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Published in:
Nanoscale

Link to article, DOI:
10.1039/c9nr02471a

Publication date:
2019

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):

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Investigation of effective media applicability for ultrathin multilayer structures

Johneph Sukham, Osamu Takayama, Maryam Mahmoodi, Stanislav Sychev, Andrey Bogdanov, Seyed Hassan Tavassoli, Andrei V. Lavrinenko and Radu Malureanu

Multilayer hyperbolic metamaterials (HMMs) are highly anisotropic media consisting of alternating metal and dielectric layers with their electromagnetic properties defined by the effective medium approximation (EMA). EMA is generally applied for a large number of subwavelength unit cells or periods of a multilayer HMM. However, in practice, the number of periods is limited. To the best of our knowledge, a comparison between rigorous theory, EMA and experiments to investigate the minimum number of layers that allow for the low error of EMA results has not yet been investigated. In this article, we compared the reflectance response of the effective anisotropic HMMs predicted by the scattering matrix method (SMM) and EMA with optical characterization data, having the unit cell twenty times smaller than the vacuum wavelength in the visible range. The fabricated HMM structures consist of up to ten periods of alternating 10 nm thick Au and Al₂O₃ layers deposited by sputtering and atomic layer deposition, respectively. The two deposition techniques enable us to achieve a high quality HMM with low roughness: the root mean square (RMS) is less than 1 nm. We showed that the multilayer structure behaves like an effective medium from the fourth period onwards as the EMA calculation and experimental results agree well having below 4% mean square standard deviation of reflectance (MSDR) for the wavelength range from 500 to 1750 nm with a wide incident angle range. These results could have an impact on the design and development of active metamaterials and their applications ranging from imaging to nonlinear optics and sensing.

1. Introduction

Hyperbolic metamaterials (HMMs) can play a key role in nanophotonics due to their controllability and tunability of the propagation of light. They are highly anisotropic media which have indefinite or hyperbolic dispersion and their properties are determined by their effective electric permittivity or magnetic permeability. Their main property is that they behave like metals in one direction and like dielectrics in the other direction in a broadband wavelength range of operation. HMMs have shown various phenomena and applications in engineering the photonic density of states, enhanced spontaneous decay and recombination rates, super lenses beating the diffraction limit, extremely high biosensing sensitivity, and polarization selectivity. They can be realized in the visible to mid-infrared wavelength region by a combination of metal and dielectrics in the form of multilayers, trenches, and nanorods. Different suitable materials to design HMMs depending on the applications for various wavelength ranges have been studied in detail.

Au is the most practical plasmonic material to fabricate HMMs in the visible and near-infrared wavelength range due to its chemical stability. However, Au does not adhere well on dielectric substrates and thus the films are fully formed only above 15 nm when not using any adhesion layers. The percolation threshold limit for Au films ranges from a thickness of around 8 to 15 nm depending on the deposition technique used and deposition parameters. Ti and Cr are the standard adhesion layers used for Au. The influence of Ti and Cr adhesion layers on ultra-thin Au films has been studied morphologically using electron microscopy and it was found that Cr inter-diffuses within the Au layer forming a Cr-Au alloy with a partially oxidized adhesion layer. Moreover, the introduction of a few nanometer thick metallic adhesion layers has shown to increase the broadening of surface plasmon polariton (SPP) resonance due to the extra absorption and scattering from the metal. As an alternative to Ti and Cr, we recently reported that the silane-based adhesion promoter (3-aminopropyl)trimethoxysilane (APTS) is a better adhesion layer between oxide and Au in...
comparison with metals regarding the support of highly confined surface plasmon modes.\textsuperscript{26}

The propagating bulk plasmon modes in HMMs are due to the coupling of short-range surface plasmon polaritons (SR-SPPs) in each metal–dielectric interface of the multilayer HMMs.\textsuperscript{27,28} This coupling implies that the electric field is localized at the interfaces, thus making the contribution of the adhesion layer even more significant. Therefore, using nonmetallic adhesion such as APTMS is the favorable option for the fabrication of high-quality Au-based HMMs.

