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Polydimethylsiloxane containing gold nanoparticles for optical applications

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ABSTRACT: A hybrid material incorporating gold nanoparticles (Au-NPs) in polydimethylsiloxane (PDMS) has been synthesized. Gold nanoparticles gained wide interest for their optical, chemical and physical properties, such as surface plasmon oscillations resonant absorption, chemical structure, imaging and sensing. Au-NPs can be conjugated with several functionalizing agents, such as polymeric chains, improving their properties and performances. PDMS composites with weight percentages ranging between 0.1 and 0.2% of spherical Au-NPs (10 nm diameter), were produced. In this work, we report on different dispersion effects of gold nanoparticles in PDMS. The surface morphology was investigated by atomic force microscopy (AFM) and wetting ability analyses. The presence of structures inside both virgin and hybrid PDMS containing Au-NPs was preliminary investigated by Small Angle X-ray Scattering (SAXS). The optical performance of such hybrid material was studied by UV-VIS spectroscopy, in transmission mode. An enhancement of the absorption was reported as a function of increasing percentage of gold nanoparticles and changing the nanoparticles dispersion approach. The presented hybrid material could be promising for many useful applications, such as optical waveguides, sensors and switches in optoelectronics.

KEYWORDS: Manufacturing; Lasers

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1 Introduction

In 1857 Michael Faraday identified the ruby gold nanoparticles (Au-NPs), which became a milestone for the modern nanotechnology. Later, Zsigmondy starting from the Faraday discovery, explored the procedure called, ‘seed mediated method’, which is used today for the chemical synthesis of NPs (bottom-up method). Another more recent common and feasible way for synthesis of Au-NPs is with laser ablation in liquid medium (top-down method) [1]. Significant assets are the accurate control of the properties by changing laser parameters (wavelength, pulse duration and energy, repetition rate) and the type of medium and material, spot focusing, fluence and irradiation time. Polydimethylsiloxane (PDMS) [2] is a polymeric material characterized by high visible transparency, gas permeability, outstanding elasticity, hydrophobicity and ease way to be synthesized. The PDMS surface is inert, resulting in the difficulty in introducing biomolecules. Its un-reactivity is a limit for instance in the use of PDMS in biomedicine as on-chip bioassays, i.e. to evaluate the concentration or efficacy of a substance by its effect on living cells or biological tissues. To overcome this problem and to spread the employment of PDMS, chemical modification layers [3] sol-gel [4] and nano- or micro-particles [5] have been embedded into PDMS surface and bulk. The generation of nanocomposites, consisting of metal nanoparticles embedded into polymer matrices, have gained high interest, due to the chemical and physical properties acquired by the polymer, enhancing its potential applications, especially in optical field [6], micro-electro-mechanical devices [7] and biosensors [8]. Metal nanoparticles–PDMS composite films can be used to fabricate surface-enhanced Raman scattering (SERS) substrate [9], thin films with peculiar optical properties [10], coloured emissive transition devices [11], and others. Several research groups have been exploring the use of metallic nanoparticles (NPs) for developing optical strain sensors, for instance, through a theoretical study based on Mie theory as reported in ref. [12]. The optical properties of gold nanoparticles were investigated as a function of the tensile and compressive strains changing [12] as well as their far-field optical properties as consequence of free electron dielectric function changes. Several parameters contribute to the feature changes in the produced hybrid material: amount and molecular weight of the added filler, dispersion level, type and size/shape of the filler [13], concentration, use of molecular functionalized nanoparticles and of added solutions. It is well known that matter in the nanometric scale shows surface features different

and sometimes opposite of those intrinsic of the bulk. These unique properties are related to the quantization of the electronic states, which are relevant to the optical and magnetic properties that, generally, are size-dependent. This dependence is more evident in nanomaterials and nanostructure embedded in different polymeric matrices, as reported in the literature [14]. Moreover, the higher surface to volume ratio of NPs, compared to bulk materials, is significant in terms of thermal, mechanical and chemical properties in nanomaterials [13]. An important feature of Au-NPs is their strong interaction with light resulting in extraordinary efficiency of absorbance, resonance and scattering of the light. The Au-NPs are excited by light at specific wavelength bands inducing the conduction electrons on the metal surface undergoing a collective oscillation, i.e. surface plasmon resonance (SPR). This effect is responsible for the absorption and scattering intensities of gold nanoparticles which are higher than corresponding sized non-plasmonic nanoparticles. Modification of the particle size, shape of gold nanoparticle, refractive index of the medium in which the NPs are embedded and SPR effects can be controlled with the tuning of the absorption band and scattering properties. Presently, we focused on the optical, morphological properties of PDMS matrix improved by the presence of Au-NPs considering two different types of mixing processes based on wet and dry preparation methods.

