Investigation of Biodegradable Metals for Green and Sustainable Temperature Sensors

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***Abstract*— The management of electronics waste and the development of sustainable end-of-life strategies are key aspects of the green evolution of the electronics industry. To address this global issue, we implemented thin-film resistance temperature detectors (RTDs) using green sensing metals, such as Mg, Mo, and Zn, and poly-ether ether ketone (PEEK), as a biocompatible, flexible, and thermally resistant substrate. The environmentally friendly RTDs were characterized in a range of temperature, from 25 °C to 70 °C, showing consistent response and average sensitivities of 1.1×10^{-1}%/°C, 7×10^{-2}%/°C, and 5.8×10^{-2}%/°C for Mg, Mo, and Zn, respectively. At a constant temperature 25 °C, the effect of humidity variation from 10% to 90% on the resistance of the sensors was observed to be 2.0×10^{-5}%/relative humidity (RH), 3.4×10^{-2}%/RH, and 5×10^{-3}%/RH, respectively, for Mg, Mo, and Zn RTDs. Furthermore, the sensor's response to mechanical strain was evaluated by bending the devices down to a 10-mm bending radius. In addition, the dissolution of the green RTDs in water allows the reusability of the substrate for a new fabrication batch, minimizing the amount of electronics waste generated. Through this study, a promising solution to environmental concerns, realizing is endowed for realizing temperature sensors, with applications in green and sustainable wearable systems is demonstrated.**

***Index Terms*— Flexible electronics, green electronics, poly-ether ether ketone (PEEK), sustainable thin-film devices, temperature sensors.**

I. INTRODUCTION

FLEXIBLE electronics are gaining much attention due to their excellent mechanical deformability and integrated functionality, which satisfy the demands of a diversified application market [1]. Although these devices have been widely employed in biomedicine and space, as well as in agriculture and military domains, one main concern is represented by the electronic waste generation once their end-of-life is reached [2], [3], [4], [5].

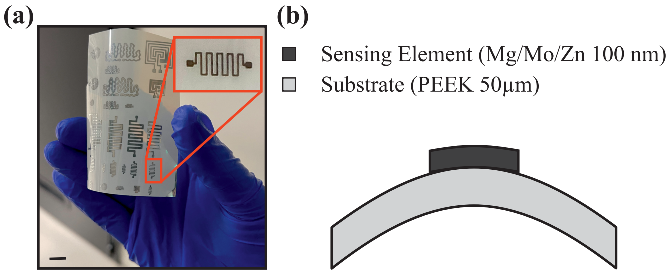

Fig. 1. (a) Array of RTDs on biocompatible PEEK substrate. Inset: the optical image of the temperature sensor under analysis (scale bar: 7.5 mm). (b) Two-dimensional cross-sectional sketch of the flexible and green RTDs.

To realize devices with minimal impact on the ecosystem [1], urgent actions are required, such as the use of innovative materials, which are easily accessible and widely present in nature. In this respect, a broad variety of biodegradable metals, such as Mg, Zn, Mo, Fe, and W, are of interest for their electrical properties, mechanical endurance under stress, as well as controlled dissolution in green media [6].

Similarly, for the choice of sustainable, yet reliable substrates, polymers have been chosen as suitable alternatives. In this respect, the most widely used carriers are polyimide (PI) [7], polydimethylsiloxane (PDMS) [8], and polyethylene terephthalate (PET) [9]. Alternatively, poly-ether ether ketone (PEEK) is a thermoplastic matrix with excellent thermal and chemical resistance. Its excellent heat, radiation, chemical, and impact resistance make it a valuable coating material in industries such as electronics, medical, aerospace, energy, and paints [10].

Among the wide plethora of electronics devices, temperature sensors, and especially resistance temperature detectors (RTDs), have a wide variety of applications, including food logistics [2], [11], robotics [12], and physiological parameters monitoring [6], [13], [14], requiring reliable functionality [8], biocompatibility [15], and mechanical flexibility [16]. Mg metal-based RTDs were already fabricated by sputtering on Ecoflex [17], and polylactic acid (PLA) [14] substrates and zinc-based RTDs were printed on paper substrate [18]. Nevertheless, these biodegradable substrates show several drawbacks, including high moisture uptake and low thermal stability, limiting their use for high-temperature fabrication

