Technology and Features of a Thermal Flowmeter for Liquid Microflows

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In recent years, due to advances in manufacturing processes and more stringent quality requirements, highly accurate measurement of microflows has become necessary. We have now developed a thermal flowmeter for liquid microflows, model F7M, capable of measuring with an accuracy of ±5 % of reading for micro flow rates of 30 mL/min and less. Moreover, this device has low pressure drop, high reliability, and a compact size. In order to create a model with these features, we developed several technologies: a flow sensor module using fused quartz glass tubes and a MEMS (microelectromechanical systems) sensor; a small straight pipe flow-path structure, and equipment capable of measuring actual micro flow rates with a high level of accuracy. We discuss the features of the F7M and the technological developments that made it possible.

1. Introduction

Liquid flow measurement has been used in various applications in various industries.[1] In recent years, due to advances in manufacturing processes and more stringent quality requirements, applications of flow measurement have expanded and the number of flowmeters installed has been steadily increasing.

The measurement of microflows is one of the areas that has particularly attracted attention in the field of flow rate measurement.[2] There are many applications, such as injection of electrolyte solution in the manufacturing process of lithium ion batteries, coating of photoresist solution in semiconductor manufacturing, mixing and dilution of reagents in biochemical examinations, spraying of special solutions, and mixing of cleaning solutions. In this paper, a flow rate of 30 mL/min or less is defined as a micro flow rate. Several methods have been proposed for measuring microflows: volumetric, differential pressure, Coriolis, ultrasonic, etc., but they are all faced with the following problems.

1. High measurement accuracy cannot be achieved for microflows, or it is expensive.
2. There is a large pressure drop when measuring.
3. Moving parts raise concerns about reliability.
4. A flowmeter with a compact size is difficult to make.

In order to solve these problems, Azbil Corporation has developed a thermal flowmeter for liquid microflows, model F7M.

This paper describes the features and technical key points of the F7M, and reports the results of performance checks.

2. Overview of the Flowmeter

2.1 Appearance and Specifications

Model F7M is shown in figure 1 and its specifications are listed in table 1.

Figure 1. Model F7M

Table 1. Specifications

Model number	F7M9010	F7M9030
Measurable flow rate range (for water)	0.1 to 10 mL/min	0.3 to 30 mL/min
Measurement accuracy (for water)	±5 % rdg. (at 20 % or more of full scale) ±1 % FS (at less than 20 % of full scale)	
Repeatability (for water)	±1 % rdg. (at 20 % or more of full scale) ±0.2 % FS (at less than 20 % of full scale)	
Size	22 (W) × 60 (H) × 122 (D) mm	
Weight	Approx. 85 g	
Fluid pressure range	0 to 500 kPa	
Flow-path pressure resistance	700 kPa	
Protection rating	IP65	

2.2 Features

Model F7M has the following features.

(1) Highly accurate measurement of microflows

The F7M has a high accuracy of ±5 % of the reading (rdg.) for flow rates of 20 % full scale (FS) or more (for the 30 mL/min range, from 6 to 30 mL/min; for the 10 mL/min range, from 2 to 10 mL/min), and a high repeatability of ±1 % rdg. This makes it possible to control microflows that were conventionally difficult to measure, helping to improve processes and yields.

(2) Low pressure drop

Inside the product, a fused quartz glass tube with a sensor module (sensor chip) attached to its outer surface and a fluorocarbon resin coupling used to input/output fluid are coaxially arranged, so there are no obstacles in the flow path. Therefore, even at the maximum flow rate, the pressure drop is less than 1 kPa, it is a negligible level for practical purposes. This makes it possible to reduce the supply water pressure of the fluid, which helps to make the equipment simple.

(3) High reliability

There are no moving parts, so the flowmeter is durable. In addition, since the only liquid-contacting parts are made of quartz glass or fluorocarbon resin, the product is highly corrosion resistant to various fluids. This makes it possible to use the flowmeter reliably in a wide range of applications.