2 Materials and methods

For the polymeric matrix, polydimethylsiloxane (PDMS), Sylgard 184 base and thermal curing agent were purchased from Dow Corning Corporation (Germany). Commercial Au-NPs 99.9% purity dispersed in water, spherical sized, 10 nm average diameter, were obtained from Sigma-Aldrich (Czech Republic). PDMS sample synthesis by standard procedure involves mixing of prepolymer and curing agent in a 10:1 ratio by weight. Herein two approaches have been adopted to fabricate metal nanoparticles-PDMS composite films. One approach involves removing the solvent from the Au-NPs under light illumination and later dispersion of the obtained dried NPs in PDMS matrix by stirrer, for 10 minutes and at room temperature. The second approach entails mixing of the PDMS prepolymer and curing agent in a 10:1 ratio by weight and then dispersion in the aqueous solution of Au-NPs in the desired quantity by stirring for 15 minutes at room temperature. The composites, with an average thickness of 1 mm, were poured on Petri dishes. This step was followed by thermal curing at 100°C for 1 hour and detachment from the Petri dishes. One sample with Au-NPs weight percentage of 0.2% was realized using the first (dried) approach and will be indicated as containing dried Au-NPs (D-AuNPs). Samples with Au-NPs weight percentages of 0.1% and 0.2% were realized using the second (wet) approach (W-Au-NPs). Three different sets of PDMS/Au-NPs samples are fabricated to support this study as displayed in figure 1.

A Dimension ICON (Bruker Corp.) microscope, in quantitative nanoscale mechanical (QNM) mode in air was used for evaluation of the surface morphology of both PDMS, virgin and with Au-NPs. Silicon tip on Nitride Lever SCANASYST-AIR with spring constant 0.4 N/m was used. The data were processed employing the NanoScope Analysis software [15]. The analysis was employed in the atmosphere environment and at room temperature to examine the micro-structures present in the surface morphology and the roughness of PDMS. The analyzed area was of about 300 nm² and 3 μm². Surface average roughness (Ra) and the mean roughness parameter (RMS) were calculated

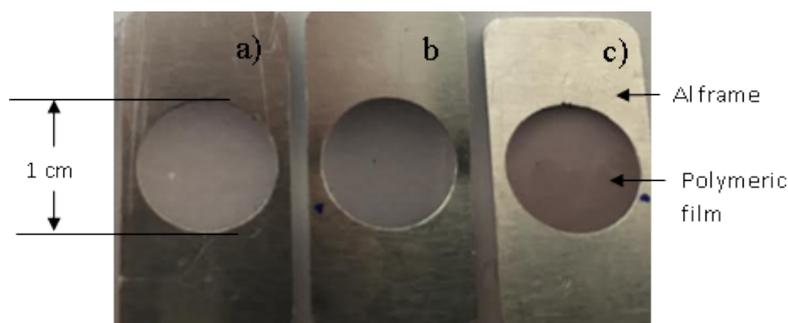


Figure 1. Set of produced samples of PDMS mixed with Au-NPs in concentration of 0.1% a), 0.2% wet b) and 0.2% dried c) magnification $\times 4$.

based on $3 \mu\text{m}^2$ three topography images measured at different areas. The choice of such a wide area aims to make AFM analysis consistent with wettability probing of the same areas.

The surface wettability of PDMS as a function of the Au-NPs size and concentration was determined by measuring the contact angle of a pure water drop over the solid surface. Lower contact angle means that the material exhibits a hydrophilic nature (contact angle lower than 45°). On the contrary, higher contact angles, higher than 90° , indicate the hydrophobic nature of the surface. The wetting property was determined by direct measurement of the tangent angle at three-phase equilibrium interfacial point using sessile drop method [16]. A $1 \mu\text{l}$ droplet of deionized water was dispensed on the solid surface using a micrometric syringe.

