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Full Length Article

Photoconductivity of tellurium-poly(methyl methacrylate) in the ultraviolet–visible-near infrared range

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ABSTRACT

Fine powders composed of tellurium grains of average size < 10 nm were produced by dry vibration milling combined with liquid-phase sedimentation techniques, starting from polycrystalline powders with average grain diameter of ca. 30 μm. Nanocomposite films were obtained by binding the nanosized tellurium grains with poly (methyl methacrylate). Raman spectroscopy revealed that the films were based on the coexistence of tellurium and tellurium oxide crystalline phases due to a partial oxidation in air of the grains. The optical measurements of the fabricated material showed that the absorbance was nearly constant in the 310–2200 nm range and that a typical UV absorption peak of the nanostructured tellurium was centered at around 260 nm. An extensive characterization of the photoconductivity properties was carried out by illuminating the tellurium-poly(methyl methacrylate) films with white light or radiations of different spectral composition selected from the UV–Vis-NIR region. Data analysis has allowed to demonstrate that the photoresponse is closely related to the optical absorption and is independent of the spectral composition of the incident radiation in the wavelength range from 310 to 2200 nm, while the photocurrent increases linearly as a function of the optical power density over about three orders of magnitude.

1. Introduction

Tellurium is a p-type semiconductor with a narrow band-gap of 0.34 eV. The highly anisotropic crystal structure consists of covalent bonded atoms forming unique helical chains which are bound in turn by weak Van der Waals interactions, producing hexagonal lattices [1]. The anisotropic properties of Te facilitate the growth of attractive nanostructures that can be used in many potential applications such as gas sensors [2–4], thermoelectric [5], piezoelectric [6,7], field effect devices [8] and nanogenerators [9] as well as photoconductors and solar cells [10–15].

The most common techniques developed for the synthesis of Te nanostructures, including nanobelts, nanotubes, nanowires and nanoflakes, are: solvothermal method [16], chemical vapour deposition [17], hydrothermal synthesis [18,19], physical vapour deposition [20], laser irradiation [21], microwave–assisted synthesis [11,22], electrochemical synthesis [10,23] and layer by layer method [13].

Recently Carotenuto et al. [14], by exploiting the very brittle nature of tellurium, produced, by dry vibration milling technique, nanostructures in the form of nanosized grains and obtained a functional material by binding the tellurium grains with poly(methyl methacrylate) (PMMA). This method allows to prepare Te nanostructures without performing chemical reactions or controlling the process temperature and it makes possible the fabrication of tellurium/polymer nanocomposites, suitable for flexible electronic applications.

In this paper, the morphology and structure of Te powder obtained by dry vibration milling followed by liquid phase sedimentation were analyzed by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Fourier transform infrared (FT-IR) spectroscopy. In addition, the morphological, structural and optical properties of tellurium/poly(methyl methacrylate) (Te/PMMA) material were investigated by SEM, Raman and UV–Vis-NIR spectroscopies. Finally, results on photoconductivity properties were derived from an extensive characterization carried out by irradiating the nanocomposite samples with light of different spectral composition and varying the optical power density.

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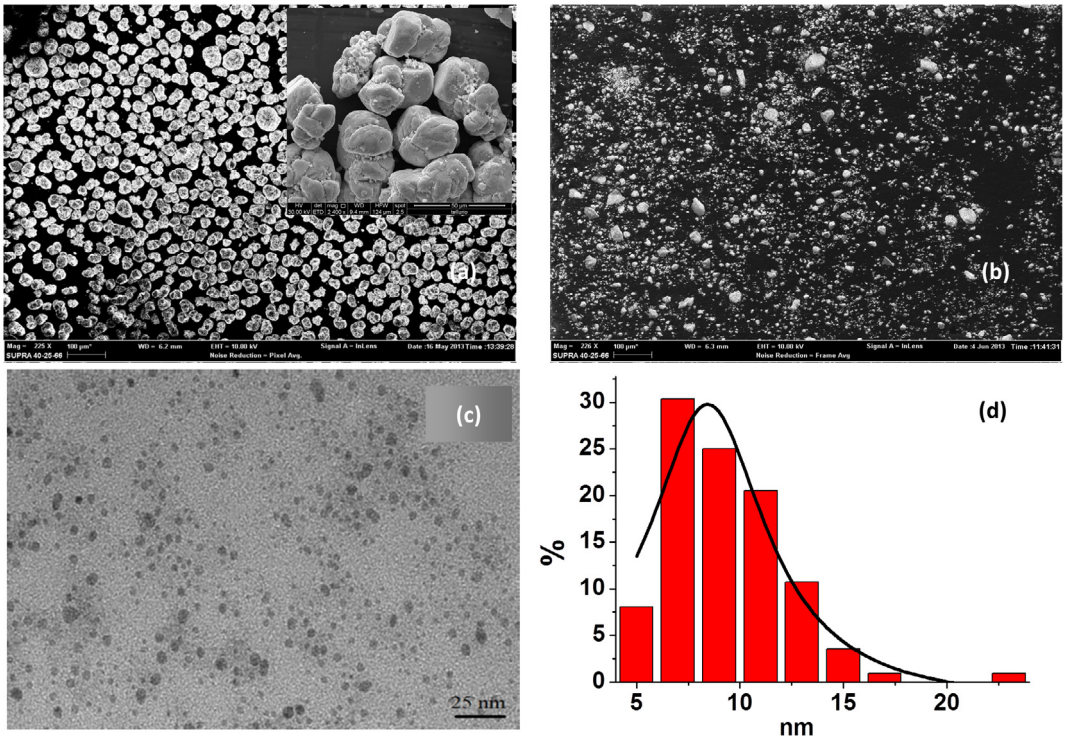

