



Noble metal nanostructures for double plasmon resonance with tunable properties

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ABSTRACT

We report and compare two vacuum-based strategies to produce Ag/Au materials characterized by double plasmon resonance peaks: magnetron sputtering and method based on the use of gas aggregation sources (GAS) of nanoparticles. It was observed that the double plasmon resonance peaks may be achieved by both of these methods and that the intensities of individual localized surface plasmon resonance peaks may be tuned by deposition conditions. However, in the case of sputter deposition it was necessary to introduce a separation dielectric interlayer in between individual Ag and Au nanoparticle films which was not the case of films prepared by GAS systems. The differences in the optical properties of sputter deposited bimetallic Ag/Au films and coatings consisted of individual Ag and Au nanoparticles produced by GAS is ascribed to the divers mechanisms of nanoparticles formation.

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1. Introduction

In the last decades, noble metal nanoparticles (NPs) attracted the considerable attention of scientific community, which is connected with their strong localized surface plasmon resonance (LSPR) that appears at optical frequencies. This makes noble metal NPs highly valuable for various applications, e.g. for biosensing [1–3], plasmonic solar cells [4–6], decorative coatings [7,8] or for surface enhanced Raman spectroscopy [9–13]. In these applications the noble metal NPs are commonly deposited onto supporting substrate or embedded into a matrix material that protects them from the surrounding environment. The key parameter of such materials is often the position of the plasmon resonance peak that can be tuned over a wide spectral region by the NPs size and shape, their distance, by the surrounding dielectric environment of nanoparticles and, naturally, also by the metal type.

Furthermore, from the point of view of possible technological applications it is highly interesting to fabricate materials with double or even multiple plasmon resonance peaks. The most used approaches that were developed for production of such materials are based either on tuning of geometry of produced NPs [14–17] or on production of core-shell or multi-shell nanoparticles that

combine different metals and/or dielectric materials [18–24]. However, these techniques are in most of the cases rather laborious as they require multiple steps or are not compatible with the demand on green process as they use harsh chemicals for NPs fabrication. Because of this there is growing interest in vacuum based physical routes of nanoparticles production. In this study we investigate two different strategies for production of Ag/Au surfaces – magnetron sputtering and gas aggregation source of NPs – with main focus given to the possibility to produce coatings with double localized surface plasmon resonance with tunable position of individual LSPR peaks.

2. Material and methods

2.1. Magnetron sputtering

The first method for production of Ag and Au nanoparticles was magnetron sputtering. In this case DC, planar, water-cooled magnetrons equipped with either Ag or Au target (2-inch in the case of Au deposition and 3-inch in the case of Ag deposition) were attached onto a vacuum chamber pumped by scroll and turbomolecular pumps (the base pressure in the deposition chamber was lower than 1×10^{-2} Pa). Both Ag and Au targets were sourced from Safina a.s. and had declared purity 99.99%. The sputtering was performed in argon atmosphere at pressure of 3 Pa and with DC current 40 mA.

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In addition, thin fluorocarbon (C:F) interlayer films were deposited in between Ag and Au nanoparticle films in one set of experiments. C:F films were produced by RF magnetron sputtering from polytetrafluorethylene target (81 mm diameter, 3 mm thickness, Goodfellow). The RF frequency was 13.56 MHz and the applied RF power was 40 W. Argon was used as the working gas (pressure of 3 Pa). The deposition time was varied from 10 s up to 80 s, which led to the thicknesses of sputtered C:F films ranging from 2 nm up to 11 nm, as measured by spectroscopic ellipsometry (Woolam M-2000DI). More details regarding C:F deposition may be found in Ref. [25].

2.2. Gas aggregation sources

The second approach for deposition of Ag and Au NPs was based on the utilization of gas aggregation sources (GAS). These GAS systems that were described in more details in previous studies [26–31] consisted of Ag and Au magnetrons (2-inch in the case of Au deposition and 3-inch in the case of Ag deposition) that were placed into water-cooled gas aggregation chambers. The aggregation chambers of both employed GAS systems were ended by an exit cone with an orifice of 2 mm in diameter. Entire GAS setups were mounted onto a vacuum chamber. Argon was used as the working gas. In the case of Ag NPs, the pressure in the aggregation chamber was 40 Pa and the DC magnetron current was 200 mA. In the case of Au NPs, the pressure in the aggregation chamber was 66 Pa and the DC magnetron current was 40 mA.

2.3. Samples characterization

Optical properties of produced nanoparticle coatings were studied by UV–Vis spectrophotometry (Hitachi U2900) in the range 325–900 nm. For these measurements microscopic glass slides (76 × 26 mm, soda lime glass, Marienfeld) were used as substrates.