UV-Vis measurement [17] was performed in different areas of each sample to quantify the effect induced by the presence of Au-NPs in PDMS. An AvaSpec-2048 spectrophotometer with UB-600 lines/mm grating and a bandwidth from 195 nm to 757 nm and a dual light Halogen/deuterium was used. The optical behavior of the PDMS/Au-NPs was monitored in the range 400–750 nm in transmission mode.

The structural features of the PDMS containing Au-NPs resulting from wet and dried Au-NPs and different filler concentration were examined by small-angle X-ray scattering (SAXS). The experiments were carried out at XEUSS 2.0 facility [18] of Nanocent Lab of Charles University in Prague [19]. The measurements were performed using $\text{CuK}\alpha$ radiation (wavelength $\lambda = 1.5418 \text{ nm}$) with exposure time of 3000 s. Measured 2D patterns were azimuthally integrated and normalized into 1D SAXS patterns using Foxtrot program.

At Tandetron lab (CZ), the Litron Nd:YAG laser system (1064 nm wavelength, 560 mJ pulse energy, 6 ns pulse duration) [20] was employed to irradiate the sample as a guidance of the effect of the sample absorbance through the evaluation of the energy of emitted ions. The samples were placed in a scattering chamber and a Faraday cup located at 88 cm along the normal to the target surface monitored the signal of the emitted ions in time of flight (TOF) approach [21].

3 Results and discussion

The reaction between silicon hydride (Si–H) groups in the curing agent and vinyl groups (Si–CH=CH₂) in the monomer is the ground of the crosslinking of PDMS [22]. In the cured PDMS, the concentration of Si–H groups depends on the mass ratio of the curing agent and the monomer (η). The greater monomer concentration (η) results in a higher concentration of the residual Si–H groups.

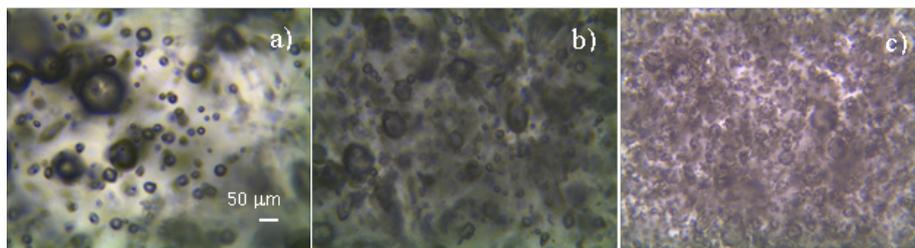


Figure 2. Optical microscope pictures of PDMS with different weight percentage of Au NPs 0.1% (a), 0.2 (b), and D-AuNPs 0.2% (c) 10× magnification.

In the present work Au-NPs were used as filler in PDMS. Gold nanoparticles, dispersed in water, when illuminated by light convert light to heat by SPR plasmonic effect [23]. The heat generated from the interaction of light with the nanoparticles in SPR absorption regime induces heat transfer from nanoparticles to the surrounding material (water or polymer). The increase of the localized temperature generates a vapor film at the particle-liquid interface, which tends to increase under continuous illumination, and eventually coalescing nanoparticles [24]. In the first approach Au-NPs dried under light exposition were mixed with PDMS forming a violet composite due to the aggregation of Au-NPs during the drying process. As the size of the nanoparticles increased, longer wavelengths would be absorbed and so a dark violet colour would be reflected. In the second approach when PDMS was mixed in wet Au-NPs solution, the nanoparticles did not have enough energy and time to aggregate and the colour was preserved, then, the composites became pink. A frontal view of PDMS with different percentage (0.1% and 0.2%) of Au-NPs both wet and dried at 10 × magnification is presented in figure 2. The optical microscopy shows the PDMS film with different weight percentage of Au-NPs: 0.1% (a), 0.2% (b), and D-Au-NPs 0.2% (c).

These pictures, acquired in the same conditions of magnification, show that bubbles/voids together with the Au-NPs are well dispersed and randomly oriented into PDMS. The diameter of the displayed structures is micrometric, i.e. larger than that of Au-NPs, probably due to the coalescence of voids and formation of microbubbles in liquid during the thermal drying in oven. They give rise to microporous material and surface roughness.

The composition and roughness of the solid surface steers and the interaction with water and air molecules, influences the wetting ability.

