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Application of the Self-Made Flexible Three-in-One Microsen-Sor to the Laboratory Oven for Immediate Micro-Monitoring of the Roll-to-Roll Process of Polarizing Films

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Abstract: The main purpose of this study is to carry out the immediate micro-monitoring of the roll-to-roll (R2R) process of polarizing films. Therefore, a self-made flexible three-in-one (temperature, humidity, and flow) microsensor is developed. The temperature and flow sensing area are 585 μm × 450 μm, the humidity sensing area is 1065 μm × 1035 μm, and the minimum line width is 15 μm. The micro-electro-mechanical systems (MEMS) technology was applied to integrate temperature, humidity, and flow sensors on a 50 μm thick polyimide substrate. A 100 Å thick chromium (Chrome, Cr) section form the adhesion layer. A 1000 Å thick gold section forms the sensing layer. A self-made flexible three-in-one microsensor set up in a laboratory oven for immediate micro-monitoring of the R2R process of the polarizing film. Since it is not advisable to set up signal lines in a clean room, the analog signals of the sensor should be transmitted via wireless means. Thus, a monitoring module should be connected to the back end of the self-made flexible three-in-one microsensor to receive the analog signals of the sensor, convert them into digital signals, send them out in the form of wireless signals, and store the data on the server-side. Through these measures, both the R2R process and yield can be improved. Therefore, the focus of this study is the environmental monitoring of drying process ovens. However, commercially available all-in-one sensors cannot handle the temperature of high-temperature factory ovens, and commercial flow sensors are rarely used in high-temperature applications. Some are also expensive and cannot be widely distributed, so this study intends to develop an integrated sensor to measure the internal environment of the drying oven.

Keywords: R2R process; self-made flexible three-in-one microsensor; immediate micro-monitoring through a laboratory oven

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1. Introduction

In order to realize highly sensitive and reliable temperature sensors with diverse forms, various types of sensors have been developed in conjunction with investigations related to emerging materials and fabrication processes. Four types of sensors with different sensing mechanisms have been widely studied: resistive temperature sensors, capacitive temperature sensors, transistor-based, and PN junction-based temperature sensors. Among these, resistive-type temperature sensors are one of the most promising candidates owing to the following reasons: (i) their simple geometry is compatible with various materials, substrates, and fabrication processes, such as solution processed materials, flexible substrates, and environmentally friendly fabrication processes, respectively; and (ii) their simple and straightforward mechanism allows facile and direct measurement of temperature with high sensitivity [1]. The capacitive sensors have many inherent advantages such as high sensitivity, excellent stability, fast response, high resolution, good frequency response, less loading effect because of high input impedance, no self-heating, and less power requirement [2]. The Seebeck effect is a basic thermoelectric effect. It refers to a

self-created potential difference around a closed loop formed by two conductive materials when there is a temperature difference between their contact points. The thermoelectric material with the thermoelectric effect is a new functional material that interconverts the thermal and electric effects. Due to the excellent energy conversion efficiency based on the high Seebeck coefficient and high electrical conductivity, many common inorganic materials have been widely studied [3]. Furthermore, increasing attention has been paid to organic materials due to their low thermal conductivity, low density, and easy processing. Additionally, the excellent mechanical flexibility of organic materials also creates huge potential for manufacturing flexible sensors [4,5].

The Internet of Things (IoT) and trillion sensor universes have developed remarkably in recent years, bringing about the advanced requirements for fast response, low power consumption and low-cost sensor devices. As the crucial devices of measuring environmental parameters, humidity sensors have been widely used in a multitude of fields, including industrial process control, medical diagnosis, agricultural production, aerospace, automotive and home appliances [6]. Humidity sensors have been conventionally fabricated using micro-electro-mechanical systems (MEMS)-based technology. However, this manufacturing process involves the use of complex fabrication steps along with the use of relatively expensive facilities [7]. Humidity sensor possesses many unique features such as low cost, high accuracy and sensitivity, simplicity, and fast response and recovery times [8]. Moreover, the humidity sensors are often fabricated on rigid substrates, thereby resulting in sensors that are not conformal enough for flexible platforms. The drawbacks associated with the conventional humidity sensors can be overcome by using additive print manufacturing processes such as screen and gravure printing which enable the fabrication of electronic devices on flexible platforms with roll-to-roll processing capabilities [9]. Flexible humidity sensors made from humidity-sensitive materials are those of which the interdigitated electrode parameters and flexible substrates play a special boosting role in developing wearable devices. However, there are certain limitations, such as the difficulty in sensing the humidity generated by different parts of the human body and the impermeability of substrates [10]. The electrode form of the capacitive micro-humidity sensor used in this study was an interdigitated electrode structure. In this type, there is a humidity sensitive thin film above the electrode [11].

