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Preliminary Results on Carbon-Based Thermistors Produced by Aerosol Jet Printing

Tiziano Fapanni
Department of Information Engineering
University of Brescia
Brescia, Italy
tiziano.fapanni@unibs.it

Stefano Bellotti
Department of Information Engineering
University of Brescia
Brescia, Italy
s.bellotti004@studenti.unibs.it

Michela Borghetti
Department of Information Engineering
University of Brescia
Brescia, Italy
michela.borgetti@unibs.it

Emilio Sardini
Department of Information Engineering
University of Brescia
Brescia, Italy
emilio.sardini@unibs.it

Mauro Serpelloni
Department of Information Engineering
University of Brescia
Brescia, Italy
mauro.serpelloni@unibs.it

Abstract— **Electronics became pervasive in the production sites generating the now-occurring fourth industrial revolution. In this paradigm, different information technologies are employed in order to achieve better control over the overall production process and better plan each maintenance intervention. In the monitoring processes, different physical quantities are constantly monitored with sensors connected to the acquisition electronics and therefore to the communication network. Among those, temperature sensors are relevant since they can provide a set of information useful to track the production process and the workers' wellbeing, and to compensate for the output of other sensors. Thus, in this work, the behavior of a carbon-based ink on different substrates was evaluated to produce thermistors. The devices were evaluated using both an oven and a climatic chamber and both positive (PTC) and negative (NTC) temperature coefficients were observed. The devices resulted promising with sensitivities comparable with the ones of standard Pt100 sensors in selected parts of the temperature measurement range.**

Keywords— temperature sensors, printed electronics, printed thermistors, carbon-based sensors

I. INTRODUCTION

Thanks to the technological improvement, electronics became pervasive in production sites generating the now occurring fourth industrial revolution. According to this paradigm, factories and companies integrate both industrial automation and information technologies in order to achieve better control over the overall production process improving its repeatability and reproducibility, as well as to increase efficiency in the maintenance intervention planning [1], [2]. In order to achieve the abovementioned goals, a whole set of technologies are currently investigated to handle, aggregate and elaborate the data through big data and machine learning techniques. On the other hand, another open issue is related to the collection of the data itself by means of sensors. Indeed, sensors need to be embedded in (often) pre-existing devices and machines in order to monitor their behavior and efficiency by cyber-physical systems. Those systems are often based on different kinds of variables such as accelerations, vibrations, torques, adsorbed power and temperature [3]. Temperature in particular is interesting since it is a relevant parameter that can significantly change according to friction between mechanical components, period of the year and hour of the day, potentially affecting both the production process as well as the health and safety of the workers. Moreover, temperature measurement is important since it is often a variable of influence for other kinds of sensors and thus it may interfere with the lecture of other relevant quantities [4]–[6]. In the literature, temperature sensors based on different transduction principles are thus proposed. Among those, resistive temperature detectors (RTD) are quite widespread thanks to their measurement range, quick response times, accuracy and repeatability [7]. Other types, such as the thermocouple-based ones, are able to measure variations in the thermal flux (e.g. difference in temperature) between two points, but require knowing the temperature at the reference junction and therefore this is not feasible in many wearable applications [8]. Positive and negative temperature coefficient thermistors (PTC and NTC respectively) have non-linear relationships with temperature that can be interesting to achieve high sensitivity without introducing expensive instrumentation. Different techniques have been explored in the literature for the development of thermistors and, in general, temperature sensors. Among the classical techniques, both thick film and microfabrication are widely used and accepted in industry. On the other hand, printed electronics techniques are lately arising since they allow a quick development process, are suitable in a plethora of applications thanks to their flexibility in the use of different functional materials and substrates, which can also be non-planar. [7] In this work, the possibilities offered by printed electronics are explored for the development of fully-printed carbon-based temperature sensors with a wide set of possible applications. In particular, aerosol jet printing (AJP) was selected for this study thanks to its fully digital and maskless production process that allows a quick and cheap prototyping that is ideal in the research frame. AJP bases its working principle on three gas flows that allow the generation of an ink mist through the atomizer (ATM) flow. The particles that compose the mist are then selected according to their size using a pressure difference imposed by an exhaust flow (EX). Lastly, the mist is focused on the substrate through a nozzle by means of a sheath flow (SH) [1]. The devices were fabricated on different plastic and metallic substrates and then characterized in temperature.