In figure 3 is shown the viewing of the drop profiles to measure the contact angle. High hydrophilic nature is observed in PDMS (figure 3(b)) because it adsorbs water molecules due to the presence of active polar functional groups. In PDMS figure 3(b) the high roughness decreases the surface energy of the material and reduces the wettability of the molecular liquids and consequently increases the contact angle which is 116°. In figure 3(c) and (d) a slight reduction and a slight increase of the contact angles are revealed. The reason is probably, the low number of Au-NPs reducing the average surface roughness, decreasing the contact angle to 95° in the first case at 0.1 wt% and increasing the contact angle to 107° at 0.2 wt%. The highest hydrophobicity of 121° was observed in PDMS containing 0.2 wt% of dried Au-NPs (figure 3(d)) probably because of the greater size of the filler.

The optical features of virgin and PDMS containing Au-NPs as filler were measured by direct incident light in transmittance mode. UV-Visible absorption spectroscopy was used to monitor the

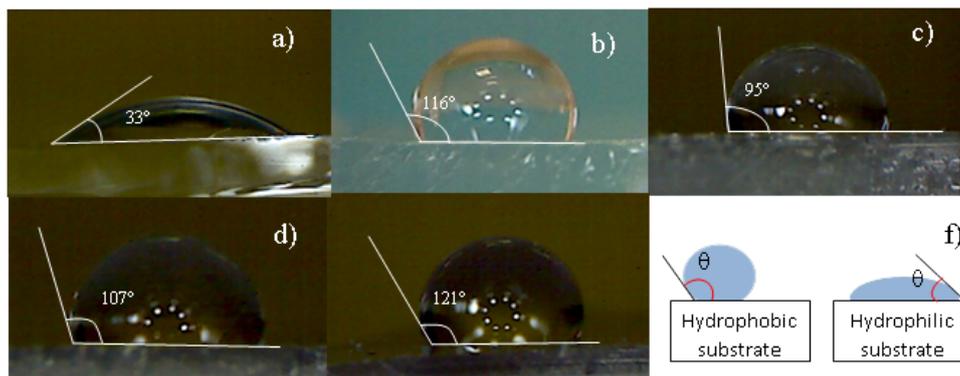


Figure 3. Optical images for wettability measurements of virgin Glass (a), virgin PDMS (b), PDMS containing 0.1% wt NPs (c) PDMS containing 0.2% wt wet Au-NPs (d) PDMS containing 0.2% wt dried Au-NPs (e) and schematic representation of water molecules on different substrates (d).

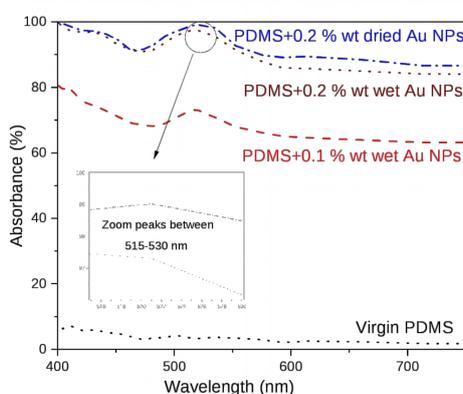


Figure 4. Spectrum of absorbance of virgin and treated PDMS and in the insert the zoom of the peaks related PDMS/Au-NPs 0.2 wt% and PDMS/Au-NPs 0.1 wt% around 515 nm and 530 nm wavelengths.

plasmon absorption SPR of gold nanoparticles. The dependence of the UV-Vis spectra of the PDMS mixed to dried or wet Au-NPs is shown in figure 4. It was observed that the measure repeated in several different areas of each sample follows the same trend presented in figure 4.

The absorption band centered at 515 nm–520 nm results from the surface plasmon band of gold nanoparticles [25]. Broad absorption peaks were observed in UV-Vis spectra. The negligible absorbance of PDMS was used as baseline. Absorption is related to the number of particles per unit volume. If the NP size increases the number of NPs in the sample decrease. The absorption intensity reported in figure 4 comparing the same Au-NPs concentration is higher in wet Au-NPs than in dried Au-NPs probably due to their smaller size and consequent increased population of Au-NPs.

The different percentage of Au-NPs in PDMS increased the absorbance up to 90% for the inclusion of 0.2% wt Au-NPs. The spectra of PDMS/Au-NPs at 0.2 wt% (wet Au-NPs) and 0.2% (dried Au-NPs) are adjacent. In the insert of figure 4 a distinct gradual decrease in the absorbance is visible from the PDMS/Au-NPs 0.2 wt% to the PDMS/Au-NPs 0.1 wt% showing at 600 nm 62% of absorbance.