The electromagnetic properties of HMMs are routinely predicted by EMA, which is generally applied when the vacuum wavelength of light is much larger than the unit cell or period of the hyperbolic metamaterial.\textsuperscript{29–32} In ref. 32, the authors analyse the applicability of EMA when the number of periods varies and show theoretically that EMA can be applied only from a certain number of periods. Since we use a ZnSe prism to excite high-k waves in the system, we believe that the limits we find are stricter than the ones obtained in the cited work. However, it is a natural question whether EMA is applicable on a fabricated structure with a limited number of periods. The minimum thickness or the number of periods for a structure sufficient to behave like an effective medium is still unclear. Understanding this limit could help in the design of the HMM structures needed for various applications.

To study the applicability of EMA, we fabricated a set of multilayer structures with various periods of HMM samples: 1, 2, 3, 4, 6, 8, and 10 consisting of 10 nm thick Au and Al$_2$O$_3$ layers respectively, using APTMS as an adhesion promoter in between each Au and Al$_2$O$_3$ interface. In terms of the quality of the fabricated multilayer structure, the HMM with four periods has a RMS roughness of 0.40 nm and the final RMS roughness of the 10$^{th}$ period sample is 0.80 nm. We characterized the samples optically using the prism coupling scheme to study their reflectance spectra. Experimental results were then compared with EMA calculations. We used the mean square deviation of reflection (MSDR) to compare the reflection difference between EMA with multilayers and experiment using mean square deviation calculation as shown in eqn (1) and (2), respectively. Comparison certifies that the reflection spectra in this multilayer case start matching the expectation MSDR < 2.5%. The MSDR is defined as

$$MSDR = \sqrt{\frac{1}{N} \sum_{\lambda, \phi} (R(\lambda, \phi)_{\text{multilayer}} - R(\lambda, \phi)_{\text{EMA}})^2}.$$  \hspace{1cm} (1)

Table 1 Table of mean-square error reflectance, MSDR < 2.5%, between multilayer structures and EMA-based HMM structures. Green shaded area represents MSDR < 2.5%, where EMA approximately holds and the red shaded area is where the behavior of HMMs is different from EMA.

![Fig. 1 Schematic illustration of the metal–dielectric multilayer structure, excitation and detection of SPPs using the Otto configuration. Symmetric plasmonic waveguide structures are made of SiO$_2$–adhesion layer–Au–adhesion layer–SiO$_2$. The adhesion layers under consideration are 1.0 nm thick APTMS layers.](image)

2. Results and discussion

2.1. Theoretical study

We numerically studied the influence of the thickness of layers ($d_m$ and $d_d$, $A = d_m + d_d$) and a number of periods on the behavior of multilayer HMM properties (reflectance) in comparison with the ones predicted by EMA. For simplicity, we consider an HMM composed of Au and Al$_2$O$_3$ with a metal filling factor of 0.5, without an APTMS adhesion layer. Table 1 shows the summary of numerical analysis of reflectance difference between the multilayer HMM calculated by 1D SMM and 1D EMA. We considered TM-polarized incident light with the magnetic field parallel to the plane of the interface to calculate the reflectance. The reflectance calculated by 1D SMM and 1D EMA is based on the model as shown in Fig. 1. The details of the procedure are presented in the Numerical Simulation section in the Methods section. Here, we define the criteria for the applicability of EMA as the standard deviation of reflectance MSDR < 2.5%. The MSDR is defined as

$$MSDR = \sqrt{\frac{1}{N} \sum_{\lambda, \phi} (R(\lambda, \phi)_{\text{multilayer}} - R(\lambda, \phi)_{\text{EMA}})^2}.$$  \hspace{1cm} (1)
where reflectance is a function of two variables, namely the angle of incidence and wavelength, as shown in eqn (1). The angle of incidence is varied between 27° and 81° and the vacuum wavelength spans from 500 to 1750 nm. $R(\lambda, \phi)$ of the multilayer HMM and $R(\lambda, \phi)$ of the EMA are the simulated reflectance spectra and $N = 57,600$ is the number of points used for the simulation. The analysis results shown in Table 1 imply that in the case of Au and Al2O3 films of 10 nm each, multilayer structures behave similarly to effective media from 4 periods (a total thickness of 80 nm). For 15 nm thick films, the applicability criteria are satisfied from 5 periods onwards. In the case of 20 nm thick layers for Au and Al2O3, the HMM structures behave differently to their EMA counterpart and do not satisfy the standard deviation condition MSDR < 2.5% until at least 10 periods. It should be noted that even the generally accepted condition of $\lambda > 10 \times (d_{\text{Au}} + d_{\text{Al2O3}})$ is still fulfilled, and the EMA condition, as defined by maximum admissible deviation, is not.