II. MATERIALS AND METHODS

A. Sensor Design and Process Considerations

Among the possible materials that should be taken into account in the sensor design, three different classes should be taken into account. Substrates are the base where the device will be fabricated and provide the main mechanical characteristics of the device. The functional material acts as the transductor and should be sensitive to the physical quantity of interest. Lastly, a coating layer can be added to shield the printed device from environmental agents and to prevent corrosion, scratching and the overall aging of the device. As regards the substrates, mostly plastic materials are reported in the literature to be feasible for temperature sensors [11] thanks to their reduced cost and their dielectric behavior. Metals on the other hand are more durable and with superior mechanical characteristics that make them ideal in specific application fields [12]. In this frame, five different substrates were selected to be tested. Polyether ether ketone (PEEK), acrilonitrile butadiene styrene (ABS) and polyimide (Kapton) were selected as plastic substrates, while two aluminum alloys (AlSi7Mg and HE30TF) were selected among the metallic ones. As the sensing material, the EXP2652-28 ink (Creative Materials Inc., Ayer, MA, USA) was selected. This material is composed of carbon black and a polymeric resin and its datasheet declares a resistivity of 0.25 Ω·cm. Lastly, as shielding materials two different materials were taken into consideration: a laquer (RS Pro Printed CCT Board Lacquer RS 136-8533, RS Components Sesto San Giovanni, Italy) and a silicon-based resin (RS Modified Silicone Conformal Coating Aerosol RS 494-714, RS Components Sesto San Giovanni, Italy).

Using the abovementioned materials, the proposed thermistors were designed to be a serpentine of 174.5 mm with two rectangular pads of 2 mm x 1 mm to easily connect the devices to the measurement instrumentation. The target linewidth selected is 200 µm that can be easily fabricated with the chosen technology. This geometry was chosen for these preliminary samples, but AJP permits high flexibility in the geometry design.

In order to evaluate the possible different resistances obtained with this design setup, a preliminary set of sensors was printed to tune the process parameters and to evaluate the thickness of the printed tracks considering changing the number of stacked printed layers. The profile of the printed tracks of each sensor was measured through an Alpha-Step IQ Surface Profilometer (KLA-Tencor, Milpitas, CA, USA) on three different positions. The collected data were processed in MATLAB to estimate the average linewidth, thickness and section area of the tracks. The achieved information was then used to calculate the average resistivity of the material along with the resistance of the sensors that was measured using an HP34401A (Hewlett-Packard, Palo Alto, CA, USA) multimeter. After those preliminary evaluations, the process parameters (shown in Table I) to be used in the production of the final devices were selected.

The first step of the production process was the cleaning of the substrates to ease the adhesion between the ink and the substrate removing all the impurities that may be present on the surface. Then, on the aluminum-based substrates a bi-component epoxy resin (193.R7042, Vernici Caldart S.R.L., Bellusco, Italy) was deposed in order to provide a dielectric layer where the sensor can be deposed. This layer was then cured for 30 minutes at 80 °C before deposing the functional carbon ink by AJP according to the previously described design and process parameters. After the printing phase, the printed samples were then oven-cured at 175°C for 10 minutes for all the samples but the ones on ABS that were cured at 100°C for 10 minutes to avoid damaging the substrate material. The sensors were then wired using a conductive epoxy CW2400 (Chemtrionics, Kennsaw, Georgia, USA). Lastly, the coating layer was spray coated on the top of the printed tracks and then cured according to the specifications provided by the manufacturers. A total of 30 sensors were produced in order to propose a minimum of 3 sensors for each substrate-coating combination. An example of the fully produced samples is shown in Fig. 1. Then, the resistance of the produced sensors was evaluated using an HP34401A (Hewlett-Packard, Palo Alto, CA, USA) multimeter in order to assess the goodness of the overall production process.

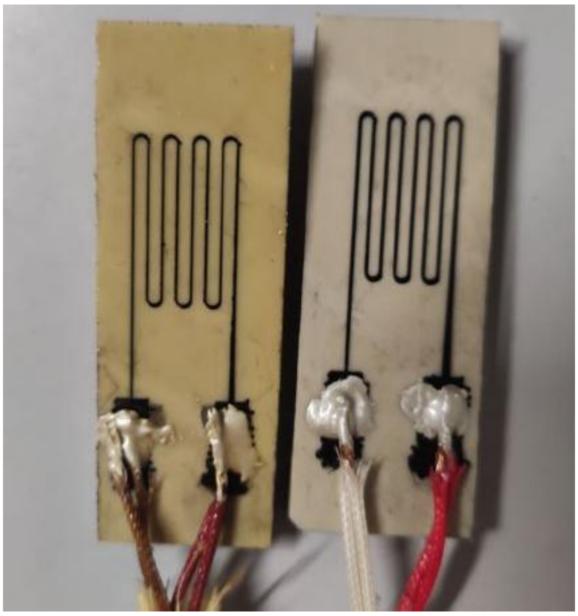

Fig. 1. Example of the produced samples on PEEK substrate coated with the two coatings: resin (left) and lacquer (right). It can be note that the resin changes slightly the color of the overall material.

