Detection of water-ice transition using a lead zirconate titanate/brass transducer

Xiaoping Li and Wan Y. Shih^{a)}

Department of Materials and Engineering, Drexel University, Philadelphia, Pennsylvania 19104

James Vartuli, David L. Milius, Robert Prud'homme, and Ilhan A. Aksay Department of Chemical Engineering and Princeton Materials Institute, Princeton University, Princeton, New Jersey 08544

Wei-Heng Shih

Department of Materials and Engineering, Drexel University, Philadelphia, Pennsylvania 19104

(Received 21 November 2001; accepted for publication 2 April 2002)

We have examined experimentally and theoretically the resonance frequency of a lead zirconate titanate (PZT)/brass unimorph disk transducer with a water (ice) layer on the brass surface. We showed that the flexural resonance frequency decreased with the presence of a water layer and the decrease in resonance frequency increased with an increasing water amount. Upon lowering the temperature, the freezing transition of the deposited water layer was detected when the resonance frequency of the transducer increased abruptly at the freezing temperature. In contrast to water, an ice layer increased the resonance frequency and the increase in the resonance frequency increased with the ice layer thickness. Theoretically, an analytic expression for the flexural resonance frequency of a unimorph transducer in the presence of an ice (water) layer on the brass surface was obtained in terms of the Young's moduli, densities, and thickness of the PZT, brass, and ice (water) layers. The theoretical predictions were shown to agree with the experimental results. © 2002 American Institute of Physics. [DOI: 10.1063/1.1481191]

I. INTRODUCTION

Ice formation on aircraft wings, space shuttle fuel tanks, and highway bridges can create hazardous conditions. For example, ice buildup on the leading edge of a fixed wing increases the drag and decreases the lift of an aircraft. Dislodged ice can damage the aircraft skin, antennas, or other instruments, leading to catastrophic engine failures if ingested. Such potential hazards have led to the development of various ice sensing techniques^{1–8} such as ultrasonic, laser, and time domain. Many of these techniques have the disadvantages of high costs, complicated structures, and difficulty to miniaturize and integrate with the main structure.

A flexural d_{31} -gradient transducer is a piezoelectric transducer with a gradient in the piezoelectric coefficient, d_{31} , in the thickness direction. Due to the axial gradient in the piezoelectric coefficient, d_{31} , the transducer undergoes bending when an electric field is applied in the axial direction. For example, a "unimorph" transducer has a piezoelectric layer bonded to a nonpiezoelectric one and thus has a stepwise d_{31} distribution in the thickness direction. The term unimorph is used to distinguish from a "bimorph" transducer that consists of two piezoelectric layers bonded to gether. The flexural resonance frequency of a flexural d_{31} gradient, e.g., unimorph, transducer is sensitive to mass accumulations and bending-modulus changes in the transducer.⁹ Based on the earlier work,⁹ a solid layer deposited on the surface of the transducer should increase the flex-

ural resonance frequency of the transducer because a deposited solid layer not only increases the mass per unit area but also the bending modulus. On the other hand, a liquid layer should decrease the flexural resonance frequency because the presence of a liquid does not increase the bending modulus but only increases the mass per unit area of the transducer.

The purpose of this study was to examine both theoretically and experimentally how the flexural resonance frequency of a flexural d_{31} -gradient disk transducer changes due to the solidification of a deposited liquid layer. For the present study, water was our model liquid system. The result of the present study can be readily applied for, say, aircraft ice detection. Specifically, we deposited water on the brass side of a lead zirconate titanate (PZT)/brass unimorph disk transducer [Fig. 1(a)]. In this case, PZT is the piezoelectric layer and brass is the nonpiezoelectric one. Freezing of the deposited water layer was examined by measuring the resonance frequency change in the transducer. The advantage of using a piezoelectric unimorph transducer is that both actuation and sensing can be achieved electrically. A dual top electrode design, i.e., a sensing top electrode separated from a driving top electrode allowed the resonance to be detected simply as demonstrated in a previous study with a piezoelectric unimorph cantilever liquid viscosity and density detector.¹⁰ When an ac voltage was applied to the driving electrode the converse piezoelectric effect caused the cantilever to vibrate. At resonance, the large resonant vibration amplitudes gave rise to large piezoelectric voltages detectable at the sensing electrode via the direct piezoelectric effect.

