



Polycarbonate films metalized with a single component molecular conductor suited to strain and stress sensing applications

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ABSTRACT

The paper reports all-organic strain and stress sensitive films that use electrical monitoring approach. The films were prepared by self-metallizing polycarbonate films with the single component molecular conductor $[\text{Au}(\alpha\text{-tpdt})_2]^0$ (tpdt = 2,3-thiophenedithiolate). It was shown that $[\text{Au}(\alpha\text{-tpdt})_2]^0$ by its nature is able to form metallic solid material with low crystallinity. Electromechanical tests demonstrated that the developed films are strain-resistive materials with advanced elastic properties: their electrical resistance varies linearly with uniaxial elongation up to relative strain being of 1.0% that is about five times larger than that for conventional metals. The gauge factor of the films is 4.4 and stress sensitivity is 30 Ω /bar. The processing characteristics of polycarbonate films, self-metallized with a metallic $[\text{Au}(\alpha\text{-tpdt})_2]^0$ -based layer, make them potentially useful for engineering flexible, lightweight, strain and pressure sensors. Due to electromechanical characteristics these films are suited to strain sensing applications requiring miniature strain control in a wide deformation range.

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1. Introduction

The development of metalized plastics, whose electrical transport properties strongly respond to delicate strain, brings great opportunities in the field of strain, pressure or bending sensors for their applications as human-machine interfaces, intelligent textiles, robotic interfaces and body sensing devices [1–3]. In this context the polycarbonate films covered with a thin layer of an organic molecular conductor are attracting large attention due to their ability to combine properties such as electrical conductivity, high sensitivity to strain (pressure), lightweight, and flexibility

[4–7]. Recently it was also shown that these films can be integrated in textiles [8]. In relation to this topic, the single component molecular conductor $[\text{Au}(\alpha\text{-tpdt})_2]^0$ (tpdt = 2,3-thiophenedithiolate) (Fig. 1), which was early reported from some of us, [9] is very attractive for metallization of polymeric films for two reasons. First its powder-like sample displays some properties of a metallic system with a very high value of room temperature electrical conductivity and, second, by its very nature, the single component conductor $[\text{Au}(\alpha\text{-tpdt})_2]^0$ is able to form a conductive covering layer in a low crystallinity state [9]. An amorphous conducting layer is an important purpose to be achieved because an amorphous state will be able to keep conductive covering layers free from problems associated with polymorphism and grain boundaries [10].

Here we demonstrate that metallization of polycarbonate films with the single component molecular conductor

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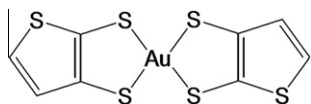


Fig. 1. Chemical structure of $[\text{Au}(\alpha\text{-tpdt})_2]^0$.

$[\text{Au}(\alpha\text{-tpdt})_2]^0$ permits engineering flexible lightweight strain (pressure) sensing organic materials that use electrical monitoring approach. We also present experimental data on the metallic nature of resistance temperature dependence of $[\text{Au}(\alpha\text{-tpdt})_2]^0$ -based covering layers. Additionally, the tensile tests showing that developed films reveal advanced electromechanical and elastic properties are presented and discussed.

2. Experimental

2.1. Materials

Experimental Materials from “Aldrich”, such as pellets of Poly(Bisphenol A carbonate) (Polycarbonate, PC, average M_w ca. 64,000), 1,2-chlorobenzene and dichloromethane, were used as received. Both solvents were of an anhydrous standard (99.8%) packed under nitrogen.

Precursors of the single component molecular conductor $[\text{Au}(\alpha\text{-tpdt})_2]^0$, such as tetra alkyl ammonium salts: $[(\text{C}_2\text{H}_5)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$ and $[(\text{C}_4\text{H}_9)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$, were synthesized as previously described [9].