Flow sensing finds applications in many industries and can be achieved using a variety of sensing principles including Bernoulli's principle (e.g., pitot tube), thermal transport (e.g., thermal anemometry), ultrasonic waves, electromagnetic induction, and drag force measurement [12]. Most of the existing flow sensors are expensive and limited in their capabilities for sensing bidirectional flow. Low-cost and accurate flow sensors with bidirectional sensing capability have numerous applications in the residential and irrigation sectors [13]. Among various flow sensors, the thermal flow sensor is the most common due to its simple structure and simple electric readout. The hot-wire flow sensor uses one simple temperature sensor with a heater, while the thermal flow sensor uses two temperature sensors with a heater between them, which uses the heat source, the heater structure, or the changes in the location and amount of temperature sensors via the conversion method of the calorimetric principle [14,15]. Thermal MEMS (micro-electro-mechanical systems) flow sensors are composed of a heater and temperature sensors fabricated on a thin film (membrane). Sensor detection changes in the heater temperature distribution caused by the fluid are able to determine flow. The advantages of the thermal type are small size, high sensitivity, and responsivity to low flow rates [16].

An asthma patient monitoring system can be categorized as an IoT-based device. It can use an ESP8266 microcontroller and different sensors to monitor heart rate, room temperature, humidity, air quality, nostril temperature, and oxygen saturation (SpO_2). All data are sent to Firebase by Wi-Fi, and then are sent to patients and doctors by mobile applications and websites, respectively. The software of the system is divided into two parts. One of them is a mobile application that was designed by the Android Studio. The second one is the website [17].

The development board applied in this study was Arduino Uno Rev 3, which is commonly seen. Bluetooth can only receive signals. It must be connected to Raspberry Pi for conversion. Arduino needs to write a program to receive signals and convert them at the same time. With the employment of Arduino, in combination with the program for the circuit board wiring simulation provided by Fritzing, the readout program and circuit diagram of the sensor were developed and designed, thereby finally completing the monitoring module.

2. Research Method

2.1. Sensing Principle of Micro Temperature Sensors

Temperature is a vital parameter that is measured in all process industries. Specifically, resistance temperature detectors (RTDs) are used in process industries where accuracy is an important criterion. Thermocouples do not provide such accuracy due to nonlinearity and cold junction compensation. Thermocouples also require signal conditioning at the sensor end or lead-wires of the same material. On the other hand, RTDs are known for their linearity, accuracy, and stability. Numerous analog techniques are currently available to interface an RTD with the control room. One major problem when using an RTD is the resistance of lead-wires connecting the sensor to the control room. Analog compensation methods, namely four- or three-wire methods, are used to nullify both lead-wire resistance and the change in resistance of lead-wires. Lead-wire compensation is inevitable for accurate measurement. These analog techniques are effective in compensating lead-wire resistance, but result in additional costs for the wires [18].

2.2. Sensing Principle of Micro Humidity Sensors

Regarding capacitive humidity sensors, by coating the interdigitated electrode structure of such a sensor with polyimide (PI), a humidity-sensitive film is created. Additionally, the humidity can be calculated based on the measurement of the capacitance change of the film in the water. The polymer materials of a micro humidity sensor must feature the low permittivity (approximately three to four) and the high electric resistance. The permittivity of water is 80, and that of the material rises with the improvement of the hygroscopicity and with the increase of the ambient humidity [19].

2.3. Sensing Principle of Microflow Sensors

The hot-wire microflow sensor is simple in structure and easy to design in the process, drive, and electric circuit for signal output. It has become mainstream in microflow sensor research. The main measuring structure is an electric resistance heater, and the constant pressure input generates a heat source and then forms a temperature field. If the external heat of the heater is constant, the electric resistance value will decrease as the fluid flow increases. If the heat of the supply hot wire is controlled, along with the temperature difference between the hot wire and the flow constant, the heating power will convert the flow into the electric signal via the constant temperature circuit for output as the fluid flow increases. In short, the hot-wire flow sensor is designed based on the positive correlation between the dissipation rate of the thermal energy of the hot wire and the fluid flow [20].

2.4. Process Development of Flexible Three-in-One Microsensors

In the manufacture of polarizing film, since the polarizing film roll is quite sensitive to temperature and humidity, the environmental temperature and humidity in the manufacturing process are quite demanding. This study uses MEMS technology to integrate three sensing structures of temperature, humidity, and flow. In order to make the integrated microsensor bendable, not damaged for a long time, and measure for a long time in any environment, this research selects polyimide film material as the substrate of the integrated microsensor. In order to avoid damage to the substrate, surface micromachining in the MEMS process is used, which is mainly formed by stacking multiple layers of films by means of evaporation, sputtering, or chemical deposition. In addition, any

