Advanced fabrication of hyperbolic metamaterials

Hyperbolic metamaterials can provide unprecedented properties in accommodation of high-k (high wave vector) waves and enhancement of the optical density of states. To reach such performance the metamaterials have to be fabricated with as small imperfections as possible. Here we report on our advances in two approaches in fabrication of optical metamaterials. We deposit ultrathin ultrasmooth gold layers with the assistance of organic material (APTMS) adhesion layer. The technology supports the stacking of such layers in a multiperiod construction with alumina spacers between gold films, which is expected to exhibit hyperbolic properties in the visible range. As the second approach we apply the atomic layer deposition technique to arrange vertical alignment of layers or pillars of heavily doped ZnO or TiN, which enables us to produce hyperbolic metamaterials for the near- and mid-infrared ranges.

Effect of substrate on optical bound states in the continuum in 1D photonic structures

Optical bound states in the continuum (BIC) are localized states with energy lying above the light line and having infinite lifetime. Any losses taking place in real systems result in transformation of the bound states into resonant states with finite lifetime. In this work, we analyze properties of BIC in CMOS-compatible one-dimensional photonic structure based on silicon-on-insulator wafer at telecommunication wavelengths, where the absorption of silicon is negligible. We reveal that a high-index substrate could destroy both off-Γ BIC and in-plane symmetry protected at-Γ BIC turning them into resonant
states due to leakage into the diffraction channels opening in the substrate.

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**Organisations:** Department of Photonics Engineering, Plasmonics and Metamaterials, DTU Danchip, ITMO University

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High aspect ratio titanium nitride trench structures as plasmonic biosensor
High aspect ratio titanium nitride (TiN) grating structures are fabricated by the combination of deep reactive ion etching (DRIE) and atomic layer deposition (ALD) techniques. TiN is deposited at 500 °C on a silicon trench template. Silicon between vertical TiN layers is selectively etched to fabricate the high aspect ratio TiN trenches with the pitch of 400 nm and height of around 2.7 µm. Dielectric functions of TiN films with different thicknesses of 18 - 105 nm and post-annealing temperatures of 700 - 900 °C are characterized by an ellipsometer. We found that the highest annealing temperature of 900 °C gives the most pronounced plasmonic behavior with the highest plasma frequency, ωp = 2.53 eV (λp = 490 nm). Such high aspect ratio trench structures function as a plasmonic grating sensor that supports the Rayleigh-Woods anomalies (RWAs), enabling the measurement of changes in the refractive index of the ambient medium in the wavelength range of 600 - 900 nm. We achieved the bulk refractive index sensitivity (BRIS) of approximately 430 nm/RIU relevant to biosensing liquids.

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Authors: Shkondin, E. (Intern), Repán, T. (Intern), Takayama, O. (Intern), Lavrinenko, A. (Intern)
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Highly ordered Al-doped ZnO nano-pillar and tube structures as hyperbolic metamaterials for mid-infrared plasmonics
Fabrication of large area metamaterial structures in a reproducible manner is a tremendous challenge. Here, we realize the fabrication of plasmonic metamaterials for the mid-infrared wavelength region composed of Al-doped ZnO (AZO) pillars by a combination of atomic layer deposition and reactive ion etching.

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Highly Ordered Transparent Conductive Oxide Nanopillar Metamaterials for Mid-Infrared Plasmonics

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High-Quality Ultrathin Gold Layers with an APTMS Adhesion for Optimal Performance of Surface Plasmon Polariton-Based Devices

A low-absorption adhesion layer plays a crucial role for both localized and propagating surface plasmons when ultrathin gold is used. To date, the most popular adhesion layers are metallic, namely, Cr and Ti. However, to the best of our knowledge, the influence of these adhesion layers on the behavior of propagating plasmon modes has not been thoroughly investigated nor reported in the literature. It is therefore important to study the effect of these few- to several-nanometers-thick adhesion layers on the propagating plasmons because it may affect the performance of plasmonic devices, in particular, when the Au layer is not much thicker than the adhesion layers. We experimentally compared the performances of the ultrathin gold films to show the pivotal influence of adhesion layers on highly confined propagating plasmonic modes, using Cr and 3-aminopropyl trimethoxysilane (APTMS) adhesion layers and without any adhesion layer. We show that the gold films with the APTMS adhesion layer have the lowest surface roughness and the short-range surface plasmon polaritons supported on the Au surface exhibit properties close to the theoretical calculations, considering an ideal gold film.

