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READOUT BEAM COUPLING STRATEGIES FOR PLASMONIC CHEMICAL OR BIOLOGICAL SENSORS

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Abstract: Plasmonic devices are among the most sensitive contemporary sensors of chemical or biological analytes, in some cases even reaching single molecule sensitivity. These are refractometric devices making use of extreme light concentrations and offering real-time, label-free operation. For their proper operation one needs efficient coupling between the propagating interrogation beam and bound surface plasmons polaritons (SPP) whose wave vector is much larger. An external prism in Kretchmann or Otto configuration can be used for such coupling, or some kind of diffraction coupler or fiber-based endfire coupler. In this contribution we investigate theoretically and experimentally possibilities to fabricate sensor structures that simultaneously exhibit plasmonic properties and ensure diffraction-based coupling. To this purpose we fabricated different micrometer-sized two-dimensional metal-dielectric arrays that can match wave vectors of propagating beams and of SPP and at the same time show plasmonic properties tunable by design. We investigated structures functioning in reflection or in transmission mode, with gold or aluminum as the basic material and with deep subwavelength details. Our structures can be made much more compact than the conventional ones, thus being convenient for monolithic on-chip integration with light source and detector and offering a larger degree of design freedom for multianalyte CORN sensing.

Keywords: CBRN sensing, plasmonic sensors, diffractive optical elements, metasurfaces.

1. INTRODUCTION

Plasmonics is a field of electromagnetics dealing with evanescent waves propagating at metal-dielectric interfaces, often in the context of nanocomposite materials. Plasmonic structures ensure extreme localizations of electromagnetic fields in subwavelength volumes, thus opening a path toward many useful applications [1-3]. One of the key fields

of use of plasmonics is the chemical, biochemical and biological (CBB) sensing [4-6].

Plasmonic sensors offer very high sensitivities, up to the single molecule level [7]. They are refractometric devices, mostly but not exclusively based on the propagation of surface plasmons polaritons (SPP). The presence of analyte in minute amounts in the zone of the strongest evanescent field causes changes of refractive index at the metal-

dielectric interface of the sensor. This change results in modulation of the propagation of the evanescent wave which is probed by an outside beam. The compactness, simplicity, all-solid design and all-optical nature of such sensors is what makes them useful for CBRN agent sensing [8-10].

Coupling between propagating modes (interrogation beam) and bound evanescent modes (surface plasmon polariton or some other kind of evanescent wave like e.g. Dyakonov wave [11-12]) in a plasmonic element is a non-trivial task. An evanescent wave will have large to very large wave vector, which is the main reason behind its ability to localize electromagnetic field into subwavelength volumes. At the same frequency, a propagating wave will have much smaller wave vector. In order for these two to couple, the two wave vectors must match. This can be done by external means [13].

The most often used structure for propagating-to-evanescent wave coupling is the prism in Kretschmann or Kretschmann-Raether configuration [14]. This is basically a transparent prism placed on the surface of the plasmonic sensor in attenuated total reflection configuration. Light incident under certain angle is refracted under the critical angle at the total internal refraction (TIR) surface in parallel with the CBB sensor surface, thus ensuring coupling of the propagating wave to surface plasmon polariton. A variation of this method is the Otto configuration [15], where the TIR side of the prism is divided from the plasmonic surface by a lower refractive index material (i.e. there is a gap between the two).

Another method for efficient coupling between propagating and evanescent modes is to place a diffractive structure at the plasmonic surface. This may be a conventional diffraction grating [2, 13], or some more complex periodic structure. This includes the extraordinary optical transmission apertures [16], basically two-dimensional arrays of holes in an optically opaque metal layer. Another geometry with similar function is the complementary structure, an array of metal islands at the surface of a thin dielectric film.

Geometries of diffractive couplers can vary greatly. Different 2D metal surface corrugations can be used, including various pyramids, cubes, cylinders, etc. Stochastically roughened metal surfaces [13] also belong to diffractive couplers, since a random profile can be actually represented as a spatial superposition of a large number of ordered diffractive arrays with different unit cells. Such couplers offer a relatively low efficiency, but are operational in a wide frequency band, which makes them convenient for white-light applications. The most complex case of diffractive couplers are 3D plasmonic crystals and 3D metamaterials [17, 18]

Both prism and grating coupling belong to phase-matching techniques. Wave vectors can be also coupled by spatial mode matching, by the end-fire configuration [2], where the propagating wave is guided to coincide with the plane of the metal-dielectric plasmonic interface. A disadvantage of this configuration is its rather low coupling efficiency when standard fiber optics is used to guide the propagating beam to the plasmonic structure.

Other methods of coupling include the use of sharp near-field probes like e.g. SNOM microscope tips and the application of charged particle beams [2].

