NIM_NIL Report Summary

Project ID: 228637
Funded under: FP7-NMP
Country: Austria

Final Report Summary - NIM_NIL (Large area fabrication of 3D negative index metamaterials by nanoimprint lithography)

The aim of NIM_NIL was the development of a production process for three-dimensional (3D) negative index materials (NIMs) in the visible regime combining ultraviolet-based nanoimprint lithography (UV-NIL) on wafer scale using the new material graphene and innovative geometrical designs. This project went beyond state of the art in three important topics regarding NIMs: the design, the fabrication using UV-NIL and the optical characterisation by ellipsometry. Different designs of negative index structures were simulated like split ring resonators, Swiss cross and fishnet taking into account different materials like silver and graphene. We simulated NIMs with response in the infrared as well as in the visible regime and investigated the response for single layer and multilayer NIMs at normal and oblique incidence. In addition, micro-optical devices made of NIM were simulated and the optical behaviour investigated.

According to the suggested design from simulations, NIL stamps were fabricated by e-beam lithography with feature sizes down to 50 nm as well as micro optical structures by variable dose exposure for realising NIM prisms.

UV-NIL imprint processes were developed to fabricate fishnet structures in the beginning in the infrared and later on in the visible regime. To achieve this goal a NIL based lift-off process was developed which was demonstrated to be feasible to realise 200 nm period structures on 2 x 2 cm². All process steps are NIL based and therefore easily upscalable to large areas and therefore mass production. To achieve NIM response in the visible, silver was the material of choice. We developed an annealing and passivation method for the silver structures to improve the optical response of the NIM material and to make it stable more than one month. By applying graphene on top of the structures, we even achieved stable silver structures tested up to now for several months. We further investigate the coupling of graphene on top of NIM material and its optical response. The optimised single layer NIMs were stacked on top of each other by a NIL based process to achieve 3D NIMs. The fabricated samples were measured with ellipsometry and the negative refraction was measured and demonstrated also under oblique incidence. The measurements and comparison of annealed and not annealed samples clearly show the improvement of the silver properties.

The interpretation of data was supported by RCWA simulations and Berreman formalism. The resonance frequency for the final design of the 200 nm fishnet grating was around 650 nm and negative refraction was detected for normal and oblique incidence for single layer as well as multilayer NIMs.

At the end of the project, a 3D micro optical NIM prism was fabricated with NIM layers working in the visible regime. Variable angle transmission measurements illuminating multiple NIM prisms showed clearly the influence of the prism shape and the resonances of the fishnet layers. The measurement of a single NIM prism proofed that the light refracts in negative direction for a wavelengths around 650 nm.

Project context and objectives:
The NIM_NIL project aimed at three main objectives concerning NIMs, the design of NIMs, the fabrication of NIMs and the characterisation of NIMs.

The design of NIMs:
Within NIM_NIL, new geometrical structures of NIMs and materials like silver and graphene were explored to extend the existing frequency limit of nanostructured NIMs of about 900 nm (1) into the visible regime.

Fabrication of NIMs: We extended the existing capability of fabricating NIM single layer structures on small areas (100 µm x 100 µm) mostly by e-beam lithography to 3D structures like prisms fabricated on large area substrates (2.5 x 2.5 cm²) with the option of up-scaling to 300 mm diameter substrates. The technology of choice was the NIL to assure the scalability for mass production.

Characterisation of NIMs:
Exploiting spectroscopic ellipsometry, the negative refraction and its dispersion was directly measured as a function of wavelength using the infrared and visible frequency region. The experimental results were compared to theory and to usual measuring techniques like reflection and transmission measurements of NIMs. It turned out that ellipsometry is a valuable tool to control the fabrication process without destroying the samples therefore it can be used for production in-line control.

The output of NIM_NIL was a micro-optical NIM prism (base 10 µm, height 2 µm) fabricated by NIL to demonstrate for the first time directly the negative refractive index in the visible regime, which is an important step towards industrial applicability. NIL guarantees a 50 - 100 times faster and more cost efficient fabrication process of NIMs on large areas compared to conventional e beam lithography. This experiment using a micro-optical device verifying Snell's law was the first one directly demonstrating negative refraction in the visible regime.

