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A Review on Heat Flux Measurement Techniques in the Nano and Microscale

An Undergraduate Honors Thesis Submitted in Partial Fulfillment of the University Honors Program Requirements University of Nebraska- Lincoln

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Abstract

Heat Flux measurements represent an important step in obtaining an accurate heat transfer profile in many engineering problems. Sensors capable of measuring this quantity have been around for decades, however, the increasing focus in nano and microscale applications in the industry and academia demands more accurate and smaller devices. This paper reviews some of the methods used to develop heat flux meters targeted to nano and microscale, as well as some calibration processes for the same. A combination of Luminescence and non-luminescence methods for direct and indirect measurement of heat flux will be discussed. A glimpse of what the future of this technology could look like will also be explored in the form of biomolecular-based thermometry.

Key concepts: Heat flux sensor, temperature gradient, direct measurement, thermoelectricity, microfabrication, luminescence, nanoscale, temperature measurements.

1. Introduction

Heat transfer can be defined as the transit of thermal energy due to a temperature gradient and it is an important physical process to consider in many engineering applications [1]. Most of the time, measuring the rate of heat transfer per unit area, or heat flux, through the desired body is the preferred way to describe heat transfer. In the industry and academia, heat flux measurement is usually performed by measuring the temperature at two given points along the temperature gradient. Since heat flux and temperature are complementary thermal measurements [2], heat flux can then be calculated using the measurements and concepts like the thermoelectric effect. This is known as an indirect measurement of heat flux.

However, a more accurate and reliable method is to directly measure the point temperatures and the heat flux with one single device called a heat flux meter (HFM). The Seebeck thermoelectric effect (refer to section 2.1. for further information), for example, is one of the concepts used to directly measurement of heat flux [3]. Overall, many methods have been developed to measure this vector and the selection depends on specifications like the application, spatial scale, and materials. Azar, K.,[2] in his book "Thermal Measurement in Electronic Cooling" summarizes the most popular methods to measure heat flux as well as some possible applications. In general, these sensors can be categorized based on their targeted measurement. These can be the rate of change of temperature of a thermal capacitance, temperature gradient, differential temperature, and power dissipation in relationship with heat transfer [2].

Independent of the method used, a calibration for the HFM must be performed. There are two calibrations widely used in the industry and academia. The first one involves the application of uniform heat flux between two plates of known thickness and thermal conductivity at different temperatures, with the HFM located in the middle. Based on the temperature drop and the parameters of the plates, the heat flux can be calculated [4]. The second one requires the use of a two-sided guarded hot plate and the null detection technique to calculate the applied heat flux. The plate's temperature is adjusted to achieve zero heat flow through the HFM. The heat flux can then be calculated based on the change in power of the heating plate and the active area [4]. The development of better calibration techniques has been in high demand in the last decades. An increase in applications in the nano and microscale yield more complex and increasingly smaller HFM, contributing to this need.

This paper focuses on some of the methods that can be used to measure heat flux in the nano and microscale. When the measuring scale is comparable or even smaller than the mean free path of the energy carriers (like phonons and electrons), the assumptions of continuum and thermodynamic equilibrium are no longer reliable [5]. Sensors relying on these assumptions would be no longer useful, creating the need for adapting classical methods or developing new ones. Hence, state-of-the-art devices designed for nano and microscale thermometry and some new calibration methods will be reviewed.

2. Luminescence Methods

Heat flux measurement methods for nano and microscale can generally be divided into two distinctive categories: luminescent and non-luminescent [5]. Luminescence is essentially the refraction and reflection of light caused by the movement of electrons from one energy level to another [6]. Excess energy is irradiated in the form of electromagnetic radiation in the ultraviolet, visible, and near-infrared spectral range. When the temperature of a material increases, the intensity of luminescence can decrease in a process called quenching. Devices specially fabricated to measure this change in luminescence can then correlate it to the change in temperature experienced by the material. In this section, two luminescent methods will be reviewed. The selection of these specific methods exposed here depended on their popularity and/or novelty. Section 3 reviews three non-luminescence methods that were chosen based on the same parameters.