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Large-scale high aspect ratio Al-doped ZnO nanopillars arrays as anisotropic metamaterials.

High aspect ratio free-standing Al-doped ZnO (AZO) nanopillars and nanotubes were fabricated using a combination of advanced reactive ion etching and atomic layer deposition (ALD) techniques. Prior to the pillar and tube fabrication, AZO layers were grown on flat silicon and glass substrates with different Al concentrations at 150-250 °C. For each temperature and Al concentration the ALD growth behavior, crystalline structure, physical, electrical and optical properties were investigated. It was found that AZO films deposited at 250 °C exhibit the most pronounced plasmonic behavior with the highest plasma frequency. During pillar fabrication, AZO conformally passivates the silicon template, which is characteristic of typical ALD growth conditions. The last step of fabrication is heavily dependent on the selective chemistry of the SF6 plasma. It was shown that silicon between AZO structures can be selectively removed with no observable influence on the ALD deposited coatings. The prepared free-standing AZO structures were characterized using Fourier transform infrared spectroscopy (FTIR). The restoration of the effective permittivities of the structures reveals that their anisotropy significantly deviates from the effective medium approximation (EMA) prognoses. It suggests that the permittivity of the AZO in tightly confined nanopillars is very different from that of flat AZO films.

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Midinfrared Surface Waves on a High Aspect Ratio Nanotrench Platform

Optical surface waves, highly localized modes bound to the surface of media, enable manipulation of light at nanoscale, thus impacting a wide range of areas in nanoscience. By applying metamaterials, artificially designed optical materials, as contacting media at the interface, we can significantly ameliorate surface wave propagation and even generate new types of waves. Here, we demonstrate that high aspect ratio (1:20) grating structures with plasmonic lamellas in deep nanoscale trenches, whose pitch is 1/10 – 1/35 of a wavelength, function as a versatile platform supporting both surface and guided bulk infrared waves. The surface waves exhibit a unique combination of properties: directionality, broadband existence (from 4 μm to at least 14 μm and beyond) and high localization, making them an attractive tool for effective control of light in an extended range of infrared frequencies.

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A surface wave (SW) in optics is a light wave, which is supported at an interface of two dissimilar media and propagates along the interface with its field amplitude exponentially decaying away from the boundary. The research on surface waves has been flourishing in last few decades thanks to their unique properties of surface sensitivity and field localization. These features have resulted in applications in nano-guiding, sensing, light-trapping and imaging based on the near-field techniques, contributing to the establishment of the nanophotonics as a field of research. Up to present, a wide variety of surface waves has been investigated in numerous material and structure settings. This paper reviews the recent progress and development in the physics of SWs localized at metamaterial interfaces, as well as bulk media in order to provide broader perspectives on optical surface waves in general. For each type of the surface waves, we discuss material and structural platforms. We mainly focus on experimental realizations in the visible and near-infrared wavelength ranges. We also address existing and potential application of SWs in chemical and biological sensing, and experimental excitation and characterization methods.
Transition from Optical Bound States in the Continuum to Leaky Resonances: Role of Substrate and Roughness

Optical bound states in the continuum (BIC) are localized states with energy lying above the light line and having infinite lifetime. Any losses taking place in real systems result in transformation of the bound states into resonant states with finite lifetime. In this Letter, we analyze properties of BIC in CMOS-compatible one-dimensional photonic structure based on silicon-on-insulator wafer at telecommunication wavelengths, where the absorption of silicon is negligible. We reveal that a high-index substrate could destroy both off-Γ BIC and in-plane symmetry protected at-Γ BIC turning them into resonant states due to leakage into the diffraction channels opening in the substrate. We show how two concurrent loss mechanisms, scattering due to surface roughness and leakage into substrate, contribute to the suppression of the resonance lifetime and specify the condition when one of the mechanisms becomes dominant. The obtained results provide useful guidelines for practical implementations of structures supporting optical bound states in the continuum.

