

TiO₂ colloidal nanocrystals functionalization of PMMA: A tailoring of optical properties and chemical adsorption

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Abstract

Colloidal TiO₂ rod shaped nanocrystals with different surface ligands (oleic and phosphonic acid) are used for tailoring the optical properties and tuning the chemical adsorption of bare poly(methyl metacrylate) (PMMA) thin films. Refractive index and absorption edge of the hybrid TiO₂/PMMA thin films have been determined, for both oleic and phosphonic acid capped nanocrystals, as a function of the nanorod concentration. The swelling of the nanocomposite samples in presence of acetone and ethanol vapours has been investigated. Our results show that nanocomposites with phosphonic acid as surface ligands are more transparent and have a higher refractive index with respect to the films with oleic acid ligands. Furthermore, the presence of inorganic nanorods (NRs) modifies the vapour adsorbing ability of the PMMA and this modification is strongly influenced by the nature of the surface ligands and by the TiO₂ nanorod concentration.

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1. Introduction

Polymer nanocomposites, consisting of inorganic nanocrystals (NCs) embedded in polymer matrix, are complex materials in which is possible to combine the properties of organic and inorganic materials in a unique compound for potential applications in many fields, such as optics, electronics, mechanics, chemical sensing and biology. These hybrid materials couple the structural flexibility and the convenient processing of the polymers with the high carrier mobility, band gap tunability, thermal and mechanical stability of the inorganic component, resulting in synergical effect on their individual properties. Hence, they can allow a versatile design of their physical and chemical properties, to meet the needs of the final end user applications [1–6].

Among the polymers, the PMMA is very interesting for optical applications as optical fibres, optical disks and lenses [7], thank to the easy processability and the high transparency in the

visible range. The incorporation of inorganic TiO₂ NCs with high refractive index and wide energy gap allows to obtain a modified polymer with new optical properties, tunable by varying the composition, size and concentration of the embedded inorganic part, and better thermo-mechanical stability. In addition the PMMA, as the majority of polymers, is able to adsorb organic vapours producing a reversible change of polymer volumes (swelling) [8].

In thin and transparent films, the swelling changes the reflectance and transmittance spectra of the polymer and these modifications can be efficiently used as optical responses for new sensing applications as it has been shown recently [9]. Furthermore, it has been found that the presence of inorganic NCs can modify the swelling behaviour of the polymers [10,11] suggesting the possibility to tailor the sensing properties of the nanocomposite polymers by engineering the NC composition, size, shape, concentration and surface chemistry.

The aim of this work is two-fold. Firstly, to obtain TiO₂ NRs/PMMA nanocomposites with high refractive index and optical transparency for the development of new optical devices with tailorable optical responses. The second is to exploit the

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sensing properties of TiO₂ NRs [12–14] in order to modify the vapour adsorbing behaviour of the PMMA towards the design of innovative optical sensors for solvent detection. In this context, we first investigated the changes of the refractive index and the absorbance in PMMA films filled with TiO₂ NRs capped by two different surface ligands, as a function of the NRs content. In addition, we performed preliminary studies on the swelling in presence of acetone and ethanol vapours with the NR concentration.

TiO₂ NRs in anatase phase were prepared by hydrolysis of tetraisopropoxide using technical oleic acid (OLEA) as surfactant at low temperatures (80–100 °C) [15]. The OLEA surface ligand was exchanged with dodecylphosphonic acid (DPA) in order to obtain NRs with alternative surface chemistry characteristics. Both the as-prepared and the surface exchanged particles were successively dispersed, at different concentrations, in PMMA based chloroform solutions. The advantages of this polymer nanocomposite preparation route, with respect to the other methods which are currently used, such as in situ polymer-inorganic composites synthesis processes [16], rely on its simplicity and the great versatility and flexibility to tune the final material properties. Ex situ nanocomposite fabrication does allow an accurate control both on the NCs properties, namely crystalline phase, shape, size and, very crucially, their tuneable surface chemistry and on the polymer moiety choice, by exploiting the chemical affinity of the two components in a suitable solvent [15]. For instance, the choice of a suitable ligand for NCs can ensure a good dispersion and a close interaction between NC and host matrix, or alternatively the tailoring of the polymer characteristics, i.e. by properly functionalizing the polymer structure with suitable chemical groups, able to effectively encapsulate nanoparticles.