2.1. Quantum Dots

Semiconductor particles such as quantum dots (QDs) have been an object of interest for thermal measurements in the nanoscale due to their temperature-dependent luminescence in form of photon emission (photoluminescence) [6]. Depending on parameters like composition, these QD-based thermometers can be tuned for a specific range of light emissions and applications. For example, Khan et al. [7] proposed a nano-thermometer based on fluorescent carbon quantum dots (CDs) to monitor the temperature of living systems comprised of cells. The CDs are developed using a hydrothermal synthesis process on a solution made out of L-cysteine, trisodium citrate, and water. Figure 1 shows a visual representation of the fabrication process as well as an image of the CDs from transmission electron microscopy (TEM) used for surface characterization.

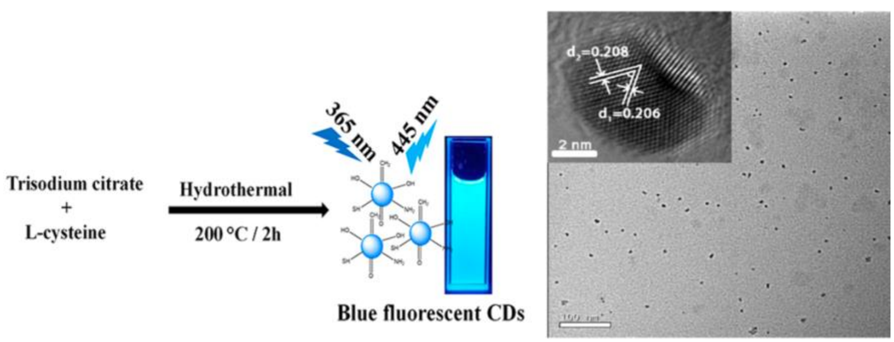

Figure 1. Fabrication process of CDs via hydrothermal synthesis (left) and TEM image of CDs (right) [7].

The two important factors that determine whether a CD-based solution, like the one being described, are photoluminescence (PL) intensity and lifetime. To investigate how the PL intensity changes with temperature, the solution was subjected to an increase in temperature from 25 to 70˚C. It was found that the PL intensity decreases linearly with temperature while keeping the same wavelength for emission. In a similar fashion, PL lifetime was found to linearly decrease with increasing temperature as well. This linearity is desired for the calibration, stability, and repeatability of the system. To test the thermometer capabilities of this method, incubated T-ca. cancer cells were merged with the solution. Cell behavior remained healthy discarding possible toxicity from the CDs. Upon excitation from a light source with a 420-480 nm wavelength, the cells display blue coloration which, as the temperature decreases, becomes lighter and lighter. Thus, the temperature can be recorded based on the PL intensity and half-life of the light emitted by the cell-CDs solution.

Liu et al. [8] also reported the use of a QD-based nano-thermometer, this time using dual-emission colloidal QDs, which exhibit two emission peaks in the near-infrared range. The fabrication of the PbS/CdS/CdSe core/barrier/shell QDs used started with the synthesis of bare PbS QDs using the organic compound oleylamine (OLA) as a ligand on $PbCl_2$ and purged with an influx of N_2. The cation exchange process was then used to synthesize CdS with the PbS QDs. The resulting QDs were scattered in toluene. Lastly, CdSe was synthesized with the PbS/CdS QDs and, again, dispersed in toluene to later be mixed with poly (methyl methacrylate) (PMMA) to form a film with a concentration of 1 µM. Because of its composition and shell-core structure, the film with QDs emits at two distinctive peaks which are around 670 and 910 nm. Upon thermal excitation, the peaks remain constant in the range of 100 to 300 K, while the PL intensity linearly increases with decreasing temperature. Having two emission peaks that behave linearly with temperature decreases the chances of errors in the system that could come from the refractive index of the surroundings, for example. Thus, dual-emission colloidal QDs offer improvements compared to single-emission methods, and make this technology more reliable and repeatable for heat flux measurements.

2.2. Organic Dyes

The main characteristic of an organic dye is that in response to a temperature change, their fluorescence intensity, fluorescence lifetime, and emission wavelength change as well. Specifically, temperature and fluorescence intensity are inversely proportional [9]. Many different organic dyes have been developed and it is currently a widely used method for nanoscale thermometry mainly because of its tunability capabilities and ease of fabrication compared to other luminescence techniques. One of the most recurrent applications is temperature measurement on living systems or microorganisms such as cells. An example of this is the thermometer developed by Arai et al. [10] to measure a temperature gradient in mitochondria, the well-known heat-producing organelle in a cell. Figure 2 shows the schematic of the thermometer system developed.