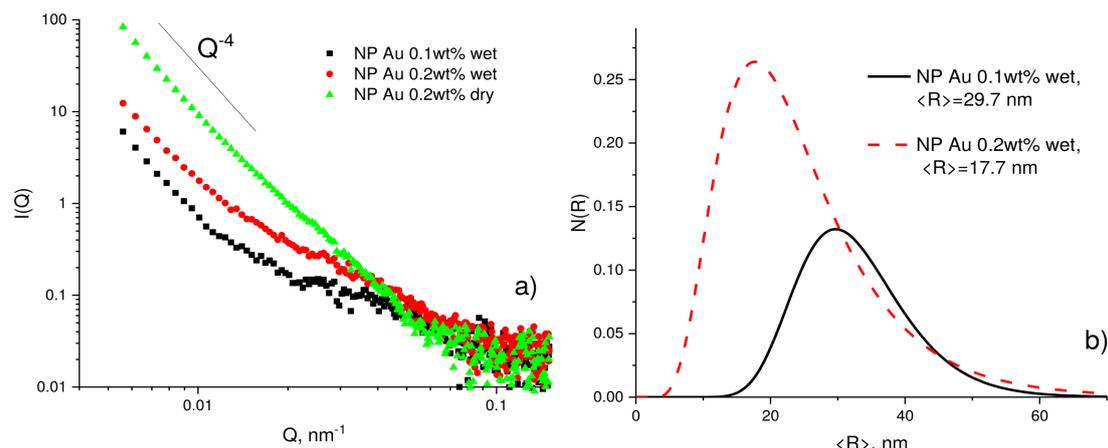


Figure 5. SAXS functions of hybrid composites containing Au NPs after subtracting the virgin PDMS scattering (a) and resulted fitted number density distributions (b).

In figure 4 is observed a wide absorbance peak near 517 nm wavelength due to the presence of nanospheres, with dimension of 10 nm, absorbing the green light. In PDMS containing D-Au-NPs the scattering increases, showing a broader peak shifted towards longer wavelength of about 528 nm, due to the particle's aggregates. This results in the conduction electrons near each particle surface becoming delocalized and shared between nearby particles. The nanoparticles responsible for the red-shifting should have a size of about 60 nm, in agreement with ref. [26].

To know how the shape and size of Au-NPs change, SAXS technique [27] was employed. In a SAXS experiment the soft X-ray beam hits a nanostructured sample and the low angle scattered photons are detected at sample-to-detector distance of 2.5 m. SAXS curve measured at virgin PDMS was subtracted from each SAXS curve for samples containing NPs in order to obtain pure contribution of NPs to the scattering. Resulted SAXS data are showed in figure 5.

SAXS data in low Q region follows power law $I \sim Q^{-4}$ dependence for all samples (figure 5). Such behavior is caused by Porod scattering from large clusters with sharp surface formed from NPs. SAXS curves measured samples with wet NPs were fitted using spherical model with log-normal size distributions. In sample prepared using dry NPs the small ungrouped NPs were not detected in SAXS. The intensity for PDMS containing 0.2% wt dried Au-NPs is higher than for wet Au-NPs with same concentration due to larger cluster size for wet NPs material. The lowest intensity measured for PDMS containing 0.1% wt wet Au-NPs is due to twice lower concentration of Au-NPs. It is might be indirect evidence that microstructure of NPs clusters in PDMS is mostly dependent on type (wet or dry) of NPs but not so much on NPs concentration. In figure 5(b), the number density distribution showing a mean radius of 29.7 nm in wet PDMS/Au-NPs at 0.1 wt% and 17.7 nm in wet PDMS/Au-NPs at 0.2 wt%. However, for verifying this statement SAXS scattering curve should be extended to lower Q by using an ultra-small-angle X-ray scattering (USAXS) technique.

To visualize in real space the shape and size of Au-NPs contained in a PDMS matrix, atomic force microscopy (AFM) on surface was performed. Figure 6 reports 1 μm square AFM images of virgin PDMS and PDMS containing low and high concentration of wet W-Au-NPs and high D-Au-NPs. In addition, 300 nm square AFM images for virgin PDMS and high wet and high dried Au-NPs are presented for a better view of the nano-structures.