2. Experimental

Organic capped anatase TiO₂ NCs were synthesized by hydrolysis of titanium tetraisopropoxide using technical OLEA as surfactant at low temperatures (80–100 °C). The resulting OLEA-capped rod-like TiO₂ NCs were homogeneously dispersed in chloroform solutions. The OLEA coating on TiO₂ nanoparticles was exchanged with dodecylphosphonic acid following a procedure previously reported [15]. By this treatment, exceptionally concentrated transparent solution of TiO₂ nanorods have obtained, stable over months without further addition of phosphonic acid molecules.

NRs with 30 nm of average length and 6 nm of diameter were observed by transmission electron microscopy (TEM), carried out on samples deposited on a carbon-coated 400 mesh copper grids.

Nanocomposite films with dispersed titania nanoparticles in polymer were fabricated by adding high molecular weight PMMA to a chloroform solution of TiO₂ nanorods at increasing nanocrystals concentration (from 0.05 to 0.5 M), corresponding to a NR load percentage in polymer ranging from 8 to 80 wt.%. A polymer concentration of 0.05 g/ml was selected to achieve optically clear nanocomposite solutions. Thin films were prepared depositing by spin-coating the nanocrystal–polymer

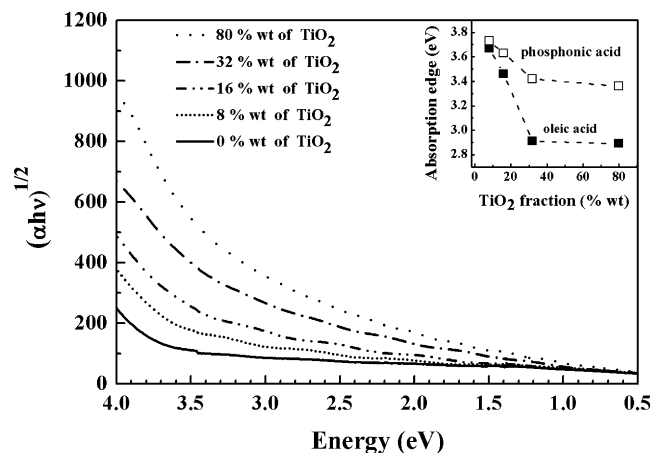


Fig. 1. $(\alpha h\nu)^{1/2}$ vs. the photon energy plots for PMMA based nanocomposites containing oleic capped TiO₂ NRs at different NRs content. Inset: The absorption onset is plotted as a function of the NRs content, either for oleic acid and phosphonic acid capped TiO₂.

mixture onto properly cleaned glass and silicon substrates. The film thickness, measured by means of Alpha-Step 500 surface profiler, was found in the range from 400 nm up to 1 μm, strongly depending on the NR concentration and surface ligand compositions.

The surface morphology and roughness of the TiO₂ NRs/PMMA films were investigated by atomic force microscopy (AFM) operating in tapping mode by using high resonance supersharp Si tips. For each sample, images have been recorded at different scan sizes ranging from 1 μm × 1 μm to 50 μm × 50 μm.

The absorption edge and the refractive index of the nanocomposites were determined by means of reflectance and transmittance spectra performed on the films deposited on glass substrates using Cary 5 UV-Vis-NIR spectrophotometer.

The effects of surroundings vapours on the TiO₂ NRs/PMMA films were tested by reflectance measurements using a Cary 5 UV-Vis-NIR spectrophotometer. The samples were inserted into a glass tube through which flowed synthetic air (20.5% O₂ in N₂) as carrier gas with a flow of 200 sccm. The organic vapour was introduced by bubbling synthetic air through the organic liquid and the mixing the saturated synthetic air with the carrier gas to produce the desired concentration. A mass flow controller system was automatically direct by Omicron apparatus. The measurements were carried out at room temperature.