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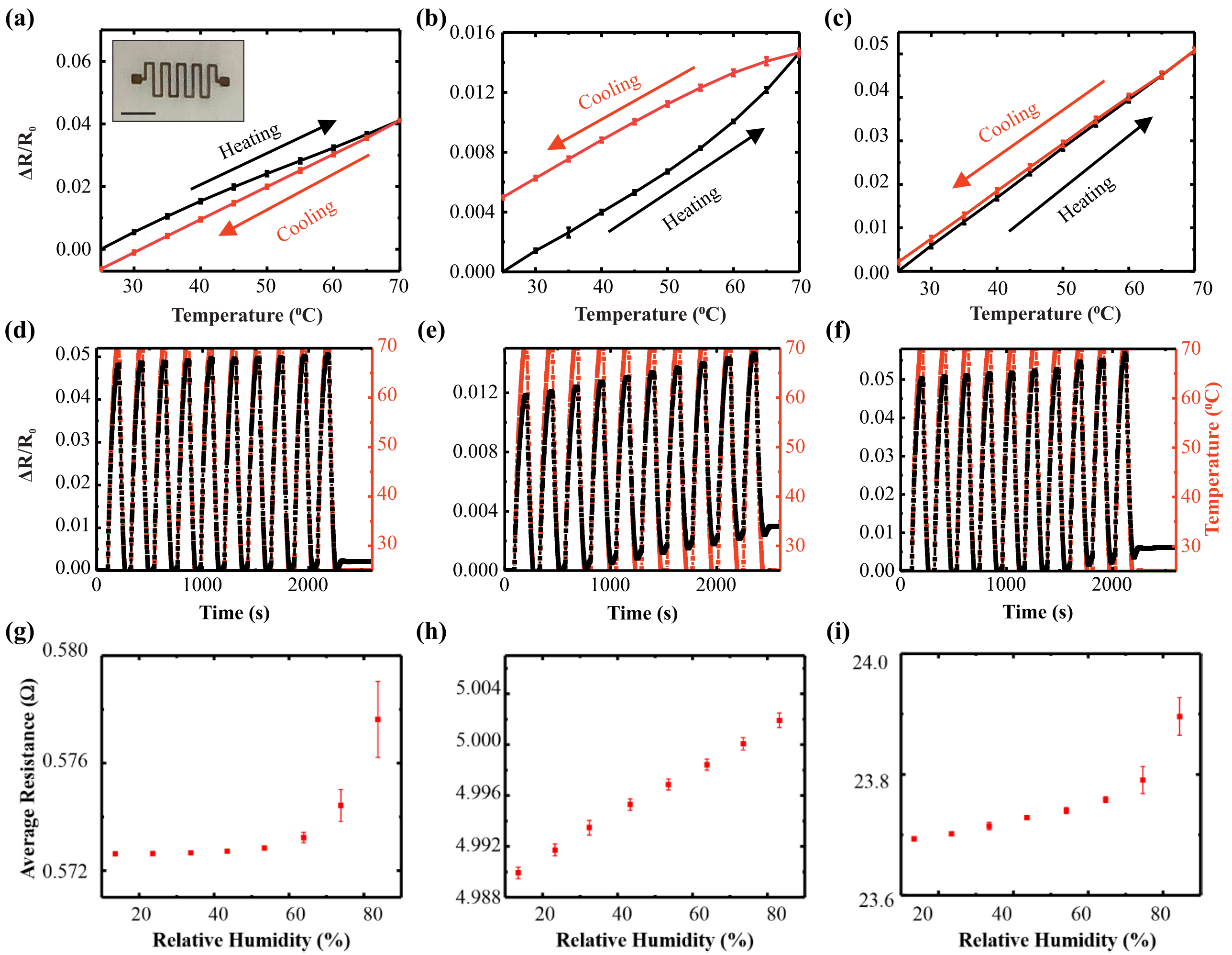

Fig. 2. Temperature and humidity responses for thin-film RTDs. Normalized resistance with standard deviation as a function of temperature for (a) Mg RTD (inset: sensor during measurement, scale bar: 7.5 mm), (b) Mo RTD, and (c) Zn RTD. Cyclic measurements from 25 °C to 70 °C for (d) Mg RTD, (e) Mo RTD, and (f) Zn RTD. Normalized resistance as a function of humidity variation for (g) Mg RTD, (h) Mo RTD, and (i) Zn RTD.