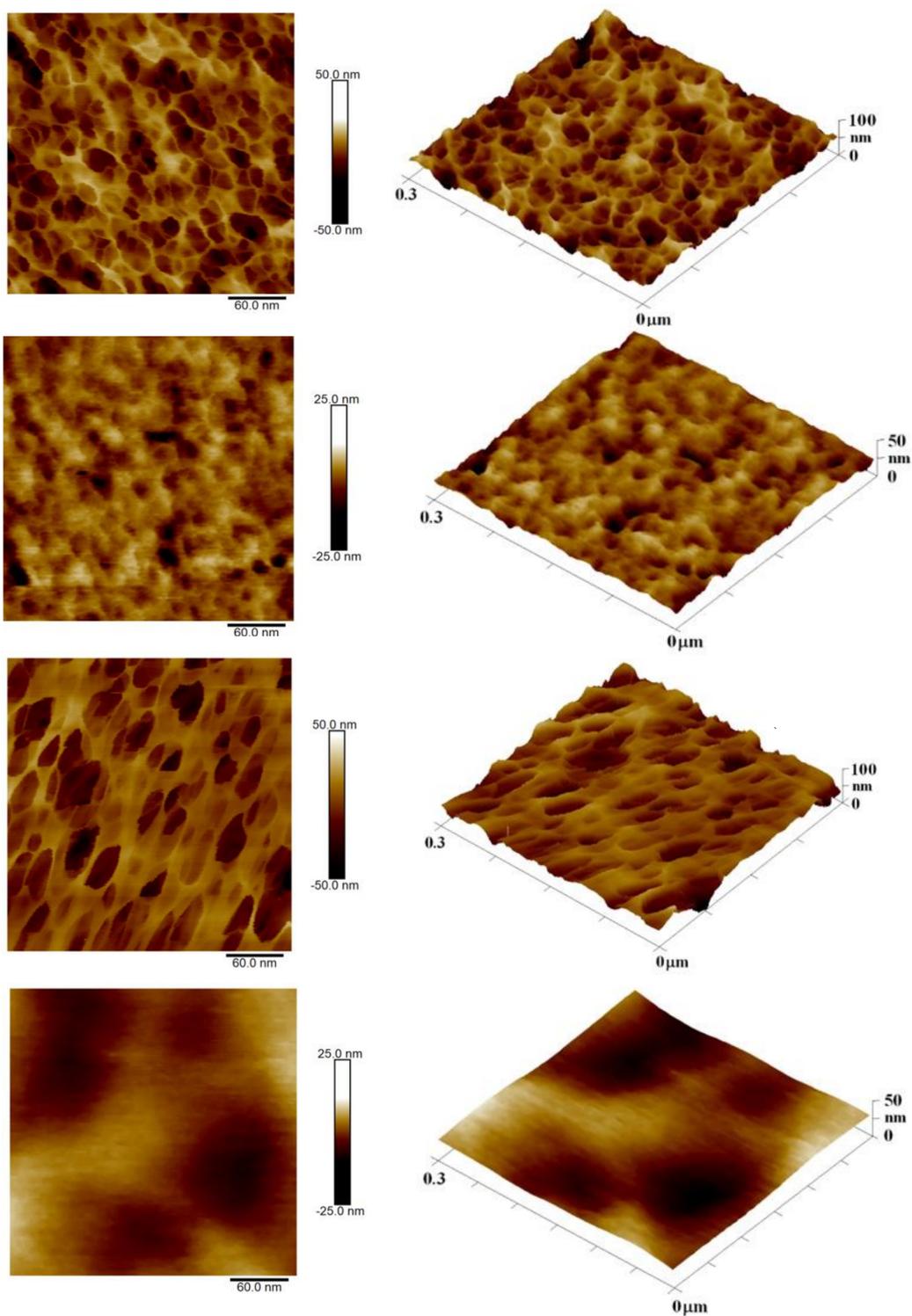


Figure 6. AFM images of surfaces in an area of 300nm square of virgin PDMS 2 D (a) and 3D (b); PDMS containing low concentration of wet Au-NPs in 2D (c) and 3D (d); PDMS containing high concentration of wet Au-NPs in 2D (e) and 3D (f); PDMS containing high concentration of dried Au NPs in 2D (g) and 3D (h).