Additionally, we observe an unexpected feature of EMA vs. rigorous theory comparison. For the 20 nm : 20 nm case, the MSDR seems to increase after 8 periods, hinting towards the possibility that, for a large number of periods, the applicability criteria might not be satisfied. However, this observation is not fully verified. Therefore, we carry out the experimental demonstration with the multilayer HMM of $d_{\text{Au}} : d_{\text{Al2O3}} = 10 \text{ nm} : 10 \text{ nm}$.

### 2.2. Experimental study

To carry out the experiment, different HMM samples having 10 nm : 10 nm (Au : Al2O3) as a unit cell were fabricated. Each sample had a different number of periods, from 1 to 10, on 4-inch glass wafers, as shown in Fig. 2. Due to the poor adhesion between the oxide and noble metals, we used an APTMS adhesion layer between Au and Al2O3 to obtain smooth and continuous films. The fabrication routine consisting of a combination of two deposition techniques, sputtering for Au and atomic layer deposition for Al2O3, was used to maintain a roughness as low as possible, thus keeping it below 1 nm even after the 10th period. The details of the fabrication procedures of the HMMs are presented in the HMM fabrication section under the Methods section.

We investigated the surface morphology of the Au nanofilms for each period using an atomic force microscope (AFM). The surface roughness of each Au layer is shown in Table 2. The roughness of the multilayer sample increases with the number of periods, thus putting a practical limit on the maximum number of periods achievable. The films will form pin holes and defects if the roughness is more than 1. The scanning electron microscopy (SEM) and AFM images of the final 10th period Au layer are shown in Fig. 3a and b. The images confirm that the Au layer is continuous without any defects or pinholes. The final Au layer has a RMS roughness of 0.80 nm. The increase in the surface roughness from the 8th period to the 10th period is due to the accumulation of roughness by more periods. Since the roughness is below 1 nm in this work, the effect of scattering is not visible in the optical characterization of the samples even after the 10th period.

We conducted a series of reflection measurements on structures with a different number of periods using the Otto configuration setup with TM-polarized light, as schematically described in Fig. 1. We used TM-polarized light to excite the plasmon modes supported by the multilayer hyperbolic metamaterial. On top of the HMMs, we sputtered a 50 nm SiO2 layer to maintain the symmetry of the modes and also to allow for the excitation of high order modes. TM polarized light is used to excite the modes. This way, we can identify the SPP-type modes and analyze their behavior. The reflection measurements are shown in Fig. 4d-f and j-l for periods 1, 2 and 3 and 4, 8 and 10, respectively. The noise close to 1100 nm visible in the experimental dispersion diagram is due to normalization errors arising from the spectral characteristics of the light source and not related to the sample’s response.

We performed 3D simulations of the reflectance of the multilayer HMMs of various periods as described in the Numerical simulations section in the Methods section to compare with the experimental reflection measurements. In our 3D simulations, we also considered the APTMS adhesion layers. The

![Fig. 2](Image) Images of various HMM samples with 1, 4, 8 and 10 periods denoted by the roman numerals. Each sample is a quarter of a 4-inch wafer in size.

![Fig. 3](Image) Topological characterization of the 10th period Au layer of the HMM. (a) SEM image showing a continuous layer with no interruptions. (b) AFM image for quantifying the roughness.

<table>
<thead>
<tr>
<th>Periods</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>6</th>
<th>8</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roughness (nm)</td>
<td>0.30</td>
<td>0.32</td>
<td>0.36</td>
<td>0.40</td>
<td>0.50</td>
<td>0.54</td>
<td>0.80</td>
</tr>
</tbody>
</table>
For the single period structure, the internal reflection (TIR) condition between the ZnSe prism reflection region beneath 35° is due to the violation of total internal reflection (TIR) condition. This leads us to state that the 3D EMA simulations are applicable only after the 4th period. This conclusion further supports the one obtained from Table 1.

One should note that in Fig. 5 there is a clear difference between the 1D simulation results used for the theoretical study and the 3D simulation and the experimental ones. Therefore, it is more relevant to use 3D simulations to compare with the experimental data, while the 1D SMM simulations may be used for comparing the two theoretical approaches.

