## A Thermally Actuated MEMS Viscosity Sensor

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## **Outline**

- Motivation
- Viscosity
- MEMS viscometers
- Proposed thermally actuated MEMS viscometer
  - Operation principles
  - Design
  - Evaluation
  - Results
- Proposed work

## Motivation

Fluid viscosity applications

- Automotive
  - Motor oil changes
    - Several factors determine when to change oil:
      - Contaminants, soot, water
      - Viscosity changes (shear, oxidation and soot)
  - Drive-train lubricants
- Medical
  - Blood coagulation rates (point-of-care treatment)
- Industrial
- Small, reliable and inexpensive -- MEMS

## In-situ monitor of lubricant quality

• Multisensor diagnostics

Nyquist Plot of BP Oils for Sensor

- Contaminants
  - Water, soot
  - Electrochemical
     Impedance
     Spectroscopy (EIS)
- Viscosity and density
  - Both change as oil degrades over time



Marx et al, "Micro-Sensor for Monitoring Oils", IEEE 2006

 Temperature and relative humidity sensors are also desired

## Viscosity

- Viscosity
  - Internal resistance to flow or shear
  - Measured with a viscometer using a small sample of lubricant
  - In-situ measurement is desired



## Viscosity and oil viscosity

•Dynamic viscosity is measured in Pa\*s or centiPoise 1 cP = 0.001 Pa\*s

•Kinematic viscosity takes into account density of fluid

$$v = \frac{\eta}{\rho}$$
  
l cSt = 0.0001 m/s<sup>2</sup>

#### •Oil Viscosity depends on temperature





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## Stokes to SAE standard

www.widman.biz/uploads/Corvair\_oil.pdf



Viscosities can be related horizontally only. For example, the following oils have similar viscosities: ISO 460, AGMA 7 and SAE GEAR OIL 140.

The viscosity/temperature relationships are based on 95 VI oils and are usable only for mono grade engine oils, gear oils and other 95 VI oils.

Crankcase oils and gear oils are based on 100°C viscosity. The "W" grades are classified on low temperature properties. ISO oils and AGMA grades are based on 40°C viscosity. IVAN PUCHADES 11/20/2009

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## Oil viscosity at room T

www.widman.biz/uploads/Corvair\_oil.pdf



## **Degradation of oil**



Effect of Shear and Oxidation on Viscosity

Oil will deteriorate from 10.3 cSt to 13.3 cSt at 100 C (operating T) Corresponds to a change from 65.2 cSt to 110 cSt at 40 C - Wang, 2001

Approximately a 50 cSt resolution is needed at 40 C.

## **Cantilever MEMS Viscometers**

•Cantilever Beam resonators •Change in natural frequency is correlated to viscosity •Electromagnetic or PZT actuation - Complex to integrate and fabricated •Optical readout •Reliability in harsh environments?

•CMOS compatibility?



Zhao et al, 2005





Fig. 3b: Experimental amplitude and phase of deflection for a silicon cantilever (70µm x 200µm x 3000µm) oscillating in various viscous fluids.



Fig. 2. Optical photograph of the fabricated device and SEM of polysilicon resistors.

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## **Cantilever MEMS Viscometers**





S. Boskovic, J. Chon, P. Mulvaney, and J.E. Sader, "Rheological measurements using microcantilevers," *Journal of Rheology*, vol. 46pp, 2002, pp. 891-899.

$$m\frac{\partial^2 x}{\partial t^2} + c\frac{\partial x}{\partial t} + kx = 0$$

$$A(\omega) = \frac{A_0 \omega_R^2}{\sqrt{(\omega^2 - \omega_R^2)^2 + \frac{\omega^2 \omega_R^2}{Q^2}}}$$

$$\omega_{R} = \frac{\omega_{vac}}{\sqrt{1 + \frac{\pi\rho b^{2}}{4\mu}\Gamma_{r}(\omega_{R})}} \qquad Q = \frac{\frac{4\mu}{\pi\rho b^{2}} + \Gamma_{r}(\omega_{R})}{\Gamma_{i}(\omega_{R})}$$

