2023 Joint Conference of the European Frequency and Time Forum and IEEE International Frequency Control Symposium (EFTF/IFCS) | 979-8-3503-1142-6/23/$31.00 ©2023 IEEE | DOI: 10.1109/EFTF/IFCS57587.2023.10272199

PZT-based Multi-Mode Cantilever for Viscosity Sensing

Javed N[1*], Priyanka Singh[1*], Sudhanshu Tiwari[2], Pavitra Jain[1], Kongbrailatpam Sandeep[1], Rudra Pratap[1] and Gayathri Pillai[1] 1
Center for Nanoscience and Engineering, Indian Institute of Science, Bengaluru, India
[2]The Elmore Family School of Electrical and Computer Engineering, Purdue University
* *Equal contribution*

***Abstract* — We report a thin film Lead Zirconate Titanate (PZT) on Silicon-on-Insulator (SOI) based microcantilever for fluid dynamic viscosity (η_{fluid}) sensing using optical and electrical characterization schemes. The viscosity of the medium is modulated by adding glycerol to deionized (DI) water. The measurements are carried over a viscosity range of 0.98-24.24 cP, which covers the operational range for biomedical applications. The fundamental cantilever mode yields a quality factor (Q) of 630 and 15 in air and DI water, respectively, whilst the second mode exhibits a Q of 840 and 30 in air and DI water, respectively. Sensor characterization using Laser Doppler Vibrometer (LDV) shows a power law relation between Q and η_{fluid} ($Q = a\,\eta_{fluid}^{b}$). A high relative responsivity of 0.38 and 0.41 with respect to η_{fluid} is observed for the first and second flexural modes of the microcantilever, respectively. These results indicate the development of a good platform for on-chip viscosity measurements for biomedical and industrial applications.**

Keywords— MEMS, Viscosity, Microcantilever, Sensor, Modes.

I. INTRODUCTION

Micro-Electromechanical Systems (MEMS) based fluid property sensors are emerging as promising candidates for disease diagnostics and real-time fluid health monitoring in industries because of their small size and low sample volume [1]. Viscosity variation in biological fluids can be used to diagnose the health condition of human beings. Synovial fluid is present in the space between the joints. The low viscosity of synovial fluid is related to rheumatoid arthritis (RA) and osteoarthritis (OA) [2]. In-line monitoring of wine fermentation was investigated using a piezoelectric MEMS resonator [3]. Piezoelectric MEMS resonators immersed in fluid are known to provide essential information about fluid properties through their vibrational characteristics [4][5][6][7]. Efforts are ongoing to develop a stand-alone fluid property sensor with electrical read-out using the self-actuating and self-sensing capability of piezoelectric MEMS resonators [4][5]. Resonance based fluid property sensing using higher order modes has been investigated using electrical characterization [6]. However, the resonant magnitudes are a function of the inverse product of dynamic viscosity and density. Decoupling of dynamic viscosity and density is restricted for higher-order modes [6]. Lower order flexural modes up to third mode has been used to sense dynamic viscosities (η_{fluid}) based on the change in Q-factor [5] in the low viscosities (0.31cP to 2.57cP). In this work, a PZT-based microcantilever is used to develop a dynamic viscosity sensing platform with high relative responsivity and range. A schematic of the Microcantilever device is shown in Fig.1. The change in Q is used as a marker for tracking dynamic viscosity.

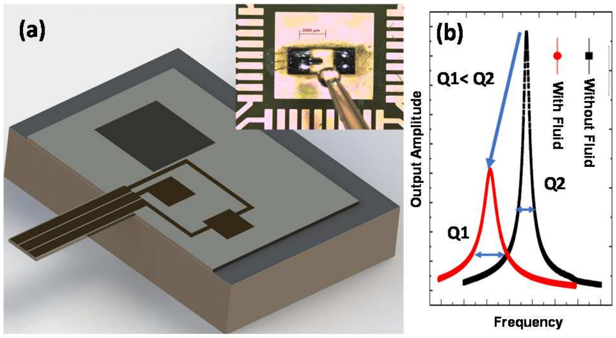

Fig. 1(a) Schematic of Microcantilever device. (b) Effect of fluid on frequency response of cantilever

