Development of clamp-on type thermal mass flow meter using near infrared absorption method for micro flow applications

Seok Hwan Lee, Seongchong Park, Woong Kang
Division of Physical Metrology,
Korea Research Institute of Standards and Science, South Korea
Contents

1 Background

2 Purpose

3 In-situ measurement of micro flow rate using FTNIR (Fourier Transform Near Infrared Spectroscopy)

4 In-situ measurement of micro flow rate using laser diode

5 Conclusion
Medical drug injection
- Small amount of drug
  (pain treatment : 21.6 ug/day, insulin pump : 70 nL/min)
- Patient treatment through accurate drug injection monitoring and control
- Limitation of infusion and syringe pumps (low accuracy, environment dependency)

Chemical analysis (chromatography)
- Micro flow liquid chromatography (250 – 600 µL/min)
- Column diameter : 0.25 – 1 mm, total volume : 2-10 µL
- Accuracy flow rate control and monitoring

Semiconductor and fuel cell industries
- Semiconductor process (precursor delivery, cleaning..)
- Fuel flow rate inside fuel cell
Micro flow meters

- Coriolis mass flow meter
  - High accuracy (±0.2 %, maximum flow rate : 5 g/h)
  - Expensive price, relatively heavy size

- Thermal mass flow meter
  - Low flow rate limit : nL/min order
  - 10 % flow rate accuracy, low price and high portability

- Ultrasonic flow meter
  - Can measure in-situ condition (none contact)
  - Low accuracy in micro flow rate
  - No commercial flowmeter which can measure micro flow rate in-situ condition.

<table>
<thead>
<tr>
<th>Flow meters</th>
<th>Coriolis</th>
<th>Thermal mass</th>
<th>Ultrasonic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy</td>
<td>*****</td>
<td>***</td>
<td>**</td>
</tr>
<tr>
<td>Price</td>
<td>*</td>
<td>****</td>
<td>**</td>
</tr>
<tr>
<td>Micro flow rate</td>
<td>*****</td>
<td>*****</td>
<td>*</td>
</tr>
<tr>
<td>In situ measurement</td>
<td>none</td>
<td>none</td>
<td>****</td>
</tr>
</tbody>
</table>
Laser based flow measurement tool

- LDA (Laser Doppler Anemometry)
  - Flow velocity measurement using Doppler effect
  - Point measurement, seeding particle necessary

- PIV (Particle Image Velocimetry)
  - Most popular flow velocity field visualization method
  - Seeding particle necessary

- MTV (Molecular Tagging Velocimetry)
  - Visualization of Raman scattering signal
  - Simultaneous measurement of concentration and velocity
  - Low signal intensity

- Seeding particle is necessary
In-situ measurement of micro flowrate without seeding

- Micro-interferometric back-scatter and phase detection
  - D. A. Markov et al, Noninvasive fluid flow measurements in microfluidic channels with backscatter Interferometry, *Electrophoresis* 2004

**Fourier spectrum of (a) the shutter control waveform, and (b) the corresponding bicell output**

**Waveforms of the shutter and bicell output**

**Calibration curve for the flow rate with the heating zone 1 mm away from the detection region**
Micro-interferometric back-scatter and phase detection
- L. F. G Geers et al, A liquid-independent volume flow measurement principle, MST 2010

Schematics of principle

Flow measurement data for pure liquids at different flow rates.

Ratio between measured and reference flow rate for IPA–water mixtures
Thermal mass flow meter with IR absorption

- Thermal mass flow meter
  - Temperature profile change for different flow rate
  - Temperature difference between upstream and downstream positions → obtaining flow rate

- IR absorption and temperature
  - Spectra of IR absorption change for different temperature
  - IR absorption spectra can be used for temperature measurement

How about replacing the heater and thermometer in a thermal mass flow meter using the IR absorption method?