 $\omega_{vac}$  – resonance in vacuum  $\omega_R$  – resonance in fluid  $\mu$  – mass per unit length of cantilever  $\rho$  – density b – beam width  $\Gamma$  – hydrodynamic function (Navier-Stokes, density, viscosity and geometry)

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## MEMS Viscometer Design considerations

- •CMOS compatible
- •Precise amplitude control
- •Simple read out (non-optical)
- •Easy to fabricate
- •Robust and reliable
- •Actuation at resonant frequency is not needed

•Measure power required to maintain a constant precise amplitude

### •Thermal actuation?

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## **Thermally Actuated Beams**

- •Large displacement
- •Slower movement
- •Lots of power
- •Applications
  - •Switches
  - •Latching
  - •Optical
  - •Micro-robots
  - •Micro-grippers



Fig. 6. SEM images of two of the fabricated actuators.

Jorge Varona et al. *Design of MEMS vertical-horizontal chevron thermal actuators*, Sensors and Actuators A: Physical, Volume 153, Issue 1, 25 June 2009, Pages 127-130,



Figure 1. A SEM micrograph of a thermally actuated 3D micromirror developed at the Institute of Microelectronics (IME).

J Singh, J H S Teo, Y Xu, C S Premachandran, N Chen, R Kotlanka, M Olivo and C J R Sheppard, *A two axes scanning SOI MEMS micromirror for endoscopic bioimaging* Journal of Micromechanics and Microengineering, February 2008, V18, p. 025001

## **Thermally Actuated Plates**

- •Large displacement
- •More power
- •Applications
  - •Valves
  - •Optical
  - •Ultrasound



Oliver Brand, Mark Hornung, Henry Baltes, Member, IEEE, and Claude Hafner, *Ultrasound Barrier Microsystem for Object Detection Based on Micromachined Transducer Elements*, JOURNAL OF MICROELECTROMECHANICAL SYSTEMS, VOL. 6, NO. 2, JUNE 1997 151

## Microvisk Viscosity Sensor





Figure 2: Time-varying signals obtained from fluids of different viscosity.



### **Proposed: Electrothermal MEMS Viscometer**



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•In-situ P+ Si heater (joule heating). •In-situ poly-silicon piezoresistor bridge to monitor membrane deflection Vout=V2-V1

•Vertical displacement due to thermal coefficient of expansion difference between Si/SiO2 and Al (bimetallic effect)

•Resistance to motion is related to the viscosity of the fluid.



## Oil viscosity testing

Vsupply=14V

Osc=9V, 5Hz

G=41

Measurements taken at room temperature 22C

5W30 – 115.4 cSt 10W40 – 239.4 cSt SAE60 – 758.4 cSt



DelV(5W30)=407mV DelV(10W40)=388mV 19mV difference – not consistent

#### Need to amplify resistance of fluid motion to improve resolution

## **Cover for Viscometer**





Cover amplifies
resistance to movement
of membrane.
Cover is smaller to
allow for wirebonds.
Gap can be easily
adjusted with KOH etch
time.

### Oil viscosity testing – with cover

Vsupply=14V

Osc=9V, 5Hz

G=41

Measurements taken at room temperature 25C

5W30 – 124.3 cSt 10W40 – 146.5 cSt

20 cSt resolution



DelV(5W30)=545mV DelV(10W40)=471mV 74mV difference

#### Improved resolution.

### Conclusions

- Cooling effect of oil
- Local heating
  - Quick measurements avoid heating the oil.
- Front plate to increase sensitivity
  - Need to determine best gap distance.
- Frequency of interrogation
  - Need to determine optimal frequency to avoid the membrane heating up to steady state.
- Need to interrogate without affecting liquid under test.