micromechanical structure is made by thin film deposition, so this method is better than bulk micromachining technology in terms of processing accuracy or resolution. Therefore, in this study, the surface micromachining technology in the MEMS process technology was selected to integrate the three sensing structures into the polyimide film. The Chemical Abstracts Service (CAS) number of methanol is 67-56-1. The CAS number of isopropanol is 67-63-0. The overall fabrication process is shown in Figure 1. Figure 2 is the size design diagram of the integrated micro-sensor designed by this research, which also integrates the temperature, humidity and flow sensing structure. The temperature and flow sensing area are 585 μm × 450 μm, the humidity sensing area is 1065 μm × 1035 μm, and the minimum line width is 15 μm. The temperature and flow microsensors are all wire-wound structures, and the wire-wound structure process is the key to the process yield in this study. In order to improve the process quality and yield, the line width of the temperature and flow microsensor is 15 μm, the wire winding spacing is increased from the original 10 μm to 15 μm, and the overall process yield can reach more than 90%. The MEMS technology was applied to integrate temperature, humidity, and flow sensors on a 50 μm thick polyimide substrate. A 100 Å thick chromium (Chrome, Cr) section forms the adhesion layer. A 1000 Å thick gold section forms the sensing layer. The lithography process which is shown in Figure 3 in this study is divided into three layers, namely gold/chromium wet etching, protective layer definition, and moisture-sensitive film (dielectric layer) definition. The self-made flexible three-in-one microsensor has features including thinness, a small structural area, a high sensitivity, an immediate measurement, and can be placed anywhere. The sensor can monitor three physical quantities at the same time, and can integrate the three physical quantities together.

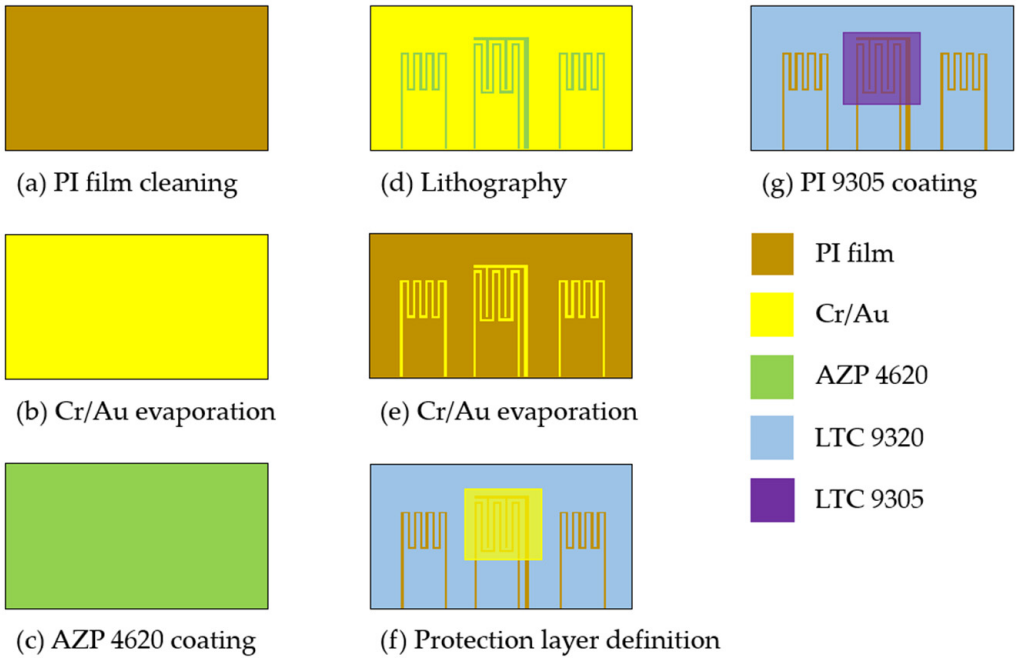


Figure 1. Fabrication process diagram of the flexible three-in-one microsensor: (**a**) is soaking PI film in Acetone, Methanol and DI water to clean, (**b**) is depositing Cr/Au with Electron beam evaporation, (**c**) is coating AZP 4620 with spin coater, (**d**) is exposure to define pattern, (**e**) is depositing Cr/Au on sensor, (**f**) is defining protection layer, (**g**) is coating PI 9305 with spin coater.