0021-8979/2002/92(1)/106/6/\$19.00

106

^{a)}Electronic mail: shihwy@drexel.edu



FIG. 1. A schematic of the cross section of the PZT/brass unimorph disk transducer with (a) an ice layer and (b) a water layer on the brass surface.

Theoretically, analytic expressions were obtained for the resonance frequency of a PZT/metal unimorph transducer in presence of an ice (water) layer on the metal surface. The theoretical predictions were shown to agree with the experimental results. In addition to simple electric actuation and detection, a PZT/metal unimorph disk transducer can also be easily miniaturized, mounted on, and integrated with the main structure.

II. THEORY

A. With a solid layer

Consider a circular disk transducer composed of three different layers: a piezoelectric layer of thickness, t_p , density, ρ_p , and Young's modulus, E_p , a metal, e.g., brass, layer of thickness, t_b , density, ρ_b , and Young's modulus, E_b , and a solid, e.g., ice layer of thickness, t_i , density, ρ_i , and Young's modulus, E_i , deposited on the brass surface as schematically shown is Fig. 1(a). In this design, the brass layer not only served as the nonpiezoelectric layer of the unimorph but also protected the piezoelectric layer and the driving and sensing electrodes from the environment. The bending-mode resonance frequency of a transducer can be written as⁹

$$f_r = \frac{2\lambda^2}{\pi L^2} \sqrt{\frac{D}{m}},\tag{1}$$

where λ^2 is the eigen value that depends on the geometry and boundary conditions of the transducer, *L* is the diameter of the transducer, *D* is the bending modulus per length of the transducer, and *m* is the mass per unit area obtained as

$$m = \rho_p t_p + \rho_b t_b + \rho_i t_i \,. \tag{2}$$

The bending modulus per length depends on the Young's modulus and thickness of each layer. Because the deposited solid layer is rigid, it contributes to the bending modulus as well. Denoting D_i as the bending modulus of the transducer

with a solid layer of thickness, t_i , on the top of the brass layer [see Fig. 1(a)] and assuming perfect bonding between the deposited solid layer and the brass, D_i can be obtained by

$$D_{i} = \int_{-t_{p}}^{0} E_{p}(z-t_{n})^{2} dz + \int_{0}^{t_{b}} E_{b}(z-t_{n})^{2} dz + \int_{b}^{t_{b}+t_{i}} E_{i}(z-t_{n})^{2} dz, \qquad (3)$$

where z is the position variable in the thickness direction, and

$$t_n = \frac{E_b t_b^2 + E_i (t_i^2 + 2t_i t_b) - E_p t_p^2}{2(E_i t_i + E_b t_b + E_p t_p)},$$
(4)

the position of the neutral-strain plane as determined by the condition that the total bending stress in the transducer is zero, i.e.,

$$\int_{-t_p}^{0} E_p \frac{(z-t_n)}{r} + \int_{0}^{t_b} E_b \frac{(z-t_n)}{r} + \int_{b}^{t_b+t_i} E_i \frac{(z-t_n)}{r} = 0,$$
(5)

with *r* being the radius of curvature. Plugging t_n in Eq. (3), the bending modulus per length can be obtained as

$$D_{i} = [E_{p}^{2}t_{p}^{4} + E_{b}^{2}t_{b}^{4} + E_{i}^{2}t_{i}^{4} + 2E_{p}E_{b}t_{p}t_{b}(2t_{p}^{2} + 2t_{b}^{2} + 3t_{p}t_{b}) + 2E_{p}E_{i}t_{p}t_{i}(2t_{i}^{2} + 2t_{p}^{2} + 3t_{i}t_{p}) + 2E_{b}E_{i}t_{b}t_{i}(2t_{i}^{2} + 2t_{b}^{2} + 3t_{i}t_{b}) + 12E_{p}E_{i}t_{p}t_{i}t_{b}(t_{i} + t_{b} + t_{p})]/12(E_{i}t_{i} + E_{p}t_{p} + E_{b}t_{b}).$$
(6)

