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Multilayer UV-cured organic capacitors

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ABSTRACT

An organic flexible capacitor was prepared via a multilayer UV-curing process of an acrylic based formulation. The outermost conductive layers were obtained by adding CNTs to PEG-DA resin up to a percolation threshold, found to correspond in between 1 and 2 phr CNTs.

The presence of the filler did not significantly influence the photopolymerization rate, while a slight decrease on final acrylic double bond conversion was observed. An increase of Tg values was measured for the films containing CNTs and attributed to an hindering of the polymer chain mobility. A flexible layer was anyway obtained in the presence of 2 phr CNTs, with a Tg value around −25 °C. The three-layer capacitors were electrically characterized, studying impedance module and phase of the system as a function of frequency. Capacitances values are in the range of the shelf standard available discrete components.

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1. Introduction

Capacitors are important elements in electrical circuits, and in this context, polymer networks offer the obvious advantage of being light in weight, flexible, elastic, and less fragile with respect to inorganic supports such as glasses, ceramics, or metals [1,2]. These features open the possibility to flexible organic capacitor for applications in plastic electronic devices.

One of the promising embedded capacitor materials for organic substrates is a polymer–ceramic composite, which is a ceramic particle-filled polymer [3]. It is a hybrid material that presents both the high dielectric constant of ceramic powders and the good processability of polymers. In the case of ferroelectric composites, for instance, it is possible to couple the high dielectric and piezoelectric features of ceramics with the good mechanical properties and the easy processability of polymers [4,5]. These materials have been exploited in various electronic applications, such as ultracapacitors for energy storage, transducers, piezosensors, hydrophones [6].

Besides microelectronics, polymers can also be applied to sensors, actuators and artificial muscles [7–9]. Very often the polymeric capacitors are prepared by complex multistep methods [10]. On the contrary, UV-curing is a simple technique and usually does not require solvents or temperature treatments.

UV-curing has already been used to prepare polymeric-composites capacitors [11,12]. For instance, very recently our group produced flexible organic capacitors by UV-curing: the capacitors were obtained by graphene–polymer transfer and UV-induced bonding [13]. SU8 resin was employed for realizing a well-adherent, transparent, and flexible supporting layer. The achieved transparent graphene–SU8 membrane presented two distinct surfaces: one homogeneous conductive surface containing a graphene layer and one dielectric surface, typical of the epoxy polymer. Two graphene–SU8 layers were then bonded together by using an epoxy photocurable resin. This strategy allowed to obtain a parallel plate flexible capacitor consisting of two parallel conducting electrodes separated by a dielectric material. The obtained system showed a stable and clear capacitive behaviour.

With a similar idea, in this paper, we report the preparation of a flexible organic capacitor through a multilayer photopolymerization technique. Acrylic formulations were employed in the presence of carbon nanotubes (CNT), up to a percolation threshold, to prepare the two parallel conducting electrodes, while the dielectric layer was the same pristine acrylic resin. The reactivity of both the pristine and loaded systems was investigated and the properties of the assembled capacitor tested.

2. Experimental

2.1. Materials

The following bifunctional acrylic resin was used: polyethyleneglycol diacrylate (PEG-DA, Cytec, Mw ≈ 740 g/mol,

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density = 1.12 g/cm^3). 2 wt% with respect to the acrylic resin of the photoinitiator 2-hydroxy-2-methyl-1-phenyl-propan-1-one (Darocur® 1173, BASF®) was added. The CNTs conductive fillers were gently supplied by ELICARB (Thomas Swan, UK).

2.2. Sample preparation

CNTs were dispersed into the acrylic resin in the range between 1 and 2 phr (per hundred resin). The mixtures were stirred with Ultraturrax until a uniform dispersion was achieved (30,000 rpm for 10 min).

After adding the photoinitiator, the filled formulations were coated on PP substrates in 50 μm thick films and the curing reaction was performed by irradiation with UV lamp (medium pressure mercury lamp Hamamatsu LC8) for 1 min under nitrogen. The UV light intensity was about 30 mW/cm^2. A 50 μm thick layer of pristine acrylic formulation, always containing 2 wt% of the photoinitiator, was coated on the photocured composite surface and UV-crosslinked in the same conditions as the previous layer. Afterwards, another layer (50 μm thick) of the acrylic formulation containing the CNTs was coated on the filled acrylic UV-crosslinked surface and UV-cured. By this way we obtained a polymeric three-layer conductive–dielectric–conductive acrylic UV-cured material of approximately 150 μm thickness. A schematic representation of the preparation method of the capacitor is reported in Fig. 1.