3. Results and discussions

3.1. Optical properties

In Fig. 1, $(\alpha h\nu)^{1/2}$, being α the absorption coefficient of the nanocomposite calculated by reflectance and transmittance spectra, is plotted versus the photon energy for different content of oleic capped TiO₂ NRs in the polymer matrices. The figure highlights a strong absorption in the UV region upon incorporation of NRs in the PMMA, characteristic of TiO₂ NRs, together with an increasing red shift of the absorption edge when TiO₂

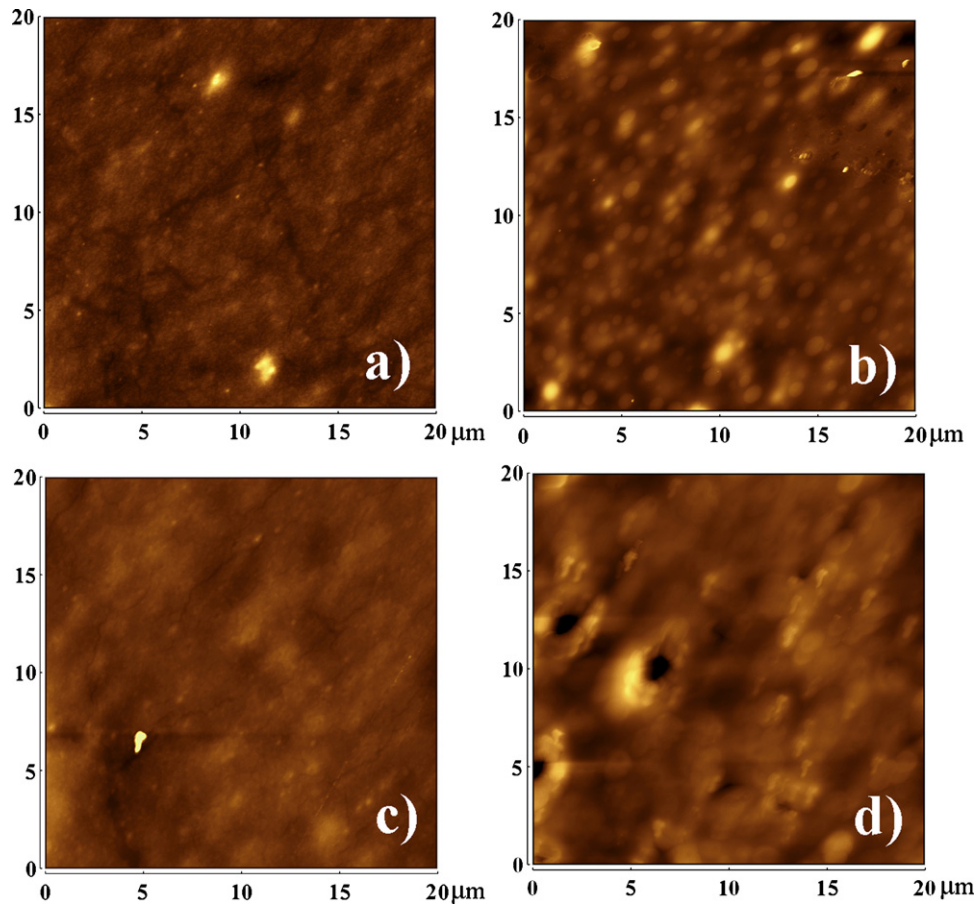


Fig. 2. $20\ \mu\text{m} \times 20\ \mu\text{m}$ AFM plan view images of the PMMA based nanocomposite as a function of the NRs concentration and surface capping: (a) 8 wt.% DPA capped TiO_2 NRs, $z = 49\ \text{nm}$; (b) 80 wt.% DPA capped TiO_2 NRs, $z = 280\ \text{nm}$; (c) 8 wt.% OLEA capped TiO_2 NRs, $z = 49\ \text{nm}$; (d) 80 wt.% OLEA capped TiO_2 NRs, $z = 533\ \text{nm}$.

content augments. A similar behaviour, is observed in films with phosphonic acid capped NRs.