In this work we consider the possibility to modify the geometry of extraordinary optical transmission arrays in order to obtain an additional degree of design freedom. To this purpose we utilize complex shapes of apertures instead of the conventional square or circular ones. These shapes are obtained as a superposition of two or more simple forms and ensure the appearance of nonlocality effects due to field localization at deep subwavelength level [19-20]. Thus we are able to use our structures as couplers in the well-known manner of diffraction gratings and simultaneously to tailor their dispersion in a wide range.

We present here a theoretical, numerical and experimental consideration of our nonlocal structures for plasmonic CBB sensors. We analyze two batches of experimental arrays of micrometer-sized apertures, one of them in aluminum and the other one in gold.

2. THEORY

Extraordinary optical transmission arrays (EOT) represent ordered 2D arrays of apertures with subwavelength dimensions in an optically opaque metal layer [14, 21, 22]. According to the conventional theory, no light should be able to pass through them, since the apertures are too small to permit any polarization to be transmitted. In reality, near 100% can pass through EOT at certain wavelengths. Propagating beams are coupled with plasmon modes by diffraction, and the much shorter wavelength of the latter allows them to pass the subwavelength holes if resonant conditions are satisfied.

Pendry observed [23] that surface electromagnetic waves can be formed in EOT films even at much larger wavelengths than those at which plasmon resonance exists and that their spectral dispersion has remarkable similarity to the one obtained by Drude model. Thus electromagnetic waves at metal surfaces with EOT holes are mimicking surface plasmons polaritons — such surface waves are denoted as spoof plasmons. For a metal film with a thickness h and square holes with a side a ordered in a square photonic lattice with the side d the following dispersion relation is obtained from the coupled mode theory if the effective medium approximation is valid [24]:

$$k = k_0 \sqrt{1 + \frac{1}{\varepsilon_h} \left(\frac{a}{d}\right)^2 \tan^2 \left[k_0 \varepsilon_h^2 h\right]}, \tag{1}$$

assuming that the EOT material is perfect electric conductor. Here h denotes the metal film thickness, a is the side of the square aperture and d is the square lattice constant, k is the wave vector of the spoof plasmon, k_0 is the wave vector in free space, ε_h is permittivity of dielectric material filling the holes, ε_d is the permittivity of the ambient (for the above equation $\varepsilon_d = 1$). By properly choosing materials and geometry, one can tailor the spectral dependence of spoof plasmons to cover any desired wavelength range, regardless of the fact that surface plasmons polaritons cannot exist in it.

In reality metals must be lossy, and their skin depth is

$$d_s = \frac{1}{k_0 \operatorname{Re}\left(\sqrt{-\varepsilon_m}\right)},\tag{2}$$

where ε_m is the dielectric permittivity of real metal.

In this case a generalized form of the dispersion relation for spoof plasmons is valid [25]:

$$k^{2} = \varepsilon_{d}k_{0}^{2} + \left(\frac{\varepsilon_{d}a}{\varepsilon_{h}d}\right)^{2} \frac{k_{0}^{2}\varepsilon_{h}}{a} \left[a + d_{s}(1+i)\right]$$

$$\times \tan^{2}k_{0}h\sqrt{\frac{\varepsilon_{h}}{a}\left[a + d_{s}(1+i)\right]}$$
(3)

Coupling of propagating modes to evanescent ones by an EOT is defined by the lattice constant d. For an arbitrary incident angle θ_i and in the direction of a lattice side the coupling angle θ is determined according to the well known expression

$$\theta = \arcsin\left(\frac{m\lambda}{d} - \sin\theta_i\right). \tag{4}$$

where m is the diffractive order and λ is the wavelength of the incident propagating beam.

According to the effective medium approximation (EMA) (e.g. [26]) used to derive (1) and (3) the exact shape of the EOT aperture is unimportant as long as the effective amount of metal and dielectric remains constant. The reason are the subwavelength dimensions of the aperture, i.e. the fact that the incident light is too "short-sighted" to discern any details. Along one of the in-plane directions one can utilize the simple mixing formula

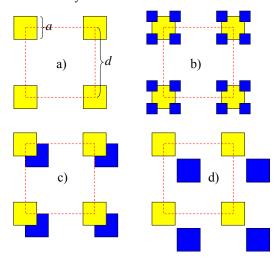
$$\varepsilon_{eff} = \frac{\varepsilon_m (d-a) + \varepsilon_h a}{d},\tag{5}$$

while more complex EMA expressions like Maxwell-Garnet or Bruggeman equation [27] essentially lead to the same conclusion.