Fig. 4 Dispersion diagram of the HMM modes for ZnSe prism–SiO₂ (50 nm)–HMM–SiO₂ (500 μm) structures. 3D EMA simulations (a–c) and (g–i) and measurements (d–f) and (j–l) with HMMs of periods 1, 2, and 3 and 4, 8, and 10, respectively. The roman numerals represent the number of periods of the HMM.

Fig. 5 Dispersion diagram of the HMM modes for ZnSe prism–SiO₂ (50 nm)–4th period HMM–SiO₂ (500 μm) structures. (a) Experiment, (b) 3D FDTD simulation on EMA HMM, (c) 1D Scattering Matrix Method (SMM) on EMA and (d) 1D SMM on the multilayer HMM.

Table 3 Table of mean square error reflectance between 3D EMA simulation and experiment for various periods of the HMM

<table>
<thead>
<tr>
<th>Periods</th>
<th>3D EMA vs. experiment</th>
<th>1D SMM on multilayer structures vs. experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.9</td>
<td>4.5</td>
</tr>
<tr>
<td>2</td>
<td>5.5</td>
<td>3.4</td>
</tr>
<tr>
<td>3</td>
<td>3.6</td>
<td>1.5</td>
</tr>
<tr>
<td>4</td>
<td>3.8</td>
<td>1.1</td>
</tr>
<tr>
<td>8</td>
<td>3.5</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Simulation results are shown in Fig. 4a–c for periods 1, 2, and 3 and Fig. 4g–i for periods 4, 8, and 10, respectively.

For the first period, as shown in Fig. 4(a and d), the low reflection region beneath 35° is due to the violation of total internal reflection (TIR) condition between the ZnSe prism and the 50 nm SiO₂ layer. For the single period structure, the experimental reflection dip is observed at lower wavelengths between 900 and 1100 nm for the same angle of incidence between 60° and 80° as compared to the broad reflection dip between 1300 and 1600 nm from the simulation. This leads to a mean square reflection difference between effective medium prediction and experimental data of 16%. We used MSDR to calculate the reflection difference between the simulation and experiment as defined in numerical simulations, as shown in Table 3. The MSDR between experiment and simulation in Table 3 was calculated by

$$ \text{MSDR} = \frac{1}{N} \sum_{\lambda, \phi} \left[ R(\lambda, \phi)_{\text{experimental}} - R(\lambda, \phi)_{\text{simulation}} \right]^2, $$

where $\phi = 27°–81°$ and $\lambda = 500–1750$ nm and $R(\lambda, \phi)_{\text{experimental}}$ and $R(\lambda, \phi)_{\text{simulation}}$ are the experimental and simulation reflectance spectra, respectively. $N = 28028$ is the number of points used in this case.

With increasing the number of periods, the reflection dip at long wavelengths blue shifts and a new mode appears at short wavelengths, as shown in Fig. 4(b, c, e and f). This new mode red shifts and this tendency is consistent in both experiments and theory. However, before reaching a thickness equivalent to four periods, the simulations consistently show the long wavelength mode red shifted compared to experiments.

From the fourth period onwards, the reflection dips in experiment and simulation start to match very well, within 4% MSDR. The reflection diagram on the 8th period also shows that the two modes start to merge, and they completely overlap for the 10th period.

In the calculation for the reflectance of 1D SMM on the multilayer HMM, we considered APTMS layers in Table 3. From Table 3, it can be observed that the reflection difference of the experimental and 3D EMA simulations decreases from 15.9% (the 1st period) to 3.5% MSDR (the 10th period) with an increasing number of periods. This supports the prediction that the effective medium approximation starts to be accurate enough from a minimum number of periods. When comparing the accurate SMM theory with experimental results, one can see that, although generally decreasing, the difference between the spectra is maximum 4.5% MSDR. Since the 1D SMM simulations are assuming the complete geometry of the structure, we can consider that the maximum 4.5% MSDR is the worst case scenario error, a combination between the experimental errors and the uncertainties in the modelling. Therefore, we can consider this number as a threshold for determining the applicability of the 3D EMA simulations. This leads us to state that the 3D EMA simulations are applicable only after the 4th period. This conclusion further supports the one obtained from Table 1.
2.3. HMM field profile

To further study the experimentally observed optical modes of the structure, we investigated the field profiles from the reflection dips of various periods of the HMM.

