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Investigation of sensing capabilities of organic bi-layer thermistor in wearable e-textile and wireless sensing devices

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A B S T R A C T

This study is stimulated by the discovery of high sensitivity of nanostructured layers of organic semiconductor α'-BEDT-TTF)$2I_xBr_{3\text{-}x}$ [BEDT-TTF = bis(ethylendithio)-tetrathiafulvalene] to heat radiation. We present the development and assessment of the flexible lightweight highly sensitive film-based thermistor as (i) a separate sensor, (ii) a sensor integrated in e-textile and (iii) a sensor embedded in a wireless sensor node. Wireless Sensor Networks (WSN) and Internet of Things (IoT), being two promising technologies, have already been applied in a number of monitoring scenarios. In spite of great progress achieved in sensing technologies and wireless embedded systems there is a gap in multidisciplinary research aimed at investigating the aggregate potential of these technologies. Experimental results demonstrate that the developed bi-layer organic thermistor has high potential for environmental and biomedical monitoring. They can be used as a part of wearable units or as sensing units on board of wireless sensing devices.

1. Introduction

S.R. Forest and M Thompson indicated conclusively in their well known "Introduction: Organic Electronics and Optoelectronics" that the use of organic compounds as active materials in electronic and optoelectronic devices opens the door to a large number of efficient and potentially low-cost methods for fabricating useful, and, in some cases, complicated structures that are inaccessible by conventional methods using conventional semiconductors [1]. It should be noted that one of the important areas for innovation in the field of organic electronics is sensing [2]. For example, it was shown that some of organic molecular conductors, such as trihalides of bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF), are able to give to electronic devices extraordinary strain, pressure and temperature sensing properties impossible to achieve with metal-based electronic structures, thereby enabling a broad range of innovative "out-of-the-box" applications [3–6]. On the other hand, the attraction of $(BEDT\text{-}TTF)_2Hal_3$-organic molecular conductors is the ability to directly impact the sensing properties of the material when deposited in thin film form [3,7].

As it has been noticed earlier, sensing is an emerging technology which can be significantly upgraded by information and communication technologies that are able to send measured data to the user over the network including the Internet. The term Internet of Things (IoT) was coined by Kevin Ashton in 1999 and was about empowering computers with gathering information by means of sensors and RFID technology [8]. Nowadays, there is no a single definition for IoT, but its common understanding is about the devices, objects and services joined in a global network with some processing and cognitive capabilities [9]. Since the time the IoT was first mentioned, sensing, networking and processing technologies have greatly evolved.

For example, there is noticeable progress in film based electronic components, e.g. sensors and storage [10], that can be deposited right on the surface of the embedded systems. This progress has been due to newly available materials that can ensure

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better performance, full customization of printed/deposited devices and, in most cases, a simple fabrication process. However, there is still a gap between the printed devices and their integration with embedded electronics and, in particular, with Wireless Sensor Network (WSN) devices. In fact the WSN paradigm is considered as a pillar technology in the forthcoming era of the IoT and was recently applied to a number of monitoring scenarios [11,12] including the medical ones [13]. The IoT is a promising technology in terms of addressing a number of problems [14] including a remote medical assistance [15,16] which appears to be an important challenge in the growing urban areas.

With the development of WSN and IoT there is a growing interest in the sensing technology on plastic substrates. This technology allows for a significant reduction in production cost and for adding new functionalities [1,17]. Hence there is a high potential for integration of lightweight temperature sensing materials into human wearable interfaces, e.g. fabrics. Wearable electronics is relevant since it offers personalized healthcare, security and comfort [18].

Covering polymeric films with conventional metals and semiconductors is one of the traditional approaches to building up flexible lightweight sensors [19]. For instance, Chun-Chih Huang et al. [20] proposed a flexible thermistor fabricated by printing a square NiO thin film on a polyimide film. Such flexible bi-layer (BL) sensors are characterized by good sensitivity and fast response time. However, these flexible sensing materials have a common drawback: poor adhesion of conventional metals, as well as their oxides, to polymers. A disadvantage of this kind leads to a low binding between inorganic sensing layers and plastic supports.