The absorption edge of the nanocomposite thin films versus the TiO_2 content with oleic acid and phosphonic acid ligands has been obtained from the intercept of the photon energy axis with the straight lines, fitting the $(\alpha h\nu)^{1/2}$ versus $h\nu$. The figure inset shows that the absorption edge shifts at lower energy by increasing the TiO_2 content in the polymer matrix. This red-shift is more marked in the nanocomposite films containing NRs capped by OLEA than in those containing DPA capped NRs. The absorption edge of the samples with OLEA capping and high NR contents (32 and 80 wt.%) is lower than the energy gap of bulk anatase TiO_2 ($E_g = 3.2\ \text{eV}$). Furthermore, the spectra of the samples with high NR contents show a remarkable absorption tail at lower energy.

The optical behaviour observed in the TiO_2 NRs/PMMA samples could be ascribed to different phenomena induced by the formation of TiO_2 NR aggregates in PMMA, also for samples at the lowest NR concentration. Indeed, we expect that the formation of collective electronic states between interacting nanocrystals in the ensemble of close-packed NRs leads to a red shift of the absorption edge, like it has been observed for CdS nanoparticles in Ref. [17]. Moreover, the optical losses due to scattering of the light by aggregates of inorganic particles

and polymeric matrices with high refractive index mismatch can explain the presence of the absorption tails in the more concentrated samples. Finally, the structural characteristic of the aggregates (number of NRs and distance between NRs) are expected to depend on the capping molecule properties and this could cause different values in the red shifts observed in the PMMA with OLEA and DPA capped NRs. Indeed, for DPA capped TiO_2 NRs, a more ordered titania alignment can be accounted, due to a stronger Van der Waals interaction through interdigitation of linear aliphatic chain of DPA molecules with respect to *cis*-double bond structure of OLEA molecules. Further experimental and theoretical investigations are in progress in our laboratories in order to fully understand the behaviour of the absorption onset in TiO_2 NRs/PMMA nanocomposites.

The evidence of NR aggregates in the PMMA matrix is supported by the morphology of the samples recorded in AFM measurements. Topographic plan view of the TiO_2 /PMMA nanocomposite thin films at different NR capping and load percentage is reported in Fig. 2. At low TiO_2 content (8 wt.%), notwithstanding a quite smooth surface is observed, small aggregates randomly distributed on the film surface are evident independently of the surface capping (Fig. 2a and c). In agreement with the larger red-shift observed in the absorption edge, by increasing the TiO_2 contents to 80 wt.%, a huge increase in

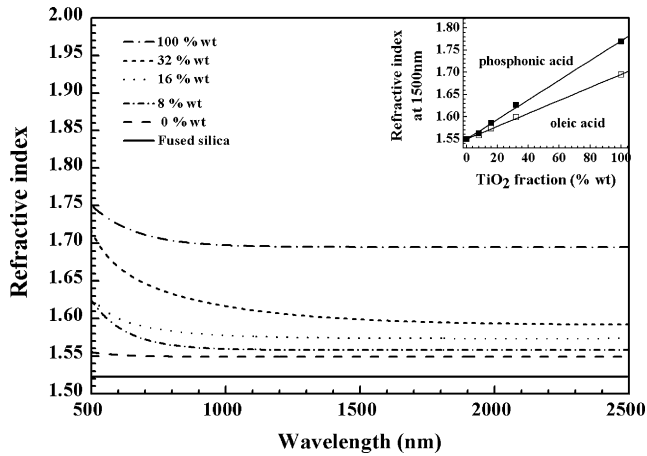


Fig. 3. Wavelength dispersion of the refractive index n for PMMA based nanocomposites containing OLEA capped TiO_2 NRs at different NRs content. *Inset*: The value of n at 1500 nm is plotted as a function of the NR content, either for oleic acid and phosphonic acid capped TiO_2 .