2.3. Characterization

The kinetics of the photopolymerization was determined, both for the pristine and filled formulations, by real time FT-IR spectroscopy employing a Thermo-Nicolet 5700 instrument. The liquid formulations were coated onto a silicon wafer with a thickness of 50 μm and exposed simultaneously to the UV light (medium pressure mercury lamp Hamamatsu LC8, light intensity on the surface of the sample of about 30 mW/cm^2), which induces the polymerization, and to the IR beam, which analyzes *in situ* the extent of the reaction. Acrylic double bond conversion was followed by monitoring the decrease in the absorbance due to acrylic double bonds centred at around 1640 cm^{-1}.

The gel content of UV-cured acrylic films was determined by measuring the weight loss after 24 h extraction with chloroform at room temperature, according to the standard test method ASTM D2765-84.

DSC measurements were performed on cured films under nitrogen flux, in the range between −120 °C and 80 °C, with a DSCQ 1000 of TA Instruments equipped with a low temperature probe.

The morphology of the cured films was investigated by Scanning Electron Microscopy (SEM) analysis using LEO (ex LEICA, ex CAMBRIDGE) S260. Accelerating voltages of 300 V–3 kV (accelerating rate of 100 V) and 4 kV–30 kV (accelerating rate of 1 kV) were applied. Prior to investigation (analysis of the cross-section), the samples were cryofractured with liquid nitrogen and metallized with carbon, under vacuum.

For electrical characterization, the polymeric capacitors were cut to match the shape of a rectangle of about 6 by 40 mm. Then, a CH Instrument 700D potentiostat was employed to perform the electrical characterization through impedance spectroscopy. Impedance Spectroscopy (IS, a.k.a. dielectric spectroscopy) measures the dielectric properties of a medium as a function of frequency. In particular, a sinusoidal tension signal is applied to the working electrode with constant amplitude and variable frequency between 1 Hz and 1 kHz. By measuring the current flowing between the contacts we got insights on the resistive and capacitive components of the materials under test.

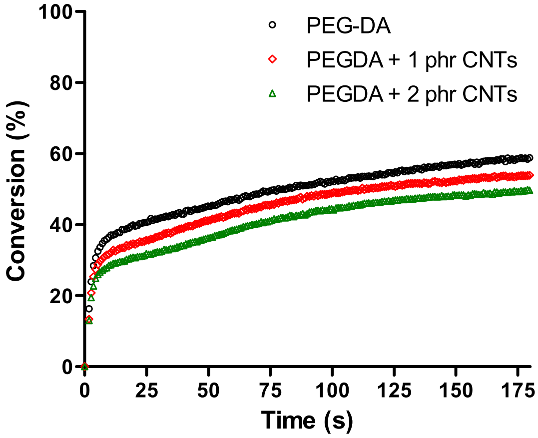

Fig. 2. Real time FT-IR acrylic double bond curves as a function of irradiation time for the pristine PEG-DA monomer and for the formulations containing 1 and 2 phr CNTs. Radical photoinitiator concentration = 2 wt%; light intensity = 30 mW/cm^2; film thickness = 50 μm.

3. Results and discussion

The organic capacitor was prepared via a multilayer UV-curing process. The conductive layer was obtained by adding into the polymeric resin CNTs up to a percolation threshold, found to correspond in between 1 and 2 phr CNTs. First of all the photopolymerization kinetics was investigated by real time FT-IR analysis, in order to evaluate the effect of the presence of the filler in the photocuring process. In Fig. 2 it is reported the acrylic double bond conversion as a function of irradiation time for the pristine PEG-DA resin and for the same resin containing 1 and 2 phr CNTs. It is possible to observe that the presence of the filler did not significantly influence the photopolymerization rate, while a slight decrease on final acrylic double bond conversion was achieved. The curves registered in real-time are performed in air, so it is expected a certain oxygen inhibition that can reduce the final acrylic double bond conversion (59% for the pristine PEG-DA resin). For this reason, the same materials were also prepared under inert atmosphere (nitrogen) and the completeness of the UV-curing process was assessed by FT-IR before and after 1 min of irradiation. In Fig. 3 the FT-IR spectra for the pristine PEG-DA are reported, showing the complete consumption of the acrylic double bond after 1 min of irradiation under nitrogen. A very similar conversion was also

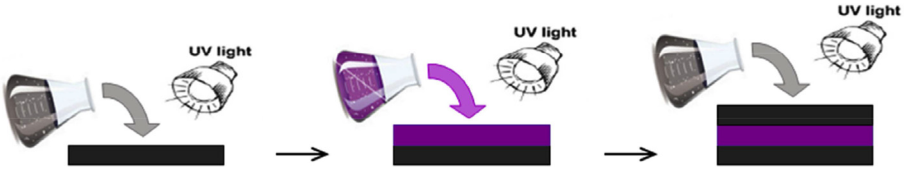

Fig. 1. Schematic representation of the preparation of UV-cured multilayer devices, where the first and the third layers are made of PEG-DA containing 1 phr or 2 phr CNTs, and the intermediate layer is the PEG-DA pristine resin (each layer is 50 μm thick).

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