(4) Compact size

The flowmeter is a compact 22 × 60 × 122 mm (W × H × D), including the printed circuit board. This makes flexible installation of the product possible, contributing to downsizing of equipment and facilities.
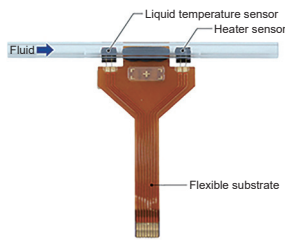
2.3 Measurement Method

Figure 2. Sensor module

The F7M is classified as a thermal flowmeter, and measures flow using power consumption. As shown in figure 2, a temperature sensor for the process fluid is located at the upstream side of the glass tube constituting the flow path, and a heater sensor for simultaneous measurement of the heater's temperature and power consumption is located at the downstream side. The temperature of the heater sensor is controlled so as to be higher by a fixed amount than the fluid temperature measured by the liquid temperature sensor. The temperature distribution when the fluid is flowing in the glass tube is shown in figure 3. The larger the flow rate, the larger the amount of heat that is taken away from the heater by the fluid. Since the heater is controlled so as

to keep a constant temperature difference compared to the temperature of the liquid, the amount of power consumed by the heater correlates with the flow rate. Therefore the flow rate can be obtained by measuring the power consumption of the heater.
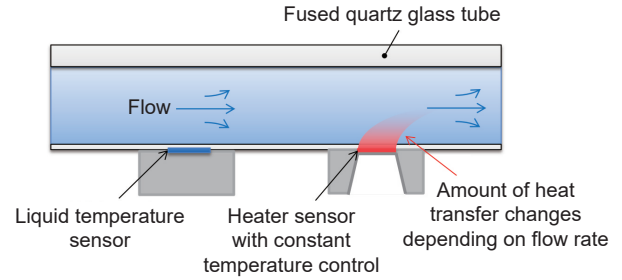

Figure 3. Model F7M's flow rate measurement method

It is in the nature of a thermal flowmeter that its output is affected by the type of fluid, due mainly to thermal conductivity. For that reason the F7M has an internal correction function. By setting a correction factor that depends on the thermal conductivity of the fluid, it is possible to measure the flow rate regardless of the type of fluid.

3. Technical Challenges

This section describes the problems we faced in realizing the features described in section 2, and the technologies used to solve the problems.

3.1 A Highly Accurate Thermal Flowmeter Sensor Module[3]

The thermal flow sensor is a MEMS sensor on an approximately 3 mm square silicon chip containing a temperature-measuring resistor and a heater. The sensor has the advantage of allowing easy miniaturization of the flowmeter, and it has excellent responsiveness due to its small thermal capacity.

The relationship between the flow rate and the power consumption of the heater is given by equation 1.

$$\Delta P = \left(\frac{1}{A/\sqrt{q}+B} \right) (T_1 - T_\infty)$$

$$A \propto \eta^{\frac{1}{6}} \times \lambda^{\frac{2}{3}} \times P^{\frac{1}{2}} \times C_P^{\frac{1}{3}} \qquad B \propto \left(\frac{l_2}{\lambda_2} + \frac{l_1}{\lambda_1} \right) \cdots \text{Equation 1.}$$

Where: ΔP is power consumption, q is flow rate, η is viscosity of the fluid, λ is thermal conductivity of the fluid, λ_1 is thermal conductivity of the glass, λ_2 is thermal conductivity of the adhesive, P is density of the fluid, C_P is specific heat of the fluid, l_1 is glass tube thickness, l_2 is adhesive thickness, T_1 is heater temperature, and T_∞ is fluid temperature

The results of actually measuring the relationship between the flow rate q and the power consumption ΔP with this sensor are shown in figure 4. In the manufacturing process, the relationship between the flow rate q and the power consumption ΔP is measured for each sensor, and a correlation equation (curve) is stored inside the sensor. The flow rate is calculated based on the stored correlation equation.
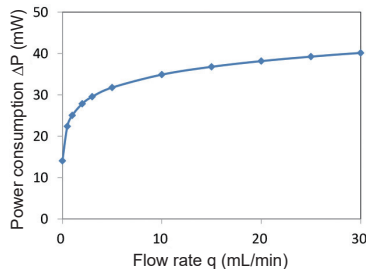
Figure 4. Relationship of flow rate and power consumption

In order to measure a liquid flow with high accuracy, thermal coupling of the sensor chip and the fluid is important, so optimal design and manufacture is necessary for the items corresponding to the parameters in equation 1: λ_1 (thermal conductivity of the glass), l_1 (glass tube thickness), λ_2 (adhesive thermal conductivity), and l_2 (adhesive thickness). In addition, for bonding of the sensor chip to the glass tube, it was necessary to devise a means so that the sensor would not be adversely affected by stress caused by thermal deformation of the glass tube and adhesive or by mechanical stress.

