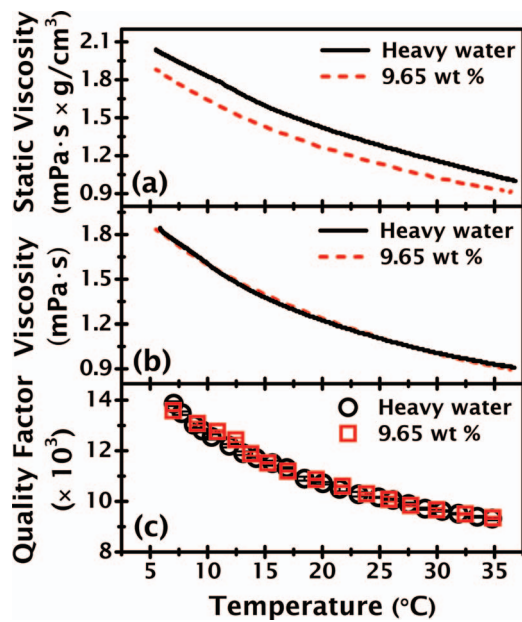


FIG. 3. (a) Measured static viscosity and (b) dynamic viscosity of heavy water and 9.65 wt.% glycerol-water mixture using a bench-top tuning fork viscometer. (c) Measured Q-factor of the SMR with heavy water and 9.65 wt.% glycerol-water mixture.

at half maximum, the Q-factor should be affected by the mass density unless its contribution is negligibly small. To investigate the effect of mass density on the measured Q-factor, heavy water (deuterium oxide, Sigma Aldrich) and 9.65 wt.% glycerol-water mixture are chosen since they have very similar viscosities of 1.25 mPa s but different densities at 20 °C (1.1073 g/cm³ for heavy water and 1.0237 g/cm³ for 9.65 wt.% glycerol-water mixture). A commercial viscometer (SV-10, A&D) utilizing the tuning fork vibration was employed for crosschecking. Figure 3(a) shows temperature-dependent static viscosity, i.e., products of the density and dynamic viscosity, directly measured with the SV-10 for the temperature range from 6 to 36 °C. Since the density of heavy water is greater than that of 9.65 wt.% glycerol-water mixture at the tested temperature range, higher static viscosities are expected for heavy water. However, the dynamic viscosities of both samples are in good agreement as shown in Fig. 3(b), indicating that SV-10, or the tuning fork viscometer in general, actually measures the static viscosity thus requires the mass density correction to obtain the dynamic viscosity. Figure 3(c) shows Q-factors of the SMR for the two samples as the temperature changes from 5 to 35 °C. The Q-factors

measured for heavy water and 9.65 wt.% glycerol-water mixture are in good agreement, suggesting that the mass density make a minor contribution to the Q-factor in viscosity measurement in the tested range and thus the SMR can measure the dynamic viscosity without the density correction.

This note reports two SMR-based methods for viscosity sensing in a low viscosity regime (<10 mPa s). Both Q-factor and amplitude-based methods show bilinear behaviours as the liquid viscosity increases. SMR measurements are validated with a commercial tuning fork viscometer. While the tuning fork viscometer measures static viscosity thus requires density correction, the SMR can directly measure dynamic viscosity. Moreover, SMRs require only a small amount of sample volume on the order of 10 μL including the waste, and can be repeatedly used with physical and chemical robustness. The small size and vacuum packaging of the SMRs allow fast measurement with the temporal resolution of 0.1 ~ 1 ms during the amplitude-based measurement, and precise measurement with the viscosity resolution of 0.035 mPa s during the Q-factor-based measurement. We believe that SMRs are well suited for studying rheological behaviors of rare and valuable samples.

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