Sensors and Actuators A 243 (2016) 71–80

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

journal homepage: www.elsevier.com/locate/sna

# Mechanical behavior of strain sensors based on PEDOT:PSS and silver nanoparticles inks deposited on polymer substrate by inkjet printing

Michela Borghetti[a], Mauro Serpelloni[a,*], Emilio Sardini[a], Stefano Pandini[b]

[a] Dip. di Ingegneria dell'Informazione, University of Brescia, Brescia, Italy
[b] Dip. di Ingegneria Meccanica e Industriale, University of Brescia, Brescia, Italy

ARTICLE INFO

*Article history:*
Received 22 February 2016
Received in revised form 16 March 2016
Accepted 21 March 2016
Available online 22 March 2016

*Keywords:*
Strain sensor
Inkjet printing
Silver nanoparticles
PEDOT:PSS
Tensile tests
Mechanical behavior

ABSTRACT

Recently, inkjet printing technology has received growing attention as a method to produce low-cost large-area electronics, sensors, and antennas on polymer substrates. This technology relies on printing techniques to deposit electrically functional materials onto polymer substrates to fabricate electronic components or sensing elements. In this paper, we applied an inkjet printed technology for the development and characterization of films on a polymer substrate aiming at giving design considerations for the optimization of strain sensors or printed electronics obtained by inkjet printing. Two inks were tested over a polyimide substrate, a water-based conductive polymer, PEDOT:PSS, and a silver nanoparticles ink. Their sensing capabilities were investigated under tensile conditions and various strain histories (strain ramp; cyclic loading-unloading tests; application of constant strain over prolonged time) aiming at highlighting the correlation between electrical response, applied strain, time and mechanical histories. Furthermore, the mechanical viscoelastic response of the substrate was investigated under similar strain histories interpreting the results at the light of the substrate deformational characteristics and evaluating its influence.



## 1. Introduction

In recent years, sensors printed on polymer substrates represent an increasing area of research and development due to the growing demand for biosensors [1], artificial skin [2], chemical sensors [3], force [4] and strain sensors [5–9]. In particular, resistive strain sensors on polymeric substrates are employed, in general, for the measurement of forces [6], movements [7] and displacements [8]. Therefore, they are used in many different fields, and not least, in the biomedical field [9]. Novel systems have been recently developed adopting polymers instead of the more conventional strain gauges, leading to sensors based on conductive polymers [10], polymer composites [11], polymer elastomers [12], thermoplastics [12], epoxies [13]. As a benefit, sensors based on polymers allow the use of conventional and convenient polymer processing and post-processing techniques.

Resistive strain sensors are typically manufactured depositing a specific resistive ink on a polymer substrate. The polyimide films are often favored as substrate due to their high durability, the wide range of working temperatures and the stability to environmental factors [14]. Ink deposition can be easily carried out through simple and low cost printing technologies such as inkjet printing. This technique is one of the latest methodologies developed for the implementation of electronic components, and recently it is becoming increasingly popular, as evidenced by its adoption in numerous scientific publications [15–17]. This technique offers the advantage of being fast and relatively inexpensive, therefore suitable especially for the rapid development of prototypes to be tested. It also gives the possibility to deposit inks on various, rigid or flexible, substrates [15]. The main advantage of inkjet printed strain gauges, over the traditional ones, lies in their simple rapid prototyping process, the wide range of electric resistance, its compatibility with the mechanical properties of the material and its built-flexibility for the measurement of large deformations.

Two typical examples of conductive materials adopted as ink for inkjet-printing strain sensors are non-polymeric inks incorporating silver nanoparticles and the intrinsically conductive polymer poly(3,4-ethylenedioxythiophene)-poly(styrene sulfonate) (PEDOT:PSS). As example, a strain sensor based on silver ink, which could play a strategic role for electrodes and sensing structures due to the extremely low resistivity and mechanical properties, was recently proposed by Andò et al. [18]. On the other hand, piezoresistive sensors based on PEDOT:PSS can be used

* Corresponding author.
*E-mail address:* mauro.serpelloni@unibs.it (M. Serpelloni).

http://dx.doi.org/10.1016/j.sna.2016.03.021
0924-4247/

as strain sensors for various applications, also in the biomedical and health fields, due to its high biocompatibility [19]. Thermal and electrochemical stability and its high transparency [20] make PEDOT:PSS an interesting material, which also presents a certain versatility, such as the possibility of deposition on different substrates. Some examples of sensors manufactured with PEDOT:PSS deposited by inkjet printing are reported in Refs. [21] and [22], providing a characterization of their electrical and mechanical properties. In Ref. [22], a sensor based on PEDOT:PSS is adopted to measure the bending angle of knee flexion and wrist rotation. The results suggest that bending sensors fabricated by inkjet printing adopting PEDOT:PSS or silver nanoparticles ink offer many advantages with respect to other systems for human movement monitoring (inside and outside the human body), such as, the small footprint, low cost and versatility. However, to obtain satisfactory results from the use of these sensors, it is necessary to characterize properly their mechanical and electrical response, so to properly correlate, at the light of the substrate deformation, sensor response and movement.

In this paper, we present the results of tensile testing protocols, properly designed to evaluate the response of PEDOT:PSS and silver nanoparticles inks deposited by inkjet printing under monotonic and cyclic strain histories. The aim is to improve the knowledge on the design of strain sensors on polymer substrate or printed electronics manufactured by inkjet printing, with particular attention for what concerns the relationship between applied strain and measured electrical resistance, and stability of the response over time and for prolonged cyclic employ.