With a solid layer, both the bending modulus per length, D_i , [Eq. (6)] and the mass per unit area, m, [Eq. (2)] increase with an increasing deposited layer thickness. The mass per unit area, m, is linear in the deposited layer thickness whereas the bending modulus per length, D_i , increases more rapidly with the deposited layer thickness [see Eq. (6)]. Consequently, deposition of a solid layer will result in an increase in the bending-mode resonance frequency.

B. With a liquid layer

When a liquid, e.g., water layer of thickness t_w is deposited on the brass side of the transducer as schematically shown in Fig. 1(b), the liquid layer contributes to the mass per unit area, m, as

$$m = \rho_p t_p + \rho_b t_b + \rho_w t_w, \tag{7}$$

where ρ_w is the density of the liquid. Because the liquid layer has no rigidity, it does not contribute to the bending modulus per length of the transducer. Denoting the bending modulus per length of the transducer with a liquid layer as D_w , D_w can be obtained by

$$D_{w} = \int_{-t_{p}}^{0} E_{p}(z-t_{n})^{2} dz + \int_{0}^{t_{b}} E_{b}(z-t_{n})^{2} dz, \qquad (8)$$

where

$$t_n = \frac{E_b t_b^2 - E_p t_p^2}{2(E_b t_b + E_p t_p)}$$
(9)

Downloaded 12 Jul 2002 to 128.112.140.74. Redistribution subject to AIP license or copyright, see http://ojps.aip.org/japo/japcr.jsp

TABLE I. Properties of PZT, brass, water, and ice used in the calculations of the resonance frequency of the PZT/brass sensor with or without a water/ice layer.

	Young's modulus (GPa)	Density (kg/m ³)	Thickness (cm)	Diameter (cm)	λ_{eff}^2
PZT	62.5 ^a	7500 ^a	0.0254	4.0	9.2
Brass	110 ^a	8900 ^a	0.0127		
Water	0	1000	Vary		
Ice	10 ^b	900	Vary		

^aProvided by the manufacturers.

^bE. M. Schulson, J. Miner. Met. Mater. **51**, 21 (1999).

is the neutral strain plane obtained by solving

$$\int_{-t_p}^{0} E_p \frac{(z-t_n)}{r} + \int_{0}^{t_b} E_b \frac{(z-t_n)}{r} = 0.$$
 (10)

Plugging Eq. (9) in Eq. (8), the bending modulus of the transducer with a liquid layer can be written as

$$D_{w} = \frac{E_{p}^{2}t_{p}^{4} + E_{b}^{2}t_{b}^{4} + 2E_{p}E_{b}t_{p}t_{b}(2t_{p}^{2} + 2t_{b}^{2} + 3t_{p}t_{b})}{12(E_{p}t_{p} + E_{b}t_{b})}.$$
 (11)

Thus, with a liquid layer, the bending modulus per length of the transducer, D_w , does not change with the liquid layer thickness while the mass per unit area increases with an increasing amount of liquid [see Eq. (7)]. This indicates that the bending-mode resonance frequency should decrease with an increasing amount of deposited water. Therefore, a flextensional transducer, quite unlike other techniques, not only can detect the presence of a layer on the transducer surface but also can distinguish whether the deposited layer is a solid or a liquid.