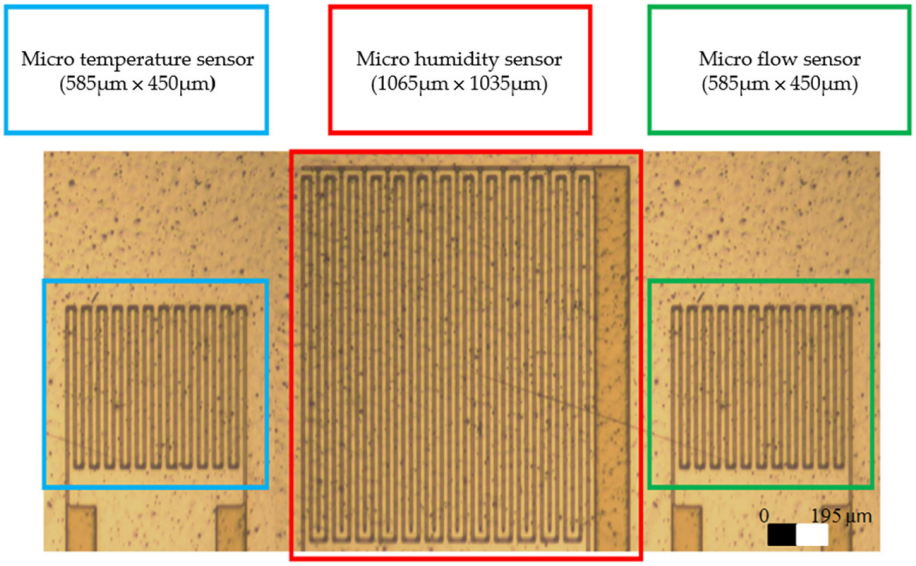


Figure 2. Image of the flexible three-in-one microsensor under the optical microscope.

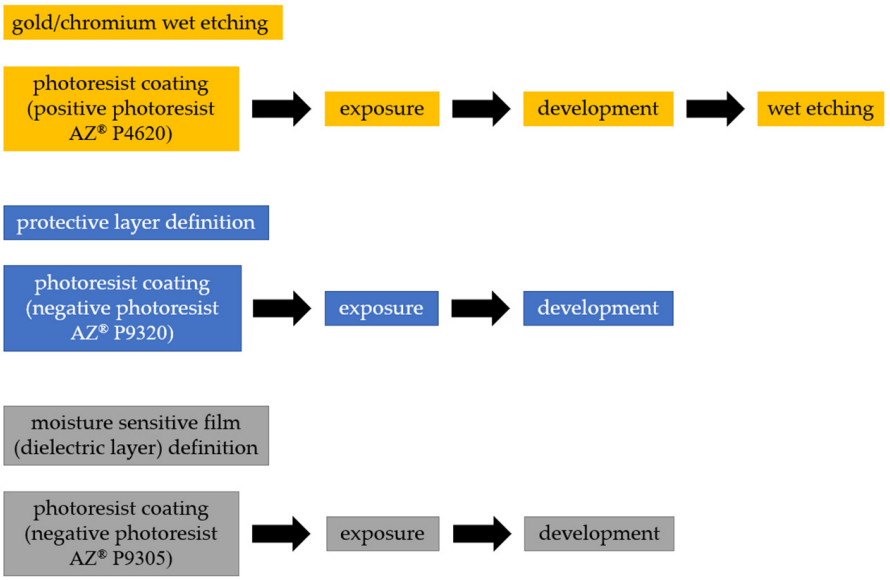


Figure 3. Lithography process.

3. Monitoring Module and Calibration of the Self-Made Flexible Three-in-One Microsensor

3.1. Selection of the Development Board of the Monitoring Module

The development board applied in this study was Arduino Uno Rev 3, which is commonly seen. Bluetooth can only receive signals. It must be connected to Raspberry Pi for conversion. Arduino needs to write a program to receive signals and convert at the same time. With the employment of Arduino, in combination with the program for the circuit board wiring simulation provided by Fritzing, the readout program and circuit diagram of the sensor were developed and designed, thereby completing the monitoring module finally.

3.2. Calibration of the Micro Temperature Sensor

To make the environment for simulating calibration closer to the interior environment of the laboratory oven, a programmable temperature and humidity testing chamber (Hung Ta HT-8045A Environmental Chamber) was applied in this study as a reference for the calibration environment. However, as the cavity body of the testing machine was large,

it was necessary to wait for another ten minutes after the temperature and humidity stabilized to ensure that the temperature and humidity could be spread evenly throughout the whole cavity body and were close to the temperature and humidity sensors inside the testing chamber. When the signal line NI PXI 2575 data retrieval device was connected, the micro temperature sensor was calibrated with a range between 20 and 105 °C, and 5 °C as the interval unit. When the temperature was stabilized, the average value of the signals of the micro temperature sensor in one minute was taken, and the measurement was carried out three times. Then the measurement results were output in the form of a curve diagram. Finally, the temperature-resistance value curve of the micro temperature sensor was obtained and presented as the original resistance value in Figure 4. Standard deviation is the distribution of a set of values relative to the mean. A larger standard deviation means that most values are far from the mean; a smaller standard deviation means that the values are closer to the mean.