## 2. Preparation of sensing films

Two types of sensors were prepared and investigated, they were obtained depositing conductive inks on poly(imide) film (Kapton® HN DuPont [23]) by a proposed inkjet printing technique. For the first sensor type, the conductive layer is an ink based on silver nanoparticles. In particular, the ink is commercialized by Sigma Aldrich under the trade name 736465; it has a volume resistivity of 11 μW/cm, a nanoparticles content of 30–35% and a particle size less than 50 nm [24]. For the second type of sensor, the conductive layer is obtained by the deposition of PEDOT: PSS, Orgacon IJ-1005, marketed by AGFA-Gevaert [25] having 0.8% of solid part.

The employed film presents a thickness of 25 μm. The choice of polyimide was mainly motivated on its high adhesion to the chosen inks and biocompatibility [26]. Furthermore, it presents an enhanced thermal stability, when exposed to relatively high temperature, thanks to its high glass transition temperatures (about 360 °C). This is an important feature for the substrate, since sensors manufacturing process involves thermal treatment to promote the evaporation of the solvents. The thermal treatment helps towards increasing the conductivity of the ink and improving adhesion to the substrate: the moderate thermal expansion of the substrate on the range of temperature allows avoiding significant geometrical distortions of the substrate.

The sensors were made using the proposed inkjet printing method. A low-cost desktop printer has been used for the film depositions. The selected printer is one of the cheapest models of Epson, XP-215, having four separate cartridges with 128 nozzles for black and 42 nozzles for each color, and a print resolution up to 5760 × 1440 dpi.

Before the ink printing, the sheet of polyimide (155 × 21 mm$^2$) was cleaned by placing it in an ultrasonic bath of acetone for 15 min at room temperature. After cleaning, the strip was dried using dry compressed air. An additional oxygen plasma treatment (Colibrì, by Gambetti) under vacuum was carried out for the PEDOT:PSS-type substrates for 180 s at 35 W RF power in order to improve the adhesion between ink and polyimide. In fact, the plasma modifies chemical state of the polyimide surface, which becomes hydrophilic.

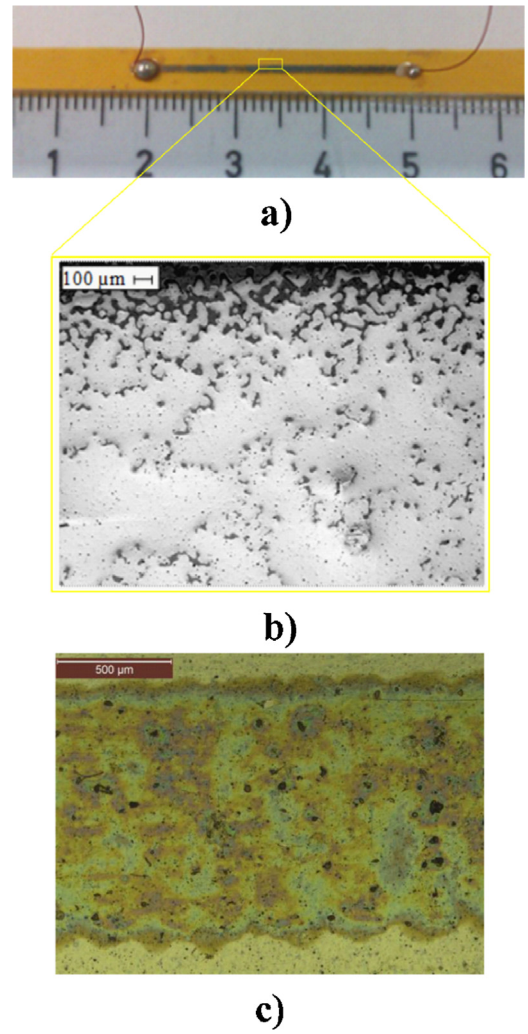


**Fig. 1.** (a) Example of a sensing strip for the tensile tests; observations at the optical microscope of the conductive track (b) for the silver nanoparticles ink and (c) for PEDOT:PSS.

An empty printer cartridge was refilled by the silver nanoparticles or PEDOT:PSS ink. Ink paths were created by repeating the printing process for three times for silver-based sensor and five for the PEDOT:PSS-based sensor, this ensures a good conductivity. After printing each of the layers, the sheet was dried in a static oven for 1 min at 50 °C to prevent the spread of ink with the following printing. After depositing all the layers, the sheet with devices in silver nanoparticles was placed in the oven for 30 min at 150 °C (6 min at 130 °C for PEDOT:PSS). To create contacts a silver paste (Dupont 5028) has been deposited manually and dried at 130 °C for ten further minutes, after which copper wires were soldered onto the silver paste, allowing measuring easily the electrical resistance.

A typical specimen prepared for the tensile testing, representative of the sensor, is represented in Fig. 1a, and is obtained by cutting rectangular strips (155 × 7 mm$^2$) from the printed sheet. This figure shows the sensitive track consisting of a single rectangular stripe of conductive ink, aligned with the specimen length and placed in the mid-span. For all the sensors, the conductive stripe has a nominal length of 30 mm and a nominal width of 1 mm.

We measured the resistance ($R$) of the deposited stripes with a 6½ digit digital multimeter. The average resistance is 6900 Ω of the stripes based on the PEDOT:PSS ink and 30 Ω of the ones based on