II. FABRICATION AND CHARACTERIZATION SET-UP

PZT-based MEMS cantilevers are fabricated using a 5-mask process [8]. The microcantilever, shown in Fig.1, is designed with two top electrodes configuration on the active piezoelectric layer. The peripheral electrode is used for actuation and the middle electrode is used for sensing. Fig.1(a) inset figure shows the addition of fluid to the device. The effect of fluid on the frequency response of the cantilever is shown in Fig.1(b). A brief schematic showing the steps involved in fabrication is shown in Fig.2(a) and an optical image of the fabricated cantilever with dimensions: 1000µm×300µm×21.6µm (length, width, thickness) is shown in Fig.2(b). The piezoelectric cantilevers are fabricated on SOI wafers with 20µm thick device layer Silicon and 300nm thick thermal oxide, Titanium, and Platinum (Ti/Pt) of thicknesses 20nm and 130nm respectively, and 1µm thick PZT. Using the

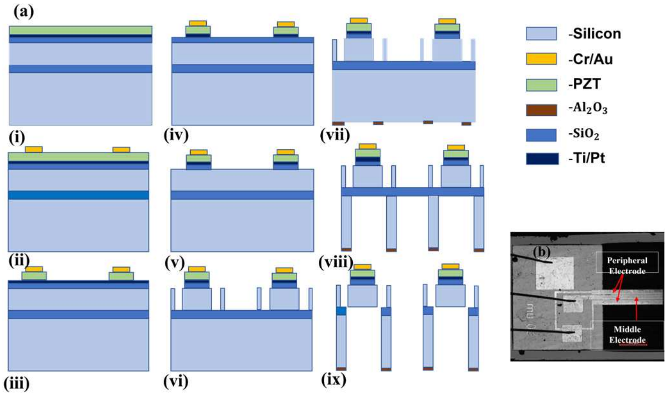

Fig. 2: (a) Fabrication process flow of PZT based microcantilever. (i) PZT on thermally oxidized and Ti/Pt deposited SOI (ii) Cr/Au Sputtering and lift-off (iii) Wet etching of PZT (iv) Wet etching of Pt (v) Dry etching of Thermal Oxide using RIE (vi) Dry etching of Device layer Si using DRIE (vii) Sputter Deposition of Al_2O_3 hard mask for backside etching (viii) Backside etching of Handle layer Si using DRIE (ix) Release of cantilever; and (b) Optical Micrograph of the Microcantilever.

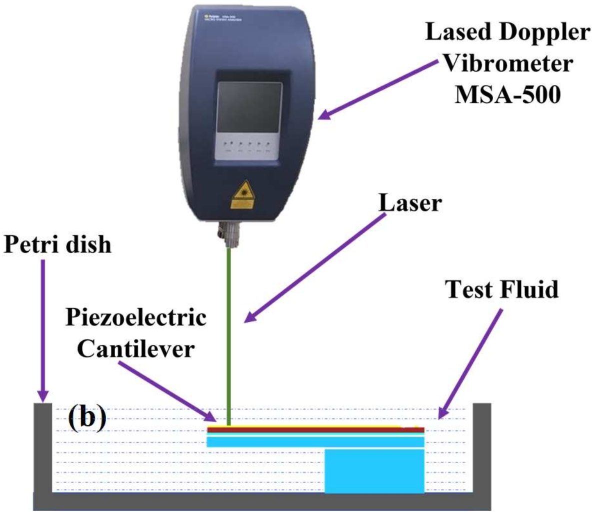

Fig. 3: Measurement setup of LDV-MSA 500 shows the cantilever device submerged in fluid.