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## Microvisk Update - 2009

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•Pulse heat



V. Djakov, "Fluid Probe," 2009, p. 45. WO2009022121A2 Microvisk Limited



RIT- Microsystems Engineering Ivan Puchades W-water, B-Brine

## **Thermal resonator**

1997 paper by Brand
Thermal resonator vibrates with heat burst
Used to monitor polymer formation in PDMS solutions Passivation Passivation Physical Aluminum Thermal Oxide Thermal Oxide P-Diffusion for Heating Resistor and Piezoresistors Silicon Substrate Etch Mask

Fig. 1: Schematic of the membrane resonator with heating resistor for excitation and piezoresistors for detection.



<sup>7</sup>ig. 4: Output signal of the membrane resonator subject to a burst excitation for (a) a 1 cSt (8.2·10<sup>-4</sup> Pa·s) and (b) a 10 cSt (9.4·10<sup>-3</sup> Pa·s) PDMS solution. Note that trace (a) was measured at a lower burst rate because of the longer decay time in the less viscous solution.



Fig. 2: (Left) Photograph of a fabricated 1.5 mm by 1.5 mm membrane resonator with the driving resistor in the center and the piezoresistors arranged in a Wheatstone bridge close to the edge. (Right) Complete membrane structure after wire bonding but prior to sealing



Fig. 5: Quality factor of the piezoelectric transducer (circles) and a 1.4 mm by 1.4 mm membrane resonator (triangles) as a function of the shear viscosity in air (open symbols) and different PDMS solutions (solid symbols).

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Actuators, vol. 1, 1997, pp. 121-124.

O. Brand, J.M. English, S.A. Bidstrup, and M.G.

Allen, "Micromachined viscosity sensor for real-time

polymerization monitoring," Proceedings of the 1997

International Conference on Solid-State Sensors and

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### Proposed: Electrothermal MEMS Viscometer



•In-situ P+ Si heater (joule heating).

•In-situ poly-silicon piezoresistor bridge to monitor membrane deflection

Vout=V2-V1

•Short thermal pulse to set diaphragm in motion.

•Damped simple harmonic oscillator with initial displacement determined by thermal pulse.

•Initial vertical displacement due to thermal coefficient of expansion.

•Viscosity of the fluid dampens vibration

• Q changes, also natural frequency.



### **Operation Principle**

•Plate behavior to suddenly applied heat

- Theory developed in the 1950's for jet propulsion.
- Static and dynamic (vibration) components
- Dynamic vibration is at natural frequency of the plate
- Goal was to minimize vibrations

B. Boley and J. Weiner, Theory of Thermal Stresses, Malabar, Florida: Robert E. Kreiger Publishing Company, 1985.

•Natural frequency of a square plate due to a heat pulse

- Due to the inertia term
- Depends on both thickness and size of the diaphragm
- Amplitude depends on temperature
- Natural frequency does not depend on temperature

$$\omega_{n} = \frac{B^{2}\pi^{2}(m^{2} + \frac{a^{2}}{b^{2}}n^{2})\tau}{t} = \frac{\left(\frac{h}{a\sqrt{\kappa}}\left(\frac{D}{h\rho}\right)^{1/4}\right)^{2}2\pi^{2}\frac{\kappa t}{h^{2}}}{t} = \frac{2\pi^{2}}{a^{2}}\sqrt{\frac{D}{h\rho}}$$

 $\frac{Eh^3}{12(1-\nu^2)}\frac{\partial^4 w(x,y,t)}{\partial x^4} + \rho h \frac{\partial^2 w(x,y,t)}{\partial t^2} = -\frac{1}{1-\nu}\nabla^2 M_T$  $w(x, y, t) = w_{et} - w_{dyn}$ 10 7.94 - h=10um h=15um h=30um 7.92 7.9 ≥ 7.88 7.86 7.84