Accordingly, as shown in figure 5, we developed special manufacturing processes for the sensor module. A portion of the circumference of the quartz glass tube is cut so that it is flat, and a special adhesive that is not easily affected by conditions such as temperature and humidity is applied at a fixed thickness.

Because of this structure and the manufacturing processes, no stress is applied to the sensor chip, and heat can be conducted between the fluid and the sensor efficiently and constantly under the same conditions. In other words, it is possible to manufacture sensor modules that can measure changes in power consumption of the heater due to flow rate changes, while also being highly resistant to disturbances and having high repeatability.
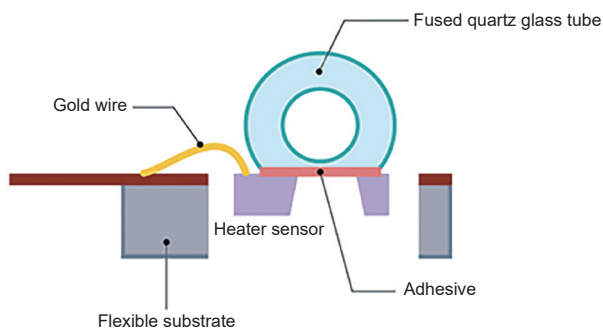

Figure 5. Cross section of the heater sensor

3.2 Straight-Pipe Structure for Low Pressure Drop and High Reliability

It is necessary to connect the glass tube and the coupling coaxially in order to achieve the low pressure drop and highly reliable flow path which take advantage of the strong points of the sensor module. The coupling must fulfill two functions: connection with the glass tube and connection with the tube on the user's equipment. Although fluorocarbon resin couplings with these functions are commercially available, there are two problems with their application to this product.

Problem 1. An internal space is required to clamp and fasten the glass tube, necessitating enlargement of the housing.

Problem 2. The glass tube has thin walls to improve thermal conductivity, so it is easily broken. When tightening and fastening the coupling, a load can easily be applied to the glass tube due to shaking of the hands, so it was necessary to take countermeasures to prevent the glass tube from being damaged during assembly.

Consequently, as shown in figure 6, we devised a special structure that solves the two aforementioned problems.
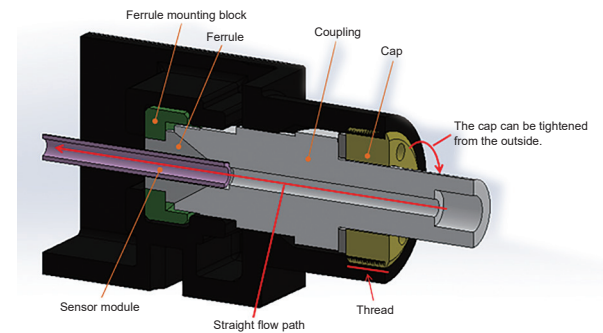

Figure 6. Cross section of the coupling (enlarged on one side)

The coupling assembly is composed of the housing, sensor module, ferrule, ferrule mounting block, coupling, and cap. In product assembly, first the sensor module, ferrule, and ferrule mounting block are assembled inside the housing, and then the coupling is inserted from the outside, and the whole flow path is fastened by screwing the cap into the housing for each coupling. Since the coupling has an anti-rotation structure combined with the housing, when the cap is screwed on, all the rotational force is transferred to the tapered connection surface between the ferrule and the coupling, so the cap alone rotates. Due to the force of rotation, the ferrule is deformed toward the center to hold the entire circumference of the glass tube, forming a closed flow path.

With this structure, the cap can be screwed from the outside of the case, allowing the case to be miniaturized.

Furthermore, since there is only a simple positioning operation of the glass tube before the cap is attached, and the force of rotation is converted only to axial force, no unnecessary load is applied to the glass tube. Our assembly process is therefore highly productive, with no concern about breakage of the glass tube.