To address this problem, organic conductors can be used. A number of research works reported on the fact that conducting polymers can be successfully applied to sensing technology [21]; the demonstrated flexible capacitive-type humidity and temperature sensors - cellulose–polypyrrole nanocomposites – are the promising examples of all-organic flexible sensors [22]. At the same time, electronic properties of conducting polymers are not stable enough since they are sensitive to atmospheric moisture [23]. Moreover, high manufacturing costs prevent the usage of expensive conductive polymers.

As mentioned earlier, molecular conductors has been recently proposed: $(BEDT\text{-}TTF)_2X_3$ [BEDT-TTF = Bis(ethylenedithio)tetrathiafulvalene, X_3 = trihalide anion]. It can be successfully combined with plastic films by relying on a low cost simple synthetic procedure described in Refs. [24,25]. This procedure shows that conducting bi-layers polycarbonate/$(BEDT\text{-}TTF)_2X_3$ are highly effective as inexpensive lightweight strain [3], temperature [4] and humidity sensors [26] in terms of their application to monitoring of a remote patient. Another significant advantage is integration of the strain sensing polycarbonate/polycrystalline (001) oriented (-(BEDT-$TTF)_2I_3$ bi layer film into a polyester textile. The resultant conducting textile ensures high flexibility and an easy detectable electrical response signal even to small strain [27].

In our recent work, we have reported on the bi layer film: polycarbonate/polycrystalline (001) oriented α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ molecular conductor [hereafter it will be as polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$] (Fig. 1) which can be used both as a direct-contact thermometer and as a passive noncooled infrared sensor [4]. This sensor is capable of identifying even negligible temperature changes with accuracy of 0.005° [4]. The obtained results improve those obtained from widely used thermistors: the accuracy of a Pt-1000 detector is 0.01° [28].

In this work we investigate the sensing capabilities of the polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ bi-layer film with the goal of figuring out whether it can be used in wearable biomedical and environmental monitoring scenarios.

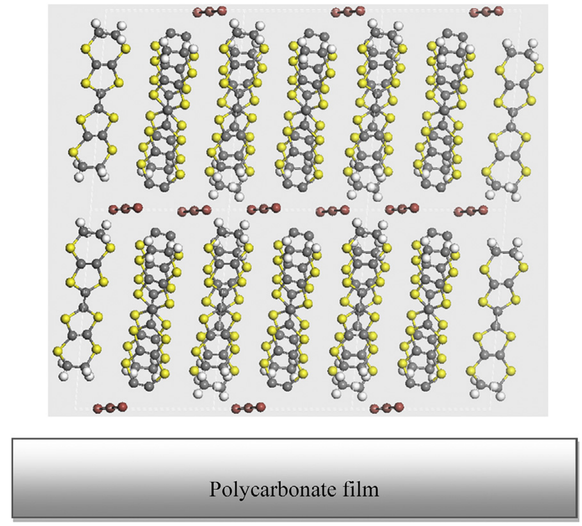


Fig. 1. Schematic presentation of the highly temperature sensitive bi-layer film: polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ showing the (001) orientation of α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$.

In particular, we present and discuss processing of the thermistor: polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ into polyester textile. This process enables engineering of e-textile which can detect even negligible temperature changes. For the sake of feasibility of this research, we implement the associated ‘proof-of-concept’ by designing a wireless sensor node with the proposed thermistor. The resultant monitoring system is capable of precise monitoring of ambient temperature changes in an office environment depending on the presence of working personnel.

2. Experimental materials and methods

2.1. Preparation and characterization of the flexible lightweight bi layer thermistor: polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$

For fabricating the BL thermistor we followed the synthetic procedure described in Ref. [29]. To be more specific, we first prepared a 25 μm thick polycarbonate (PC) film which contains an 8 wt % of BEDT-TTF. To effectuate this task, the film is cast on a glass support at 130 °C from a 1,2-dichlorobenzene solution of polycarbonate and BEDT-TTF. Afterwards, to perform covering of the film with the (001) oriented layer of α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$, we exposed the film surface to the vapors of a 0.5 M solution of IBr in dichloromethane for 3 minutes at 30 °C and relative humidity 40%. For the reasons of temperature and humidity control, we used a climatic chamber MEMMERT HPP. A binary mixture system: IBr/dichloromethane solution-IBr/dichloromethane vapor was used at equilibrium.