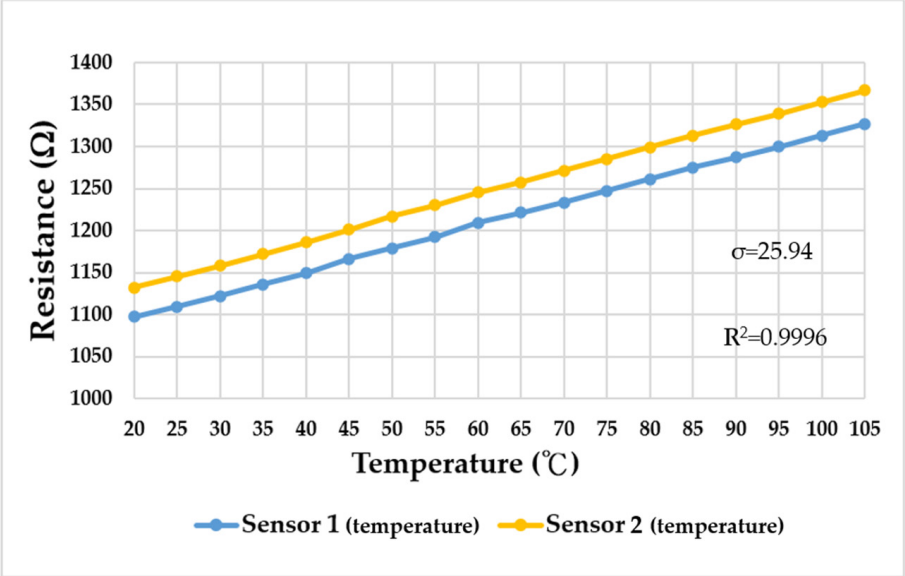


Figure 4. Calibration curve of the micro temperature sensor (original resistance value).

3.3. Calibration of the Micro Humidity Sensor

Likewise, the programmable temperature and humidity testing chamber was also applied to the measurement of the calibration of the micro humidity sensor. The humidity calibration ranged between 20% RH and 98% RH, with 5% RH as the interval unit. However, considering that capacitance values of the same humidity at different temperatures might vary slightly and that capacitance values during the humidification and dehumidification might also be slightly different, the humidity calibration should measure the capacitance values during the humidification and dehumidification between 20% RH and 98% RH at different temperatures. Regarding the humidity calibration operation, the temperature was set between 20 and 65 °C and rose every 5 °C; the humidity was set between 20% RH and 98% RH, and the humidification and dehumidification were carried out every 5% RH. Additionally, the NI PXI 2575 data retrieval device was applied to record the average of the capacitance values in one minute when the temperature and humidity stabilized. The measurement was carried out three times to take the average value. Then the measurement results were output in the form of a curve diagram. Finally, the relative humidity-capacitance value curve of the micro humidity sensor was obtained and presented as the original capacitance value in Figure 5.

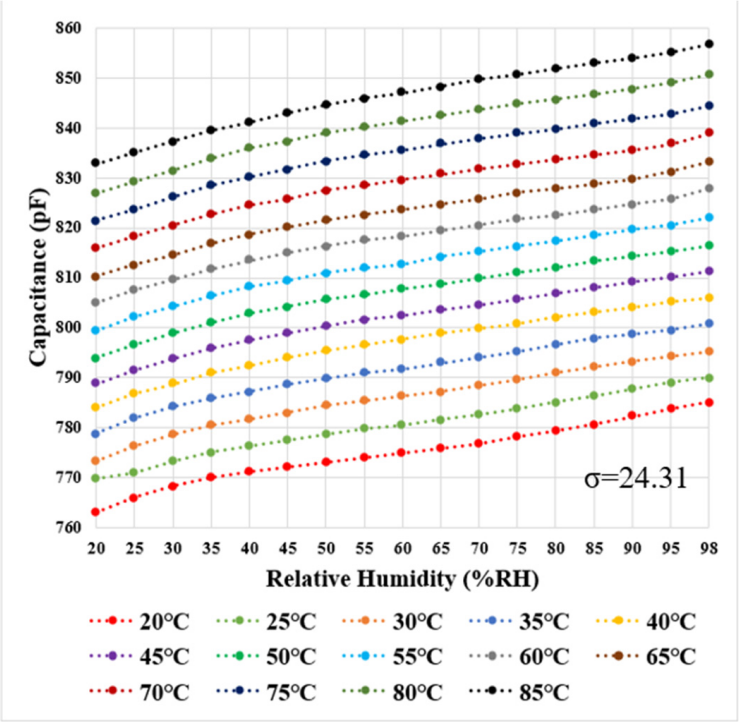


Figure 5. Average calibration curve of the micro humidity sensor (original capacitance value).

3.4. Calibration of the Microflow Sensor

The stable and variable flow input provided by the 850E Fuel Cell Test System was applied to the calibration of the microflow sensor (see Figures 6 and 7).

