Hyperbolic metamaterials for terahertz applications

A.V. Atrashchenko^{1,2}, I.V. Shadrivov^{2,3}, V.P. Ulin¹, E.V. Astrova¹, P.A. Belov², and Yu.S. Kivshar^{2,3}

¹Ioffe Physical-Technical Institute, Russian Academy of Sciences, St. Petersburg 194021, Russia

²National Research University of Information Technologies, Mechanics and Optics, St. Petersburg 197101, Russia

³Nonlinear Physics Center, Research School of Physics and Engineering, Australian National University, Canberra ACT 0200, Australia
astron86@mail.ru

Abstract – We develop a method for fabricating hyperbolic metamaterials for terahertz (THz) applications. We prepare a porous silicon matrix with a triangular lattice of holes, which we fill with copper by means of electrochemical deposition. We study transmission properties of this wire medium using THz time-domain spectroscopy, and demonstrate hyperbolic media properties.

I. INTRODUCTION

Numerous theoretical and numerical studies show exotic properties of hyperbolic metamaterials based on a wire medium [1]. Experimentally, wire media have been realised for microwave and optical frequencies, however at THz frequency it is still a challenge to make such structures. For fabrication of wire metamaterials operating at THz, infrared and visible frequencies, the most promising techniques are based on self-organization processes, which determine the arrangement and structure of metal wires. An example of such technology is the method of electrochemical etching, which allows fabrication of porous matrix of semiconductors [2, 3] and dielectrics [4]. The pores of matrices can be filled with different materials, including metals. This makes such matrices a basis for many composite dielectric-dielectric, metal-dielectric, metal-semiconductor and semiconductor-dielectric structures. The matrices can be fabricated with different hole sizes from several nanometres to several microns with a lattice constant ranging from tens of nanometres to several microns.

In this work, we fabricate *large area* macroporous matrix with a triangular lattice in lightly doped silicon and filled pores with copper using electrochemical deposition. As a result, the this medium exhibits hyperbolic media properties in the terahertz frequency range.

II. FABRICATION OF THE WIRE MEDIUM BASED ON MACROPOROUS SI

To fabricate a triangular lattice of macropores in n-type Si(100) wafer of the thickness of 250 μm , and resistivity of $\rho=5 \Omega \cdot \text{cm}$, we use a photoelectrochemical etching technique [5, 6]. Employing this method, we fabricate a macroporous matrix with through pores (of the same length as the substrate thickness, 250 μm) with the pore diameter of 2 μm and lattice constant of 4 μm (see Fig. 1a). To create a wire medium, we use the method of electrochemical deposition of metals [7]. First, one side of the macroporous matrix is covered by a gold layer using vacuum deposition, so that the whole surface including pores is covered by gold. Then, we perform the metal deposition of copper for 3000 minutes of an alcoholic solution of CuCl_2 at the potential of -1 V in the potentiostatic regime. Next, gold is removed by nitro-hydrochloric acid. The filling fraction of the macroporous matrix is then measured by gravimetric method, and it is found to be approximately 70%. As a result, we create the wire medium (wire diameter of 2 μm , length of 250 μm , lattice constant of 4 μm) made of copper enclosed in the matrix of lightly doped Si (see schematics in Fig. 1b). Surface of the wire medium is shown in Figure 1c. The manufactured sample area is 2.5cm by 2.5cm.

Fig. 1: Wire metamaterial based on a composite Si-Cu. a) Optical microscope image of the surface of the macroporous Si matrix. b) Schematic view of the wire metamaterial comprised of copper wires (diameter of $2 \mu\text{m}$) forming the triangular lattice inside the Si matrix, with the lattice constant of $4 \mu\text{m}$. c) Holes filled with copper are seen in the dark field image.

III. EXPERIMENTAL CHARACTERIZATION

Characterization of the THz response of the fabricated wire medium is performed using EKSPLA THz time-domain spectroscopy setup (THz-TDS). Transmission spectra of the wire medium are measured for different angles of incidence (deviation from the normal of 0, 5, 10 and 15 degrees). Measurement of the transmission spectra for larger angles is not possible in our setup due to a dramatic increase of absorption of THz waves in the wire medium, because of the increase of the electric field component directed along the wires. As a reference, we also measure the transmission spectra for the lightly doped Si substrate and for the macroporous matrix without copper infiltration. All measured spectra are normalised to the free-space transmission.

Assuming purely dielectric response of the metamaterial, we use measured complex transmission data for retrieving the index of refraction of the composite medium, by numerically inverting the Fresnel equations. The intensity I of the THz radiation transmitted through the wire medium can be found as

$$I = I_0 \left[\cosh^2 \chi d + \left(\frac{\chi^2 - \alpha^2}{2\chi\alpha} \right)^2 \sinh^2 \chi d \right]^{-1}, \quad \chi = \frac{\omega}{c} \sqrt{n_1^2 \sin^2 \varphi - n_2^2}, \quad \alpha = \frac{\omega}{c} n_1 \cos \varphi, \quad (1)$$

where d is the thickness of the sample ($250 \mu\text{m}$), φ is the angle of incidence, n_1 and n_2 are the refractive indices of the wire medium and air, respectively, ω is angular frequency, and c is the speed of light.