The X-ray analysis of the resulting surface-modified film indicates the presence of only (00l) reflections being characteristic of conducting layers formed by oriented crystallites (see Fig. 2). The measured values of the interplanar spacing d_{00l} (16.355 Å) along with intensities of (00l) reflections confirm the formation of the (001) oriented layer of α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$, were 0.66 > x > 0.33 [29]. These α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ linked crystallites contain set of the IBr_2^-, I_2Br^-, and I_3^- trihalide anions in which IBr_2^- anion is a main component [29].

2.2. Integration of the bi layer thermistor into polyester textile

For forming the rigid support for the bi-layer thermistor, the

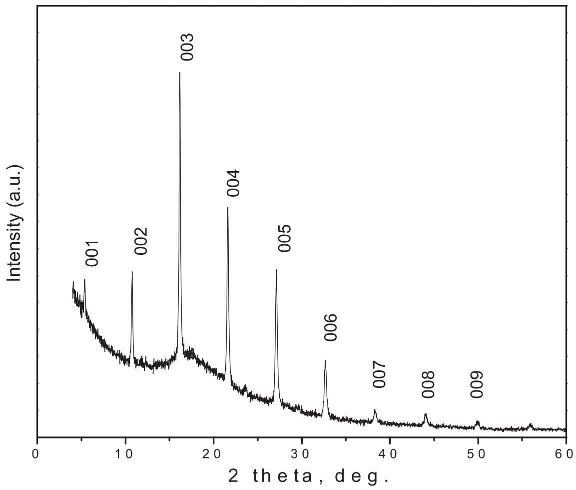


Fig. 2. X-ray diffraction pattern of the conductive sensing layer of the BL thermistor: polycarbonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$, showing (00l) orientation of the conducting α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ crystallites; 2θ = 5.43 deg.

part of the textile is sandwiched between two glass slides and heated up to ≅250 °C (the textile melting point) using soldering iron; the plastification process of polyester fabric is visually controlled. This procedure results in the formation of a smooth rigid polyester film in a heated part of textile. The polycarbonate-based layer of the thermistor: polycarbonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ is attached to the rigid polyester-based support using the glue. This glue is used as it is unable to destroy the polycarbonate layer of the polycabonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ film. To make electrical contacts, four Pt-based wires are attached to the conductive temperature sensing layer of the thermistor using graphite paste. This process is schematically shown in Fig. 3.

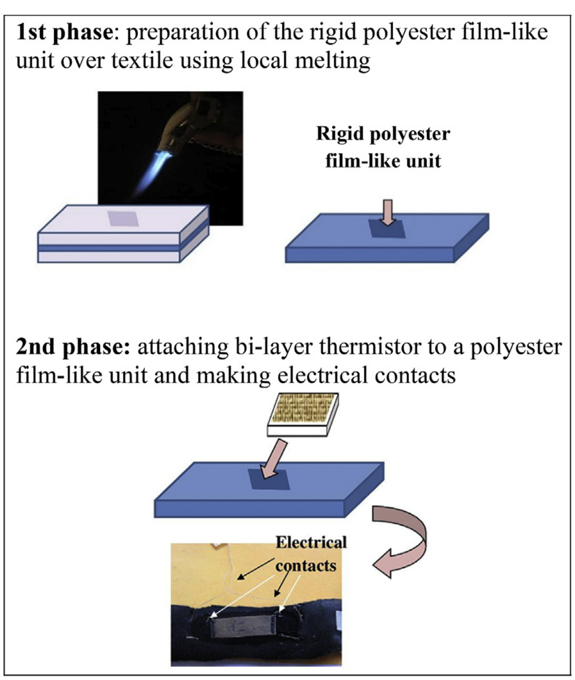


Fig. 3. Schematic presentation of the thermistor processing into textile using a local melting procedure.

3. Results

This section shows how the developed bi-layer thermistor: polycarbonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ can be processed into polyester textile to make a temperature sensing fabric. Fabric-based sensing is a large field of research in the biomedicine. For instance, temperature sensing textile is a promising technology for detecting early signs of breast cancer [30,31]. The Circadian Biometric Recorder sensors integrated in the bra pick up the minute fluctuations of circadian rhythm-based temperature variances of cell cycles on a 2–12 h bases to identify any abnormality at an early stage of cell augmentation [30,31]. E-textile can be used in many state-of-the-art sport, military and aerospace technologies as they can guarantee unobtrusive sensing.