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Conductive Oxides Trench Structures as Hyperbolic Metamaterials in Mid-infrared Range

Nanostructures that possess hyperbolic iso-frequency contours exhibit unique properties of high anisotropy and extremely large wavevectors, which are the key issue to numerous photonic applications from subdiffraction imaging and superlenses to sensing and spontaneous emission enhancement [1,2]. Moreover plasmonics for mid-infrared offer unique applications such as bio-sensing, thermal imaging and quest for novel materials and structures has been continuing [3]. In this report we show that vertical trench structures made of, for example, aluminum-doped ZnO (AZO) or other transparent conductive oxides can function as hyperbolic metamaterials (HMMs) for the mid-infrared wavelength region.

We fabricated a probe sample by a combination of atomic layer deposition (ALD) and dry etch techniques. We templated a Si wafer with deep UV photolithography and made trenches by deep reactive ion etching. Subsequent deposition of an AZO layer by the ALD technique forms vertical AZO trenches with a high aspect ratio (Fig.1a). After homogenization procedure the structure exhibits hyperbolic dispersion (Fig.1b) serving as an attractive platform for light manipulation [4]. Characterization results will be reported at the conference.

Effective medium approximation for deeply subwavelength all-dielectric multilayers: when does it break down?

We report on theoretical analysis and experimental validation of the applicability of the effective medium approximation to deeply subwavelength (period λ/30) all-dielectric multilayer structures. Following the theoretical prediction of the anomalous breakdown of the effective medium approximation [H. H. Sheinfux et al., Phys. Rev. Lett. 113, 243901 (2014)] we thoroughly elaborate on regimes, when an actual multilayer stack exhibits significantly different properties compared to its homogenized model. Our findings are fully confirmed in the first direct experimental demonstration of the breakdown effect. Multilayer stacks are composed of alternating alumina and titania layers fabricated using atomic layer deposition.

For light incident on such multilayers at angles near the total internal reflection, we observe pronounced differences in the reflectance spectra (up to 0.5) for structures with different layers ordering and different but still deeply subwavelength thicknesses. Such big reflectance difference values resulted from the special geometrical configuration with an additional resonator layer underneath the multilayers employed for the enhancement of the effect. Our results are important for the development of new homogenization approaches for metamaterials, high-precision multilayer ellipsometry methods and in a broad range of sensing applications.
Fabrication of Al-doped ZnO high aspect ratio nanowires and trenches as active components in mid-infrared plasmonics

In this work, we report on fabrication of deep-profile one- and two-dimensional lattices made from Al-doped ZnO (AZO). AZO is considered as an alternative plasmonic material having the real part of the permittivity negative in the near infrared range. The exact position of the plasma frequency of AZO is doping concentration dependent, allowing for tuning possibilities. In addition, the thickness of the AZO film also affects its material properties. Physical vapor deposition techniques typically applied for AZO coating do not enable deep profiling of a plasmonic structure. Using the atomic layer deposition technique, a highly conformal deposition method, allows us to fabricate high-aspect ratio structures such as one-dimensional lattices with a period of 400 nm and size of the lamina of 200 nm in width and 3 µm in depth. Thus, our structures have an aspect ratio of 1:15 and are homogeneous on areas of 2x2 cm² and more. We also produce two-dimensional arrays of circular nanopillars with similar dimensions. Instead of nanopillars hollow tubes with a wall thickness on demand from 20 nm up to a complete fill can be fabricated.