Fig. 6 shows the real part of the tangential component of the transverse magnetic field profile at the reflection dips with the angle of incidence from 33° to 85° for various periods in the structure: 2, 4 and 10 respectively calculated by the transfer matrix method as discussed in the Numerical simulations section under the Methods section. From Fig. 6a–c, we can observe that the symmetrical mode appears at higher angles and the anti-symmetric mode at lower angles. The modes are due to the existence of metal-dielectric and the anti-symmetric mode at lower angles. The modes are observed that the symmetrical mode appears at higher angles and above the HMM. The roman numerals represent the period of the HMM. For the 10th period, as shown in Fig. 6c, the two modes observed at $\phi = 45.6^\circ$, $\lambda = 603$ nm and $\phi = 43.4^\circ$, $\lambda = 570$ nm merge into one.

3. Methods

3.1. HMM fabrication

The HMM samples were fabricated on 500 μm thick glass wafers. The wafers were precleaned in Piranha solution (70% H2SO4 + 30% H2O2) for 20 minutes to remove any organic dust residues. The unit cell consists of four-layers: 1 nm APTMS – 10 nm Au – 1 nm APTMS – 10 nm Al2O3. Fabrication starts with the immersion of the cleaned wafers into 2.5% APTMS in isopropyl alcohol (IPA) solution for 3 hours to deposit the APTMS adhesion layer. Then, the 10 nm Au layer was sputtered at 2 mtorr pressure with a deposition rate of 10 Å per second. Another layer of APTMS is deposited on top of the 10 nm Au layer in a similar way before the deposition of Al2O3. The deposition of 10 nm Al2O3 was done using atomic layer deposition at a temperature of 200 °C for 93 cycles, where each cycle consists of 0.1 second pulse time of the precursors trimethyl-aluminum (TMA) and H2O. We continued the fabrication of various samples with different periods following the same process steps.

3.2. Optical characterization

The reflection measurements were performed using a high index ZnSe semi-cylinder prism of a refractive index ranging from 2.4 to 2.7 in the visible and near infrared wavelength regime. TM polarized light was used to excite the modes. The light source was a Super-K high continuum broadband laser from NKT Photonics, allowing us to measure from a wavelength of 500 nm to 1750 nm. The samples were placed on the ZnSe prism with the help of a tightening screw with an unavoidable air gap between the sample and the prism. The incidence angles were varied from 27° to 81° with a 2° interval by rotating the mechanical stage. Each incidence angle was measured five times when ascending and five times when descending the angle to avoid the mechanical stage uncertainties. The reference spectrum is the measure of the reflectance without the sample at 45° incidence. Note that the noise close to a wavelength of 1100 nm visible in the experimental dispersion diagram is due to normalization errors arising from the spectral characteristics of the light source and not related to the response of the sample.

3.3. Numerical simulations

A series of calculations listed in Table 1 were performed by the scattering matrix formalism to calculate the reflectance from the multilayer structures. We considered TM-polarized incident light with the magnetic field parallel to the plane of the interface and for the same range of wavelengths of $\lambda = 500$–1750 nm and the angle of incidence as in the experiment, $\phi = 27^\circ$–$81^\circ$ for the case of calculation in Table 1 and $\phi = 33^\circ$–$85^\circ$ for the simulation in Fig. 6.

![Fig. 6 Transverse magnetic field profile of the HMM for (a) 2, (b) 4, and (c) 10 periods, where the red asterisk (*) marks the reflection dips corresponding to the angle, $\phi$ and wavelength, $\lambda$ of the HMM dispersion diagram. The HMM is represented by the yellow and green color for Au and Al2O3, respectively, and the red color represents the APTMS adhesion layer. The grey area represents the SiO2 layer beneath and above the HMM. The roman numerals represent the period of the HMM.](image-url)
The model structure consists of ZnSe prism–SiO₂ (50 nm thick)–HMM–SiO₂ substrate. Here, the HMM is either multilayers of Au and Al₂O₃ or the one characterized by EMA.

