

## Magnetoelastic sensors in combination with nanometer-scale honeycombed thin film ceramic TiO<sub>2</sub> for remote query measurement of humidity

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Ribbonlike magnetoelastic sensors can be considered the magnetic analog of an acoustic bell; in response to an externally applied magnetic field impulse the sensors emit magnetic flux with a characteristic resonant frequency. The magnetic flux can be detected external to the test area using a pick-up coil, enabling query remote monitoring of the sensor. The characteristic resonant frequency of a magnetoelastic sensor changes in response to mass loads [L. D. Landau and E. M. Lifshitz, *Theory of Elasticity*, 3rd ed. (Pergamon, New York, 1986), p. 100]. Therefore, remote query chemical sensors can be fabricated by combining the magnetoelastic sensors with a mass changing, chemically responsive layer. In this work magnetoelastic sensors are coated with humidity-sensitive thin films of ceramic, nanodimensionally porous TiO<sub>2</sub> to make remote query humidity sensors. © 2000 American Institute of Physics. [S0021-8979(00)32708-6]

### INTRODUCTION

Figure 1 shows the basic operational principle of a magnetoelastic sensor; the sensor material is a ferromagnetic, amorphous metallic glass thick-film exhibiting large values of magnetostriction.<sup>1</sup> In response to a time varying magnetic field, magnetic energy is converted to elastic energy which acts to mechanically deform the sensor. As the sensor is magnetostrictive, magnetic flux is emitted from the sensor with the mechanical deflections. The frequency spectrum of the sensor can be obtained by sweeping an ac magnetic interrogation field over a predetermined frequency range, with the response measured using a pickup coil. If the frequency of the ac field is equal to the mechanical resonance frequency of the sensor the conversion of the magnetic energy into elastic energy is maximal and the sensor undergoes a magnetoelastic resonance. Thick-film ferromagnetic magnetoelastic ribbons, such as the Metglas™ alloys,<sup>2</sup> have been used as position sensors,<sup>3</sup> strain sensors,<sup>4</sup> and antitheft markers.<sup>5</sup>

For a thin, ribbon-shaped sensor of length  $L$  vibrating in its basal plane the resonant frequency is given by<sup>6</sup>

$$f_n = \sqrt{\frac{E}{\rho(1-\sigma^2)}} \frac{n\pi}{L} \quad n = 1, 2, 3, \dots,$$

where  $E$  is Young's modulus of elasticity,  $\sigma$  is the Poisson

ratio,  $\rho$  is the density of the sensor material, and  $n$  denotes integers. We concern ourselves with the fundamental resonant frequency,  $n = 1$ , due to its relatively larger amplitude.

A dc magnetic field, superimposed with the ac magnetic field, is used to effectively offset the magnetic anisotropy of the sensor material enhancing the magnetoelastic properties.<sup>4,7</sup> This dc biasing field can be supplied either by a field coil, or by adjacent placement of a magnetically hard thick film. Figure 2 demonstrates the frequency-dependent response of a 30 mm × 4 mm × 30 μm Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> Metglas sensor, measured at room temperature. A constant 5.5 Oe dc field was applied over the test region by a Helmholtz coil; the frequency of the 50 mOe sinusoidal ac field is swept over the predetermined frequency range, and the response of the

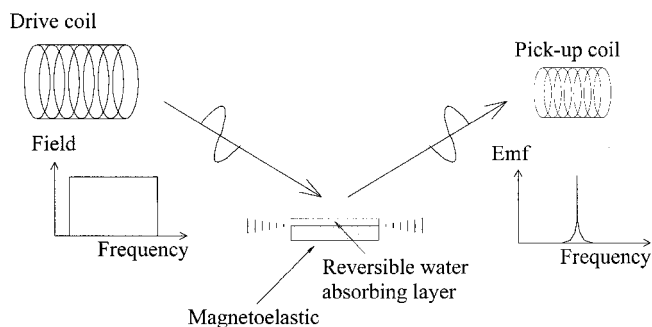


FIG. 1. Schematic drawing demonstrating remote query nature of magnetoelastic sensors.