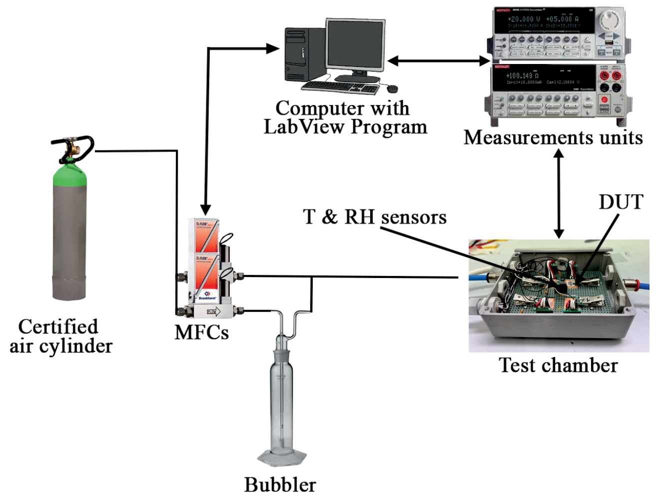

Fig. 3. Schematic of the experimental setup used for electrical characterization of the developed devices.

processes. Here, a comparative and thorough analysis of the electrical, mechanical, and dissolution performance of RTDs produced with biodegradable metals, is presented. PEEK was employed as substrate for the fabrication as this bio-compatible plastic overcomes constraints of other biodegradable substrates, offering exceptional mechanical and thermal capabilities [19]. The implementation of the bio-compatible PEEK substrate presented in this study addresses the critical limitations of Ecoflex paper and PLA substrates, offering a more robust, thermally stable, and environmentally friendly alternative for thin-film electronics fabrication. Here, Mg-, Mo-, and Zn-based temperature sensors were fabricated, showing functionality in the temperature range from 25 °C to 70 °C and in the humidity range from 10% to 90%. In addition, device flexibility was demonstrated down to 10-mm bending radii. Finally, to support temporary usage of the sensors with minimum waste generation, Mg, Mo, and Zn RTDs were fully degraded in tap water within 7 min, 7 h 30 min, and 3 h 37 min, respectively, supporting the reuse of the flexible substrate for a new fabrication batch. This article discusses the fabrication of thin-film electronics utilizing degradable sensing elements on a bioplastic PEEK substrate. At the end of the device's lifecycle, the structures atop the substrate are fully degradable, allowing the substrate to be repurposed for the fabrication of new devices. This approach aligns with the key strategies of the reduce, reuse, and recover (3R) principles of waste management, and thus significantly mitigates the environmental impact of waste from electrical and electronic

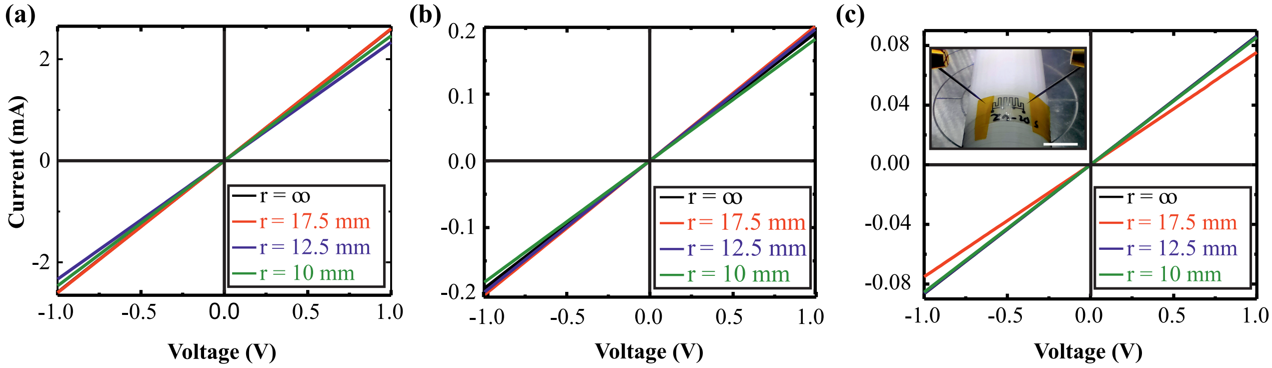

Fig. 4. Effect of the bending on the normalized resistance of the (a) Mg RTD, (b) Mo RTD, and (c) Zn RTD. Inset: sensor bent to a radius of 10 mm (scale bar: 7.5 mm).

