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Piezoelectric excited millimeter sized cantilever sensors for measuring gas density changes

Ruben Rosario, Raj Mutharasan*

Department of Chemical and Biological Engineering, Drexel University, Philadelphia, PA 19104, USA

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Rapid, *in-situ* measurement of gas density is valuable for monitoring industrial processes. We show that piezoelectric-excited millimeter-sized cantilever (PEMC) sensors exhibit precision for measuring gas density changes as small as 0.088 g/l with sensitivity equivalent to 0.049 g/(l Hz). PEMC sensors were fabricated by bonding 127 μm thick piezoelectric layer (lead zirconate titanate) to a base 160 μm thick silica layer. Gases (He, N_2, Ar) were passed continuously through an isothermally maintained flow cell, in which the PEMC sensors were mounted and the steady state resonant frequency values were monitored. The changes in observed resonant frequencies were collected and compared in multiple experiments with well-established theory for the frequency response of a dynamic cantilever surrounded by an inviscid fluid and were found to be in reasonable agreement. A finite element model of the PEMC sensor showed good agreement with the measured resonant frequency values.

1. Introduction

The density measurement of fluids is important both in industry and laboratory applications. For example, measurement of gas density can be used to interpret binary gas concentrations when conventional methods such as gas chromatography and mass spectroscopy are expensive for *in-situ* applications [1]. Density of a gas mixture is a function of composition, particularly if it is well behaved. Thus density measurement can be substituted for maintenance-intensive composition measurement. Consequently, sensitive and robust method of gas density measurement is an important area as it addresses many practical industrial needs.

Mechanical resonators in the form of microcantilevers [2], quartz tuning forks [3], and silicon microtubes [4] have been investigated for measuring both gas and liquid fluid densities. The microcantilever and quartz tuning fork sensors operate on the principle that the added mass of the fluid surrounding the resonating device (fork prongs, cantilever) cause a shift in the resonating frequency which can be conveniently measured. An alternate approach of using magnetically driven silicon tubes have been shown to respond to gas density changes. This Corialis-type mass flow and density meters were initially designed for liquids and therefore, to gases they operate at the lower end of their sensitivity range [5]. Application of these devices for measurement of gases at low pressures is problematic since they are constructed with dense materials, and thus are not sensitive [4].

Piezoelectric-excited millimeter-sized cantilevers (PEMC) have been shown to be sensitive for biosensing [6–10], and effective for measuring liquid densities with sufficient sensitivity to detect differences as small as 2 μg/cm^3 [11]. Hence, the purpose of this study is to investigate if PEMC sensors perform as well in much lighter mediums such as gases. This paper examines the effectiveness of PEMC sensors for measuring gas density. We tested both first and second order bending modes for their responses to changes in gas density. We also compared the experimental responses obtained with theory which indicated that the added mass of the surrounding fluid causes a predictable frequency shift.

2. Theory

Vibration of cantilevers in inviscid fluid has been extensively examined theoretically by several investigators. The model published by Sader' group [12–14] predicts that the resonant frequency of a cantilever (f_{fl}) of uniform cross-section in a medium of density (ρ_f), in relation to its resonant frequency in vacuum, is given by:

$$\left(\frac{f_{vac}}{f_{fl}}\right)^2 = 1 + \frac{\pi \rho_f b}{4\rho_c t}\Gamma(n) \quad (1)$$

The term, f_{vac}, is the resonant frequency in vacuum, where there is no added fluid mass effect. The parameters b, ρ_c, Γ, n and t are the cantilever width, effective density of the cantilever, hydrodynamic function, mode number, and cantilever thickness, respectively. Eq.

* Corresponding author. Tel.: +1 215 895 2236; fax: +1 215 895 5837.
E-mail addresses: mutharasan@drexel.edu, mutharr@drexel.edu (R. Mutharasan).

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(1) holds true when the Reynolds's number defined as $(2\pi f \rho b^2/4\eta)$, is much greater than one. The hydrodynamic function is the normalized external load that fluid exerts on the moving cantilever, the force exerted by the fluid on the cantilever [12]. The hydrodynamic function is described further in the supplementary materials that accompany this paper. The hydrodynamic function and its Pade approximation [19] were calculated for Sensors A and B and are included in the supplementary materials. Eq. (1) suggests that the resonant frequency of a cantilever decreases with density of the surrounding fluid in a nonlinear fashion. Such a behavior can thus be used to determine density of the surrounding fluid from the measured resonant frequency response, and is the approach we take in this work.

The test gases (He, N_2, air, Ar) behave as ideal gases at room temperature and atmospheric pressure. Thus Eq. (1) can be modified as

$$\frac{1}{f_{fl}^2} = \frac{1}{f_{vac}^2} + \frac{\pi P M_w b}{4RT\rho_c t}\Gamma(n)\frac{1}{f_{vac}^2} \tag{2}$$

where M_w is molecular mass of the gas surrounding the cantilever, P is the pressure, and T is the absolute temperature. Eq. (2) suggests that if we plot the experimental resonant frequency in various gases on the y axis (as $1/f_{fl}^2$) and molecular mass of the gas on the x axis, we expect to obtain a straight line with a y-intercept equal to $1/(f_{vac})^2$ and the straight line slope would equal to $(\pi Pb\Gamma)/(4R\rho_c T t f_{vac}^2)$. Eq. (2) also suggests that the resonant frequency response is a function of gas pressure since density is a linear function of pressure. Similarly, the slope is inversely proportional to temperature, and thus it is paramount to maintain T and P constant in experimental investigations for obtaining the expected straight-line relationship. In the current study, this was included in the experimental design, and all experiments were conducted at constant back pressure of 1 cm water head maintained at the exit of the flow cell, and the temperature was kept constant in the incubator at 37 °C.