3.1. Embedding of the bi layer thermistor: polycarbonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ into polyester textile

For developing temperature sensing e-fabric using the α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$-based conducting layer as an active component one has consider the high sensitivity of the electrical resistance of α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ to temperature changes happens along with high sensitivity to deformation [32]. Fig. 4 presents the thermistor polycarbonate/α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ which shows a reversible electrical response to cyclic monoaxial deformation with gage factor (GF) ≅8; GF = ε(R$_0$/R$_ε$-R$_0$), were R$_0$ = initial resistance, R$_ε$ = resistance at relative strain ε.

Hence, when developing the temperature sensing e-textiles at its first phase has to be reduced to engineering a rigid textile-based support to which the thermistor can be connected at the second phase. Our material engineering principle is based on Hooke's law. The combination of soft fabric with a rigid textile-based unit may be considered as a combination of two springs in which one (textile) has the spring constant (k_1) being significantly less comparing to another spring (k_2) (rigid textile-based unit). Relying on Hooke's law, the extending (Δx) of this combination under force (F) is as follows:

$$\Delta x = \Delta x_1 + \Delta x_2 = F/k_1 + F/k_2 \quad (1)$$

when $k_1 << k_2$, $\Delta x_1 >> \Delta x_2$, the extending of a rigid textile-based unit will be extremely small and therefore the deformation effect will be reduced to a minimum. To form a textile-based rigid unit we have used an impregnation process: a part of a textile is impregnated by rigid polymer (polycarbonate) and then the bi layer

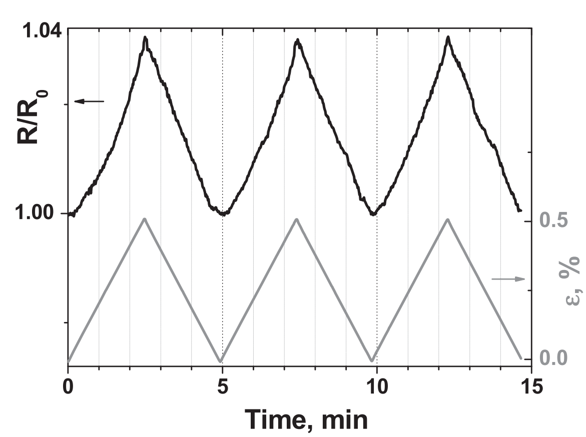


Fig. 4. Electrical resistance response of the α′-(BEDT-TTF)$_2$I$_x$Br$_{3-x}$ conducting layer to cyclic monoaxial elongations.

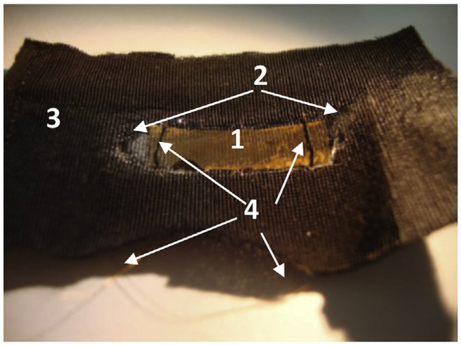


Fig. 5. Photo image of the BL thermistor (1) being attached to the rigid impregnated part (2) of the polyester fabric (3); 4 –electrical connections.

thermistor: polycarbonate/α'-(BEDT-TTF)$_2$I$_x$Br$_{3\text{-}x}$ is connected to the impregnated part (see Fig. 5) [30].

At the same time, our recent microscopic study revealed that this 'attaching' approach resulted in the formation of some cracks on the conductive sensing layer of the bi layer thermistor (Fig. 6). This result motivated us to look for another approach for processing the bi layer thermistor into polyester textiles.