B. Evaluation in Temperature

The evaluation in temperature was performed at first using a ThermoScientific VACUTherm Oven (Thermo Electron LED GmbH, Langenselbold, Germany) set to perform rising steps of 10 °C in the range [30 ; 100] °C. Those first measurements provided a set of data that required further investigation at lower temperatures. Thus, a climatic chamber UC 150/70 (Advanced Material Testing S.R.L., Limbiate, Italy) was employed to analyze the [-10 ; 40] °C temperature range. In this configuration, the climatic chamber was set to switch between the two extremes of range and waited for 40 minutes. This cycle was repeated 3 times for each sensor.

In both the experimental configurations hereby described, the devices under test were placed in a box to limit the effects of gas convection and fluctuations in humidity. Moreover, a commercial Pt100 sensor with a nominal accuracy of ±0.1 °C was placed in proximity to the devices under test to monitor the local temperature. The hereby described experimental

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setup is shown in the scheme in Fig. 2. All the collected data were processed using MATLAB in order to understand and describe analytically the behavior of the devices.

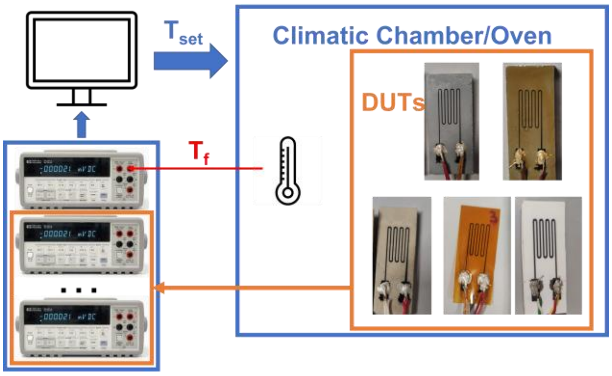

Fig. 2. Scheme of the experimental setup employed to characterize the devices under test (DUTs). The climatic chamber and the oven temperature were set by a computer and then measured in feedback by a standard Pt100 sensor. All the DUTs were measured using a set of multimeters logged by means of a LabVIEW virtual instrument.

III. RESULTS AND DISCUSSION

A. *Preliminary Design Evaluations*

In Fig. 3 the average profiles achieved with the different number of depositions are shown. There, it is possible to observe at first an increase in the average thickness of the produced lines that increases from ≈3 µm with three stacked depositions up to ≈13 µm with nine. The thickness was evaluated as the average of all points that are at least 90% of the maximum thickness of the profile. Increasing the number of layers, however, also increases the coffee ring effect, which results more evident increasing the amount of material in the printed line and introduces undesired peaks. From the measured profiles, it was possible to extract the geometrical features of the line such as the trace width. This was evaluated considering the width of the trace at 10% of the maximum thickness of the profile. Again, it is possible to observe an overall increase in the width that follows a quadratic equation. From these measurements, it was possible to estimate the section area of the tracks approximating it to a trapezoid. Then, in order to evaluate the goodness of the overall fabrication process, the resistance of all the sensors was measured and used along with the previously achieved information to calculate the average resistivity. The quantities that were obtained and used in this calculation are summarized in Table II. The resistivity was calculated by applying the second Ohm's law and found to be 0.254 Ω·cm, independent of the number of deposited layers. Since the resistivity values that were achieved are comparable with the ones provided by the ink manufacturer, the overall process was validated.

TABLE II. PRELIMINARY PROCESS EVALUATIONS USED IN THE DESIGN FLOW TO DEFINE THE PROCESS PARAMETERS

Layer #	thickness (µm)	linewidth (µm)	Area (µm²)	Resistance (kΩ)
3	3.09±0.22	191.0	664.33	772.0
5	5.53±0.87	199.0	1054.84	380.8
7	8.50±1.05	214.8	1758.82	254.6
9	12.77±2.00	238.8	2365.16	173.9