Morphology of deposited Ag and Au nanoparticle films was evaluated by scanning electron microscopy (SEM). SEM images were acquired by Tescan Mira 3 with acceleration voltage 15 kV using secondary electron detector. Cleaned, one-side polished Si wafers (10 × 10 mm, ON semiconductors) were used as substrates for SEM measurements.

3. Results and discussion

3.1. Ag and Au magnetron sputtering

The first studied deposition technique was DC magnetron sputtering. As can be seen in Fig. 1, where examples of SEM images acquired for sputter deposited gold and silver films for different deposition times are presented, this deposition method leads at initial stages to the appearance of randomly distributed and isolated Ag or Au nanoparticles. The formation of such nanoparticles is due to the diffusion of Ag and Au atoms on the surface of the substrate. Within the diffusion distance, diffusing atoms may be either captured by a surface site or encounter each other which leads to the formation of stable dimers that act as condensation nuclei and adsorb new incoming adatoms. As soon as the probability for diffusing or impinging metal atoms to attach onto already created nanoparticles is higher than their probability to meet another diffusing atom, nanoparticles start to grow in their size that is accompanied by the decrease of the mean inter-particle distance. Formed nanoparticles have hemispheroidal shape as demonstrated by recent experiments [32]. Finally, coalescence of nanoparticles occurs at longer deposition times that results in the formation of metallic network [33].

Variation of sizes, shapes and distances between individual formed nanoparticles with deposition time has strong impact on optical properties of produced coatings (e.g. Refs. [34,35]). Both the increase of NPs size and decrease of the inter-particle distance caused the red-shift in the position of localized surface plasmon resonance peak for both sputtered Ag and Au films. In the case of sputtered silver, 5 s of the deposition time led to the LSPR peak at 440 nm, with 80 s of deposition the LSPR peak shifted to 620 nm (Fig. 2a). Analogously, the gold LSPR peak shifted from 550 nm observed for 3 s of deposition to 790 nm when the deposition time was 40 s (Fig. 2b). Prolongation of the deposition time subsequently caused disappearance of the plasmon resonance peaks due to the coalescence of created nanoparticles and formation of interconnected and conductive metallic networks.

Taking into account results presented in Fig. 2, the position of LSPR peak may be easily tuned by the deposition time. However, the deposited films exhibit only one LSPR peak. The most straightforward approach for production of thin films with double peak resonance seems to be combination of optical properties of Ag and Au nanoparticle films. However, due to the similar bulk lattice constants of silver and gold [36] there is a mutual miscibility of Ag and Au atoms. Because of this optical response of Au deposited on the top of Ag nanoparticles showed only one plasmon resonance. This effect is highlighted in Fig. 3, where UV–Vis spectra of Ag film deposited for 10 s and overcoated by Au film deposited also for 10 s is presented. As can be seen, the LSPR peak of such prepared coatings is around 600 nm, which is value that is in between values observed for samples with only Au or Ag nanoparticles deposited for the same deposition time (dotted lines in Fig. 3).

In order to enable production of double LSPR peak materials, it was suggested to use thin dielectric film to separate Ag and Au nanoparticles [37]. These authors used evaporated polytetrafluorethylene as dielectric interlayer and showed that the coupling between Ag and Au nanoparticles is strongly dependent on its thickness. The very same effect may be achieved when using magnetron sputtered C:F. In order to demonstrate this the deposition time of Au and Ag nanoislands was fixed at 10 s, but Ag and Au nanoparticle films were separated by C:F interlayers with thicknesses varied from 2 up to 11 nm. As it is shown in Fig. 4, 2 nm thick C:F interlayer had no significant effect on the UV–Vis spectra as compared to the spectra obtained without any separation layer. The UV–Vis spectra were again characterized by single LSPR peak with center at 630 nm. Further increase of the thickness of separating C:F layer resulted in gradual red-shift of this LSPR peak towards the value observed for Au films deposited for 10 s. This effect was furthermore accompanied by appearance of the second LSPR peak at 440 nm whose intensity increased with increasing thickness of C:F interlayer. The new LSPR peak corresponds to Ag nanoparticles. For the thickness of separation layer 5 nm, the intensities of both LSPR peaks become equal. Above this thickness the intensity of LSPR peak that corresponds to Ag nanoparticles starts to decrease relative to the intensity of Au LSPR peak. These results clearly indicate, in agreement with previous results [37], that there is an optimal value of thickness of magnetron sputtered interlayer at which the dipole–dipole coupling between Ag and Au nanoparticle films is the strongest.

3.2. Deposition of Ag and Au nanoparticles by means of gas aggregation sources

The second method of deposition of Ag and Au nanoparticles was based on the use of gas aggregation sources of nanoparticles. As highlighted in the previous study [38], the mechanism of NPs formation is markedly different as compared to magnetron sputtering: whereas during the magnetron sputtering the created

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