Spectra of the absorption coefficient: 30, 35 and 40 °C (Lab on a chip, 2011)
Purpose

- in-situ measurement of micro flow rate using near infrared absorption method
  - Measurement of flow temperature inside of tube using IR absorption method
  - Water heating inside of tube using IR laser
  - Obtaining flow rate from the IR absorption method in-situ condition

- Development of clamp on type of thermal mass flow meter which can measure flow rate in-situ condition

Schematic of clamp on type thermal mass flowmeter

Schematic of conventional thermal mass flowmeter
In-situ measurement of micro flow rate using FTNIR (Fourier Transform Near Infrared Spectroscopy)

(S.H. Lee et al., In situ measurement of micro flow rate using near infrared absorption method, Optics Express, Vol. 26, 2018)
Experimental setup

- Reference flow rate measurement
  - Flow generation: syringe pump (1-100 mL/h)
  - IV-set polymer (3 mm Dia.) tube
  - Balance (Mettler-Toledo, max: 81 g, resolution: 0.005 mg)

- IR absorption measurement
  - IR laser heating: 1450 nm wavelength, 500 mW, 50 mm focal length lens
  - FTNIR absorption measurement: 360 nm – 2500 nm wavelength, Halogen lamp
  - Transverse FTNIR measuring position

(a) Experiment setup (b) magnification of measuring part
Temperature measurement using FTNIR

- Reference temperature measurement
  - Control temperature using heating circulator (10-60 °C)
  - Obtaining reference temperature: PT 100 sensor
  - Absorption spectra change for different reference temperature
  - Linear calibration curve between ref. temperature and averaged absorption signal (1500 – 1700 nm)

Absorption spectra for different temperature

Absorption intensity according to temperature
Flow rate change with laser heating

- Flow rate and FTNIR absorption spectrum
  - Change of absorption spectrum with change of flow rate
  - The temperature increases as the flow rate decreases

Absorption spectra for different flow rate at the 1 mm position
Temperature profile with laser heating

- Temperature profile for different flow rate and positions
  - Quantitative temperature obtained from calibration curve
  - Temperature profile shift to downstream with increasing flow rate
  - Above 20 mL/h, the difference in temperature profile is small
Better Standards, Better Life!

Heat transfer simulation

- Comparison between exp. and sim. results
  - Conductive and convective heat transfer
  - Good agreement of experimental and simulation results
  - More dominant convection with increasing flowrate

Governing equations

\[
\dot{q}_{\text{aser}} + \dot{q}_{\text{conv},i\rightarrow i-1} = \dot{q}_{\text{cond},i\rightarrow i-1} + \dot{q}_{\text{cond},i\rightarrow i+1} + \dot{q}_{\text{bs}}
\]

\[
\dot{q}_{\text{aser}} + \dot{m} C_p (T_i - T_{\text{in}}) = KA \frac{T_i - T_{i-1}}{\Delta x} + KA \frac{T_i - T_{i+1}}{\Delta x} + h P \Delta x (T_i - T_{\infty})
\]

\[
T_{i=1} = T_{\infty}, \quad T_{\text{en},N} = 0
\]

Comparison of temperature profiles between the experimental and simulation results

Calculated percentage of heat transfer (conduction and convection)

- 1 mL/h
- 10 mL/h
- 60 mL/h
Calibration curve between temperature difference (ΔT) and flow rate
- Two exponential curves based on the flow rate ranges (1-20 mL/h and 40-100 mL/h)
- The two flow rate ranges (1-20 mL/h and 40 mL/h) are significantly influenced by conduction and convection, respectively
- We measure the quantitative flow rate by using the calibration curve.

Flow rate according to the difference of room temperature (T<sub>room</sub>, 22.5 °C) and temperature (T) at 1 mm position with exponential curve fitting for different flow rate ranges (1-20 mL/h, 40-100 mL/h)
Quantitative flow rate measurement using NIR method

- Error of NIR absorption method according to the flow rate with measurement uncertainty
  - The reference flow rate was obtained from the gravimetric flow standard system in KRISS
  - The error according to the flow rate is less than 1.2 % (0.05 mL/h) and the uncertainty is less than 1 % (0.39 mL/h) at the 1-100 mL/h flow rate

\[ \text{Error} = \left( \frac{q_{NR} - q_{REF}}{q_{REF}} \right) \times 100 \]

Error of NIR absorption method according to the flow rate with measurement uncertainty
Conclusions (FTNIR)