the surface roughness together with the formation of very large aggregates with lateral size of hundredth of nanometer for phosphonic capped NRs (Fig. 2b) and of the order of micrometer for the oleic capped ones (Fig. 2d) are evident. As a further feature, holes with diameter of the order of micron and depth of several hundredths of nanometer are present in the films containing OLEA capped TiO_2 . The RMS roughness values measured on $20 \mu\text{m} \times 20 \mu\text{m}$ region range, by increasing the NR content from 8 to 80 wt.%, from 4 to 23 nm for DPA capped NRs and from 3 to 46 nm for the OLEA ones.

The dispersion of the refractive index, n , in the wavelength range from 500 to 2500 nm is reported in Fig. 3 for the samples with OLEA capped NR at different TiO_2 content. The values of n were determined by the transmittance spectra using the method proposed in Ref. [18]. As it is expected, the presence of inorganic components with high refractive index in the nanocomposite

samples increase the value of n with respect to PMMA. It is worth to note that the surface chemistry of the TiO_2 NRs influence the optical properties. Indeed in the inset of Fig. 3 are reported as comparison the calculated value of the refractive index at 1500 nm versus the TiO_2 content for the films with OLEA and DPA capped NRs. A linear relation between n and the NRs weight fractions is observed for both the capping ligands, with a slight difference in slope. This is due to a different morphology of the films induced by the capping molecules, such as the higher porosity of the films based on OLEA capped TiO_2 NRs decreases the density of the films and as a consequence n .

3.2. Sensing properties

The reversible modification of the vapour adsorbing properties in the PMMA induced by the presence of the TiO_2 NRs has been explored by using acetone and ethanol vapours at the respective saturation vapour pressure at room temperature. The choice of solvents was due to the high sensitivity of TiO_2 to this organic species. The effects of the surrounding vapours were tested by measuring the reflectance spectra of the samples deposited on Si substrates in the presence of the vapours. The swelling of the films in presence of an input signal causes the change of the fringe pattern by shifting the position of the maximum and minima. By the analysis of these reflectance spectra the degree of swelling, $\Delta t/t$, was calculated, where t is the thickness of the film and Δt is the relative thickness variation. The analysis of the spectra was performed numerically by using the standard matrix method [19]. The theoretical curves were obtained by using the values of n previously measured and assuming that the adsorption of the ethanol and acetone produces negligible modification of n of the nanocomposite PMMA.

In Fig. 4, the swelling degree versus the TiO_2 content for the nanocomposite films with both the capping in presence of