III. EXPERIMENT

In the present design, the flextensional d_{31} -gradient transducer was a PZT/brass unimorph disk. The piezoelectric PZT (APC PZT-857) disk, approximately 0.0254 cm thick and 3.0 cm in diameter with silver-paste electrodes on both sides, was purchased from APC International Ltd. (Mackeyville, PA). The APC PZT-857 is a soft PZT with a composition near the morphotropic phase boundary. The brass layer was an Alloy 260 brass sheet (McMaster-Carr Supply Company, Dayton, NJ) with 0.0127 cm thickness and a 4.5 cm diameter. The material characteristics of the PZT and that of the brass were taken from the data sheet provided by the suppliers and listed in Table I. Prior to bonding the PZT layer to the brass, the PZT disk was first poled with an electrical field of 2×10^6 V/m at 110 °C in transformer oil. After poling, the top electrode was refashioned into a ring-and-dot pattern as schematically shown in Figs. 2(a) and 2(b). The central circular electrode (the dot) that was about 0.4 cm in diameter served as the sensing electrode. The outer electrode that was shaped like a ring (the ring) served as the driving electrode. The silver paste between the central circular electrode and the outer ring electrode was chemically etched to create a gap about 0.2 cm wide. The PZT disk was then



FIG. 2. A schematic of the PZT/brass unimorph transducer: (a) viewed from the bottom PZT side and (b) viewed from the side.

glued to the brass sheet with the bottom electrode facing the brass sheet using a conductive epoxy from GC Electronic (Rockford, IL).

While the resonance frequencies of a piezoelectric transducer can be determined using an impedance analyzer,9,10 with a sensing electrode separated from the driving electrode, the resonance frequencies can be obtained without an impedance analyzer. To generate bending oscillations in the PZT/brass unimorph transducer, an ac electric field was applied at the outer ring electrode. The lateral stresses in the PZT layer associated with the bending movements in turn gave rise to induced electric fields detectable as voltage differences between the dot sensing electrode and the brass. When the frequency of the driving electric field matched one of the bending-mode resonance frequencies of the unimorph transducer, the induced voltage at the sensing electrode was maximized. Thus, the resonance frequencies of the transducer can be determined by monitoring the output electrical signal at the sensing electrode. With this approach, the resonance frequencies of the transducer can be easily determined. The advantages of such dual electrode designs include simple electrical detection, potential for integration with the microelectronic circuitry for in situ resonance-frequency monitoring, and large driving voltages that can also be used to melt the ice when de-icing is desired.

A schematic of the experimental setup is shown in Fig. 3. The brass rim of the PZT/brass unimorph disk transducer was firmly clamped with a cylindrical brass holder with an opening about 4.0 cm in diameter. The surface of the brass holder was wavy to ensure the brass rim of the disk unimorph transducer was firmly clamped by the brass holder. The PZT side of the transducer was facing down for protection and the brass side of the transducer was facing up to hold the deposited material, e.g., water or ice. A functional generator (Tektronix TM 5006, FG 501A 2 MHz function generator) was connected to the outer ring electrode to provide the driving voltage for the bending oscillation. A dualchannel digital oscilloscope (HP 54600A) was connected to the central sensing electrode to detect the piezoelectric voltage induced by the bending vibrations. When the frequency matches one of the bending-mode resonance frequencies of the transducer, the induced voltage at the sensing electrode was maximized. To avoid heating the transducer, a small driving voltage of 1 V peak to peak was used for the experi-



FIG. 3. A schematic of the experiment setup.

ment. The driving voltage can be increased to 30 V in the present setup. An Isotemp refrigerated circulator (Fisher Scientific Isotemp refrigerate circulator model 900) was used to control the temperature. The transducer was placed inside the cavity formed by the copper coil of the refrigerate circulator. A T-type thermal couple connected to a digital thermal meter (Fluke 2190A) was placed right above the transducer surface to monitor the temperature change in the transducer. The brass surface of the transducer was cleaned with detergent and water before the experiment.

IV. RESULTS

The first bending-mode resonance frequency was first measured at room temperature without water. Various amounts of water were carefully deposited on the brass surface of the transducer using a micropipette. The bending resonance frequency of the transducer with water was measured. Figure 4 shows the measured resonance frequency of the PZT/brass transducer versus water layer thickness (open circles). Also shown are the calculated resonance frequencies (dotted line) where the mass per unit area and the bending modulus per length were obtained using Eqs. (7) and (11), respectively. An effective eigen value, λ_{eff}^2 =9.2, was used for all the calculations, which was about 10% smaller than the ideal eigen value, $\lambda^2 = 10.2$.¹¹ Under ideal clamping conditions, i.e., the transducer should be clamped at the rim of the



FIG. 4. The first flexural mode resonance frequency of the PZT/brass unimorph vs the water thickness at room temperature.