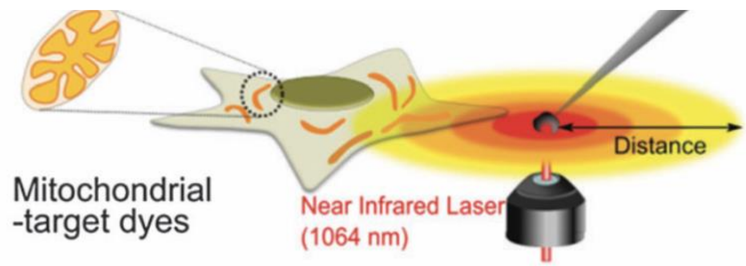

Figure 2. Schematic of a rosamines-based nanothermometer system [10].

Rosamines, a class of fluorophore, was used for this device due to a higher likelihood of localizing to the mitochondria and a higher temperature sensitivity compared to other dyes of similar classes [10]. To create the temperature gradient, a near-infrared laser was directed toward the system comprising the mitochondria and the dye, which can be comprehended as a nanodevice. The device was cycled through the heating and cooling process to test the sensitivity, reversibility, and repeatability of the dye. When the temperature increased, the intensity of the dye decreased linearly. When the laser was turned off and the temperature decreased, the intensity returned to normal in the same fashion, proving that the dye has the necessary characteristics of a thermometer.

Similarly, Huang et al. [11] used the fluorophore DyLight549 coupled with the protein streptavidin to measure the temperature distribution in the proximity of superparamagnetic nanoparticles integrated inside cells. As expected, when the temperature and magnetic field in the area closest to the nanoparticles increased, the intensity of the DyLigth549 decreased. From previous calibrations, this behavior signified an increase of around 15˚C during a time span within 15s [11]. Although these two implementations of dye-based fluorescent thermometers are very specific to the field of research involving microorganisms and cells, they shed a light to the usability of this technology.

3 Non-Luminescence Methods

3.1. Scanning Thermal Microscopy

Scanning Thermal Microscopy (SThM) is a non-luminescence method that was first developed by Williams and Wickramasinghe in 1986. It consists of probes that act as nanoscale thermometers that map the temperature distribution profile of an area creating a nanoscale thermal image of the same. Moreover, SThM is usually coupled with Atomic Force Microscopy (AFM) to cover a wider variety of samples that do not have to be conductors [6].

The probes can be tailored depending on the scale, material being studied, and resolution desired. There are two types of probes that are commonly used: a thermocouple junction and a resistive wire bent [6]. The calibration of these probes involves measuring a reference sample and comparing the measured with the expected value [5]. In the case of a thermocouple junction, the thermovoltage measured is linked to the sample temperature. For the resistive wire, the resistance measured is linked to the thermal conductivity of the sample. For the latter, the resolution is strongly dependent on the material the probe is made of.

In spite of the great development of this technology in recent decades, concerns regarding the reliability of this technology with a decreasing spatial scale are still present. For instance, for noncontact measurements, the probe is separated from the sample by a thermal resistance that prevents the probe from thermally equilibrating with the sample [12]. Moreover, the probes are also subjected to wear and tear for both contact and non-contact measurements, leading to undesired results [13].

Menges et al. [12] addressed some of these issues by developing a High-Vacuum scanning thermal microscope with a non-equilibrium scanning method. Figure 1 shows the microscope setup as well as the scanning probe. The heat flux through the surface of the tip was obtained by measuring the temperature variation of the probe. This heat flux depends on both the temperature difference and the resistance between the probe and the sample, and by measuring both in a quasi-simultaneous manner, systematic errors were avoided [12]. To achieve this, the temperature of the sensor was set above ambient temperature by applying a DC current to it. From here, both the resistance and the temperature of the sample can be calculated. An accurate temperature map of the sample can then be created by repeating this procedure at multiple points along the surface of the sample.