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0.2

0.4

0.6

0.8

1.2

× 10<sup>-4</sup>

1

### **Operation Principle Plate-fluid interaction**

- Plate vibration in fluid
  - Fluid-structure interaction theory
  - Frequency shift due to density of fluid – Virtual Added Mass
  - Viscous effects are neglected.
    - Only become important for large viscosity values

$$\omega_{fluid} = \frac{\omega_{vacuum}}{\sqrt{1+\beta}}$$

$$\beta = 0.669 \frac{\rho_{fluid} a}{\rho_{plate} h}$$

$$\beta = 0.6538 \frac{\rho_{fluid} a}{\rho_{plate} h} (1 + 1.082\xi)$$

• 2009 paper relates shifts in frequency to viscosity for microstructures

$$Q = 2\pi \frac{\text{energy\_stored}}{\text{energy\_dissipated\_per\_cycle}} \approx \frac{0.95}{\xi}$$
$$\xi = \sqrt{\frac{\upsilon}{\omega a^2}}$$

v - kinematic viscosity  $\rho$  – density

Y. Kozlovsky, "Vibration of plates in contact with viscous fluid: Extension of Lamb's model," Journal of Sound and Vibration, vol. 326, 2009, pp. 332-339.

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### **Vertical Displacement Calibration**

- Veeco Wyko White Light Interferometer
- Measure z-displacement and Vout=V2-V1



•Calibration of Vout to vertical deflection.

•Images at 0, 50, 100, 150, 200 and 250m A

### Thermal MEMS Viscometer Design Outline

- Based on operation principles
  - Determine Diaphragm Thickness
    - Thin enough for significant displacement
    - Thick enough to prevent buckling
    - Evaluate diaphragm thickness vs. vertical displacement
  - Determine Pulse Energy
    - Need enough energy to obtain significant diaphragm deflection
    - Short enough to prevent interaction with fluid
      - Temperature affects initial displacement amplitude
    - Monitor diaphragm temperature with varying pulse times
  - Dynamic Measurements
    - Natural frequency and quality factor Q in air
    - Natural frequency and quality factor Q in fluid
      - Viscosity measurement

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### Determining Diaphragm Thickness v. Linear actuation

#### •<u>h<10um</u>

Rapid increase with lower power, hystheresis effect – snapback

•*10um>h>20um* Linear relation to power

#### •<u>h>20um</u>

Rapid increase at 1W leveling off at 2W Buckling

•Good match to theoretical predictions



a = 2.5mm

#### •Keep *h* between *15* and *20 µm*

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### **Determining Pulse Energy - Theoretical**

1.2

1

Temp SiO2

1-D Transient Temperature Equation  $K_{SiO2} = 0.009 \text{ cm}^2/\text{s SiO}_2$ semi-infinitely long body x >= 0

$$\frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial x^2}$$

$$T = T_a erfc \left(\frac{x}{2\sqrt{\kappa t}}\right)$$



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### **Pulsed Diaphragm Temperature Evaluation in Fluid**



Forward biased diode to monitor temperature at 5 Hz, 200 ms pulse.
100 C swing in air
50 C swing in oil



•30 V pulse.

No temperature differences can be appreciated t<sub>pulse</sub> < 100 us</li>
Energy has to be large enough to set diaphragm vibrating at its natural frequency without damaging the device.

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    - Natural frequency and quality factor Q in fluid
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## LabView Integration



## Natural Frequency in Air

- •30V 30us square pulse
- •Theoretical natural vibration frequency SSSS

$$f_{ij} = \frac{\lambda_{ij}^2}{2\pi a^2} \left[ \frac{Eh^3}{12\gamma(1-\nu^2)} \right]^{1/2}$$

$$f_{11} = 16579Hz$$

- $\lambda$  function (boundary conditions, a/b, v)
- E Young's modulus
- h-plate thickness
- $\gamma$  mass per unit area of plate
- a length of plate
- v Poisson's ratio