2.2. Metallization of polycarbonate films with $[\text{Au}(\alpha\text{-tpdt})_2]^0$

A set of 25–30 μm thick films have been prepared on a glass support at 130 °C. The films comprising a 97 wt.% of PC and 3 wt.% of $[(\text{C}_4\text{H}_9)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$ were cast from a 1,2-dichlorobenzene solution that contains a 2 wt.% of PC and 0.1 wt.% of $[(\text{C}_4\text{H}_9)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$. The film surface was treated with vapors of a saturated solution of I_2 in CH_2Cl_2 during 6–8 min at 23 °C. Detailed data on this metallization procedure are presented in ESI (Fig. S1 and Table S1).

2.3. Composition and texture of the covering layer

- The elemental analysis of the covering layers was performed with a scanning electron microscope (SEM) “Quanta FEI 200 FEG-ESEM” with an EDX-EDAX instrument operating at 10 kV. The $[(\text{C}_4\text{H}_9)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$ salt was used as a standard.
- Surface analysis on micro scale was performed using a SEM “Quanta FEI 200 FEG-ESEM”. SEM images of different magnifications were acquired at 20 kV.

2.4. X-ray diffraction (XRD) patterns

To study the structures of the covering layers of $[\text{Au}(\alpha\text{-tpdt})_2]^0$, film samples with 2.3 cm^2 were attached to a glass support and their X-ray diffraction patterns were recorded on a Rigaku “Rotaflex” RU-200B diffractometer in reflection mode with monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 1.540598 \text{ \AA}$); The generator was activated at 50 kV and 80 mA.

2.5. Macroscopic electro-transport properties

The values of the room temperature resistance of BL films as well as their resistance temperature dependences were measured by a standard four-probe dc-method. Rectangular pieces (4 × 2 mm^2) were cut out from the film samples, and four annealed platinum wires with a diameter of 20 μm were attached to a conducting covering layer of BL films with a conductive graphite paste.

2.6. Microscopic transport properties

The resistance and topography were simultaneously mapped in current-sensing mode on a commercial Agilent 5100 scanning force microscope (AFM) by scanning a biased tip that was maintained in contact with the sample surface; I - V responses were acquired by scanning locally the dc current through the tip-sample junction while sweeping the applied bias voltage.

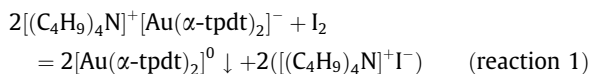
2.7. Mechanical and electromechanical test

Mechanical and electromechanical properties of the films were studied using a 5848 MicroTester with a 1 kg load cell (Instron). Tester was additionally equipped with two electrical contacts and the resistance change under tensile testing was monitored using an Agilent 34410 A; film strips in dimension of ca. 28 × 2 mm^2 and free from physical imperfections, were held between two clamps positioned at a distance of ca. 18 mm. During measurement, the strips were pulled by the top clamp with velocity being 2.0 $\mu\text{m/s}$. Measurements were run in two replicates for each film.

3. Results and discussion

3.1. Metallization of polycarbonate films with $[\text{Au}(\alpha\text{-tpdt})_2]^0$

The metallization procedure used here is closely related to a simple synthetic method developed for surfacing polycarbonate films with conducting charge-transfer salts [4,11,12]. The metallization is a single-stage procedure, which involves two consecutive processes: (1) an internal oxidation of $[\text{Au}(\alpha\text{-tpdt})_2]$ -based precursor introduced in a polycarbonate film (reaction 1) and (2) self-assembly of single component molecular conductor $[\text{Au}(\alpha\text{-tpdt})_2]^0$ as a metallic topmost layer.



In line with the method (ESI, Fig. S1) we first prepared 25–30 μm thick polycarbonate films spiced up with a 3 wt.% of the $[(\text{C}_4\text{H}_9)_4\text{N}]^+[\text{Au}(\alpha\text{-tpdt})_2]^-$ salt that is a precursor for $[\text{Au}(\alpha\text{-tpdt})_2]^0$. To metalize the films with $[\text{Au}(\alpha\text{-tpdt})_2]^0$, we exposed one of their surfaces to vapors of a saturated solution of iodine in dichloromethane. The formation of conductive covering layers was tested by direct current (dc) conductivity measurements. (ESI, Table S1). The composition of the conductive layers was studied by EDX analysis. The S: Au stoichiometry of the layers varies from 6:1 to

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