According to EMA, the effective ordinary and extraordinary permittivities, \( \varepsilon_o \) and \( \varepsilon_e \), are expressed by

\[
\varepsilon_o = f_m \cdot \varepsilon_m + f_d \cdot \varepsilon_d
\]

\[
\varepsilon_e = \left( \frac{f_m}{\varepsilon_m} + \frac{f_d}{\varepsilon_d} \right)^{-1}
\]

where \( \varepsilon_m \) and \( \varepsilon_d \) are the permittivities of the metal and dielectric, and \( f_m \) and \( f_d \) are the volume fraction of the metal and dielectric layers, respectively. In general, EMA is assumed to hold under the condition that the thicknesses of each layer are deeply sub-wavelength. We note that in our structure with a thickness of 10 nm for both Au and Al₂O₃ layers as compared to the wavelength interval of \( \lambda = 500–1750 \) nm, we maintain the ratio between the period thickness (\( \Lambda = 20 \) nm) and wavelengths of \( \Lambda / \lambda = 1/25–1/87.5 \). The permittivity of the Al₂O₃ film, \( \varepsilon_d \), was measured using an ellipsometer. The permittivity of the Au film, \( \varepsilon_m \), is characterized by the Drude–Lorentz model:

\[
\varepsilon_m = 1 + \sum_{m=0}^{m=5} G_m \Omega_m^2 / (\omega_m^2 - \omega^2 + jo\Gamma_m)
\]

where \( m = 0 \) is the Drude term, thus \( \omega_0 = 0 \).

The permittivities of the ZnSe prism and the fused silica (SiO₂) substrate are taken from Connolly et al. and Malitson et al. respectively. Reflectance spectra of the effective medium approximated HMMs, provided in Fig. 4, are conducted with the use of the commercially available Lumerical software. To compare more precisely with the experimental results, we included the APTMS adhesion layer in the unit cell and used 3D FDTD simulations. The refractive index of APTMS is assumed to be 1.46.

Here, the HMM is an indefinite uniaxial layer homogenized by EMA for a unit cell of 4 layers with three different materials, APTMS – Au – APTMS – Al₂O₃, based on the below formula, where \( f \) parameters stand for volume fractions of \( \text{Al}_2\text{O}_3 \) as \( f_{d1} \), APTMS as \( f_{d2} \) and Au as \( f_m \) in the unit cell.

\[
\varepsilon_o = f_m \cdot \varepsilon_m + f_{d1} \cdot \varepsilon_{d1} + f_{d2} \cdot \varepsilon_{d2}
\]

\[
\varepsilon_e = \left( \frac{f_m}{\varepsilon_m} + \frac{f_{d1}}{\varepsilon_{d1}} + \frac{f_{d2}}{\varepsilon_{d2}} \right)^{-1}
\]

A nested parameter sweep is used to calculate the reflection of the TM-polarized plane wave for angles between 27° and 81° and wavelengths between 500 and 1750 nm for different thicknesses of the homogenized HMM corresponding to 1, 2, 3, 4, 8, and 10 periods.

The transverse magnetic field profile in Fig. 6 is calculated using the transfer-matrix method. We use the reflectance coefficient to obtain the tangential field components at the ZnSe prism–SiO₂ interface. The obtained field was further used to calculate the fields in the structure, considering 1 nm resolution.

4. Conclusions

High-quality periodic multilayer structures with a number of periods from 1 to 10 were fabricated and optically characterized using the prism coupling experiment. The study was to compare the reflectance by the various structures with the one predicted by the effective medium approximation. The structures were fabricated by combining two deposition techniques: sputtering and atomic layer deposition, which allow control of the thickness and minimization of the roughness of each layer of the HMMs. The final RMS roughness obtained was 0.80 nm. The optical characterization also shows a very good agreement with the effective medium approximation calculation by the 3D FDTD method as close as below 4% MSDR in comparison with the experiment from the 4th period of the structure. Our findings show that we need at least 4 periods of the multilayer structure to comply with the criteria for the applicability of EMA.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

This research was supported by the Villum Fonden “DarkSILD project no. 11116”, and the Direktør Ib Henriksen’s Fond, Denmark. Theoretical analysis was carried out with the support of the Russian Science Foundation (No. 17-19-01731). Modeling was carried out with the support of the Ministry of Education and Science of the Russian Federation (3.1668.2017/4.6) and the grant of the President of the Russian Federation (MK-403.2018.2). All the fabrication work was performed at DTU Danchip.

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