Fabrication of deep-profile Al-doped ZnO one- and two-dimensional lattices as plasmonic elements

In this work, we report on fabrication of deep-profile one- and two-dimensional lattices made from Al-doped ZnO (AZO). AZO is considered as an alternative plasmonic material having the real part of the permittivity negative in the near infrared range. The exact position of the plasma frequency of AZO is doping concentration dependent, allowing for tuning possibilities. In addition, the thickness of the AZO film also affects its material properties. Physical vapor deposition techniques typically applied for AZO coating do not enable deep profiling of a plasmonic structure. Using the atomic layer deposition technique, a highly conformal deposition method, allows us to fabricate high-aspect ratio structures such as one-dimensional lattices with a period of 400 nm and size of the lamina of 200 nm in width and 3 µm in depth. Thus, our structures have an aspect ratio of 1:15 and are homogeneous on areas of 2x2 cm² and more. We also produce two-dimensional arrays of circular nanopillars with similar dimensions. Instead of nanopillars hollow tubes with a wall thickness on demand from 20 nm up to a complete fill can be fabricated.
Fabrication of high aspect ratio TiO$_2$ and Al$_2$O$_3$ nanogratings by atomic layer deposition

The authors report on the fabrication of TiO$_2$ and Al$_2$O$_3$ nanostructured gratings with an aspect ratio of up to 50. The gratings were made by a combination of atomic layer deposition (ALD) and dry etch techniques. The workflow included fabrication of a Si template using deep reactive ion etching followed by ALD of TiO$_2$ or Al$_2$O$_3$. Then, the template was etched away using SF$_6$ in an inductively coupled plasma tool, which resulted in the formation of isolated ALD coatings, thereby achieving high aspect ratio grating structures. SF$_6$ plasma removes silicon selectively without any observable influence on TiO$_2$ or Al$_2$O$_3$, thus revealing high selectivity throughout the fabrication. Scanning electron microscopy was used to analyze every fabrication step. Due to nonreleased stress in the ALD coatings, the top parts of the gratings were observed to bend inward as the Si template was removed, thus resulting in a gradual change in the pitch value of the structures. The pitch on top of the gratings is 400 nm, and it gradually reduces to 200 nm at the bottom. The form of the bending can be reshaped by Ar$^+$ ion beam etching. The chemical purity of the ALD grown materials was analyzed by x-ray photoelectron spectroscopy. The approach presented opens the possibility to fabricate high quality optical metamaterials and functional nanostructures.
Highly doped InP as a low loss plasmonic material for mid-IR region
We study plasmonic properties of highly doped InP in the mid-infrared (IR) range. InP was grown by metal-organic vapor phase epitaxy (MOVPE) with the growth conditions optimized to achieve high free electron concentrations by doping with silicon. The permittivity of the grown material was found by fitting the calculated infrared reflectance spectra to the measured ones. The retrieved permittivity was then used to simulate surface plasmon polaritons (SPPs) propagation on flat and structured surfaces, and the simulation results were verified in direct experiments. SPPs at the top and bottom interfaces of the grown epilayer were excited by the prism coupling. A high-index Ge hemispherical prism provides efficient coupling conditions of SPPs on flat surfaces and facilitates acquiring their dispersion diagrams. We observed diffraction into symmetry-prohibited diffraction orders stimulated by the excitation of surface plasmon-polaritons in a periodically structured epilayer. Characterization shows good agreement between the theory and experimental results and confirms that highly doped InP is an effective plasmonic material aiming it for applications in the mid-IR wavelength range.
Hyperbolic Metamaterials with Complex Geometry

We investigate new geometries of hyperbolic metamaterials such as highly corrugated structures, nanoparticle monolayer assemblies, super-structured or vertically arranged multilayers and nanopillars. All structures retain basic properties of hyperbolic metamaterials, but have functionality improved on particular purpose: increased absorption, radiative rate engineering, and selective control over volume plasmon-polariton or directional surface waves.

General information

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Organisations: Department of Photonics Engineering, Plasmonics and Metamaterials, DTU Danchip, Technical University of Denmark
Surface waves on metal-dielectric metamaterials

In this paper, we analyze surface electromagnetic waves supported at an interface between an isotropic medium and an effective anisotropic material that can be realized by alternating conductive and dielectric layers with deep subwavelength thicknesses. This configuration can host various types of surface waves and, therefore, can serve as a platform allowing many applications for surface photonics. Most of these surface waves are directional and their propagation direction is sensitive to permittivities of the media forming the interface. Hence, their propagation can be effectively controlled by changing a wavelength or material parameters. We discover that two new types of surface waves with complex dispersion exist for a uniaxial medium with both negative ordinary and extraordinary permittivities. Such new surface wave solutions originate from the anisotropic permittivities of the uniaxial media, resulting in unique hyperbolic–like wavevector dependencies.