- In-situ measurement of micro flow rates was conducted using the NIR absorption method.
- We used the IR absorption method to measure the temperature under in-situ conditions instead of using a contact temperature sensor.
- The temperature profiles were obtained by the NIR absorption method via laser heating for different flow rates.
- The shape of the temperature profile changes resulting from conduction and convection heat transfer were presented based on the range of flow rates.
- A simulation was conducted considering conductive and convective heat transfer for analysis of temperature profiles.
- The calibration curves between the flow rate and the difference in room temperature (Troom, 22.5 °C) and the temperature (T) obtained from the NIR absorption method exhibit two exponential curves based on the flow rate ranges (1–20 mL/h and 40–100 mL/h).
- The error and the uncertainty of the NIR absorption method were approximately 1.2 % and 1% in the 1–100 mL/min flow rate range, respectively.
- Thus, we confirmed for the first time that the NIR absorption method quantitatively measures the flow rate in the in-situ condition.
In-situ measurement of micro flow rate using laser diode

Laser diode based flow measurement

- In-situ measurement of micro flow rate using IR diode
  - Portable instrument for micro flow rate measurement
  - IR diode ($10) is much cheaper than FTNIR ($40,000)
  - Compact size (less than 5 mm)
  - Accurate measurement of flow rate using temperature at up and downstream

Measurement setup using FTNIR ($40,000)  Measurement setup using IR diode ($10)
Experimental setup

- Reference flow rate measurement
- Flow generation: Coriolis MFC
- IV-set polymer (3 mm Dia.) tube
- Balance (Mettler-Toledo, max: 81 g, resolution: 0.005 mg)
- Laser diode: 1550–1650 nm, 2 mW, d=5 mm
- Lock-in Amp.: to enhance S/N
- Heating laser: 1450 nm, 500 mW
Temperature measurement using the NIR absorption method

- Temperature measurement using laser diode
  - Light from 1550 to 1650 nm with a laser diode
  - Calibration using temperature controlled bath
  - Uncertainty of temperature measurement: 0.001 %

Spectrum of laser diode

Absorption spectra for different temperature

Transmission intensity according to temperature for laser diode based temperature measurement
Different measurement positions
- A heated spot created with a heating laser
- Changing the position of heating spot
- Increase of flow reduced the temperature of the upstream
- Temperature change not large with flow rate in downstream
Different measurement position

- Temperature difference decreases exponentially
- When the measurement position is moved from -1 mm (upstream) and 9 mm (downstream) to -3 mm (upstream) and 7 mm (downstream), the curve shifts downward with almost the same slope.
- Where the upstream measurement position is close to the laser heating position, we can measure a wider range of flow rates.

Temperature difference measured upstream and downstream
Different heating laser energies at the fixed measurement position (-1,9)
- When the heating laser energy increases, the maximum temperature increases
- When the flow rate increases, the temperature upstream of the heated spot decreases, and there is little change in the temperature downstream.
- As the heating laser energy decreases, the temperature curve according to the flow rate at each position is shifted downward.

Comparison of temperature profiles for different heating laser energies

Temperatures at two measuring positions according to the flow rates for different heating laser energies
Different heating laser energies

- The heating laser energy decreases, the slope of the temperature curve decreases.
- The higher the heating laser energy, the better the accuracy of the flow measurement.
- The lowest flow rate measured in this study is 0.2 ml/h, but if the laser energy is controlled, lower flow rates can be measured in the future.
Uncertainty analysis

- Mathematical model is based on the comparison calibration \( E = \frac{m_{DUT} - m_{REF}}{m_{REF}} \)

- The relative uncertainty of \( E \)

\[
u(E) = \sqrt{u(E_A)^2 + (c_{rW} u_r(\delta W))^2 + (c_{rt} u_r(\delta t))^2 + (c_{re} u_r(\delta e))^2 + (c_{rD} u_r(\delta \rho_{DUT}))^2 + (c_{rD} u_r(\delta q_{DUT}))^2}
\]