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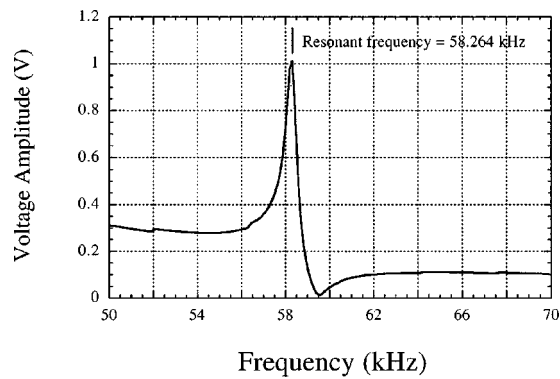


FIG. 2. Frequency spectrum of a  $30\text{ mm} \times 4\text{ mm} \times 30\text{ }\mu\text{m}$   $\text{Fe}_{40}\text{Ni}_{14}\text{P}_{14}\text{B}_6$  alloy 2826 MB (Ref. 2) sensor, measured at room temperature.

sensor monitored by use of a pickup coil. Since it is the frequency response of the sensor that is monitored, rather than the amplitude, the relative orientation of the sensor with respect to the detecting coil is unimportant. The sensor material was obtained from Allied Signal Corporation,<sup>2</sup> alloy 2826MB, and used without further processing; similar sensors were used for all measurements described herein.

Changes  $\Delta m$  to the mass  $m$  of the sensor correspond to an increase of its density by a factor  $1 + \Delta m/m$  which, in turn, changes the resonant frequency by a factor of  $(1 + \Delta m/m)^{-0.5}$ . If the mass increase is small compared to the mass of the sensor, the shift in the resonant frequency is given by  $\Delta f = -f(\Delta m/m)$ . Thus small increases in mass can be detected by monitoring the downward shifts in the resonance frequency of the sensor.

In this work, we report on application of the sensing principle to fabrication of remote query humidity sensors. Thick film magnetoelastic sensors, approximately  $25\text{ mm} \times 4\text{ mm} \times 30\text{ }\mu\text{m}$ , are combined with sol-gel deposited  $\text{TiO}_2$  ceramic layers approximately  $2\text{ }\mu\text{m}$  thick. The  $\text{TiO}_2$  layers reversibly absorb-desorb water moisture, in turn reversibly gaining or losing mass.

## EXPERIMENTAL RESULTS

The humidity sensors were fabricated by coating alloy 2826MB sensors with sol-gel deposited, porous,  $2.0\text{ }\mu\text{m}$  thick

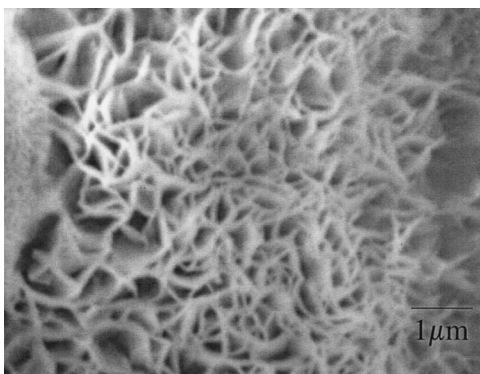


FIG. 3. SEM image of the  $\text{TiO}_2$  layer, the fabrication details of which are described in Ref. 11, deposited on 2826MB alloy sensor. Magnification is  $\times 25\text{ }000$ .

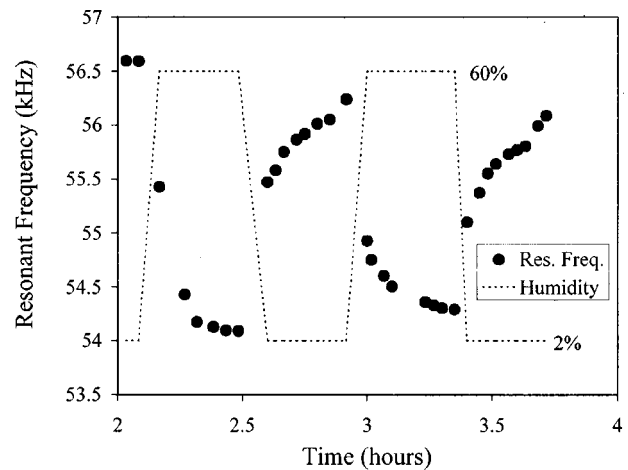


FIG. 4. Measured resonant frequency of magnetoelastic humidity sensor cycled between 60% and 2% relative humidity levels.

layers of  $\text{TiO}_2$ . Two different sol-gel recipes were used, that of Refs. 8–10 and 11, with each resulting  $\text{TiO}_2$  layer responding rapidly to changes in relative humidity. Interestingly enough, the approach followed in Ref. 11 for fabrication of the  $\text{TiO}_2$  layer resulted in a nanoscale porous honeycomb structure as shown in Fig. 3. Presumably the honeycomb structure, with a pore size of approximately 80 nm, boosts the ceramic layers ability to trap moisture. The sensors were tested in a 50 cm diam cylindrical humidity chamber (1 m length), about which a ten-turn pick coil was wound. Figure 4 shows the resonant frequency of a magnetoelastic humidity sensor in response to alternating high and low relative humidity levels, 60% and 2%, respectively; the mass change of the sensor between the two extremes is approximately 1.7 mg, or 3.7%. The dashed line in Fig. 4 corresponds to the humidity cycle the sensor was exposed to. It is interesting to note that there is an immediate jump in the resonant frequency of the sensor with the humidity changes.

