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Interfering surface and localized plasmon: Tuning the Wood anomaly for biosensing

A.N. Shaimanov[a,b], N.A. Orlikovsky[a,d], E.M. Khabushev[a,c], A.V. Zverev[a,d], A.A. Pishimova[a,d], G.V. Sharonov[a,e], G.M. Yankovskii[a], I.A. Rodionov[a,d], A.V. Baryshev[a,c,*]

[a] All-Russia Research Institute of Automatics, 127055 Moscow, Russia
[b] Moscow State University, 119992 Moscow, Russia
[c] Moscow Institute of Physics and Technology, 141700 Dolgoprudny, Moscow Region, Russia
[d] Bauman Moscow State Technical University, 105005 Moscow, Russia
[e] Shemyakin–Ovchinnikov Institute of Bioorganic Chemistry, 117997 Moscow, Russia

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ABSTRACT

We demonstrate spectra of slabs of plasmonic 1D nanostructures and show their changes when detecting specific biomolecular binding. The slabs were fabricated by electron-beam lithography, they had sub-millimeter dimensions and allowed us to detect a specific binding of low-density lipoproteins. Optical spectra of the slabs exhibiting a spectrally sharp resonant peak have been analyzed numerically to interpret their features and to define structural parameters governing the quality factor of the resonance. We show a comparison between the sensing performances of the different slabs under study, thus discussing their better designs and experimental geometries.

1. Introduction

For detecting and monitoring diseases at early stages and in development, when investigating concentrations of biomarkers in the interval from negligible to reference values, highly sensitive, reliable diagnostic approaches are necessary [1,2]. Optical biosensing techniques–the enzyme-linked immunosorbent assay (ELISA) [3,4] and the Biacore technology utilizing surface plasmon resonance (SPR) [5]–are up to date the most commonly used in healthcare and science. A comparison of the mentioned methods shows that the Biacore assay platform has a competitive advantage over ELISA when probing low-affinity interactions [6].

Recently, various plasmonic and photonic nanostructures are discussed for probing biomolecular reactions illustrating markers of diseases [7,8]. Measurements of lipoprotein fractions in plasma has been integral part of clinical diagnostics for decades. Concentrations of low density and high density lipoproteins (LDL and HDL, respectively) are considered as one of the major risk factor for cardiovascular diseases. Yet, in routine clinical practice, lipoprotein measurements are indirect and use concentrations of cholesterol and triglycerides to calculate apparent lipoprotein composition [9]. Calculated in such way these values have much lower predictive values compared to direct measurements of lipoprotein particles [10]. Moreover, modified forms of lipoproteins, such as oxidized low density lipoprotein, has superior prognostic value compared to their unmodified counterparts. However, concentrations of such modified lipoproteins are aprox. 1000 times lower than that of normal LDL [11,12]. This is why direct detection of low concentrations of normal and modified LDL has great promise for clinical use. SPR sensing has a potential to fulfill this demand [13,14], but there are still no simple and robust clinical devices based on this principle. For an optical biosensor based on a microstructure or nanostructure, the key feature is a resonance probing analytes–the Q-factor and spectral (or intensity) sensitivity of the resonance [15–23]. And, of course, these characteristics are defined by technological perfection of constituent elements and used materials [24–26].

Technology for fabricating large area sensor elements by the so-called hot-embossing and dielectric-heating nanoimprint lithography (NIL) methods is demonstrated [27]. The authors discuss nanostructures supporting a Fano resonance [28–30], obtaining a high-sensitivity to biomolecular binding. It should be pointed, however, that a lab-on-a-chip technology should provide total analysis of many markers of diseases at once and, also, implies handling extremely small biofluid volumes. For this need, sensing elements (slabs) of such chips has to be miniature, cascaded or arranged compactly for multi-channel detection without any loss in sensing performances. The miniature chips can be easily made by utilizing a standard approach – the electron-beam

* Corresponding author at: All-Russia Research Institute of Automatics, 127055 Moscow, Russia.
E-mail address: baryshev@vniia.ru (A.V. Baryshev).