To test our extraction procedure, we measure the properties of the substrate and of the porous matrix. The refractive index of the lightly doped Si obtained from the analysis of the transmission spectra is found to be approximately 3.4, which is consistent with the values found in literature [8]. The refractive index of the macroporous matrix is around 2.67 which corresponds to the effective refractive index calculated from Maxwell-Garnett model [9].

The results of the retrieved refractive index using Fresnel formula for the wire medium are shown in Fig. 2a. The dependence of the refractive index on the angle of incidence is also calculated using interference fringes at 1.4 THz, and it is shown in Figure 2b. Lower accuracy of determining of the refractive index is due to the difficulty of identifying the peaks of the interference, as compared to the calculations using Fresnel formula.

For the normal incidence of the THz waves, the refractive index is equal to the refractive index of the matrix medium (Si). This result is consistent with the theoretical analysis presented in Ref. [10]. We observe an increase of the refractive index with the increase of the angle of incidence. This is due to the fact that the wire medium is a uniaxial material, and with the propagation at an arbitrary angle the effective index becomes dependent on all components of the dielectric tensor. In this case, the change of the angle involves excitation of the currents in the wires by the component of the electric field directed along the wires. We show that this effect is a clear indication that our sample shows the properties of an anisotropic medium in the form of a hyperbolic material [1]. At the same time, with the increase of the refractive index with angle, both the reflection and absorption in the material grow, making it hard to characterise the sample.

Fig. 2: Effective refractive index of the wire metamaterial. (a) Frequency dependence of the refractive index calculated by inverting the Fresnel equations. (b) Angular dependence of the refractive index at 1.4 THz calculated from the interference fringes and compared to that extracted from the Fresnel equations.

IV. CONCLUSIONS

We have fabricated large scale terahertz wire metamaterial by employing the readily available electrochemical methods. The wire medium is based on the macroporous matrix of lightly doped Si whose pores are filled with copper. We have studied experimentally the properties of this medium such as the change of the refractive index as a function of angle. Such samples fabricated by the electrochemical methods will allow to access experimentally many nontrivial properties of the hyperbolic metamaterials predicted theoretically, including subwavelength imaging, magnification of subwavelength images, ultra-high Purcell factor, and others.

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REFERENCES

- [1] C.R. Simovski, P.A. Belov, A.V. Atrashchenko, Yu.S. Kivshar, "Wire Metamaterials: Physics and Applications," *Advanced Materials*, vol. 24, issue 31, pp. 4229-4248, 2012.
- [2] V.P. Ulin and S.G. Konnikov, "Electrochemical pore formation mechanism in III-V crystals (Part I)," *Semiconductors*, vol. 41, issue 7, pp. 832-844, 2007.
- [3] H. Föll, M. Leisner, A. Cojocaru, and J. Carstensen, "Macroporous semiconductors," *Materials*, vol. 3, issue 5, pp. 3006-3076, 2010.
- [4] A. Eftekhari, *Nanostructured Materials in Electrochemistry. Chap. Highly Ordered Anodic Porous Alumina Formation by Self-Organized Anodizing*, WILEY-VCH Verlag GmbH & Co. KGaA, 2008.
- [5] V. Lehmann and U. Gruning, "The limits of macropore array fabrication," *Thin Solid Films*, vol. 297, issue 12, pp. 13-17, 1997.
- [6] E.V. Astrova, T.N. Borovinskaya, V.A. Tolmachev, and T.S. Perova, "Technique for patterning macroporous silicon and the fabrication of bars of 2D photonic crystals with vertical walls," *Semiconductors*, vol. 38, No. 9, pp. 1088-1091, 2004.
- [7] H. Föll, M.-D. Gerngros, A. Cojocaru, M. Leisner, J. Bahr, and J. Carstensen, "How to make single small holes with large aspect ratios," *Phys. Status Solidi RRL*, vol. 3, No. 2-3, pp. 55-57, 2009.
- [8] J. Dai, J. Zhang, W. Zhang, and D. Grischkowsky, "Terahertz time-domain spectroscopy characterization of the far-infrared absorption and index of refraction of high-resistivity, float-zone silicon," *J. Opt. Soc. Am. B*, vol. 21, issue 7, pp. 1379-1386, 2004.
- [9] J.C. Maxwell-Garnett, "Colours in metal glasses and in metallic films," *Philos. Trans. R. Soc.*, vol. 203, pp. 385-420, 1904.
- [10] S.J. Tyo, "A class of artificial materials isorefractive with free space," *IEEE Transactions on Antennas and Propagation*, vol. 51, No. 5, pp. 1093-1099, 2003.