Fig. 1. SEM micrograph of the "as received" tellurium powder; in the inset the magnified SEM micrograph (a); SEM micrograph of the powder after the milling treatment (b); TEM micrograph of the powder after the milling treatment and liquid-phase sedimentation (c), and particle size distribution of the nanopowder (d).

2. Experimental

Tellurium nanopowder was prepared by using the ball milling approach starting from a commercial tellurium powder provided by Aldrich (99.8% by weight, −220 mesh). The ball milling apparatus consisted of a cylindrical steel jar (inner diameter of 25 mm), containing one grinding ball (diameter of 10 mm) operating in a vibrational mode (Mix Mill apparatus, Retsch, MM-200). Highly milled powder was prepared working in air for 7 h at 25 Hz and the nanoscopic portion was separated by a liquid-phase sedimentation technique. In order to obtain films, the prepared powder was consolidated by applying the spin-coating technology using the PMMA (M_w = 996,000 $gmol^{-1}$) as polymeric binder and acetone as solvent. The typical composition of the fabricated Te/PMMA films was 89% by weight of tellurium.

The "as received" and milled tellurium powders were characterized by scanning electron microscopy with a FEI QUANTA 200 FEG apparatus while a FEI Tecnai G2 Spirit twin apparatus was used for the transmission electron microscopy measurements of Te nanopowder. TEM specimens were prepared by depositing a drop of Te nanopowder dispersed in polystyrene/aceton solution on a copper grid. Polystyrene had the function to avoid Te nanoparticle segregation during the drop drying.

The FT-IR spectra of Te powders were carried out by preparing two kinds of samples by mixing the "as received" or milled powders with KBr (1% by weight). These mixtures were homogenized and pressed at ~10–15 tons, under vacuum, for 7–10 min to obtain pellets. The IR absorption spectra were then measured by putting the pellets in a Nicolet Nexus FT-IR spectrophotometer, operating in the double-beam mode between 400 and 4000 cm^{-1}.

The inner morphology and the thickness of Te/PMMA films were determined by SEM imaging of the cross-sections. UV–Vis-NIR absorption spectra were performed on Te/PMMA samples in the 200–2500 nm range with an acquisition rate of 1 nm/s, by a Perkin-Elmer Lambda 900 spectrophotometer. Te/PMMA film strucure was characterized by Raman spectroscopy at room temperature in backscattering configuration by Jasco Ventuno μ-Raman system, equipped with a Peltier-cooled charge-coupled device camera (operating temperature: −50 °C) and He-Ne laser (λ = 632.8 nm).

The electrical properties of films were investigated at room temperature in sandwich configuration, with top and bottom contacts of silver paint having surface areas of 3 and 10 mm^2, respectively. The samples were biased using a Tektronics PS 280 DC power supply and the current was measured by a Keithley 6485 picoammeter, equipped with a GPIB interface and custom-made LabVIEW application. The photoresponse of the samples was recordered by switching on and off the white light of a 450 W Xenon lamp (ILC technology) and radiations of different spectral composition obtained by means of the following filters:

(1) narrow bandpass interference filters with 10 nm full width half maximum (FWHM) centered at 365, 400, 460, 500, 540, 600, 660, 700, 950 and 1050 nm.
(2) long pass filter with cut-on wavelength at 600 nm and transmittance in the spectral range from 610 to 1200 nm
(3) long pass filter with cut-on wavelength at 800 nm and transmittance in the spectral range from 815 to 1200 nm
(4) long pass filter with cut-on wavelength at 1000 nm and transmittance in the spectral range from 1020 to 2200 nm
(5) heat absorbing glass filter with cut-on and cut-off wavelengths at 326 and 760 nm, respectively, centered at 440 nm and FWHM = 330 nm
(6) UV band pass filter with cut-on and cut-off at 310 and 393 nm, respectively, centered at 360 nm and FWHM = 50 nm.

Also, neutral density filters were used to vary the optical power density, F, over a large range (1.7–1608 mW/cm^2). F was measured by Laser precision Rk-5720 power radiometer.

3. Results and discussion

The "as received" commercial tellurium powder contained quite monodispersed grains with a spherical shape and a tight size