To highlight the formation of the above described crack-like defects, we suggest that a rigid flat unit at the polyester textile to which the thermistor has to be attached may be prepared by locally melting of a small part of the textile being sandwiched between two glass slides. Under local melting the small part of the polyester textile (see Fig. 7, 1st phase), has to lose its textile-like texture while becoming film-like. The melted part must become much more rigid as compared to the rest of the textile. The above procedure allowed us to prepare the smoother rigid support as compared to that fabricating by textile impregnation. The bi-layer thermistor is attached to the rigid polyester-based support using the glue (see Fig. 7, 2nd phase). At the final stage of the prototype fabrication, we attach the electrical contacts to the conductive temperature sensing layer of the thermistor using graphite paste (Fig. 8). The SEM image of the surface of the α'-(BEDT-TTF)$_2$I$_x$Br$_{3\text{-}x}$ -based temperature sensing layer shows that it has no imperfectness which can be found in Fig. 6.

3.2. Temperature testing of the developed prototype e-textile

We design testbed for validating and experimenting on sensing human body temperature changes. Fig. 9 shows that the resistance of the e-textile strongly depends on temperature: the resistance decreases linearly from 34 to 28.5 kΩ when temperature increases from 30 to 43 °C. From these data the electrical response of the fabricated e-textile to temperature changes equally ≅0.42 kΩ/degree which is a good result in terms of resistance change per degree: 2.1 Ω per 0,005 temperature degree change. The temperature coefficient of resistance is calculated as the relative change of resistance per degree of temperature change, i.e. ≅ −1.2%/degree that well corresponds to early reported data [33].

We then test the sensing e-fabric at heating cycles from room temperature up to 60 °C. This temperature test (Fig. 10) showed that the electrical response of the prototype to temperature cycling is reversible, repeatable and stable in time.

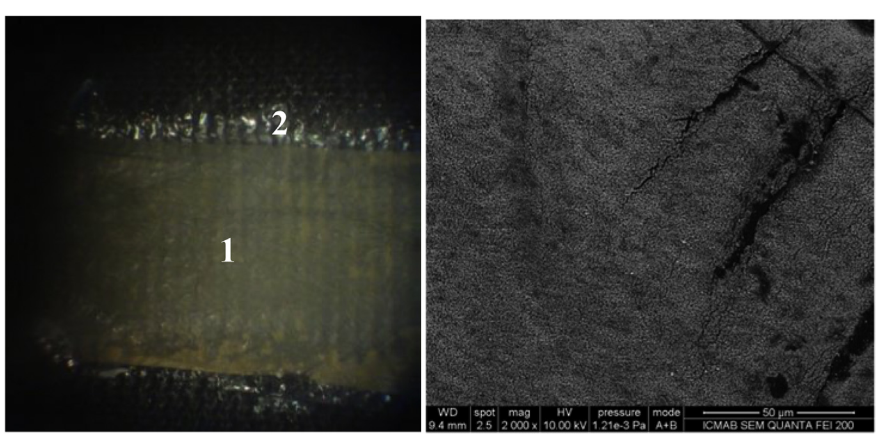
These experimental results ensure that the developed all-organic bi-layer thermistor polycabonate/α'-(BEDT-TTF)$_2$I$_x$Br$_{3\text{-}x}$ is promising for application in smart wearable fabrics. In particular, it guarantees the measurement of small changes in a body temperature fluctuations.

3.3. Interfacing BL-film sensor with wireless sensor node

In this section we present a custom made wireless sensor node and its interfacing with the BL-film sensor. We deploy this monitoring system in a real facility and conduct temperature measurements.

3.3.1. Wireless sensor node design

Fig. 11 presents the architecture of wireless sensor node with the BL-film sensor described in Section 2. The architecture consists of four units: sensing, processing, wireless communication and power supply. The processing unit is built around the ATXmega128 microcontroller unit (MCU) with a precise 24-bit Analog-to-Digital Converter (ADC). Main tasks of MCU include the management of sensor operation and sending measured data to wireless communication unit. It is based on ETRX3 wireless modem able to transmit and receive data from the sensor nodes in the network. Wireless modem has some important self-configuration features which



a) b)

Fig. 6. (a) photo image of the one of the parts of the bi layer thermistor ("*1*") that was attached to the rigid impregnated part of the polyester fabric ("2"); (b) the SEM image of the thermistor showing cracks developed on the conductive sensing layer of the BL thermistor being attached to the polyester fabric.