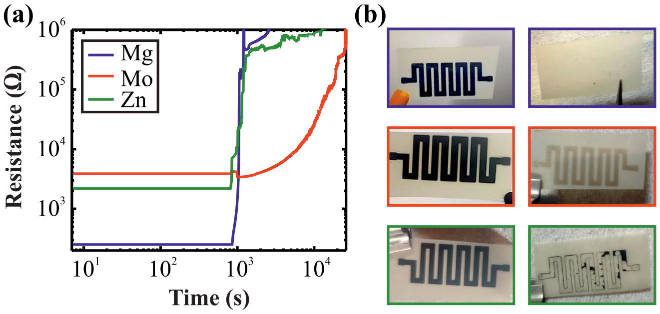

Fig. 5. (a) Variation in electrical resistance over time for the green RTDs, dipped in tap water at 812 s. (b) Optical images of each sensor before (left column) and after (right column) dissolution.

equipment (WEEE) [20]. Additionally, it supports the 17 sustainable development goals of the United Nations, which aim to make our planet sustainable and safe for future generations. Multiple structures are fabricated and characterized to evaluate the sensors' reproducibility, using different sensor geometries and materials. Multiple measurements are performed to evaluate the sensors' reproducibility, using different sensor geometries.

II. FABRICATION

A 7 × 7 cm flexible 50-μm-thick PEEK (BIEGLO GmbH) foil was employed as a flexible carrier, as shown in Fig. 1. To ensure substrate cleanliness, sonication with acetone and isopropyl alcohol was performed for 5 min each, and substrates were dried in an oven at 200 °C for 24 h. Metal shadow masks were used in a sputtering system (Kenosistec, KS 500C) for the deposition of the sensing elements. For the dc sputtering process, 99.9% pure metallic targets of Mg, Mo, and Zn were utilized. The process was carried out at a pressure of 3×10^{-6} bar in an Ar environment at room temperature. The resulting thickness for each sensing material was 100 nm. The thickness of the metal layers was monitored using Dektak 3 Stylus Profilometer.

III. RESULTS

The performance evaluation of the fabricated sensors was conducted through four separate experiments: 1) ramp and cycling measurements of the RTDs from 25 °C to 70 °C, with constant relative humidity (RH) of 30% for 36 and 98 min, respectively; 2) response to RH variation from 10% to 90%, while keeping the temperature fixed at 25 °C for 33 min; and 3) mechanical and 4) dissolution tests, to validate sensor bendability and assess degradability.

A. Electrical Performance

Ramp measurements were conducted on the three RTDs [see Fig. 2(a)–(c)]. The electrical characterization was conducted in a custom-made setup consisting of a sealed chamber, mass flow controllers, dedicated data acquisition system, including a high-performance digital multimeter (Keithley Model 2000 with a 6 1/2-digit), a measuring unit (Keithley Series 2600B), and developed LabVIEW software, maintaining a constant RH of 30%. To increase (heating cycle) and decrease (cooling cycle) the temperature between 25 °C and 70 °C (step of 5 °C), peltries module integrated with PT100 temperature sensors was used to locally heat and cool the sensors. The modules were controlled using the PID function of LabView. Commercial RS Pro Silver Conductive Paint (123-9911, 20-g TBC, ZP) was employed to extend the electrical contacts of the sensors. The schematic of the acquisition setup is reported in Fig. 3. The average and standard deviation resistance values of three individual RTDs made of each green material (Mg, Mo, and Zn) with 2.5 mm/7.5 mm width over length ratio at the beginning of the experiment were 573 ± 0.12 Ω, 4.9 kΩ ± 0.04 Ω, and 23.9 kΩ ± 2.0 Ω, respectively. From this analysis, the sensitivity was calculated for each sensor using the following equation:

$$\text{Sensitivity} = \frac{(R - R_0)}{R_0(T - T_0)} \tag{1}$$

where R is the measured resistance, R_0 is the initial resistance, T is the current temperature, and T_0 is the initial temperature of 25 °C [21]. After a complete heating-cooling cycle, the sensitivities of Mg-, Mo-, and Zn-based RTDs were determined to be 1.1×10^{-1}%/°C, 9.2×10^{-2}%/°C, and 2.9×10^{-2}%/°C during heating, and 1.1×10^{-1}%/°C, 2.3×10^{-2}%/°C, and 1.1×10^{-2}%/°C during cooling. These sensitivities, as well as the hysteresis reported for each sensor [see Fig. 2(a)–(c)], are in line with the performances of these sensing materials [5], [14], [17], [22]. To ensure the reliability of the employed sensors, ten heating-cooling cycles were performed changing the temperature between 25 °C and 70 °C, as depicted in Fig. 2(d)–(f), monitoring the resistance variation over time. Besides the reliable functionality of all the sensors during