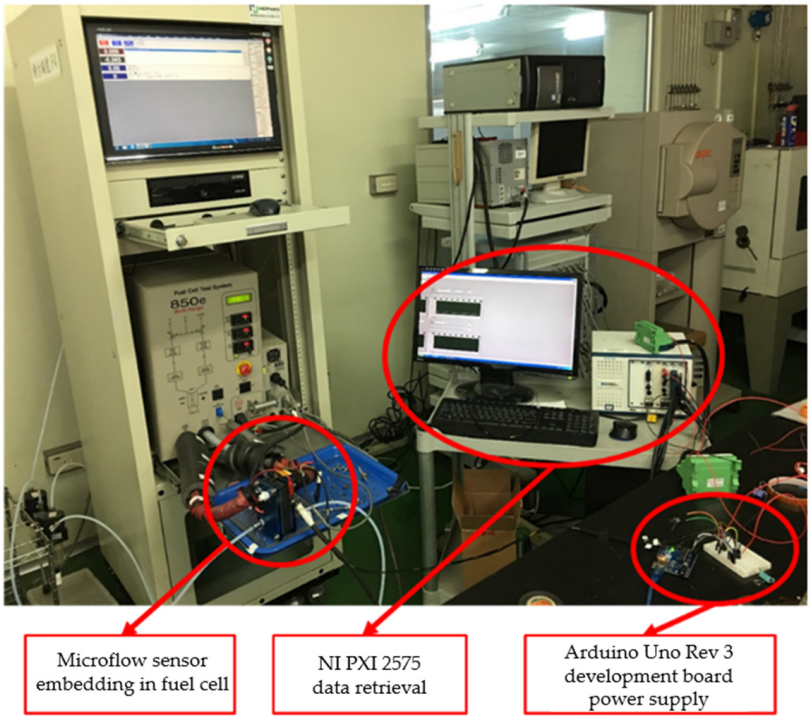


Figure 6. Calibration system diagram of the microflow sensor.

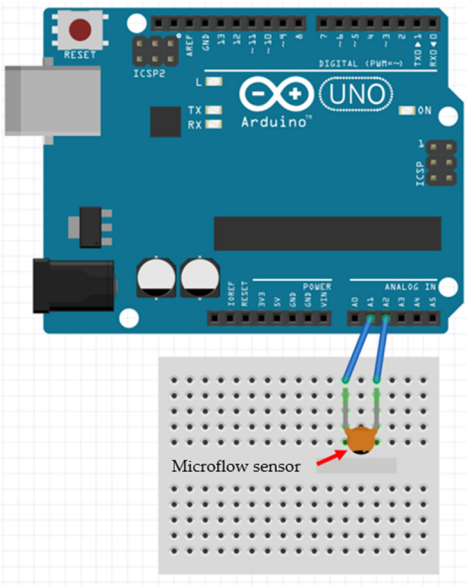


Figure 7. The Arduino Uno Rev 3 development board was connected to the microflow sensor.

Regarding the calibration of the microflow sensor, first, the sensor should be clamped between two bipolar plates and aligned with the flow channel; then, the Arduino Uno Rev 3 development board should be used as the power supply; between the power supply and the hot-wire microflow sensor, the NI PXI 2575 data retrieval device was cascaded to measure the current variation value. NI can read up to 6 decimal places. The Arduino Uno board can read up to 2 decimal places. These conditions are sufficient for application in the environment. Additionally, the power supply provided constant voltage to enable the microflow sensor to produce a stable temperature field and introduce air for calibration. In terms of the flow calibration operation, the microflow sensor was calibrated with a range between 0 cc/min and 3000 cc/min, with 300 cc/min as the interval unit. When the flow stabilized, the average of the current values in one minute was recorded. The measurement was carried out three times to take the average value. Finally, the flow-current value curve of the microflow sensor was obtained and presented as the original current value in Figure 8.

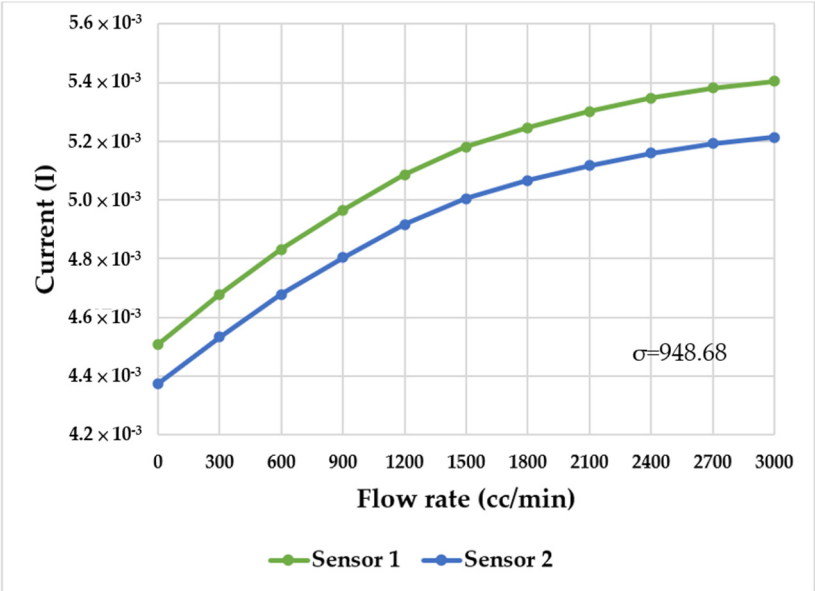


Figure 8. Calibration curve of the microflow sensor (original current value).