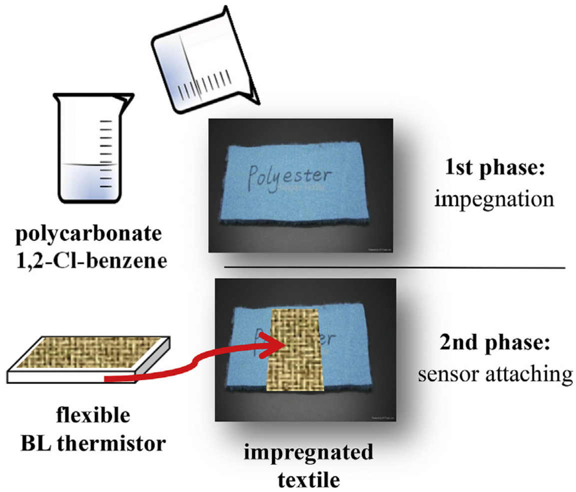


Fig. 7. Schematic presentation of the thermistor processing into textile using an impregnation procedure.

simplify WSN deployment and debugging: WSN configuration, adjustment of TX power. Thermistor is connected to the MCU via ADC and is characterized by quick response time and high sensitivity. Power supply unit is comprised of a 3.6 V, 3200 mAh AA Li-Ion cell and power management based on a DC/DC which provides the sensor nodes with a stable 3 V. Design of wireless sensing devices requires careful consideration of power management solutions [34] for guarantying smooth transition from wired sensing devices [35] to wireless options.

Fig. 12 shows the prototype of the wireless sensors node equipped with the BL-film sensor. The prototype is a compact sensing device (8 × 4.5 cm) with the state-of-the-art sensor and empowered by the wireless communication feature.

Apart from the sensor which was fabricated from scratch (see Section 2), we used off-the-shelf components for assembling the wireless sensor node. Due to autonomous nature of WSN [12] [36], the key criteria for choosing the electronic components is low power consumption to guarantee the WSN long-term operation. The novelty in the design of the thermistor is the application of "self-metallization" technology. It is helpful for depositing the sensors in the empty spaces on the printed circuit board. We note that for testing reasons we used the thermistor deposited on an extra board.

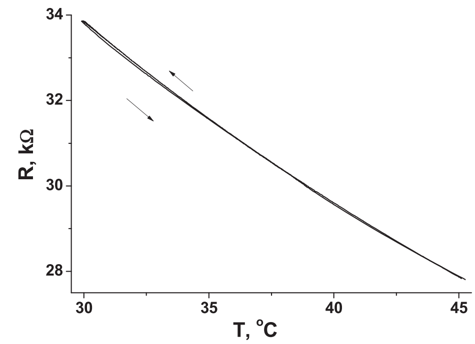


Fig. 9. Resistance temperature of the developed e-textile in the body temperature range.

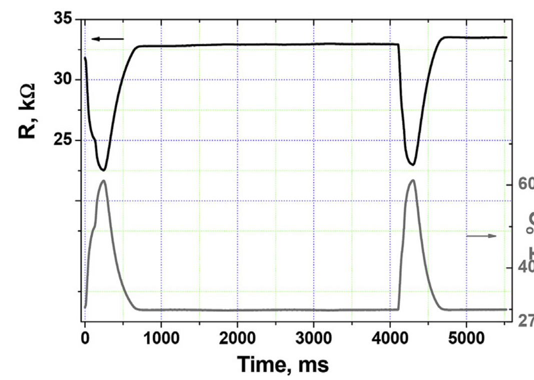
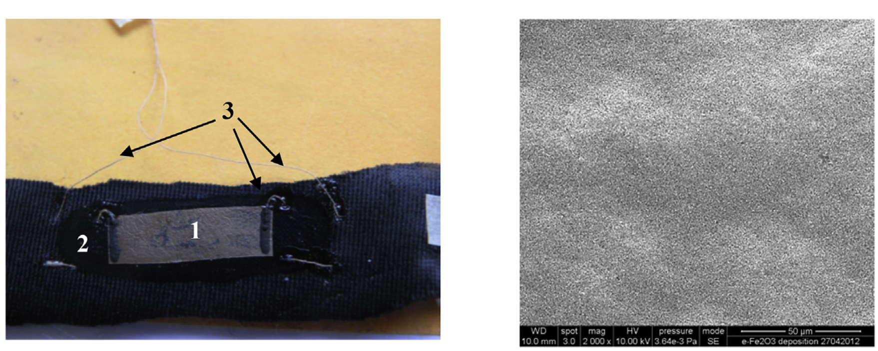


Fig. 10. Response of the electrical resistance of the developed e-textile to temperature.