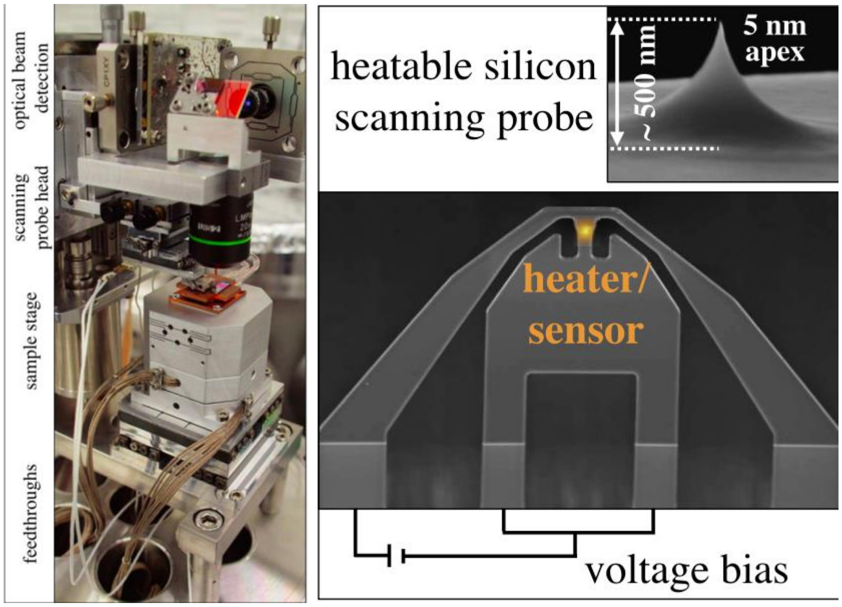

Figure 1. Scanning thermal microscope set-up (left) and silicon scanning probe (right) [12].

The ability to get the measurement of the resistance between the sample and the probe and the temperature of the sample in one single scan as opposed to two consecutive scans has major advantages. One of them is avoiding the undesired wear in the probe that can happen even after one single measurement, and it sets this technique apart compared to others like regular SThM or SThM with the null-point approach. However, some limitations with this technology that were not addressed by the authors and are likely to be persistent to this day are the contemplation of near-field radiation in vacuum settings (just as was the case for this study) and the use of this technology to model heat transfer in microscopic organisms [13].

3.2. Thermoelectric Devices

Thermoelectricity studies the relationship between electricity and heat within a material. The two phenomena in which this relationship can be experienced are the Seebeck and the Peltier effect. The first one is the production of electrical potential or voltage when a specimen experiences a temperature difference. The latter one is the movement of heat through a specimen when subjected to an electrical current [14]. Many temperature reading devices and heat pumps like thermocouples (Seebeck effect), resistance temperature detectors (RTD-Peltier effect), Peltier device, and thermoelectric coolers (TEC) work under this principle.

The Seebeck effect, in particular, is a concept commonly used in the industry and academia for heat flux thermometry. Companies like *FluxTeq* and *greenTEG* are just an example of companies that specialize in the development and commercialization of HFMs. Even though every company uses different materials, production methods, and/or sensor designs, the foundational concept is the same. The HFM with a known thermal conductivity and thickness is positioned in the “path” of the heat flux being measured. Inside this device, multiple thermocouples are connected in series creating a thermopile, with a cold junction on one side of the device and a hot junction on the opposite side. This thermopile in this specific arrangement is capable of measuring the temperature gradient between the HFM, which is proportional to the heat flux [15]. Since the conductivity is known, so is the device’s resistance that creates a voltage difference, given as the sensor's output. The more thermocouples the device has, the greater the voltage that can be measured, the more accurate the results. Increasing the thickness can increase the sensitivity of the device, representing how susceptible to the heat flux the sensor is. However, this increase in the thickness also leads to an increase in response time, which might not be desired depending on the application.

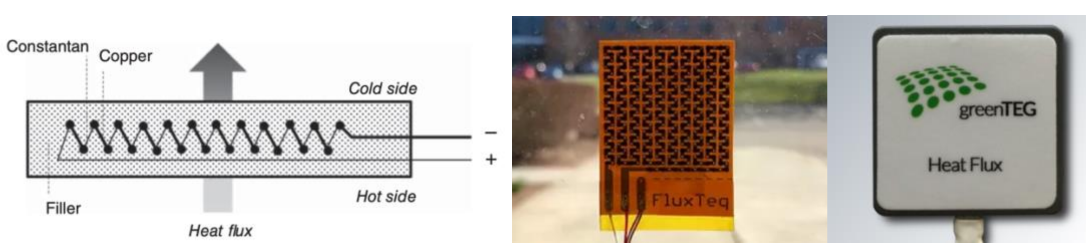

Figure 2. Schematic of a common state-of-the-art sensor (left) [15]. FluxTeq (middle) and greenTEG (right) heat flux sensors (from fluxteq.com and greenteg.com, respectively).