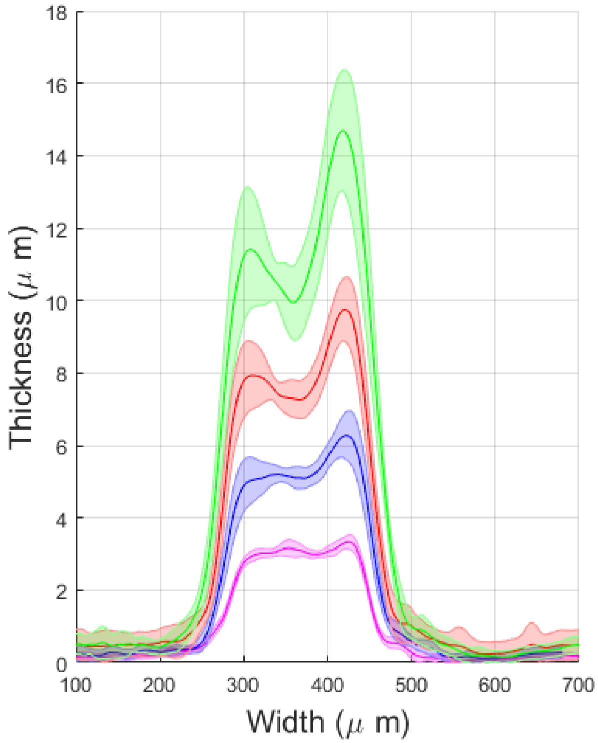

Fig. 3. Average profiles of the printed tracks printed with 3 (magenta), 5 (blue), 7 (red) and 9 (green) successive deposition layers.

B. *Evaluation in Temperature*

The evaluations in temperature were at first carried out in an oven in the range [30 ; 100] °C. The achieved data were processed in MATLAB to obtain a set of temperature-resistance characteristics. As shown in Fig. 4, even though each material had a resistance change at increasing temperature values, the relationship between the temperature and the measured resistance is non-linear and in some cases not monotonic. This is due to the ink characteristics. In fact, the chosen ink is a mixture of carbon black and polymeric resin. The behavior in temperature of such materials is described in the literature [13] where it is shown that the sensitivity can change from positive to negative.

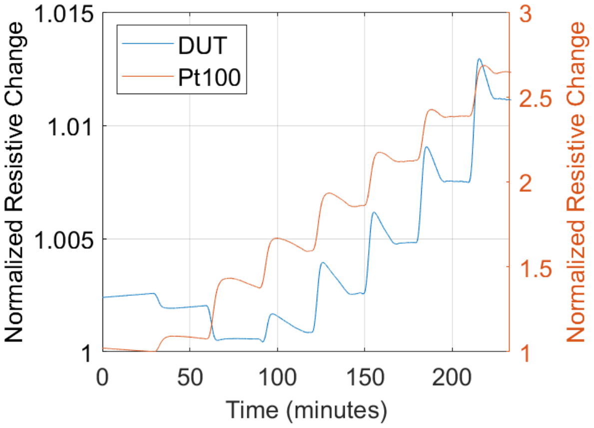

Fig. 3. Normalized resistive change achieved using the oven with one of the printed sensors (blue) compared with a standard Pt100 (red).

The negative temperature coefficient effect (NTC) is due to increased conductivity by tunnel effect [13], while the positive temperature coefficient can be observed in the proximity of the melting point of the resin and it is due to its thermal expansion that increases the distance between the different particles of carbon black [14]. To analyze the overall behavior of the ink and try to obtain the temperature in which the slope of the characteristics goes from negative to positive (Curie temperature, T_C), further analysis was performed on all the sensors using a climatic chamber to increase the temperature range of analysis. In this frame, different behaviors were observed on the different substrates (Fig. 5). In those, it was possible to separate the devices into two different groups. Kapton and aluminum samples (AlSi7 and HE30TF) presented a full change between negative and positive sensitivity even though the aluminum substrates presented a reduced NTC component. The other substrates, on the other hand, seem to show only a sharp change in the sensitivity module before and after a specific temperature for each substrate. Starting from these considerations, the Curie's