4. Application of the Self-Made Flexible Three-in-One Microsensor to the Laboratory oven for Immediate Micro-Monitoring of R2R Process

Among the many roll-to-roll processes, the manufacture of polarizing film can be regarded as a product with high precision and high output value. Polarizing film is to polarize the natural light that is generally not polarized, and convert it into polarized light. Therefore, liquid crystal display (LCD) can use this polarized light and twist characteristics of liquid crystal molecules to control the passage of light or not. In this study, the MEMS technology was applied to integrate temperature, humidity, and flow sensors on a 50 µm-thick polyimide substrate. Furthermore, the polyimide Fujifilm Durimide® LTC 9320 resisting electrochemical corrosion and acid was chosen as the protective layer. The self-made flexible three-in-one microsensor features include thinness, a small structural area, a high sensitivity, an immediate measurement, and can be placed anywhere. It was set up in the laboratory oven (see Figure 9) for immediate micro-monitoring of the R2R process.

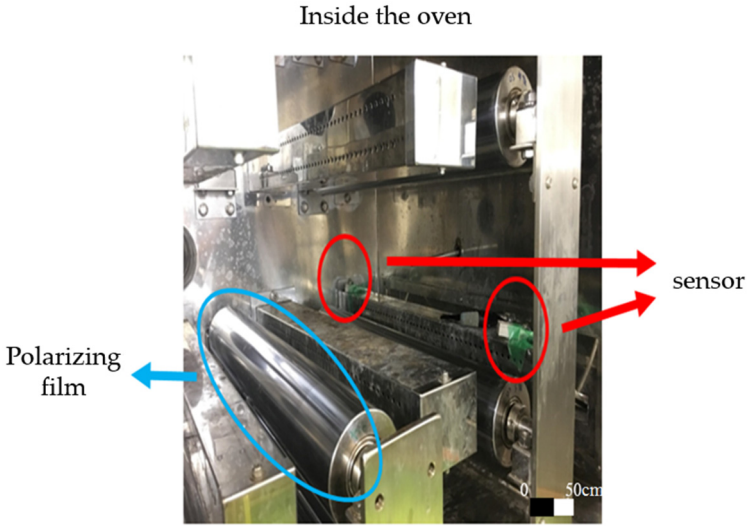


Figure 9. Setting up a flexible three-in-one microsensor in the laboratory oven.

4.1. Monitoring Data on the Self-Made Flexible Three-in-One Microsensor

4.1.1. Monitoring Data on Temperature

In the section of monitoring data on temperature, the changes in the temperature inside the oven during the processes of temperature rising from room temperature (25 °C) to 110 °C and temperature lowering from 110 °C to the room temperature (25 °C) were measured to record the temperature measurement curves of the self-made micro temperature sensor, the oven, and the commercial sensor, which are presented in Figure 10.

4.1.2. Monitoring Data on Humidity

In the section of monitoring data on humidity, we measured the humidity change inside the oven during the process of gradually heating from room temperature (25 °C) to 110 °C, and then naturally cooling from 110 °C to room temperature (25 °C). We recorded and compared the difference between the humidity measurement curves of the self-made and commercial humidity sensors. The humidity measurement curves are shown in Figure 11. It can be seen that in the measurement of humidity, the accuracy of the self-made humidity sensor is still slightly different from the expected one, and the error is larger than the humidity value measured by the commercial sensor. The selected material is not ideal in terms of humidity sensitivity changes, and there is a hysteresis in the response of humidity up and down.

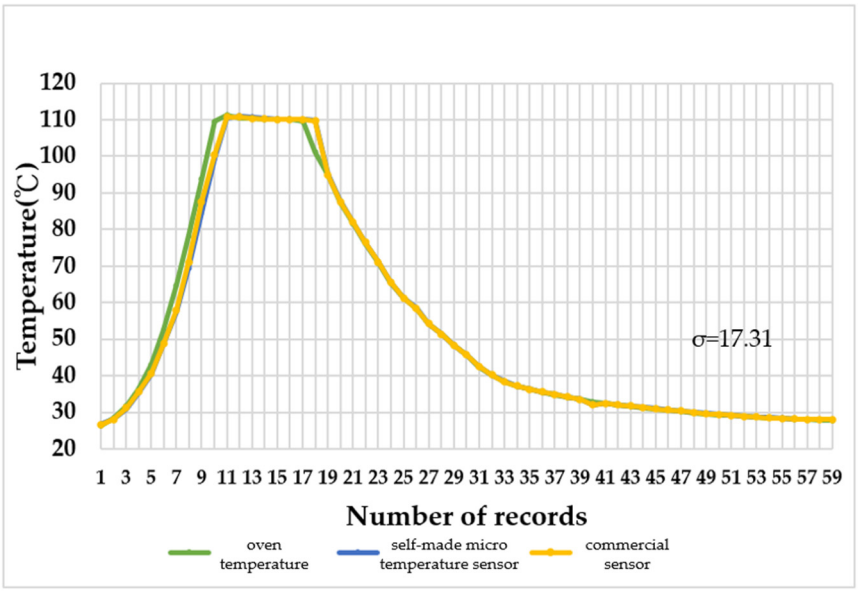


Figure 10. Monitoring data on temperature.