The three main objectives of NIM_NIL were:
(a) to extend the negative index refraction into the visible regime;
(b) the fabrication of these NIMs and their comprehension and rationalisation on the basis of
(c) the characterisation by ellipsometry and theoretical modelling will have a deep impact on the optics industries.

1. Design of artificial metamaterials
Extending the negative refraction into the visible range was achieved by designing new geometrical structures of NIMs and using an annealing and passivation method for the silver structures to improve the optical response of the NIM material and to make it stable more than one month. By applying graphene on top of the structures, we even achieved stable silver structures tested up to now for several months. We further investigate the coupling of graphene on top of NIM material and its optical response. Simulation and theoretical consideration of different designs lead to a NIM structure extending the visible regime to 650 nm at normal and oblique incidence not only for single layer but also for multilayer 3D NIMs.

2. Processing of artificial metamaterials
The NIMs were fabricated using NIL. This technology makes it possible to replicate the geometrical structures for NIMs on large areas in a fast and cost-effective way. Furthermore, new approaches were developed in structuring silver and graphene using NIL. One main outcome was an annealing and passivation method for silver structures making them stable on air - tested up to now for several months.

3. Realisation of optical, electronic and magnetic properties of metamaterials
NIL was used to fabricate features at the nanoscale exhibiting negative electronic and magnetic response on large area as predicted by the design simulation part within NIM_NIL. The NIM optical response was measured by ellipsometry and therefore the simulated optical response could be compared to the measured one. To verify and compare the results, 'traditional measurements for NIMs' i.e. transmission and reflection measurements were also performed. The characterisation was performed for single layer NIMs as well as multi layer NIMs demonstrating negative n in the visible regime at normal and oblique incidence.

Additionally, a demonstrator of these unique optical properties was delivered within NIM_NIL: A 3D NIM prism was fabricated to demonstrate the realisation of the optical properties in a direct way by verifying Snell's law.

4. Innovative collective response at elevated frequencies

Successively, ellipsometry measurements were applied to measuring the NIM response and the annealed and passivated silver structures and also the new material graphene. Single layer NIM samples exhibiting negative refraction within the visible as well as multilayer samples were characterised. Therefore, it was possible to show and understand the response at elevated frequencies in the visible regime using these non-destructive measurements in combination with theoretically calculated models.

Demonstration of results within NIM_NIL

By achieving the above described goals, applications including perfect lenses, cloaking devices and magnification of objects below the diffraction limit can come true. As demonstrator, we used a NIM prism as micro-optical device because it is a valuable tool to verify the negative refraction in an easy but still impressive way.

Project results:

Description of main science and technology (S&T) results and foregrounds:
The main S&T research results and gained foreground are:

(1) fabrication technology for 3D NIM materials and etching processes for micro-optical NIM prisms;
(2) graphene production and applications;
(3) micro and nanostructuring of graphene using UV-NIL;
(4) milling technology for micro optical devices and stamps for NIL;
(5) NIL materials and process for fabrication of micro optical devices;
(6) fabrication of metallic nanostructures using nanoimprint lithography;
(7) processing and passivation of metallic nanostructures;
(8) fabrication of large-scale stamps for UV-NIL;
(9) ellipsometry of graphene;
(10) 3D metallic nanostructures: Fabrication and optical characterisation;
(11) ellipsometry as characterisation method in mass production of optical structures/NIMs;
(12) design of 2D and 3D metamaterials with optical response in the visible regime.

Those results were also summarised by the NIM_NIL consortium in a brochure (available at [http://www.nimnil.org](http://www.nimnil.org) online).

Potential impact:

The following dissemination activities took place during the NIM_NIL project:
A dissemination workshop was organised by PRO and CNR for graphene presenting results to interested companies and discussing possible applications.

The dissemination workshop in JENA took place on 3 and 4 July and a brochure was setup by PRO with input from all NIM_NIL members as well as the other consortia i.e. NANOGOLD, METACHEM AND MAGNONICS.

At the end of the project, another dissemination workshop was organised with the partners, which research on graphene and potential industry partners PROFACTOR and Micro Resist Technology had a booth at MNE2011 and MNE2012 and presented NIM_NIL results.