There is another advantage of fastening the cap from the outside of the housing, which is that decreasing pressure on the seal due to creep (progressive deformation caused by continuous force) of the fluorocarbon resin can be easily countered by retorquing the cap. In the manufacturing process, as a countermeasure against creep, retorquing is done one day after assembly. Thus, since effects in the initial stage when the cold flow of the resin is greatest can be reduced, reliability is increased.

In order to verify the long-term reliability of the straight-pipe flow path structure, we conducted a pressure-resistance test at 1 MPa after applying a high-temperature

load equivalent to 10 years of service life. The result showed that there was no leakage and that this structure was highly reliable.

3.3 Experimental Equipment for Measuring Micro-flows with High Accuracy

In order to produce a high-precision flowmeter, it is necessary to have experimental equipment capable of measuring the flow rate with higher accuracy. For that purpose we developed special flow measurement equipment.

A schematic diagram of the equipment is shown in figure 7. For accurate measurement of the flow rate, pure water stored in a tank is extracted with compressed air, run through the device under test (DUT), and stored in an electronic balance that is a standard measuring device. The flow rate calculated from the change of mass in the water stored in the balance is compared with the flow rate measured by the DUT to evaluate the accuracy of the DUT. Both the ambient temperature and the fluid temperature can be adjusted using a thermostatic chamber and a chiller, so measurements can be carried out under various conditions.
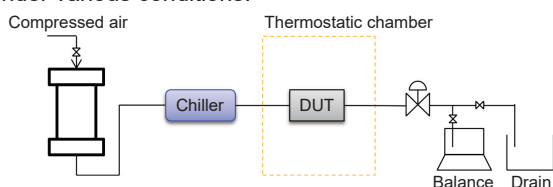
Figure 7. Schematic of micro flow rate measurement equipment

In measuring the mass of water stored in the balance, there are two problems arising from the fact that it is a measurement of a microflow.

Problem 1. Error due to evaporation of water

During measurement, a small amount of the liquid stored in the balance will evaporate even in a normal temperature environment. The amount of evaporation is correlated with the open area of the balance's tank. Evaporation of 0.1 g / 30 min was observed in our experimental environment. This is an error level that cannot be ignored in microflow measurement.

Problem 2. Error caused by droplets

Generally, the outlet of the liquid to the balance is placed in the air so as not to contact the surface of the liquid in the balance's tank, in order to avoid effects on the balance.

When measuring a microflow, however, liquid is discharged in the form of droplets due to surface tension. Measured drops weighed about 0.5 mg each. Whether or not one drop is discharged into the balance's tank can be an error factor in measuring a microflow.

We have taken the following countermeasures for these problems.

Countermeasure 1. Oil layer to prevent evaporation and submerged discharge outlet

As shown in figure 8, we designed the tank of the balance such that the end of the discharge outlet is submerged in water that is stored in the tank before-hand. Floating above the water is a layer of oil, which has a lower specific gravity than water and does not evaporate.

Since the water is underneath the oil layer, evaporation is completely prevented, and discharge by droplets does not occur. Consequently, problems 1 and 2 are solved and measurement accuracy is improved.
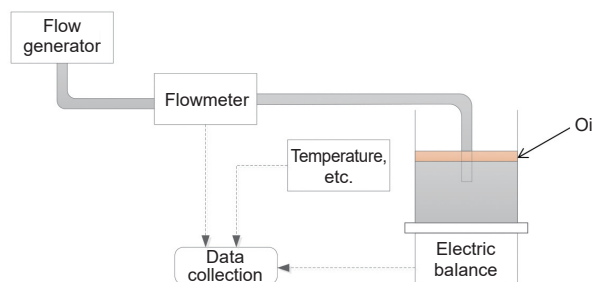
Figure 8. Submerged discharge for accurate flow measurement

Countermeasure 2. Continuous measurement of weight and time simultaneously

In order to avoid an error due to transient responses when the flow rate changes, it is necessary to carry out an accuracy test under the condition of a constant flow volume. But in the submerged structure devised in countermeasure 1, the flow path cannot be switched by a commutator or the like. Therefore, a method was devised to measure the weight and the time simultaneously, and to continuously repeat the data acquisition. As shown in figure 9, the reference flow rate is given by the slope of the straight line obtained from the weight and the corresponding time of the multiple measurement points. Using this method, we can obtain the actual flow rate, unaffected by the transient responses of the flowmeter.