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technology (EBL) for fabrication of a NIL stamp with sub-100-nm features followed by a nanoimprint lithography for fast and cheap replication.

In the present work, we demonstrate 500-μm-sized slabs of plasmonic 1D nanostructures with different designs for detecting LDL. We experimentally and theoretically study their optical spectra, elucidate main dependencies between structural parameters and quality factors of the observed optical resonances, give an explanation on resonant light coupling and demonstrate successful probing of low concentrations of LDL. For the nanostructures having optimal designs, we measured and numerically analyzed optical responses in the transmission and reflection geometries when modeling biomolecular binding for LDL with studied concentration.

2. Samples, experimental and calculation details

For fabricating sub-millimeter sized square slabs with the lateral dimensions of 500 × 500 μm, a hydrogen silsesquioxane (HSQ) negative-type e-beam resist was spincoated on square quartz substrates. Then a polymer conductive layer was spincoated on the resist to avoid a negative charge build up on HSQ, which is removed before development. Electron beam lithography of 1D structures was made using Raith tool at 50 kV acceleration voltage. After standard development, process samples were baked out for final transformation to SiO_x structure of HSQ. On this structured clean surface, we deposed in the same vacuum cycle an ultrathin titanium film (d_{Ti} = 5 nm) followed by 80-nm-thick (d_{Au} = 80 nm for samples A) or 40-nm-thick films of Au (d_{Au} = 40 nm for samples B). Thin films were deposited by multi gun e-beam evaporator (Angstrom Engineering), see Fig. 1. The RMS roughness of gold film surface measured using AFM microscopy was less than 1.5 nm for all the films. The thicknesses d_{Au} were selected to prove performances reported in [31] and as those of the sensing elements in Biacore biosensing systems.

The fabricated 1D pattern had periods of D = 600, 625, 650 and 675 nm, a height and a width of the HSQ ridge were set parameters for all samples d_{HSQ} = 80 nm and h_{HSQ} = 65 nm correspondingly. A reference lithography-made HSQ 1D patterns were measured after depositing 5-nm-thick Cr film in SEM tool in low-kV regime. After metal films deposition all the samples were statistically measured by the e-beam Raith tool with 50 linescans on each structure in automatic regime. The resulted period and line width nonuniformity were about 1% and 5% correspondingly. The final height of HSQ structures is slightly lower (for 5–7 nm) than initial HSQ resist thickness due to development and bakeout processes. The height was measured by a stylus profiler. Evaluation of line edge roughness (LER) revealed that the average LER fluctuated from 2 to 4 nm for a series of fabricated samples. It is worth noting that, in our case, LER is basically formed by the initial LER of the HSQ ridge and roughness of the Au film deposited on the vertical wall of the HSQ ridge. Twelve slabs–three identical rows of the slabs with the above-mentioned periods–were fabricated on each substrate for performing experiments on the detection of specific biomolecular binding.

The transmission spectra were measured for polarized light by a spectrometer (Ntegra Spectra, NT-MDT) in the wavelength range of 700–950 nm. The measurements were carried out for the E_x-polarized light (see Fig. 1). The samples were illuminated by a parallel beam of polarized light in the geometry of normal incidence. The exposed area did not exceed the size of the slabs and a diameter of 300 μm. Collection of the zero-order transmitted light was done by an objective, 10 × (NA = 0.3).