3.4. Experimental results

We assess the prototype of wireless sensor node in a real office environment within one working day. Fig. 13 shows how the resistance of the BL-film changes with respect to the temperature in the office which is served by a Heating, Ventilation and Air Conditioning (HVAC) system. The goal of this experiment is to evaluate the sensitivity of BL-film. Since the temperature in the office can not drastically change and for ensuring energy efficient operation of the node, it set up to conduct the measurement every 2 min and send the data to a network coordinator located in a corridor.

The results shown in Fig. 13 demonstrate that the temperature in the office fluctuates in the range 21–22 °C over time even though



a) b)

Fig. 8. (a) photo image of the developed prototype of temperature sensing e-textile (1-thermistor, 2-rigid film-like unit prepared by local melting of polyester-based textile, 3 electrical connections); (b) SEM image of the thermistor showing a very good quality of its conducting layer.

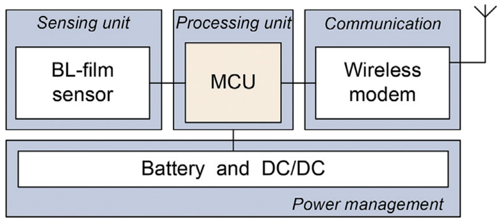


Fig. 11. Sensor node architecture.

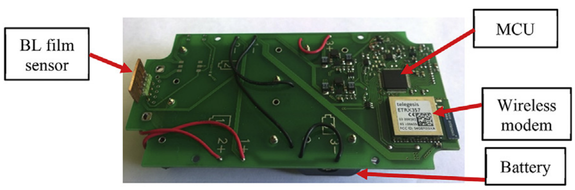


Fig. 12. Prototype of the wireless sensor node with the BL-film as a sensing element.

it is controlled by the HVAC system. For example, the temperature starts growing in the beginning of working day by 9AM. It happens due to arriving of workers to their office. Then the temperature gradually decreases by 1PM which is most likely associated with the lunch time. Afterwards the temperature starts increasing again when the office workers come back. Finally, the office temperature cools down at the end of working day.

4. Conclusions

In this work we have presented the BL thermistor: polycarbonate/α'-$(BEDT\text{-}TTF)_2I_xBr_{3\text{-}x}$ and investigated its conductive sensing layer texture and structure by using SEM and X-ray analysis. Also, we measured the resistance temperature dependence of the thermistor. We have demonstrated that all investigated properties are in agreement with earlier reported results and improve them.

Apart from reporting on the BL thermistor we have presented a new approach to integrating the BL thermistor into textile where the thermistor is attached to the smooth film-like rigid support prepared directly at the polyester textile by its local melting. We discovered that melted part of the textile is much more rigid comparing to the rest of the fabric.

The fabricated prototype can be used as a temperature sensor for detecting the temperatures in the range from RT to 60 °C. According to the experimental results the electrical response to temperature change is reversible, repeatable and stable in time.

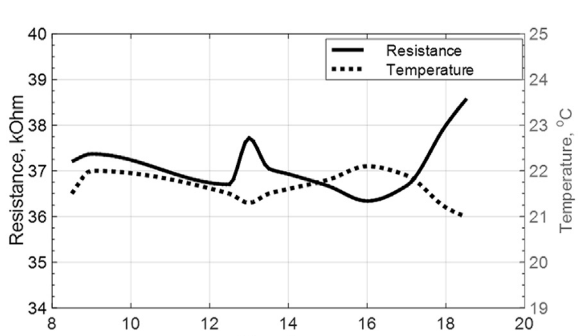


Fig. 13. Experiment showing how the film resistance and temperature change in an office environment during the working hours.

Developed textiles can detect even negligible temperature change with accuracy of 0.005 °C. This results improves one obtained for widely used thermistors where accuracy of a Pt-1000 detector is 0.01 °C.

Finally, we have demonstrated that proposed solution has a number of applications in biomedical and environmental monitoring scenarios by integrating the thermistor with the wireless sensor node for enabling timely delivery of measured data to the user.

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