PZT layer. In the present experiments, the PZT layer was smaller than the brass. The transducer was clamped at the brass rim instead of the PZT rim (see Fig. 3) and the diameter of the clamp was slightly larger than the diameter of the PZT disk (4 cm) to avoid cracking the PZT layer. These were not exactly the clamping conditions for the ideal eigen value $\lambda^2 = 10.2$ where the unimorph should be clamped at the PZT rim. To take into account of the present slightly different clamping conditions, we allowed the eigen value to be different from $\lambda^2 = 10.2$. It was obtained by fitting the calculated resonance frequency of the bare transducer without water to the measured value and is listed in Table I along with other physical properties used in all subsequent resonance frequency calculations. Figure 4 clearly shows that the resonance frequency decreased with an increasing amount of water and that the calculated resonance frequencies of the transducer with water agreed with the measured ones. This indicates that the resonance frequency of a unimorph transducer with a liquid layer on the metal side can indeed be described with Eqs. (1), (7), and (11).

After a water layer was deposited on the brass surface, the temperature was lowered. The first-mode flexural resonance frequency of the transducer was recorded at various temperatures during the cooling and the heating cycles. Figure 5 shows the resonance frequency versus temperature with 700 μ l of water (about 0.56 mm thick) deposited on the transducer. Also shown is the resonance frequency of the unloaded transducer (open squares) for comparison. Apart from the underlying increase in the resonance frequency with a decreasing temperature, which may be attributed to the increase in the Young's moduli of both PZT and brass with a decreasing temperature, there was an abrupt increase in the resonance frequency from about 1500 to about 2400 Hz when the apparent temperature of the water covered transducer was lowered below 2 °C. The sudden increase in the resonance frequency signaled the freezing transition of the water layer. With a water layer, the resonance frequency of the transducer (open circles during cooling and filled circles during heating) was lower than that of the unloaded transducer (open squares) at the same temperature. The water



FIG. 5. The first flexural mode resonance frequency vs temperature for the PZT/brass unimorph transducer with 700 μ l of water deposited on the brass surface. The background line is the resonance frequency of the bare transducer vs temperature.

layer possessed no rigidity (zero Young's modulus), the decrease in the resonance frequency due to the presence of water was attributed to the mass effect of the water layer [Eq. (7)] as we have shown in Fig. 4. As the water layer transformed into ice, the transducer's bending modulus increased abruptly [see Eq. (6)] and, hence, the resonance frequency. The resonance frequency change due to the freezing transition of the water layer was reversible. Upon heating, the resonance frequency of the transducer with an ice layer dropped abruptly from about 2400 to about 1500 Hz as the apparent temperature was increased above 4 °C, signaling the melting of the ice layer. The slight temperature hysteresis between the freezing and the melting cycles (about 2 °C in the example shown in Fig. 5) increased slightly with an increasing water thickness.

In Fig. 6 we plot the resonance frequency with respect to the deposited ice layer thickness where filled squares represent the measured resonance frequencies and the open circles calculated values using Eqs. (1)-(6). For a given ice thickness, the resonance frequency was measured when the ice layer was just formed (at around 0 °C) during the cooling



FIG. 6. The first flexural mode resonance frequency of the PZT/brass sensor vs ice thickness.

cycle. Clearly, the measured resonance frequencies agreed with the calculated ones, indicating the validity of using Eqs. (1)-(6) for calculating the resonance frequency of a unimorph transducer with a solid layer such as ice deposited on the metal surface. As can be seen from Fig. 6, the resonance frequency of the transducer increased with an increasing ice layer thickness due to the increased bending rigidity [Eq. (6)] by a thicker ice layer. This is in contrast to the case where the transducer was loaded with water that had no contribution to the bending modulus. The water layer only increased the mass of the transducer, causing the resonance frequency to decrease with an increasing water layer thickness.