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Authors: Takayama, O. (Intern), Shkondin, E. (Intern), Panah, M. E. A. (Intern), Repän, T. (Intern), Malureanu, R. (Intern), Jensen, F. (Intern), Lavrinenko, A. (Intern)
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Publication date: 2016

Surface Waves on Metamaterials Interfaces

We analyze surface electromagnetic waves supported at the interface between isotropic medium and effective anisotropic material that can be realized by alternating conductive and dielectrics layers. This configuration can host various types of surface waves and therefore can serve as a rich platform for applications of surface photonics. Most of these surface waves are directional and as such their propagation can be effectively controlled by changing wavelength or material parameters tuning.

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Deep subwavelength photonic multilayers fabricated by atomic layer deposition

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Experimental Demonstration of Effective Medium Approximation Breakdown in Deeply Subwavelength All-Dielectric Multilayers

We report the first experimental demonstration of anomalous breakdown of the effective medium approximation in all-dielectric deeply subwavelength thickness (d~λ/160-λ/30) multilayers, as recently predicted theoretically [H. H. Sheinfux et al., Phys. Rev. Lett. 113, 243901 (2014)]. Multilayer stacks are composed of alternating alumina and titania layers fabricated using atomic layer deposition. For light incident on such multilayers at angles near the total internal reflection, we observe pronounced differences in the reflectance spectra for structures with 10-vs 20-nm thick layers, as well as for structures with different layers ordering, contrary to the predictions of the effective medium approximation. The reflectance difference can reach values up to 0.5, owing to the chosen geometrical configuration with an additional resonator layer employed for the enhancement of the effect. Our results are important for the development of new high-precision multilayer ellipsometry methods and schemes, as well as in a broad range of sensing applications.

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TiO$_2$ and Al$_2$O$_3$ ALD Grown Multilayers for Subwavelength Photonics

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**Projects:**

**Dark-field hyperlens: Superresolution imaging and label-free sensing device for biological applications**
The ability to see and manipulate objects with ever decreasing size in a microscope is paramount to the ongoing development of many areas of modern science and technology, from microelectronics to biology and life sciences. The project goal is to demonstrate a technique enabling to image low-contrast nanoscale biological objects in real time without the need for scanning, fluorescent labelling, or fixation. Such a technique can have as great an impact as the invention of the optical microscope itself.

The project goal is achieved by using artificially engineered metal-dielectric nanostructures (hyperbolic metamaterials) with a unique ability to recover information contained in light waves coming from the object's subwavelength features. This is contrary to conventional optical systems where the loss of this information limits the resolution. The central idea of the project is engineering the metamaterial so that only the subwavelength information is transmitted, while any other (background) radiation is filtered out, leading to contrast enhancement similar to the dark-field microscopy. As a result, we would combine superior image resolution (a property of hyperbolic metamaterials) and high image contrast (the result of "dark-field" background filtering). This will be highly desirable for label-free biological imaging scenarios, where faint, weakly scattering objects are abundant. The project aims to verify the concept through direct experimental realization.

Department of Photonics Engineering
Plasmonics and Metamaterials
DTU Danchip
Period: 01/06/2016 → 06/09/2019
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Acronym: DarkSILD
Project ID: 70943
Number of related Ph.D. students: 1
Project participant:
Novitsky, Andrey (Intern)
Takayama, Osamu (Intern)
Shkondin, Evgeniy (Intern)
Phd Student:
Repän, Taavi (Intern)
Project Manager, academic:
Lavrinenko, Andrei (Intern)

**Relations**

Publications:
Dark-field hyperlens for high-contrast sub-wavelength imaging
Dark-field hyperlens: Super-resolution imaging of weakly scattering objects
Operator approach to effective medium theory to overcome a breakdown of Maxwell Garnett approximation
Highly doped InP as a low loss plasmonic material for mid-IR region
Project