## T=64 us, f=15,625 Hz – corresponds to natural frequency of plate

## **Experimental Responses**



#### lest Setup for Screening **Experiment** Device 2H 30V-30us pulse v. SAE 60 temperature •PCB electronics SAE60 35 SAE60\_43\_1 SAE60\_54\_1 SAE60\_75\_1 •LabView analysis for real Ê -5.00E-04 +00 0.00 + 00 5.00E-04 1.00E-03 1.50E-03 2.00E-03 -2.00E-07 Displa time monitoring •Long term analysis 1 00E-06 Time (s) 5 KOhm LabView SV FFT Device 2H Natural Frequencies vs. Dynamic Viscosity oven 0.01 57 cS 5V V heat 0.009 146 cSt Oscilloscope 0.008 0.007 249 cS1 0.006 **፪**0.005 A 0.004 419 cSt 0.003 0.002 0.00 PMO<sub>5</sub> 1000 1500 2000 2500 3000 3500 Frequency (Hz) 4000 4500 5000 Waveform **GND** Outputs: Generator Frequency Amplitude Q Temperature **IVAN PUCHADES**

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### LabView Screenshot



## LabView Code



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## **Sensor Viscosity Test**

The viscosity of Motor oil has a strong dependence on temperature.

Change temperature of motor oil to test a range of viscosities.

Temperature also affects the natural frequency of vibration. But this effect is linear and can be easily measured in air and removed from the viscosity measurements.



		5W30	10W40	SAE60
,	Density (60 F)	0.876 kg/l	0.8713 kg/l	0.8931 kg/l
	Viscosity 40C	57.2 eSt	109.7 cSt	293.4 cSt
	Viscosity 100C	10.5-11.2cSt	14.0 cSt	24.0 cSt
	Viscosity Index	176	146	104

#### Effect of Temperature on Natural Vibration Frequency

Change in dimensions (thermal expansion coefficient) and young modulus will change the resonant frequency of the vibrating plate.

This effect is linear.

$$f_{ij} = \frac{\lambda_{ij}^2}{2\pi a^2} \left[ \frac{Eh^3}{12\gamma(1-\nu^2)} \right]^{1/2}$$

 $\lambda$  – function (boundary conditions, a/b)

- E Young's modulus
- h-plate thickness
- $\gamma$  mass per unit area of plate
- a length of plate
- v Poisson's ratio

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Heat pulse time and amplitude have no effect on frequency of vibration.

They affect the amplitude of vibration but not the frequency.
 Designed plates have different material compositions.

### **Effect of Temperature on Natural Vibration**

#### Frequency

$$f_{ij} = \frac{\lambda_{ij}^2}{2\pi a^2} \left[ \frac{Eh^3}{12\gamma(1-\nu^2)} \right]^{1/2}$$

•Higher frequencies due to Poly, Passivation and Metal layers lead to a negative temperature dependence (*E* dominated). Fo decreases with T.

•Lower frequencies, without Pass and Metal, are geometry dominated  $(h^3, a^2)$ . Fo increases with T.



ID	Device	Heater	Piezo	Size	Rheater	Pass	Metal	Fo	Q/cycles	Amp(mV)	UP/DOWN	Slope Norm
35	P_2.5_0.16_No _PASS_Yes_MTL	Р	Poly	2.5	16%	No	Yes	23724	5	2	DOWN	-1.20E-03
36	P_2.5_0.02_No _PASS_Yes_MTL	Р	Poly	2.5	2%	No	Yes	20922	31	5	DOWN	-1.17E-03
47	Poly_2.5_0.16_No_PASS_No_MTL	Poly	Poly	2.5	16%	No	No	23616	62	10	DOWN	-3.30E-04
43	Poly_2.5_0.35_Yes _PASS_No_MTL	Poly	Poly	2.5	35%	Yes	No	21042	7	4	DOWN	-8.75E-04
4D18	Poly_2.5_0.02_No _PASS_No_MTL	Poly	Poly	2.5	2%	No	No	16440	100	20	DOWN	-1.90E-03
25	Poly/P+_2.5_0.35_No _PASS_Yes_MTL	Poly	P+	2.5	35%	No	Yes	18660	80	250	UP	1.39E-03
49	P_2.5_0.02_No _PASS_No_MTL	Р	Poly	2.5	2%	No	No	14505	49	5	UP	1.52E-03
59	Poly_2.5_0.35_No _PASS_Yes_MTL	Poly	Poly	2.5	35%	No	Yes	16997	60	15	UP	1.94E-03
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## **Oil testing**