<table>
<thead>
<tr>
<th>Quantity (Xi)</th>
<th>Uncertainty factor</th>
<th>Value of uncertainty</th>
<th>Value of component (k = 2)</th>
<th>Degree of freedom</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass</td>
<td>( u(\delta W) )</td>
<td>5.17E-04</td>
<td>1.96E-04</td>
<td>289</td>
</tr>
<tr>
<td>Time</td>
<td>( u(\delta t) )</td>
<td>9.80E-04</td>
<td>3.27E-07</td>
<td>210</td>
</tr>
<tr>
<td>Density</td>
<td>( u(\delta \rho) )</td>
<td>3.75E-03</td>
<td>9.83E-07</td>
<td>107</td>
</tr>
<tr>
<td>Indicator</td>
<td>( u(\delta q_{DUT}) )</td>
<td>3.46E-04</td>
<td>1.00E+00</td>
<td>354</td>
</tr>
<tr>
<td>Thermometers</td>
<td>( u(\delta T) )</td>
<td>3.33E-05</td>
<td>1.00E+00</td>
<td>1140</td>
</tr>
<tr>
<td>Temperature curve fitting</td>
<td>( u(\delta T_{curve}) )</td>
<td>1.30E-03</td>
<td>1.00E+00</td>
<td>183</td>
</tr>
<tr>
<td>Buoyancy correction</td>
<td>( u(\delta \epsilon) )</td>
<td>2.89E-06</td>
<td>-9.80E-04</td>
<td>3873</td>
</tr>
<tr>
<td>Relative error</td>
<td>( u(E_A) )</td>
<td>2.90E+00</td>
<td>1.00E+00</td>
<td>4</td>
</tr>
</tbody>
</table>

Main sources of uncertainty for the laser diode flowmeter calibration for 6 ml/h flow rate

\( u_{c}(E)=1.14 \% \) \( U(E)=2.28\% \)
Deviation analysis

- Deviations in the LD flowmeter readings according to the flow rate with measurement uncertainty
  - The maximum measurement uncertainty was 6.8% at a 1 ml/h flow rate, and the minimum measurement uncertainty was 1.78% at 8 ml/h.
  - The performance of the LD flowmeter developed in this study was confirmed to be close to that of a commercial thermal mass flowmeter (10%, full scale).

![Deviation vs Flow Rate Chart]

Deviation in the LD flowmeter readings according to the flow rate with measurement uncertainty
Conclusions

- A laser diode-based flowmeter based on this infrared absorption method that can measure in-situ micro flow rates from 0.2 to 20 ml/min using a simple diode laser and a photo detector was developed.
- We measured the temperature profiles of the flow rate by changing the temperature measurement position and the heating laser energy upstream and downstream of the heated spot, and compared the obtained values with the simulation results.
- The flow measurement range was adjusted according to the temperature measurement position. Increasing the energy of the heating laser also improved the measurement accuracy in the lower flow range.
- The developed flowmeter was calibrated by the gravimetric method, and the maximum measurement uncertainty was 6.8%, which is similar to that the conventional commercial contact thermal mass flowmeter. The lowest flow measurement uncertainty was 1.78% at a flow rate of 8 ml/h.
- Thus, it was confirmed that the flow rate can be measured through the temperature difference gauged using a simple diode laser set.
표준이 올라가면 생활이 즐거워 집니다!

Leading Group in Fluid Flow and Acoustics Metrology

Thank you for your attention

18th FLOMEKO, 26-28 June 2019 Lisbon-Portugal
Laser heating of water

- **NIR absorption heating**
  - 1450 nm: absorption of water (O-H band), transmission of polymer tube
  - Focusing laser: tens of µm hot spot generation
  - Spectral change in the range of 1500-1700 nm according to laser heating
  - Temperature increase more and more near hot spot

Absorption spectra at upstream positions

Absorption spectra at downstream positions
Quantitative flow rate measurement using NIR method

- Temperature difference ($\Delta T$) between measuring position and room temperature for varied flow rate
  - Varied $\Delta T$ according to the measuring position in up and downstream
  - Linearly increase of $\Delta T$ with increasing flow rate up to 20 mL/h @ -1 mm: conduction is dominant effect
  - Gradually decreasing the slope of $\Delta T$ from 20 mL/h to 100 mL/h @ -1 mm: influence of convection increases

Difference between room temperature ($T_{room}$, 22.5 °C) and temperature ($T$) according to the flow rate for different positions (a) upstream and (b) downstream with IR laser heating at the 0 mm position.