In this work we consider a situation when additional deep subwavelength modifications are introduced to the aperture shape (composite aperture or super-unit cells) [19, 20, 28]. According to EMA, this should not cause any noticeable changes in the spectral dispersion of the structure. In reality, however, this is not the case [19]. On the contrary, the introduced details may cause very high local field concentrations, due to, for instance, proximity effect (two shapes at very close distances are enhancing field in the gap between them) or sharp tip effect (field concentration is higher if pointed parts are sharper). If this is so, the approximation of locality is no longer valid and the EMA breaks apart. In this way one can tune the spectral behavior without changing the diffractive properties necessary for wave coupling.

Since one can shape the apertures in any form, in principle it is possible to tailor the spectral dispersion to suit a desired application. In the case of CBB sensors, this means that one could be able to design a spectral dispersion to coincide

with the spectrum of a targeted analyte and thus fabricate a target-specific device without any functionalization and receptor layers. Thus one can obtain a simpler and more compact device with improved selectivity and at the same time with inherent ability to couple with the interrogating beam without any external means.



Picture 1. Deep subwavelength modifications of square EOT array in a square lattice. a) basic EOT; b) array with edge patches; c) array with shifted overlapping squares and d) array with shifted, non-overlapping squares.

Dashed lines denote the unit cell.

Picture 1 shows some simple cases of aperture shape modifications. In Pic. 1a a simple EOT is shown with square apertures in a square array. For simplicity, only four nearest apertures are shown. The unit cell used for calculation of spectral properties is shown by dashed lines. Pic. 1b shows a composite shape where a smaller square "patch" is added to each edge of the basic aperture. Pic. 1c shows the case when the composite shape is obtained by simple overlapping of two identical squares, and in Pic. 1d one can se a composite unit cell consisting of two identical squares at a small distance from each other.

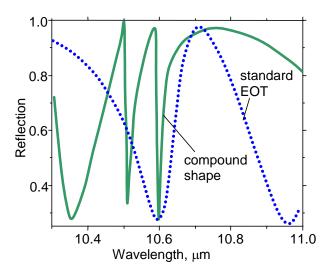
3. NUMERICAL

We used the finite elements method (FEM) to perform our electromagnetic simulations of plasmonic properties of both conventional and generalized EOT structures. We solved Maxwell's equations with the boundary conditions defined for our 2D aperture array. We utilized the RF module of the Comsol Multiphysics software package.

As an illustration, picture 2 presents the results calculated for a conventional EOT with square holes in a square lattice in a 100 nm thick gold layer (dotted line) and for a superstructure with deep subwavelength modifications. Each lattice had a unit cell side 5 μm and a hole side of 2.8 μm . The superstructure was obtained by placing smaller squares in corners of each square hole (corner patches), their side being 1.4 μm , the edge of the larger square coincident with the center of the smaller, as shown in Picture 1b. This is a simple case used for illustrative purposes. The real subwavelength geometry may include any combination of primitive patterns, as long as the composite apertures remain arranged in the same 2D lattice.

The unit cell was defined by locating its center exactly in the middle between four surrounding holes. In this way each corner of the unit cell contains a quarter of a hole. Simulations were done for a normal incidence of the interrogating beam.

It can be seen in Picture 2 that the EOT with composite apertures shows a much richer spectral behavior, with two additional peaks that do not appear in the simple EOT, their positions being dependent on the geometry of deep subwavelength modifications. Our calculations for different geometries (not shown here because of the paper length restrictions) show richer spectra, sometimes even vastly so.



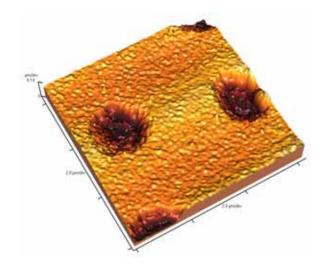
Picture 2. Reflection coefficient of a simple EOT array (dotted) and superstructure (solid)

4. EXPERIMENTAL

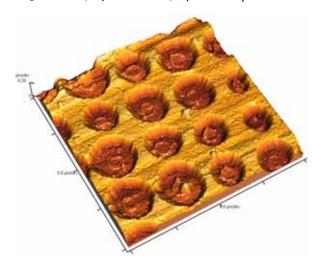
We used single-side polished n-type single crystalline silicon 375 μm thick substrates, <100> orientation, 2-5 Ωcm resistivity. In our first batch of samples 500 nm silica layer was formed on the polished surface by thermal oxidation. A 30 nm chromium layer was further sputtered (binding buffer) and a 100 nm gold layer was sputtered over the Cr buffer. 500 nm thick positive photoresist AZ1505 was spin-coated on top of the gold layer and patterns were laser drawn using LaserWriter LW 405, spot size 2 μm . After resist removal, gold and chromium layers were removed by isotropic wet etching.