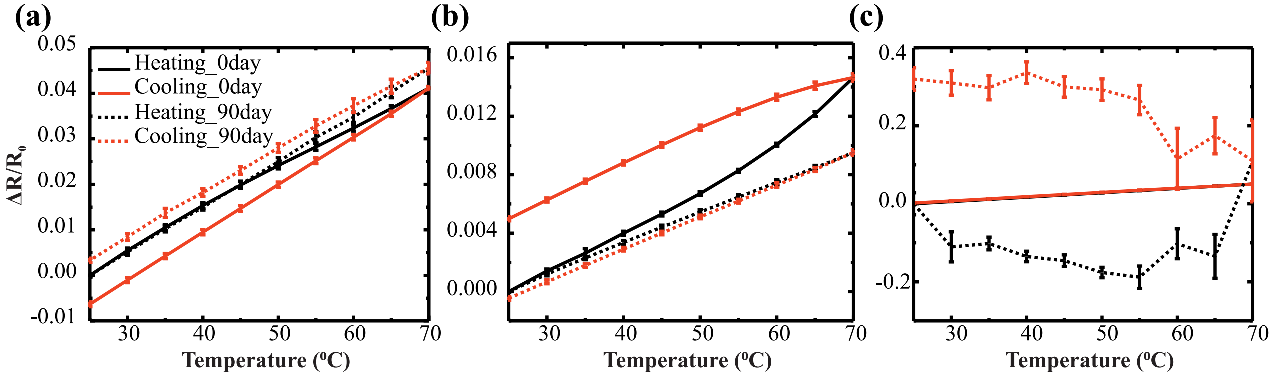

Fig. 6. Normalized resistance as a function of temperature for (a) Mg RTD, (b) Mo RTD, and (c) Zn RTD; measurement was taken after fabrication at zero day, and the second measurement was taken after three months of fabrication.

the temperature cycling, no significant resistance degradation is denoted from the collected values at the beginning of the experiment (571 Ω, 4, 9 kΩ, and 24.6 kΩ for Mg, Mo, and Zn, respectively) and the ones at the end (572 Ω, 5.0 kΩ, and 24.8 kΩ for Mg, Mo, and Zn, respectively). For Mo and Zn cycling tests [see Fig. 2(e) and (f)], a resistance drift of 2% and 0.8% is denoted at temperature equal to 70 °C, respectively. Further studies will be performed to evaluate whether this variation is due to contact loss, device aging, or material degradation.

To evaluate the sensors' responses at different humidity levels, the RTDs were kept at constant temperature of 25 °C, and the RH was swept from 10% to 90% [see Fig. 2(g)–(i)]. Two distinct regions are denoted while characterizing the Mg- and Zn-based RTDs. First, a linear trend is reported up to 52% humidity with rates of 6.6×10^{-4}%/RH for Mg and 5×10^{-3}%/RH for Zn; whereas, in high humidity condition (75% $\leq$ humidity $\leq$ 90%), a steep variation of the resistances is observed, resulting in an average rate of 6.4×10^{-2}%/RH and 4.8×10^{-2}%/RH, as shown in Fig. 2(g)–(i). These sensible variations at high humidity were due to the high response to the water concentration of these metals.

The resistance of the sensor element fluctuates with water concentration in RH. When this parameter is changed from 10% to 90%, the amount of water in the air around the sensor increases, which causes dew to form on the sensing element's surface. Water also reacts with the metal surface of the sensing element to form oxides, which in turn causes conductivity to degrade [23], [24].

On the other hand, a linear response is reported for the Mo-based sensors, while increasing the humidity in the range of 10%–90%, with a rate of 3.4×10^{-2}%/RH, as depicted in Fig. 2(h).

As the resistances of the sensors were affected by the level of humidity, the sensors worked best in environments where there was little variation in humidity [25].

However, the Mg- and Zn-based RTDs could still be used to measure humidity in constant temperature conditions, particularly in the RH range of 75%–90%, where they showed a higher rate of resistance variation with humidity. Mo-based RTDs, on the other hand, could be utilized to measure humidity in the RH range of 10% to 90%. In Fig. 2(g)–(i), the sawtooth shape of the plots is attributed to the stabilization time used during the characterization (33 min) and water absorption. Future works will evaluate the use of proper passivation layers, to achieve reliable temperature response at any humidity level, as well as functionality as a humidity sensor at different temperatures.