DC sputtering process, we deposit top electrodes of chromium and gold (Cr/Au) with 20nm and 130nm thicknesses, respectively. Subsequently, PZT and bottom electrode (Ti/Pt) is patterned using wet etching processes. Using Reactive Ion Etching (RIE) and Deep Reactive Ion Etching (DRIE), we pattern thermal oxide and device layer Silicon, respectively. Thereafter backside patterning is performed. Al_2O_3, deposited using Radio Frequency (RF) sputtering followed by lift-off, is used as a hard mask for backside etching of SOI wafer. Finally, the backside etching is done using DRIE and RIE to release the device. The fabricated device is bonded on to a Printed Circuit Board (PCB), wire bonded and coated with parylene for electrical insulation while operating in fluid medium. The dynamic response characterization of the PZT-based MEMS cantilever is done with LDV (Polytec MSA-500) and lock-in amplifier (MFLI Zurich Instruments) in different fluids. A schematic diagram of the measurement set-up using the LDV is shown in Fig. 3. The device and PCB are totally submerged in a fluid medium. The frequency response of the cantilever in terms of the optical read-out from the LDV is measured by focusing the laser beam on the tip of the cantilever. An actuation AC signal of 3V is applied on both the middle and the peripheral electrodes. The first and the second flexural modes of the cantilever, as shown in Fig.4, were used for frequency response in the fluid.

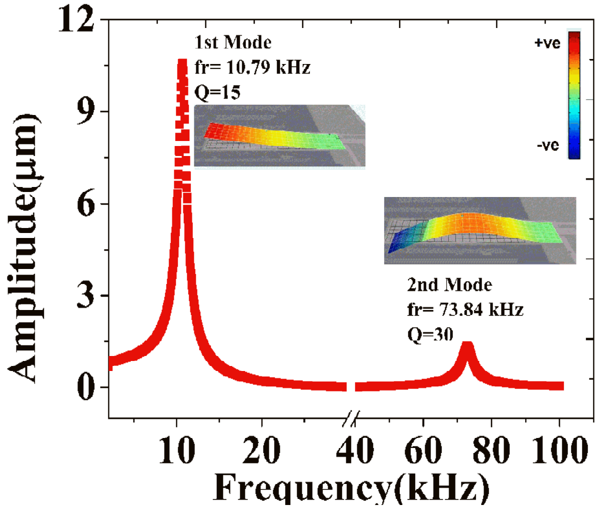

Fig. 4: Frequency response of the MEMS cantilever while submerged in DI water. The mode shape generated by LDV using the displacement mapping information is also shown in the inset.

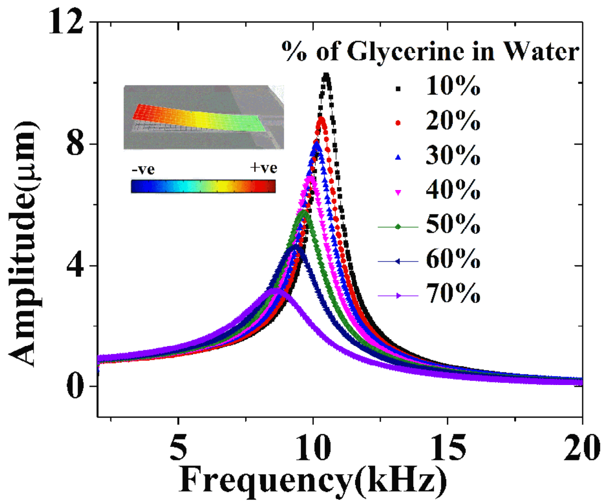

Fig. 5: LDV Measurements - Amplitude response of first mode. The inset shows the mode shape of the device in viscous medium.

III. RESULTS

The dynamic viscosity of the fluids was measured using Anton Paar Rheometer (MCR-302), and it was found that the η_{fluid} for 10%-70% (V/V) Glycerol-Water solutions ranges from

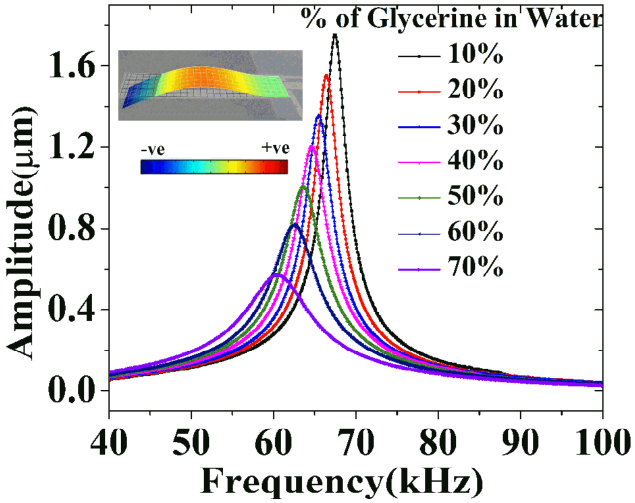

Fig. 6: LDV Measurements - Amplitude response of second mode. The inset shows the mode shape of the device in viscous medium.