Despite recent advances in this technology in the recent decades and the vast number of applications for these devices, nanoscale thermal measurements usually require more specialized equipment. For instance, whereas companies like *greenTEG* claim µW resolution in their commercially available lineup *gSKIN®*, nanoscale research almost always requires at least resolution in the nW scale. HFMs using the Seebeck effect have been reported for this type of application, but they are generally custom-made for research and development. One such example is the microfluidic heat flux sensor (HFS) developed by Nam et al. [16] as shown in Figure 3.

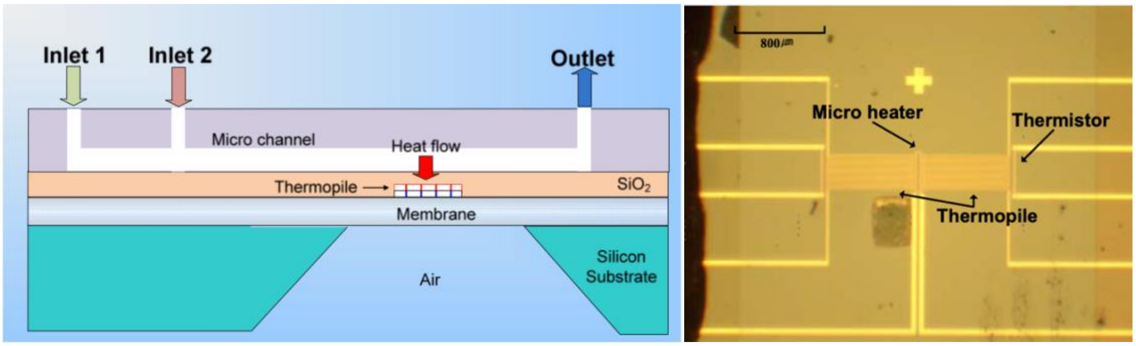

Figure 3. Schematic cross-section of the microfluidic sensor with a thermopile (left) and micrograph from the top of the sensor (right) [16].

The objective of this sensor was to measure the heat generated from living cells. A complementary metal-oxide-semiconductor fabrication process was used to create the thermopile-based device. The thermopile is a 0.2 µm thick layer of Au and Ni that was patterned and etched using an E-beam evaporation lift-off process, followed by deposition, and etching of a 0.4 µm thick layer of SiO_2 that act as electrical insulation and the junction of the thermopile. The silicon substrate is supposed to act as a heat sink and the air gap for thermal isolation. To measure the heat flux, a culture fluid is first introduced through the first inlet (Inlet 1 as shown in Fig. 3) followed by the living cells through the second inlet (Inlet 2 as shown in Fig. 3). The first flow without the cells acts as a reference value, while the flow with the cells is the objective value or value being measured.

The final sensor achieved a resolution of 20 nW and serves as a good example of adapting existing methods for nanoscale applications. Moreover, it shows how an HFM device can be used to thermally characterize flow and not only stationary specimens.

3.3. MEMS Devices

Microelectromechanical systems or MEMS have been around since the early 90s. Their applications have rapidly diversified covering a large span of industries. One of such applications is sensing physical or chemical variables [17]. Measuring heat flux and/or temperature is just one of many applications that MEMS have nowadays, and of the best examples is microfabricated thermocouples. For instance, Kim et al. [18] developed a device using thin-film thermocouples with simplified fabrication and calibration processes. The device consists of nine thin-film thermocouples (made out of alumel and chromel) and a heater (made out of nichrome) deposited on a quartz wafer with a thickness of 200 μm using the sputtering process [18]. However, instead of using photoresists masks for this process, stainless-steel reusable masks were used. Figure 4 shows the completed temperature sensor.