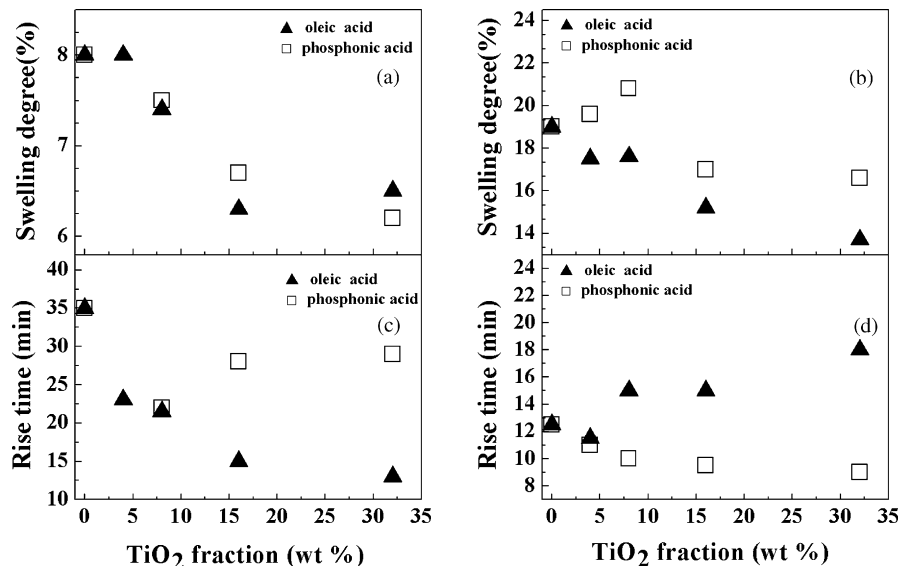


Fig. 4. Swelling degree, defined as $\Delta t/t$, vs. the TiO_2 NRs content for the nanocomposite films, based on the differently capped TiO_2 NRs, measured in presence of ethanol (a) and acetone vapours (b), respectively. The rise times are also plotted as a function of the NR content in presence of ethanol (c) and acetone (d).

ethanol (Fig. 4a) and acetone vapours (Fig. 4b) is reported. The swelling of pure PMMA in presence of ethanol and acetone vapours is high, around 8 and 19%, respectively. The incorporation of TiO₂ NRs in the polymeric matrix produces clearly a modification in the vapour adsorbing power of the PMMA and this change is strongly influenced by the kind of the capping molecules. We observe a weak decreasing of the adsorbing power by increasing the inorganic component; only in the case of DPA capped TiO₂ NR based film an initial improvement of the swelling in presence of acetone vapours, by increasing the TiO₂ content up to 8 wt.%, is noticed. In the Fig. 4c and d, we plot the rise time of the optical response as a function of the inorganic content for both the capping agents. It is evident that the presence of the NRs improves generally the response time with respect what measured for PMMA. A slight increase is obtained only for OLEA TiO₂/PMMA based films when exposed to acetone vapours. The presence of the NRs could speed up the response of the PMMA, but also in this case the capping molecules play a fundamental role. This behaviour, strongly influenced by the capping molecules, demonstrate that physical and chemical interactions between the solvent molecules and the surface of the NRs are involved in the diffusion of the molecules into the composite polymer. By changing the capping molecules these interactions can enhance or inhibit the swelling phenomenon of the composite polymer and slow down or speed up the response time of the PMMA based nanocomposites. In conclusion, these preliminary results suggest the potential to tailor the vapour adsorbing power of the PMMA nanocomposites by means of embedding NCs that can differ in composition, size, content and surface chemistry. Then, further experimental and theoretical investigations are necessary to fully understand this complex response behaviour and consequently to control the sensing properties and optical responses of the PMMA nanocomposites in view of sensing applications.

4. Conclusions

Two sets of samples of PMMA based nanocomposites prepared at increasing concentration of TiO₂ NRs, capped with oleic and phosphonic acid were spun as thin films on glass and Si substrates. The investigation of the optical properties revealed that nanocomposite based on DPA capped NRs possess a higher optical transparency and a higher refractive index with respect to OLEA capped NRs.

A preliminary investigation on the sensing properties of the TiO₂ NRs/PMMA based nanocomposites indicates that the vapour adsorbing behaviour of the PMMA is strongly influenced by the incorporation of TiO₂ NRs, highlighting also how such influence is mainly depending on the surface chemistry of the inorganic moiety in the nanocomposite, namely the presence of an organic molecule layer coordinating the surface of the TiO₂ NRs, appears to be critical in the sensing process. Indeed, the capping molecules can enhance or inhibit the swelling phenomenon of the composite polymers, being also able to modulate the response of the PMMA.

In conclusion, nanocomposite based on PMMA and colloidal oxide NCs appears to be promising materials for vapour optical sensor devices, since their optical characteristics and sensing responses can be effectively tailored by engineering the chemical status and the size dependent optoelectronic properties of the embedded inorganic nanoparticles. However, further experimental and theoretical investigations are necessary, and are currently in progress in our laboratories, to fully understand the optical behaviour of the proposed class of materials and to control the sensing properties of the PMMA based nanocomposites.