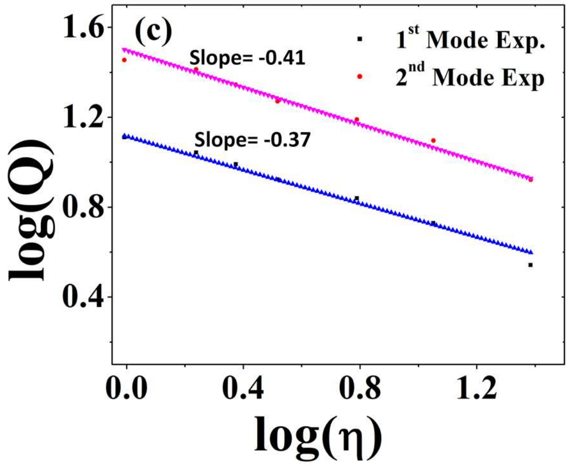

Fig. 7: Sensitivity of Q factor with dynamic viscosity of medium.

0.98 cP to 24.24 cP. Figures 5 and 6 show the frequency response of the first and second modes in different fluid mediums obtained from LDV measurements. Here, we observe that the frequency response follows the viscosity trends, i.e., the peak broadening increases for higher viscosity due to higher loss. In Fig. 7, the Q-factor of different modes with varying viscosity of the fluid is plotted. The Q-factor decreases from 13 to 3.4 and 28.5 to 8.3 for the first and second flexural modes, respectively. It follows a power law ($Q = a(\eta_{fluid}^{b})$) relationship in both modes. The coefficient b determines the relative responsivity of the resonator to viscosity. Relative responsivities of first and second flexural mode are found to be 0.37 and 0.41, respectively. For electrical measurements, a lock-in Amplifier (MFLI Zurich Instruments) was used. Figure 8(a) shows the electrical measurements set-up. The cantilever is actuated by applying a 2V AC signal to the peripheral electrode, and the middle electrode is used for sensing. To avoid excessive electrical feedthrough, test fluids of $50\mu L$ were added in a controlled manner so that the fluid just submerged the cantilever and did not come in contact with wire bonds and PCB solder pads. The first flexural mode was not visible due to electrical feedthrough. We assume that the presence of strong feedthrough, even after electrical insulation, is due to insufficient parylene. The second flexural mode was measured for 10% to 40% Glycerol-water solutions and the Q-factor decreased from 22 to 13.5. In Fig. 8(b) the response of the second mode for different viscosities from electrical measurement is shown. A baseline correction of the sensing output was done before calculating the quality factor for the second flexural mode in different fluids. Since the fluid volume in this measurement is low, the effect of evaporation of the fluid was evident in the measurement. The resonance frequency change was higher in the frequency response obtained from the Lock-in Amplifier as compared to that of LDV.