This equipment can measure with an expanded uncertainty of 0.15 % rdg. (estimate by Azbil Corporation) and has sufficient performance to evaluate product accuracy of ±5 % rdg.
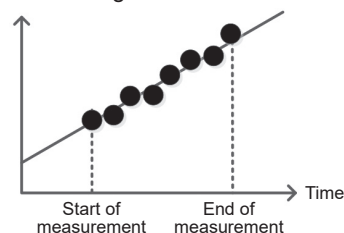
Figure 9. Flow rate calculation by multiple measurements

4. Performance Check Results

4.1 Flow Rate Measurement Results

Figure 8 shows the water flow rate measurement results for two F7M models with respective capacities of 10 mL/min and 30 mL/min. Five of each model were used to take measurements.
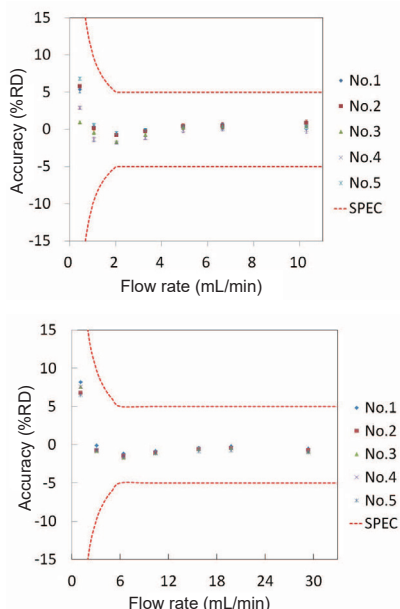

Figure 10. Flow rate measurement results for 10 mL/min model (above) and 30 mL/min model (below)

The measurements of both models were correct within ±2 % rdg., surpassing the product specification of ±5 % rdg. (at a flow rate of 20 % FS or more) and verifying the development of a high-accuracy flowmeter for liquid microflows.

4.2 Flow Rate Measurements of Fluids Other Than Water

In actual users' applications, it is often necessary to measure the flow rate of liquids other than water. Due to the operating principle, thermal flowmeters are affected by the physical properties of the fluid, and especially by its thermal conductivity, as shown in equation 1.

Model F7M measures the flow rate of fluids other than water by means of a correction factor that can be set for each type of liquid.

Flow rate measurement was carried out for 100 % isopropyl alcohol (IPA) and a 50 % mixture of IPA and water. The relationship between the actual flow rate and the flow rate measured by the F7M is shown in figure 11 for two cases: when the correction factor was not set, and when it was set.

As figure 11 shows, when the correction factor for the type of liquid is set, the flow rate can be measured regardless of the type of liquid, verifying that model F7M can be used for various fluids other than water.
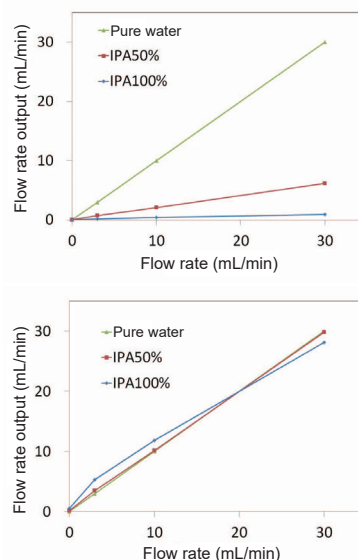

Figure 11. Flow measurements for water and IPA before applying the correction factor (above) and after applying it (below)

5. Conclusions

We have developed a thermal flowmeter for liquid microflows, model F7M, which measures micro flow rates with high accuracy. Its features include low pressure drop, high reliability, and small size. Model F7M has been available since 2017 and has been well received by various users.

In the future, we plan to augment the product lineup and the product's functions. We would like to expand the flow rate range and add an automatic correction factor setting function based on the liquid type, etc. We believe that the value of the product to its users can be further enhanced.

References
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