3. Materials and methods

3.1. Sensor fabrication

The PEMC sensor is a two-layer composite structure comprised of a 127 μm-thick piezoelectric layer (lead zirconate titanate, PZT; Type 5A, $d_{31} = -190 \times 10^{-12}$ m/V; Piezo Systems, MA) bound to a 160 μm fused silica glass layer (SPI Supplies, PA) with a cyanoacrylate adhesive (~30 μm thick). The PZT measured 5 × 1 mm (length × width) and the glass layer measured 4 × 1 mm. The glass was glued to the PZT so than the glass extended ~0.5–1.0 mm beyond the leading of the PZT layer; this featured is labeled the *overhang*. Copper wire (30 gauge) was soldered to the electrode surfaces of the PZT layer, and the soldered end of the cantilever was embedded in a non-conductive epoxy within a 6 mm-diameter glass tube. Photographs of sensor are included in Fig. 1. The characteristic dimensions of sensors A and B are indicated in panels B and D of Fig. 1, respectively. The dimensions are labeled as *gap*, *overhang*, and *PZT free length*, and they are also defined in Fig. 1 caption. The glass segment, for some sensors, was anchored in the epoxy as pictured in Panels A and B. The dimensions of all four sensors are summarized in Table 1. The bonded glass layer constrains the longitudinal extension of the PZT that result from an applied potential, and thus produces a flexural motion. After the epoxy was cured for at least 24 h, the PZT, glass, and exposed epoxy were brush-coated with a thin, insulating layer of electronic grade polyurethane (Wasser, Washington), which was allowed to cure for seven days at room temperature.

3.2. Experimental procedure

A schematic of the experimental apparatus is shown in Fig. 2. It consisted of an impedance analyzer (HP 4192A LF), flow cell, and constant temperature chamber maintained at 37 °C. The cantilever assembly was secured in the flow cell, into which various

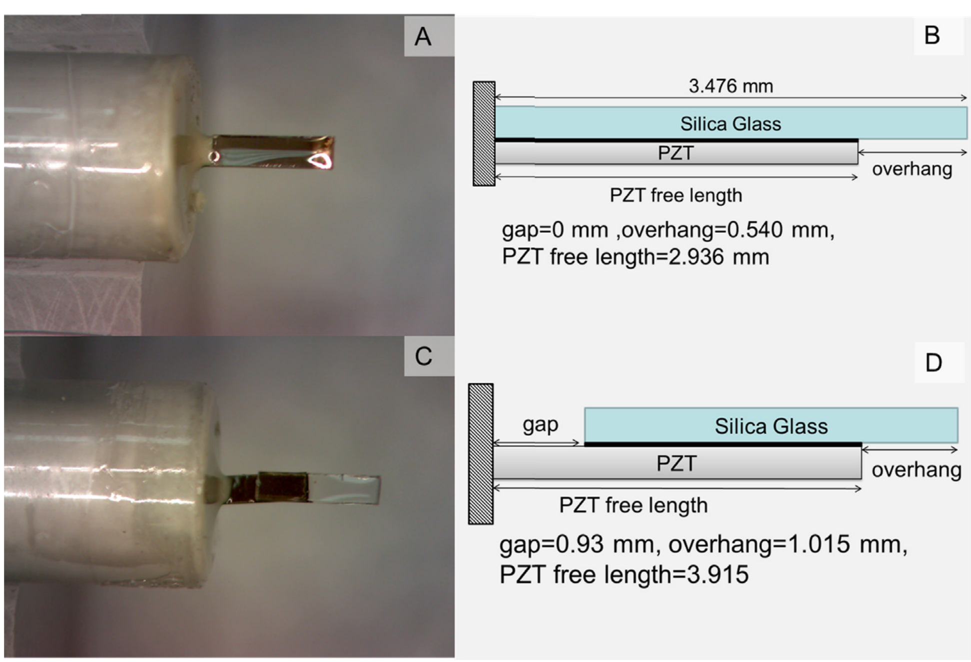

Fig. 1. A typical sensor construction is represented above. A 5 × 1 mm rectangular section of PZT was bonded to a 4 × 1 mm section of fused silica glass. After soldering copper leads to PZT electrodes, the solder end was embedded in epoxy contained within a glass tube. Panels A and B present a photo and diagram of a sensor design in which the glass layer, as well as the PZT are anchored in epoxy. Panels C and D show a photo and an outline diagram of a sensor in which the glass layer is not anchored. The characteristic dimensions of the two sensor geometries are clearly delineated in panels B and D; the dimensions given in panels B and D pertain to sensors A and B, respectively. The gap is the length between the anchor and bonded glass layer. The overhang dimension is the length of glass extending beyond the PZT layer. PZT free length is the length of PZT protruding out of the epoxy.