The following dissemination activities took place during the NIM_NIL project: NNT 2012 (see http://www.nntconf.org/ online for further details)


M. Kafesaki, ‘What are good conductors for metamaterials and Plasmonics?’ - invited talk at workshop: Novel Ideas in Optics (see http://engineering.purdue.edu/~shalaev/workshop/ online for further details)

M. Kafesaki, ‘What is the best conductor for metamaterials’ - talk, EIPBN 2012 (see http://eipbn.org/eipbn-2012-conference-site/ online for further details)


P. Tassin, T. Koschny, M. Kafesaki, and C. M. Soukoulis, ‘Dissipative loss in metamaterials and plasmonics’ - invited talk, SPIE Photonics Europe 2012 (see http://spie.org/x12290.xml online)


C. M. Soukoulis, ‘Wave propagation: From electrons to photonic crystals and metamaterials’, Workshop: Electronic Correlations and Disorder in Quantum Matter; Dedicated to Peter Wölfle's 70th Birthday - invited talk, APS March Meeting 2012
(13) P. Tassin, 'Graphene, superconductors, and metals: What is a good conductor for metamaterials and plasmonics?' - invited talk, EMLC2012 (see http://www.EMLC2012.com online for further details)


(18) C. M. Soukoulis, 'Photonic Metamaterials: Challenges and Opportunities' - oral presentation, Metamaterials2011 (see http://congress2011.metamorphose-vi.org online for further details)

(19) Thomas Oates, Babak Dastmalchi, Kurt Hingerl, Iris Bergmair, Karsten Hinrichs, 'Characterizing metamaterials using spectroscopic ellipsometry' - poster presentation

(20) Babak Dastmalchi, Thomas Oates, Karsten Hinrichs, Michael Bergmair, Kurt Hingerl, 'Spectroscopic ellipsometry of the fishnet metamaterial' - oral presentation


(24) C. Helbert, K. Dietrich, D. Lehr, T. Käsebier, T. Perscht, and E.-B. Kley, 'A dedicated multilayer technology for the fabrication of three-dimensional metallic nanoparticles' - oral presentation, SPIE Optics+Photonics 2011 (see http://spie.org/x57032.xml online)

(25) P. Tassin, T. Koschny, M. Kafesaki, and C. M. Soukoulis, 'Graphene in metamaterials: What makes a material a good conductor?' - oral presentation

(26) P. Tassin, T. Koschny, and C. M. Soukoulis, 'Understanding and reducing losses in metamaterials' - invited talk, WavePro, Crete 2011 (see http://cmp.physics.iastate.edu/wavepro/index.shtml online for further details)

(27) P. Tassin, T. Koschny, M. Kafesaki, and C. M. Soukoulis, 'What is a good conductor for metamaterials? A comparison between metals, graphene, and superconductors' - invited talk, MediNano-3 (see http://www.medinano3.ipb.ac.rs online for further details)

(28) Goran Isic, Milka Miric, Marko Filipovic, Djordje Jovanovic, Borislav Vasic, Radmila Kostic, Rado Gajic, Iris Bergmair, and Kurt Hingerl, Tom Oates, Karsten Hinrichs, Jozef Humlicek, Maria Losurdo, and Giovanni Bruno, 'Spectroscopic Ellipsometry of Few Layer Graphene', poster presentation
The following reviewed publications are available till now (four more will follow):


(3) Congwen Yi, Tong-Ho Kim, Wenyuan Jiao, Yang Yang, Anne Lazarides, Kurt Hingerl, Giovanni Bruno, April Brown, Maria Losurdo, ‘Evidence of Plasmonic Coupling in Gallium Nanoparticles/Graphene/SiC’, small 2012 (online preview); doi: 10.1002/smll.201200694


The following exploitation results were written up for the brochure of the JENA workshop and will have an impact mainly in the optics industry:

Design:
- design of 2D and 3D metamaterials with optical response in the visible regime.

Technology:
- milling technology for micro optical devices and stamps for NIL;
- e-beam lithography with resolution down to 50 nm for NIL stamps;
- NIL materials and processes for fabrication of metallic nanostructures;
- nanoimprint Lithography materials and processes for fabrication of micro optical devices;
- graphene production;
- annealing and passivation of metallic nanostructures and coupling to graphene;
- graphene structuring;
- fabrication of 3D NIM materials and etching technology for micro-optical NIM prism.

Characterisation:
- optical properties and spectroscopic ellipsometry of metamaterials;
- ellipsometry as characterisation method in mass production for optical structures / NIMs;
- ellipsometry of graphene.

Project website: http://www.nimnil.org

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