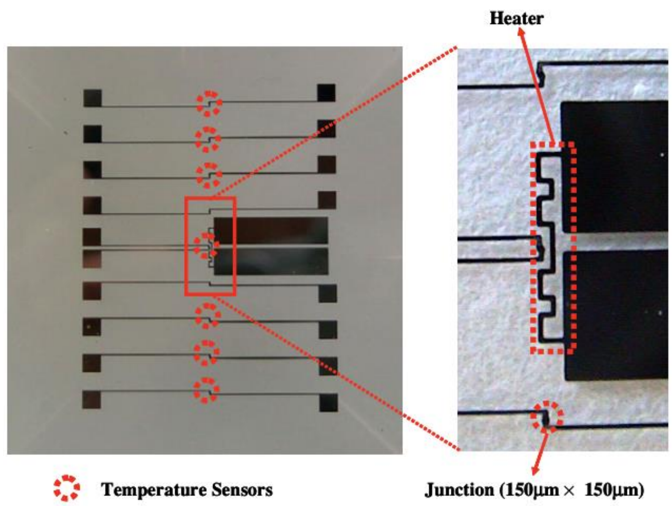

Figure 4. Completed micro-thermal sensor [18].

The thermocouple arrangement was proven to correctly measure the temperature when compared to commercial K-type thermocouples since these are essentially the same type. A microchannel made out of polydimethylsiloxane (PDMS) was also bonded to the silicon wafer using air plasma. The main purpose of this research was to fabricate an optimal microchannel device for various mass flow rates and input powers, but the simplified fabrication process described could be extended to MEMS focused solely on heat flux measurements.

On a different note, Feng et al. [19] developed a MEMS device to measure near-field thermal radiation (NFTR) between two parallel membranes. The two freestanding membranes have each a triple-layer design with two external SiO_2 layers and a middle SiN layer. The distance between them is 1 µm. Each membrane also contains a Pt line heater in a zigzag shape and with four ends that act as both a resistor and a thermometer. The fabrication was accomplished using a MEMS and a port-MEMS process on a silicon wafer, which included sputtering, plasma-enhanced chemical vapor deposition (PECVD), reactive-ion etching (RIE), lift-off technique, and the use of sacrificial Al layers. Figure 5 shows a schematic design of the device.

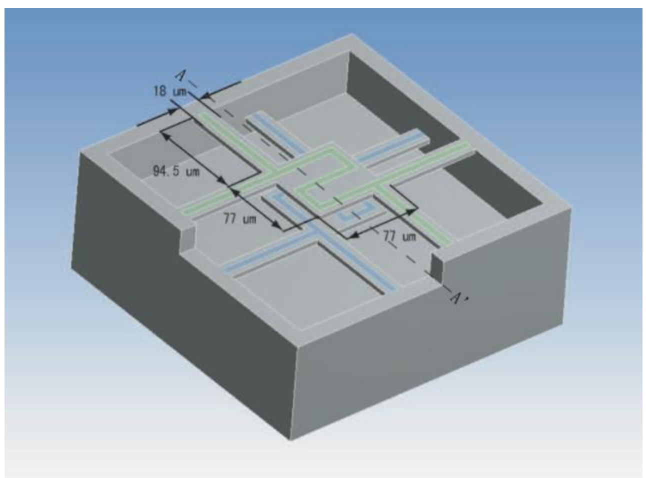

Figure 5. Schematic of the dual membrane MEMS device [19].

For the measurement process, the lower membrane heated up and acted as the emitter, while the upper membrane acted as the receiver. To measure NFTR, the setup had to necessarily be in a vacuum at ambient temperature. The emitter membrane was heated with various constant currents, then the receiver membrane was extracted, and the emitter was heated again in a similar fashion. The NFTR calculations required measuring the resistance of the Pt element for every current. Based on the difference in results obtained from using one membrane compared to using both, it was concluded that NFTR was measured successfully, although future improvements could include an actual measurement of the upper membrane (cold membrane) temperature.

4. Future Work: Biomolecular-Based Thermometry

Nowadays, many industries are looking at nature for inspiration in design, fabrication, and/or problem-solving. In the heat flux thermometry field, this philosophy is particularly expressed in biomolecular-based thermometry. It is based on the premise that living cells sense temperature changes through proteins, nucleic acids, and mRNAs. These chemical compounds and molecules either respond to temperature by changing their structure or undergoing reactions [6]. For many types of bacteria, there is one mRNA in particular that contributes to the heat sensitivity of the microorganism, an RNA thermometer called the ROSE element [20]. ROSE elements control the expression of genes when a sudden increase in extracellular temperature, or heat shock, occurs, unblocking a ribosome binding site that would be blocked at normal temperatures [21]. This means that synthetic RNA thermometers with simpler structures can be developed and used to measure and record the temperature of a nanoscale process.