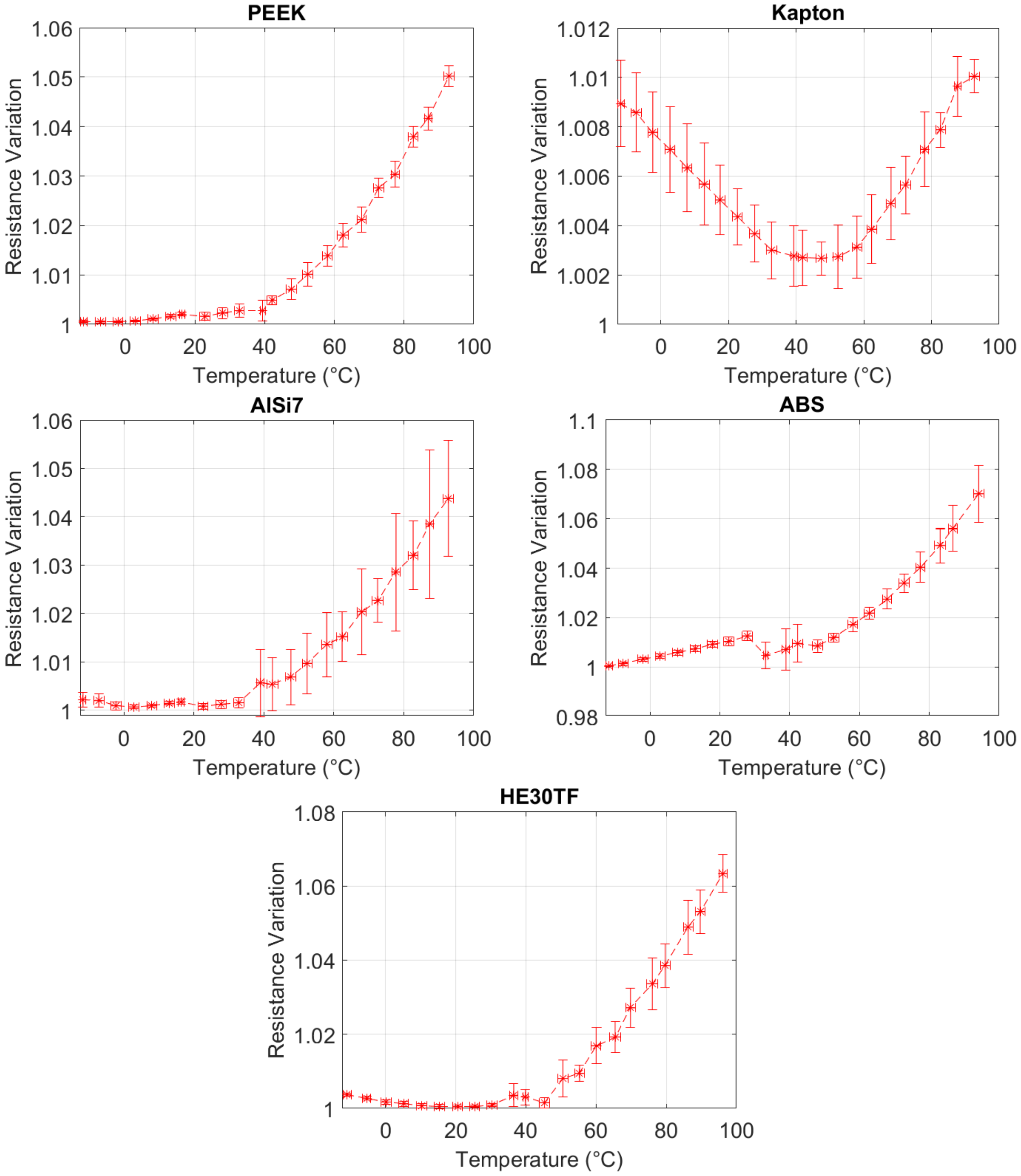

Fig. 5. Normalized resistance changes at different temperatures. The data points represent the mean value with their standard deviation achieved on 6 samples; Dashed lines is the first order hold interpolant.

temperature was evaluated for all the proposed substrates. Interestingly, the aluminum-based substrates achieved values around 28 °C, while all the plastic-based ones presented Curie temperature over 42 °C. This is probably related to the different thermal expansions produced by the different kinds of substrates and how they interact with the functional material. Interestingly, the average sensitivity at temperatures above the Curie's one, are usually one order of magnitude bigger than the ones achieved before T_C and it is comparable to the sensitivity of a standard Pt100 commercial temperature sensor.

IV. CONCLUSIONS

In this work, a preliminary evaluation of the behavior in temperature of a carbon-based ink on multiple substrates for Industry 4.0 and predictive maintenance was explored. The achieved results show how the behavior of the devices is non-linear and substrate-dependent. The former is explained considering the different components (e.g. Carbon Black and resins) that compose the functional ink that presents different overlapping behaviors. Those in some cases were able to change the thermistor from PTC to NTC after the Curie temperature (T_C) that was evaluated on each substrate. Interestingly, the aluminum-based substrates achieved T_C values around 28 °C, while all the plastic-based ones presented Curie temperature over 42 °C. Moreover, the sensitivity at temperatures higher than T_C resulted on average an order of magnitude bigger showing how at high temperature it is possible to achieve a good sensitivity.

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