B. Mechanical Properties

Bending tests were conducted to evaluate the mechanical stability of all three temperature sensors. These experiments were performed by wrapping the sensors around rods of different radii using Kapton tape (Catime, 301-10233-01SEA00). As shown in Fig. 4(a)–(c), I–V measurements of each sensor were performed in a probe station at different conditions: flat, bent to a radius of 17.5, 12.5, and 10 mm. Strain values of 0.1% for 17.5-mm bending radius, 0.2% for 12.5-mm bending radius, and 0.3% for 10-mm bending radius were calculated for the devices fabricated on PEEK [26]. The measured resistances of Mg, Mo, and Zn sensors with 2 mm/4 mm width and length ratio in flat (382 Ω, 5.2 kΩ, and 11.2 kΩ) and reflattened conditions (383 Ω, 8.2 kΩ, and 11.6 kΩ) demonstrate no loss of functionality due to mechanical strain.

C. Transient Properties

To assess the temporal functionality of our green RTDs, dissolution experiments were performed on the sensors in normal tap water. For this purpose, the electrical resistance of each sensor was monitored over time. For the three sensors, a stabilization time of 812 s was applied, before immersion in tap water [see Fig. 5(a)]. The Mg-based sensor reached resistance $\geq$1 MΩ within 7 min and completely dissolved in 15 min [see Fig. 5(a) and (b)]. Next, the dissolution of Zn RTD resulted in the loss of conductivity after 3 h 37 min. On the other hand, the Mo-based device showed the longest functionality up to 7 h 30 min before reaching a measured resistance $\geq$1 MΩ.

These metals exhibit dissolution characteristics in water. Multiple studies demonstrate that these substances meet the criteria for biodegradability and are classified as biodegradable and biocompatible metals [27], [28], [29], which is proving that trace and residue of Mo and Zn after dissolution process are safe.

Metal hydroxide and oxide [27] are formed when the metal dissolves in water, and these compounds can be recovered using the chemical electrolysis process [30]. Among these

RTDs (Mg, Mo, and Zn), Mg shows the best performance in terms of flexibility and electrical stability, making them suitable for wound healing monitoring during orthopaedical implants. Mo is sensitive to humidity changes, making it suitable for breath monitoring and bioimplant [27], [28], [29].

To evaluate the lifetime of the RTDs, the performance of the RTDs was evaluated after three months. These RTDs, fabricated using metals Mg, Mo, and Zn, were initially characterized at the time of fabrication. During this characterization, the sensor was exposed to humidity levels of 10%–90% and temperatures ranging from 25 °C to 70 °C [see Fig. 2(d)–(i)]. After three months of being kept under normal conditions, the same sensors were recharacterized through a heating-cooling cycle, to assess their lifetime and aging, as shown in Fig. 6. The Mg- and Mo-based sensors [Fig. 6(a) and (b)] are still operational, with only minor variations in performance. However, sensor Zn has degraded rapidly due to surface oxidation [31].

IV. CONCLUSION

The fabrication of flexible and green RTDs on a biocompatible PEEK substrate is reported. The use of Mg, Mo, and Zn as the sensing elements allows temperature monitoring from 25 °C to 70 °C, and humidity response from 10% to 90%. In addition, mechanical flexibility down to 10-mm bending radii, as well as sensor solubility in water as an end-of-life strategy, was demonstrated. This could support the reuse of the flexible substrate for a new fabrication run, aiming at a drastic reduction of electronics waste generated. The implementation of reliable, environmentally friendly, and yet flexible temperature sensors on an innovative and reusable PEEK substrate represents a first step toward a new class of sustainable electronic devices for applications in smart agriculture, bio-implants, and food industries. Future works will involve the demonstration of a fully circular technology, for a new generation of thin-film electronics, aiming at no electronic waste creation and low carbon footprint.

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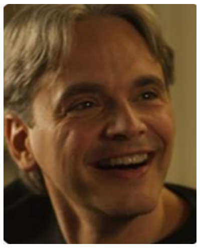

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He was a Post-Doctoral Fellow with the Nanomaterials Engineering Research Group, ETH Zurich, the Free University of Bozen-Bolzano, Bolzano, Italy, and the Sensing Technologies Laboratory, Bozen-Bolzano, Italy. Until October 2022, he was an Assistant Professor with the Free University of Bozen-Bolzano. In November 2022, he joined the University of Modena and Reggio Emilia, Modena, Italy, as a Tenure-Track Professor. His main activities include the design, fabrication, and characterization of flexible and sustainable sensors and devices, for wearables and green electronics.

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