The preliminary testing of the slabs was implemented in distilled deionized water (Figs. 2 and 3) or phosphate-buffered saline solution (PBS). In Fig. 4, differential spectra showing biomolecular binding are discussed, obtained as a difference between experimental spectra before and after analyte introduction (ΔT). The molecular deposition was performed on the slabs according to a protocol designed to maximize

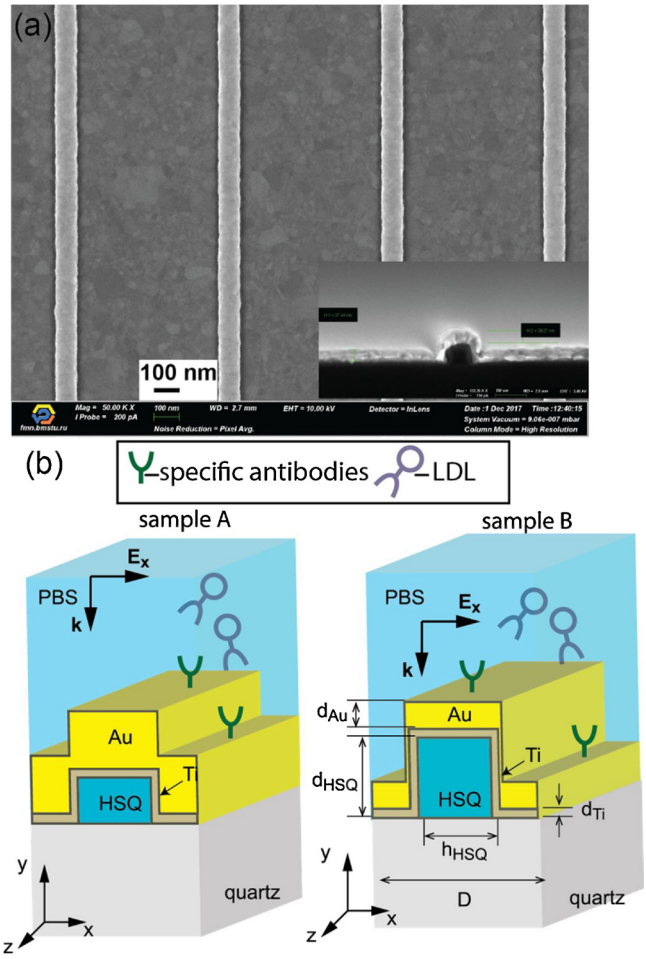

Fig. 1. Experimental samples and calculation model: (a) a scanning electron microscope image of a 1D nanostructure and (b) a sketch of the unit cell of experimental samples and calculation model. Abbreviations: HSQ–hydrogen silsesquioxane, LDL–low density lipoproteins, PBS–phosphate-buffered saline solution.

the spectral response of the sensor to the specific molecular binding. A CS_2 molecular linker was used to establish a covalent bond of the slab surface with the amino groups of used antibodies [32]. Briefly, antibodies at 100 μg/ml were mixed with 0.1% CS_2 in PBS and deposed in a drop on the slab and incubated for 2 h at room temperature. Rabbit polyclonal antibodies for ApoB-100 and ApoE (Imtec, Russia) were used for testing specific and non-specific LDL binding, respectively. LDL was isolated from blood plasma of a healthy donor by means of gradient ultracentrifugation in 12% iodixanol density gradient medium (OptiPrep™, Sigma-Aldrich). Purity of the LDL was validated by electrophoresis in 7% polyacrylamide gel and proved to be > 90%. To minimize non-specific binding of the analyte with the sensor surface, a 0.1% solution of bovine serum albumin (BSA) was used to treat the slabs' surfaces for 10 min before analyte deposition, thus filling the vacancies between immobilized antibodies. 0.1% BSA was also present in all washing, incubation and measurement solutions during further procedures.

For fitting experimental spectra, COMSOL Multiphysics models were set up for one unit cell of the 1D grating, with the Floquet boundary conditions along the *x* axis describing the periodicity and being infinite along the *z* axis [Fig. 1(b)]. The models were prepared so that to take into accounts the followings: the dielectric constants of constituent materials; epsilons of thin films of HSQ and Au were evaluated by a VASE ellipsometer (J.A. Woollam Co). It should be pointed out that calculated spectra are in a good qualitative agreement with

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