Figure 5 demonstrates the reversibility of the magnetoelastic humidity sensor, with the resonant frequency measured at increasing and decreasing humidity levels. The sensor was held at a constant humidity level for 10 min prior to each measurement.

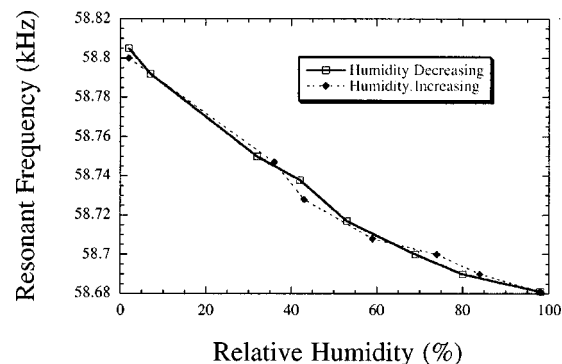


FIG. 5. Measured resonant frequency of magnetoelastic humidity sensors as a function of increasing and decreasing relative humidity levels. The sensor was exposed to a constant humidity level for 10 min prior to each measurement.

each measurement. There is a slight deviation from linearity, presumably due to rate-limited diffusion times of the ceramic.

## CONCLUSIONS

Presented herein is application of magnetoelastic sensors for remote query sensing of humidity without direct physical connections such as wires, or special alignment requirements as needed for laser telemetry. In combination with a mass changing humidity responsive TiO<sub>2</sub> ceramic layer magnetoelastic sensors can be used for remote query humidity monitoring. The sensors are passive, responding to the query field, and are quite inexpensive allowing for their use on a disposable basis. Remote query sensors such as those described would be useful for monitoring the humidity levels inside sealed containers such as food packages. In combination with different chemically responsive layers the sensing technology could be extended to other *in situ* or *in vivo* monitoring applications<sup>12,13</sup> such as gastric pH or glucose.<sup>14</sup>

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- <sup>1</sup>J. Gutierrez, J. M. Barandiaran, and O. V. Nielsen, *Phys. Status Solidi A* **111**, 279 (1989).
- <sup>2</sup>Metglas™ is a trademark of the Allied Signal Corporation. See product information at <http://www.electronicmaterials.com/products/amorph/index.htm>
- <sup>3</sup>J. M. Barandiaran and J. Gutierrez, *Sens. Actuators A* **59**, 38 (1997).
- <sup>4</sup>E. E. Mitchell, R. DeMoyer, and J. Vranish, *IEEE Trans. Ind. Electron.* **33**, 166 (1986).
- <sup>5</sup>J. Ryan, Jr., *Sci. Am.* May, 120 (1997).
- <sup>6</sup>L. D. Landau and E. M. Lifshitz, *Theory of Elasticity*, 3rd ed. (Pergamon, New York, 1986), p. 100.
- <sup>7</sup>P. M. Anderson III, *J. Appl. Phys.* **53**, 8101 (1982).
- <sup>8</sup>W. W. So, S. B. Park, and S. J. Moon, *J. Mater. Sci. Lett.* **5**, 12 (1986).
- <sup>9</sup>K. Terabe, K. Kato, H. Miyazaki, S. Yamaguchi, A. Imai, and Y. Iguchi, *J. Mater. Sci.* **29**, 1617 (1994).
- <sup>10</sup>T. Hayashi, T. Yamada, and H. Saito, *J. Mater. Sci.* **18**, 3137 (1983).
- <sup>11</sup>L. Miller, M. I. Tejedor-Tejedor, and M. A. Anderson, *Environ. Sci. Technol.* **33**, 2070 (1999).
- <sup>12</sup>P. G. Stoyanov, S. A. Doherty, C. A. Grimes, and W. R. Seitz, *IEEE Trans. Magn.* **34**, 1315 (1998).
- <sup>13</sup>C. A. Grimes, P. G. Stoyanov, Y. Liu, C. Tong, K. G. Ong, K. Loiselle, M. Shaw, S. A. Doherty, and W. R. Seitz, *J. Phys. D* **32**, 1329 (1999).
- <sup>14</sup>C. A. Grimes, K. G. Ong, K. Loiselle, P. G. Stoyanov, D. Kouzoudis, Y. Liu, C. Tong, and F. Tefiku, *J. Smart Mater. Struct.* **8**, 639 (1999).