Temperature of 10W40 oil is increased as the frequency, amplitude and Q of the sensor is measured.

$$f_{ij} = \frac{\lambda_{ij}^2}{2\pi a^2} \left[ \frac{Eh^3}{12\gamma(1-\nu^2)} \right]^{1/2}$$

$$\omega_{fluid} = \frac{\omega_{vacuum}}{\sqrt{1+\beta}}$$

$$\beta = 0.6538 \frac{\rho_{fluid} a}{\rho_{plate} h} (1 + 1.082\xi) \qquad \qquad \xi = \sqrt{\frac{\nu}{\omega a^2}}$$

$$Q = 2\pi \frac{\text{energy stored}}{\text{energy dissipated per cycle}} \approx \frac{0.95}{\xi}$$





Temperature Density Viscosity

### Removing remperature Effect

Effect of Temperature is removed.

Devices with 2% heater show very small variation when effect of Temperature is removed.

D11, D25 and D59 show similar slopes.



#### Density and Viscosity

ID	Device	Heater	Piezo	Size	Rheater	Pass	Metal	P_M	Fo	Q/cycles	Amp(mV)
11	P_2.5_0.16_Yes _PASS_Yes_MTL	Р	Poly	2.5	16%	Yes	Yes	Yes_Yes	22880	25	10
12	P_2.5_0.02_Yes _PASS_Yes_MTL	Р	Poly	2.5	2%	Yes	Yes	Yes_Yes	27250	20	10
36	P_2.5_0.02_No _PASS_Yes_MTL	Р	Poly	2.5	2%	No	Yes	No_Yes	20922	31	5
25	Poly/P+_2.5_0.35_No _PASS_Yes_MTL	Poly	P+	2.5	35%	No	Yes	No_Yes	18660	80	250
49	P_2.5_0.02_No _PASS_No_MTL	Р	Poly	2.5	2%	No	No	No_No	14505	49	5
59	Poly_2.5_0.35_No _PASS_Yes_MTL	Poly	Poly	2.5	35%	No	Yes	No_Yes	16997	60	15

### rioung vs. kinemauc viscosity

Exponential fit to experimental data.

Temperature of oil can be converted to kinematic viscosity. Takes into account change is density.

Best at low values.



### **PIOTTING VS. KINEMATIC**

### vierneitv

Similar results obtained with three different devices.

Error associated with Fo extraction algorithm and transient temperature effects.



## **Compare to theoretical**



v - kinematic viscosity  $\rho$  – density

# Testing in oils with different viscosities

Device D25 is placed in oils of different viscosities:

5W30, 10W40 and SAE60

Temperature of the oil is increased.

Frequency of resonance changes with the oil's temperature, density and viscosity.



### **Remove effect of temperature**

The effect of temperature on the resonant frequency is removed with the data obtained with the device was tested in air.

In this case the change in frequency is reduced by 0.13% / C due to this temperature effect.



### Plot resonance frequency vs. viscosity

The data is plotted against kinematic viscosity. This takes into account the change in density that the oil experiences as the temperature is increased.

The proposed sensor measured kinematic viscosity as the oil is not only sheared but also displaced.



$$v = \frac{\eta}{\rho}$$

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## Normalize frequency

0.9

0.85

8.0 0

50

Normalized at 40 cSt so that all three oils have a common viscosity value.



100

Kinematic viscosity (cSt)

150

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## Conclusion

•Successful fabrication of thermal resonator devices to measure viscosity.

•Improved understanding of factors affecting performance.

- •Good sensitivity to viscosity.
- •Further testing may improve sensitivity even further.
- •JMEMS article under review. Requested more data to support claims.
- •A second journal article in preparation.

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