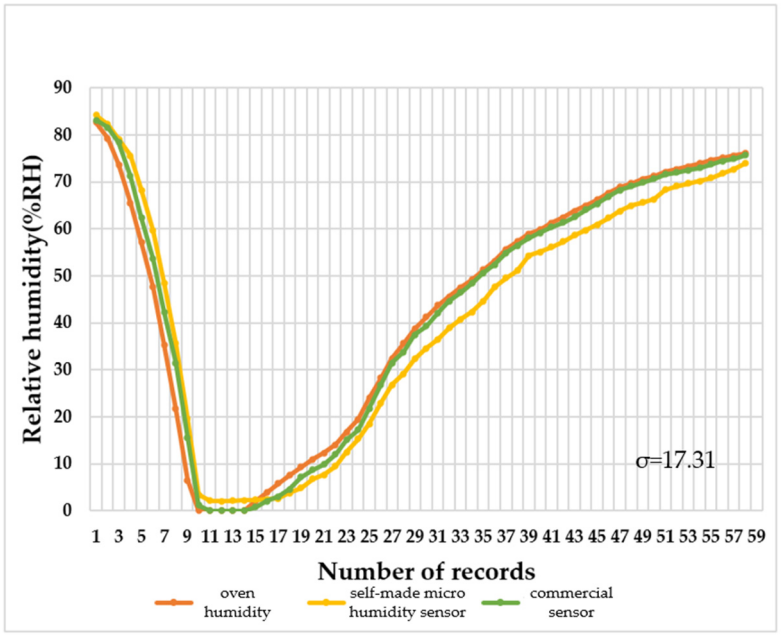


Figure 11. Monitoring data on humidity.

5. Conclusions

The self-made flexible three-in-one microsensor can capture the data on the interior environment of the laboratory oven via the self-made wireless monitoring module during the fabrication of the polarizing film without affecting the environment for the polarizing film process. This will help the operator observe and assess whether the temperature and humidity control settings and heat uniformity inside the oven need to be adjusted or improved, so as to reduce the number of defective polarizing films and improve the yield during the oven process.

In the part of temperature monitoring data, the temperature change inside the oven is measured during the process of gradually heating up from room temperature (25 °C) to 110 °C, and then naturally cooling from 110 °C to room temperature (25 °C). We recorded and compared the difference between the temperature measurement curves of self-made and commercial temperature sensors. It can be seen that although there is a slight delay

between the self-made and commercial temperature sensors with the thermometer of the oven itself, the values of the self-made temperature sensor and the commercial temperature sensor are different from each other. The difference is not big, and it can be seen that the self-made temperature sensor has considerable accuracy in temperature measurement. In the section of monitoring data on humidity, we measured the humidity change inside the oven during the process of gradually heating from room temperature (25 °C) to 110 °C, and then naturally cooling from 110 °C to room temperature (25 °C). We recorded and compared the difference between the humidity measurement curves of the self-made and commercial humidity sensors. It can be seen that in the measurement of humidity, the accuracy of the self-made humidity sensor is still slightly different from the expected one, and the error is larger than the humidity value measured by the commercial sensor. The selected material is not ideal in terms of humidity sensitivity changes, and there is a hysteresis in the response of humidity up and down.

Although the self-made flexible three-in-one microsensor can perform basic monitoring, there are still many flaws and imperfections that need to be improved. In the part of the humidity sensor, the material of the dielectric layer still needs to be strengthened, and another better humidity sensitive material needs to be found to make the response more real-time in the part of the monitoring module. It is only a preliminary and simple experimental structure. The combination of the development board and the additional accessories (Bluetooth board, SD card, power supply, etc.) is still a bit redundant. It must be gradually replaced with a smaller and function-specific board in the future, so that it can be arranged on the production line at multiple points.

Author Contributions: The work presented here was carried out in collaboration between all authors. C.-Y.L. conceived, designed, and discussed this study C.-K.L. and J.-T.L. performed the experiments and analyzed the data. All authors have contributed, reviewed, and improved the manuscript. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The authors confirm that the data supporting the findings of this study are available within the article.

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Conflicts of Interest: The authors declare no conflict of interest.

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