The second batch was fabricated using the same Si wafers with a 600 nm thick thermally oxidized silica layer. A 500 nm thick aluminum layer was sputtered over the thermal oxide and an AZ1505 resist layer 500 nm thick was spin coated over aluminum. Patterns were again defined using LaserWriter, and Al layer was removed by isotropic wet etching.

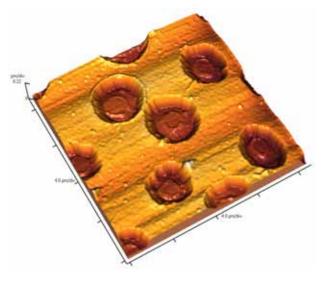
The surface morphology of our experimental samples was characterized by atomic force microscopy in contactless mode (Veeco Autoprobe CP-Research atomic force microscope) and by a dark field metallurgical microscope.



Picture 3. Apertures in optically opaque gold film on SiO_2 substrate, 2 μ m diameter, 5 μ m side square unit cell



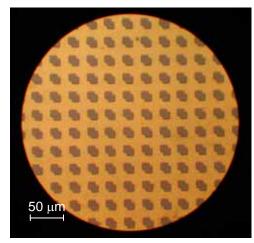
Picture 4. Subwavelength aperture array in gold film with a gradient change in hole diameter

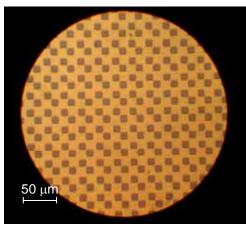


Picture 5. Subwavelength hole pairs ensuring use of nonlocality through deep subwavelength modification of electromagnetic modes

Picture 3 shows the simplest structure in our experiments. It is an array of circular apertures in a 100 nm thick gold film, and the substrate is silica on silicon as described at the beginning of this section. The diameter of the subwavelength apertures is 2 μ m and the side of the square unit cell is 5 μ m. Dark fields at the bottom of each hole correspond to silica, while the lighter areas are gold.

Picture 4 shows an experimental sample of an array of circular holes in gold film. Laser exposition was done with variable intensity from left to right, so that the diameter of the holes gradually changes. Thus a gradient structure is obtained with spatially varying properties. This kind of structures is of interest for coupling between propagating and evanescent modes where the gradients ensure an additional degree of freedom in transforming the optical space [29, 30].





Picture 6. 2D arrays of composite unit cells made of square apertures with identical dimensions in aluminum film. Top: partially overlapping squares; bottom: pairs of apertures with small gap

The case of aperture pairs with a nanometer-sized gap is shown in Picture 5. As determined by AFM measurement, the gap between the apertures in the top part of the picture is about 300 nm at the narrowest point. In this way a submicrometer bridge composed of gold is formed in the middle of the hole pair. In our other experiments, not presented here, we also fabricated complementary structures, where gold islands 2 μ m in diameter were ordered in 2D square array over the same substrate. In such structures proximity effects leading to near field

enhancement between two islands occur in the gap between two neighboring islands.

Square apertures with 8 μm sides were fabricated in aluminum films 500 nm thick. The side of the square unit cell was 24 μm . Picture 6 shows microphotographs of two experimental patterns observed on a dark field metallurgical microscope. In one pattern (top picture) square apertures are overlapping, being shifted along both axes of the square unit cell by 4 μm . The second pattern, shown on the bottom, consists of two neighboring squares, where the distance between the two closest edges in the vertical and the horizontal direction is 2 μm each.

5. CONCLUSION

We developed and fabricated planar micrometer-sized structures that are intended to simultaneously serve as a platform for chemical or biological sensors and as a coupler between propagating and evanescent modes. In this way a general and tailorable tool was developed for further fabrication of SPP CBB sensors. Electromagnetic characterization of the fabricated structures does not belong to the scope of the work and the results related to it will be published elsewhere.

Our couplers utilize Pendry's concept of spoof plasmons in two-dimensional arrays of subwavelength apertures in thin metal films. To further tailor the spectral dispersion of the obtained structures we utilized fine tuning of the position of apertures and the issuing proximity effects, as well as the field enhancement at sharp tips. Thus obtained nonlocality effects ensure the possibility to produce structures with desired transmission/reflection spectra that can be in principle made to coincide with the spectrum of a particular analyte. In this way one can obtain CBB sensors that do not necessarily need surface functionalization by receptors. At the same time we are still able to use our structures as couplers in the well-known manner of the EOT arrays since their diffractive properties are defined by the lattice layout, and not by the shape of their apertures. A kind of hybrid structure is thus obtained, retaining diffraction properties of an EOT but having customizable dispersion. The spectral range is not limited to the UV-visible range as in the conventional surface plasmon polariton sensors, and can be actually used at longer wavelengths, even reaching THz range. In this way a path is open towards more compact sensors that are rugged and more convenient for field use, and at the same time offer an inherently increased selectivity.

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