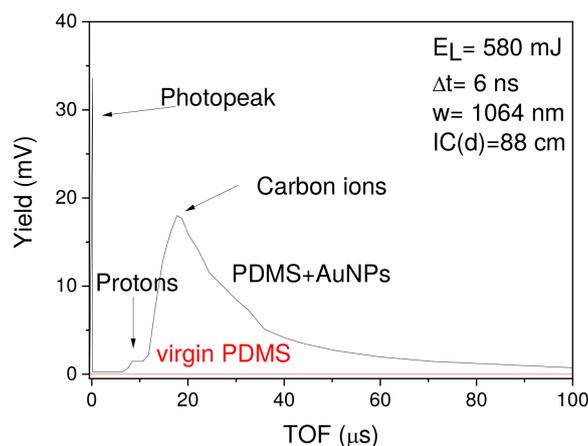


Figure 7. IC-TOF spectrum during the laser irradiation of PDMS+AuNPs.

The nanoparticle aggregation on the solid surface, which shape became irregular, can be deduced from figure 6(e) because the structures/voids contained in virgin PDMS have a maximum size of 30 nm. In figure 6(f) aggregated structures having a maximum size of about 50 nm–60 nm are depicted. In the virgin PDMS a high-density distribution of the structures/voids is observed whereas in PDMS containing 0.2 wt% W-Au-NPs and in 0.2 wt% D-Au-NPs a low-density distribution. Average roughness of 4.1 nm, 2.4 nm, 3.7 nm and 7.0 nm for virgin PDMS, 0.1 wt% W-AuNPs, 0.2 wt% W-AuNPs and 0.2 wt% D-AuNPs respectively were evaluated in areas of $3 \mu\text{m}^2$ to probe the same area explored by wettability measurement.

Lase irradiation of virgin PDMS doesn't show IC signal (as reported in figure 7) due to the high transmittance of the matrix at the laser wavelength of 1064 nm, pulse duration of 6 ns and pulse energy of 580 mJ focused on 1 mm^2 spot surface.

On the contrary, the PDMS+AuNPs 0.1 wt% shows absorbance and emission of photons, electrons and ions. The faster protons emitted from PDMS+AuNPs 0.1 wt% are at about $8 \mu\text{s}$ corresponding to a kinetic energy of about 63 eV. The broad peak around $14 \mu\text{s}$ at half maximum height is due to the emission of carbon ions at energies of about 248 eV. This result confirms that the virgin PDMS transmits the laser pulse while that containing NPs absorbs it generating a hot plasma rich in carbon ions.

4 Conclusions

Polymers containing metal nanoparticles have gained high interest in the optical field, due to their special optical properties, such as dichroism [6], switching [11] and controlled absorption coefficient [28]. The optical responses of Au-NPs embedded in PDMS films are connected to the Au-NPs features, which result from the adopted method. In this work, PDMS was mixed with wet and dried Au-NPs in different concentrations to investigate some of the acquired properties of the polymeric matrix.

The network structure of PDMS makes possible the diffusion of Au-NPs into its matrix. The PDMS network, does not protect the gold nanoparticles from aggregation as observed in the present study. The changes in the morphological and optical properties of the matrix are related

to the presence of the Au-NPs dopants in PDMS working as links uniformly distributed in the polymer. The appearance of pink and violet PDMS-AuNPs composites suggests the presence of Au-NPs aggregates with different size, as confirmed by SAXS and AFM analyses. The UV-Vis measurement shows the maximum peak absorption at around 517 nm–528 nm indicating Au-NPs with size ranging between 10 nm and 60 nm in the investigated samples. The sample exhibiting higher absorption is the PDMS containing 0.2 wt% of Au-NPs due to less aggregation of the fillers and consequent higher number of NPs per volume of matrix. The presence of Au NPs in PDMS improves the absorbance of the matrix as observed also during the laser irradiation of the sample leading to the enhancement of the emitted ion energy, electron density and plasma temperature. It is established that the higher the size of Au NPs is the higher the temperature in the surrounding area and the longer the time to reach the thermal equilibrium [29]. Work is in progress to investigate the thermal behavior of the PDMS containing Au-NPs, which is crucial for the control of the temperature distribution for applications in metallic micro- and nano-fabrication process by direct laser writing but also for material growth, material absorption control, cancer treatment and controlled drug release.

PDMS-Au-NPs composites are a promising material: as optothermal transduction elements enabling the focused localization of heating for selective ablation of cancerous tissue [30]; optical waveguides or switches in optoelectronics. The study of the refractive index and the effect of the ratio of the curing agent and the monomer effecting greatly the shape and size of the gold nanoparticles will be subject to further investigations.

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