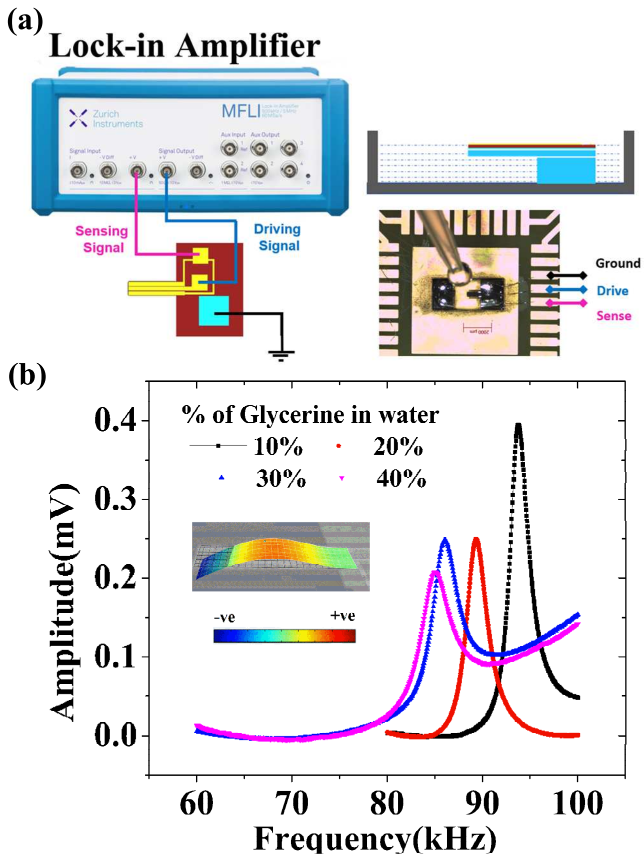

Fig. 8: (a) Electrical Characterization set-up. (b) Electrical Measurements - Frequency response obtained from Lock-in amplifier. The inset shows the mode shape of the device in viscous medium.

IV. CONCLUSION

We have successfully demonstrated the PZT cantilever as a viscosity sensor. We report a high relative responsivity of 0.41 using LDV based optical read-out. We successfully showed a similar trend in electrical read-out, albeit influenced by electrical feedthrough. By improving the electrical insulation with thicker parylene, we expect to overcome the problem of electrical feedthrough. This technique can be extended to real-time viscosity sensing applications in bio-diagnostics, automotive, and chemical processing industries.

ACKNOWLEDGEMENTS

The authors would like to thank the financial support from the Department of Science and Technology (DST)- Science and Engineering Research Board (SERB) India and National Nanofabrication Centre (NNFC) for the fabrication facility and Micro Nano Characterization Facility (MNCF) for characterization facility at CeNSE, IISc.

REFERENCE

[1] O. Cakmak, E. Ermek, N. Kilinc, G. G. Yaralioglu, and H. Urey, "Precision density and viscosity measurement using two cantilevers with different widths," *Sensors Actuators, A Phys.*, vol. 232, pp. 141–147, 2015.

[2] A. Miranda-Martínez, M. X. Rivera-González, M. Zeinoun, L. A. Carvajal-Ahumada, and J. J. Serrano-Olmedo, "Viscosity measurement sensor: A prototype for a novel medical diagnostic method based on quartz crystal resonator," *Sensors*, vol. 21, no. 8, pp. 1–15, 2021.

[3] J. Toledo, V. Ruiz-Díez, G. Pfusterschmied, U. Schmid, and J. L. Sánchez-Rojas, "Flow-through sensor based on piezoelectric MEMS resonator for the in-line monitoring of wine fermentation," *Sensors Actuators, B Chem.*, vol. 254, pp. 291–298, 2018.

[4] D. Hur and J. H. Lee, "Determination of liquid density and viscosity using a self-actuating microcantilever," *Jpn. J. Appl. Phys.*, vol. 52, no. 5 PART 1, 2013.

[5] L. Huang *et al.*, "Piezoelectric-AlN resonators at two-dimensional flexural modes for the density and viscosity decoupled determination of liquids," *Microsystems Nanoeng.*, vol. 8, no. 1, 2022.

[6] G. Pfusterschmied, F. Patocka, C. Weinmann, M. Schneider, D. Platz, and U. Schmid, "Responsivity and sensitivity of piezoelectric MEMS resonators at higher order modes in liquids," *Sensors Actuators, A Phys.*, vol. 295, pp. 84–92, 2019.

[7] S. Tiwari, A. Dangi, and R. Pratap, "A tip-coupled, two-cantilever, non-resonant microsystem for direct measurement of liquid viscosity," Microsystems Nanoeng., vol. 9, no. 1, 2023.

[8] S. Tiwari, R. Kumar, A. Dangi, and R. Pratap, "Enabling Fabrication of PZT Based PiezoMEMS Devices," *Proc. IEEE Sensors*, vol. 2018-October, pp. 1–4, 2018.