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Biographies

Annalisa Convertino received her Laurea degree in Physics at the University of Bari (Italy) in 1994 and her PhD in Physics at the University of Bari in 1999. She is currently researcher at the Istituto per lo Studio dei Materiali Nanostrutturati (ISMN)-CNR in Rome, Italy. Currently she is involved in the research on organic-inorganic nanocomposite functional materials for the development of optical gas sensors and in nanostructured semiconductors (nanorods and quantum dots) for optoelectronic and photonic applications.

Gabriella Leo was born in Bari (Italy) in 1961. She studied Physics at University of Bari where she graduated in 1986. She received her PhD in Physics at University of Bari in 1992 for work on study of implantation damage in CdTe single crystals. She is researcher at Istituto per lo Studio dei Materiali Nanostrutturati (ISMN)-CNR in Rome, Italy. Her current research interests are mainly addressed to the investigation of the morphological, optical, electronic and magnetic properties of ensemble and single epitaxial quantum dot (InAs in GaAs matrix) and colloidal nanocrystals (CdSe/ZnS, CdSe/CdS, ZnO, TiO₂, and Co) for optoelectronic, magnetic and sensing applications.

Michela Tamborra was born in Ruvo di Puglia (Italy) in 1978. She received her degree in Chemistry in 2003 from the University of Bari (Italy). She is currently a PhD student in Chemistry under the direction of Prof. A. Agostiano at University of Bari. Her research activity is carried out in collaboration with CNR-IPCF Bari Division Nanochemistry laboratory and ranges from the synthesis and characterization of colloidal semiconductor nanocrystals to their exploitation in opto-electronic applications. She is involved in the incorporation of semiconductor and oxide nanocrystals in polymers to develop novel nanocomposite materials with original and suitable properties.

Corrado Sciancalepore was born in 1975. He got his degree in Chemistry in 2006 from the University of Bari (Italy). His research activity is focused on the preparation of nanocomposites made of oxide nanocrystals and thermoplas-

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Marinella Striccoli was born in Bari (Italy) in 1966. She obtained her degree in Physics in 1992 at University of Bari (Italy). In 1995 she was visiting scientist at the AT&T Bell Labs under the supervision of F. Capasso. From 1992 to 2000 she worked as researcher at Centro Laser research center, then for 2001 she had a grant fellowship from INFN. Since 2002 she has joined CNR (Italian National Research Council) as staff researcher in the Institute for Chemical Physical Processes. She has been responsible and involved in several National and European Projects in the field of Material Science and semiconductor nanostructures. Now her research interests are mainly focussed on colloidal nanocrystals structures (semiconductors, metal and oxide), in their organization in ordinate assemblies and in the development of nanocomposite materials. She is co-author on more than 30 papers on international peer reviewed journals and several conference contributions.

M. Lucia Curri was born in Taranto (Italy) in 1968. She obtained her Chemistry degree in 1992 at University of Bari (Italy). In 1997 she got her PhD in Chemistry from the same university, with a thesis in archaeological chemistry. She had then a research assignment at University College of London (UK) and then joined CNR (Italian National Research Council) with a grant fellowship in 1997 and is working as staff researcher since 1999. She is the scientific responsible of several National and European Research Projects in nanomaterial chemistry. Her research interests include development of chemical methods to prepare nanoparticles of semiconductors metal and oxides and to organize them in mesostructures, or incorporate them in polymers for developing novel nanocomposite materials and 2/3 D nanoparticle assemblies. She has co-authored more than 60 papers on international journals.

Angela Agostiano was born in Ferrandina-Matera (Italy) in 1952, and she received her degree in Chemistry at the University of Bari in 1975. After several postlauream experiences, she was appointed full professor in physical chemistry at the University of Bari in 2000. She is the Italian Scientific responsible of cooperation projects between Germany and Italy, and between Hungaria and Italy. Since 2003 she is director of the Bari section of the National “Institute for fisico-chemical processes” of CNR. Author of more than 140 publications in the fields of electrochemistry and photoelectrochemistry of processes involved in photosynthesis, physicochemical investigation of different aggregation states of Chlorophyll a and preparation and characterization of nanosized semiconductors.