Höfinger et al. [22] propose a nanodevice using short strands of the ROSE element from a bacteria belonging to the group *rhizobia*. DINAMelt software was used to analyze random RNA sequences from the ROSE element and test their sensitivity to temperature changes. From here, critical temperatures for every sequence can be calculated and used as a categorizer for their stability in that given temperature. Based on the stability and sensitivity to the thermal behavior, some RNA sequences are selected for use in the nanodevice. Table 1 shows some of the selected RNA sequences and their respective critical temperatures.

Table 1. Some selected RNA sequences and their respective critical temperatures [22].

T_{crit} (°C)	Sequence	Colour sum
−10	CAUUACUUACG	
0	UGGGGGGUGGG	
10	AAAUCCUUUCCUU	
20	GUAAAGAAAGAUA	
30	GUCAUACACCUCCG	
40	UACAAGAUGGUU	
50	CUCGUAUCCCACUAAGAUG	
60	CCAUGAUUCUUAGUGG	

A sensor incorporating these RNA has to be fabricated to contain multiple strands that transition from fully formed hairpin structures to single coils at different critical temperatures. The design of such a device consists of a binary assembly of solid and liquid phases. The solid phase is a matrix, and the liquid phase would flow through the matrix. Figure 6 shows a schematic of this design. Strands of RNA are crosslinked to the matrix to remain stationary, while the liquid is embedded in the selected RNA sequences. The crosslinked strands are complementary to the ones in liquid form. When the temperature of the assembly reaches a given critical temperature, the selected RNA will unveil into coils and hybridized with its crosslinked partner. Since both the crosslinked and liquid strands are color-coded, and the assembly is transparent, the temperature at a given time can be read just by looking at the color of the hybridized pair.

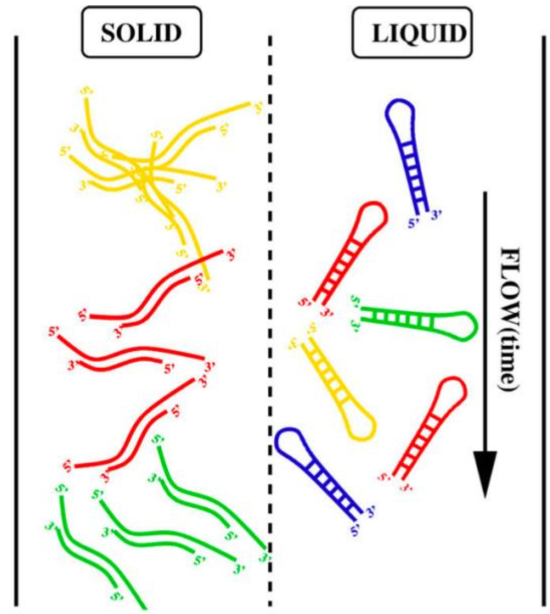

Figure 6. Schematic of the nanodevice using selected RNA sequences [22].

However, to use this method for heat flux measurements, more experimental studies demonstrating the use of this device in different circumstances and measuring a temperature gradient are necessary.

5. Conclusion

Temperature and heat flux measurements are indispensable variables to characterize many processes in different fields in the industry and academia. Although many methods to achieve this have been developed throughout the years, an increment in micro and nanoscale applications has led to a rethink of existing methods and the proposal of new ones. From the current state of the literature, it is safe to state that most of the newly developed methods rely on the complementary nature of temperature and heat flux. This means that most of the methods, luminescent and non-luminescent, measure heat flux indirectly. QDs and organic dyes, for example, measure temperature at a given area, with the possibility of measuring a temperature gradient from where the heat flux can be calculated. In a similar but more straightforward manner, temperature changes recorded using STM, and most of the MEMS can also be used to calculate the heat flux. Devices based on the Seebeck effect but for nanoscale processes, like the one proposed by Nam et al., are arguably the closest to an HFM due to its direct measurement of heat flux. It is also safe to imply that despite all the innovation in this field, it is in its early stages. A reliable method that can be commercialized and widely used in the industry is still some years down the road. Nevertheless, new methods such as biomolecular-based thermometry show exciting ways in which innovative design philosophies like biomimicry can reshape this field and bring about possibly bridge the gap between single-use methods and industry-wide solutions.

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