The thickness sensitivity could be obtained as the slope of the fractional change of the resonance frequency with respect to the thickness change¹²

$$S_{t} = \frac{1}{f_{r}} \frac{df_{r}}{dt_{3}} \approx \frac{1}{f_{r0}} \frac{df_{r}}{dt_{3}},$$
(12)

where dt_3 and df_r refer to the small change in the deposited layer thickness, and f_{r0} is the resonance frequency of the bare transducer. For the present transducer with a 0.254-mmthick PZT layer, the ice thickness detection sensitivity, S_t , was about 1.5 Hz/Hz/mm at an ice thickness of about 1 mm and the water thickness detection sensitivity was about -0.1Hz/Hz/mm at a water thickness of about 1-2 mm.

V. SUMMARY

We have examined experimentally and theoretically the resonance frequency of a PZT/brass unimorph disk transducer with a water (ice) layer deposited on the brass surface. The presence of a water layer decreased the resonance frequency and the decrease in resonance frequency increased with the water amount. We showed that a PZT/brass unimorph transducer could easily distinguish a deposited ice layer from a water layer. The freezing transition was detected by monitoring the resonance frequency with respect to temperature. As the temperature was lowered, the resonance frequency of the transducer increased abruptly as the deposited water froze into ice at the freezing temperature. In contrast to water, the presence of an ice layer increased the resonance frequency and the increase in resonance frequency increased with ice thickness.

Theoretically, an analytic expression for the resonance frequency of a unimorph transducer due to the presence of an ice (water) layer deposited on the brass surface was obtained in terms of the Young's moduli, densities, and thickness of the PZT, brass, and ice (water) layers. The theoretical predictions were shown to agree with the experimental results.

ACKNOWLEDGMENTS

This work is supported in part by the Army Research Office Multidisciplinary University Research Initiative (MURI) under Grant No. DAAH04-95-1-0102, the National Aeronautics and Space Administration (NASA) under Grant No. NAG2-1475, and the National Science Foundation under Grant No. BES-0120321.

Downloaded 12 Jul 2002 to 128.112.140.74. Redistribution subject to AIP license or copyright, see http://ojps.aip.org/japo/japcr.jsp

- ¹A. Heinrich, Aircraft Icing Handbook, Technical Center Publication DOT/ FAA/CT-88/8-2, FAA, 1991.
- ²J. J. Gerardi and G. A. Hickman, 27th AIAA Aerospace Sciences Meeting, Reno, NV, 90–12 Jan. 1989.
- ³A. A. Demo and J. C. Garnier, 23rd AIAA Aerospace Science Meeting, Reno, NV, 14–17 Jan. 1985.
- ⁴B. Magenheim and J. K. Bocks, 20th AIAA Aerospace Sciences Meeting, Orlando, FL, 11–14 Jan. 1982.
- ⁵R. G. DeAnna, M. Mehregany, and S. Roy, Proc. SPIE **3046**, 42 (1997).
- ⁶M. Lustenberger, Proceedings of the 14th Symposium on aircraft Integrated Monitoring Systems, P. 619-638, Vibrometer, Fribourg, Switzer-

land, 1988.

- ⁷K. J. Claffey, K. F. Jones, and C. C. Ryerson, Atmos. Res. **36**, 277 (1995).
- ⁸S. Roy, A. Izad, R. G. DeAnna, and M. Mehregany, Sens. Actuators A 69, 243 (1998).
- ⁹X. Li, W. Y. Shih, I. A. Aksay, and W. H. Shih, J. Am. Ceram. Soc. **82**, 1733 (1999).
- ¹⁰ W. Y. Shih, X. Li, H. Gu, and W.-H. Shih, J. Appl. Phys. 89, 1497 (2001).
 ¹¹ A. W. Leissa, *Vibrations of Plates* (NASA SP-160, Scientific and Techni-
- cal Information Division, Washington, DC, 1969).
- ¹²T. Thundat, P. I. Oden, and R. J. Warmack, Microscale Thermophys. Eng. 1, 185 (1997).