APPLIED PHYSICS LETTERS **95**, 023306 (2009)

Negative refractive index metamaterials in the visible spectrum based on MgB_2/SiC composites

Nicholaos Limberopoulos,[1] Alkim Akyurtlu,[1,a] Keith Higginson,[2] Adil-Gerai Kussow,[3] and Charles D. Merritt[4]
[1]*Department of Electrical and Computer Engineering, University of Massachusetts, Lowell, Massachusetts 01854 USA*
[2]*Triton Systems, Inc., 200 Turnpike Rd, Chelmsford, Massachusetts 01824, USA*
[3]*Department of Physics and Applied Physics, University of Massachusetts, Lowell, Massachusetts 01854, USA*
[4]*Optical Physics Branch, Code 5610, US Naval Research Laboratory, Washington, DC 20375, USA*

(Received 1 April 2009; accepted 20 May 2009; published online 14 July 2009)

An isotropic three-dimensional negative refractive index metamaterial has been fabricated and characterized in the visible regime. The metamaterial is based on a structure consisting of polycrystalline magnesium diboride (MgB_2) as the host, providing negative permittivity, and silicon carbide (SiC) nanoparticles embedded randomly within the host, providing negative permeability. The metamaterial was fabricated using hot isostatic pressing to produce a fully dense solid with well-dispersed SiC nanoparticles. The properties of the resulting bulk metamaterial were evaluated using surface plasmon excitation, which showed coupling of both magnetic and electric plasmons, signifying both negative permeability and permittivity at 632 nm. © *2009 American Institute of Physics*. [DOI: 10.1063/1.3152793]

Negative refractive index metamaterials (NIMs) demonstrate propagation characteristics that are dramatically different from those in a medium with positive refractive index and may enable some exotic applications such as superlensing[1,2] and cloaking. In a NIM, the Poynting vector and the phase velocity have opposite directions, which should reveal reversal of both the Doppler shift and Cherenkov radiation.[3] The theoretical and experimental research on NIMs began in the microwave regime,[2] followed by terahertz,[4] and finally, recently, has moved into the infrared[5–9] and optical regions.[10–18]

In this paper, we present experimental validation of the negative index effect in an isotropic metamaterial in the visible regime (632 nm). The metamaterial consists of a matrix of MgB_2 (in a normal state, at room temperature), with nanoparticles of SiC randomly (or regularly) embedded within it.[12] The effective permeability is negative at some frequency range due to a Mie resonance associated with the SiC inclusions, and the effective permittivity is negative below the plasma frequency of the MgB_2 host material. The volume fraction, f, and the radius of the SiC spheres, r_{SiC}, were adjusted to make the regions of $\varepsilon_{\mathrm{eff}}<0$ and $\mu_{\mathrm{eff}}<0$ overlap to obtain negative refraction index within the visible region.[12] The main advantages of this design are the intrinsically low electron scattering losses of MgB_2[19–23] and optical isotropy. Moreover, the random (as opposed to regular) arrangement of SiC enables simpler fabrication approaches.

Bulk MgB_2 can be made by either heating powders of MgB_2[24,25] or the constituent elements[26–28] under similar conditions of temperature and pressure. Fabrication challenges include high vapor pressure of magnesium at sintering temperatures and low oxidative stability of the elements. In the present case, an even distribution of SiC particles is also required to preserve the resonance effects that cause negative effective permeability.

Solid billets of MgB_2 and the MgB_2/SiC metamaterial were made by hot isostatic pressing (HIPing). MgB_2 and β-SiC powders were first milled together, and then hermetically sealed in small niobium cans. Pressurized argon was used to compress the filled can, at a temperature of 600 °C for pure MgB_2 and 700 °C for the mixed powder. The resulting billets were cut with a diamond blade to ~1 mm thickness and polished with 0.25 μm diamond grit immediately before all optical measurements. No changes in surface optical properties were observed up to 30 h after polishing.

For pure MgB_2 billets, ellipsometric measurements were made using a spectroscopic ellipsometer (J. A. Woollam). The data reveals a plasma energy of about 2.1 eV, in accordance with previously published data,[19–23,29] and with the values used for the theoretical studies,[12] validating that design approach.

Scanning electron micrographs of the polished cross section of the MgB_2/SiC composite are shown in Fig. 1. For the majority of the material, a continuous MgB_2 phase surrounds submicron inclusions of SiC (verified by energy dispersive X-ray spectroscopy), consistent with the original metamaterial design. At lower magnification, inclusions of pure MgB_2

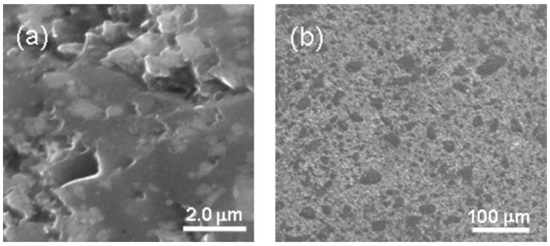

FIG. 1. Scanning electron micrographs of MgB_2/SiC metamaterial: (a) the surrounding matrix is a composite of MgB_2 (dark gray) and SiC (light gray), as per the metamaterial design; (b) at lower magnification, isolated regions of pure MgB_2 (dark gray) are also visible.

---

[a] Electronic mail: alkim_akyurtlu@uml.edu.

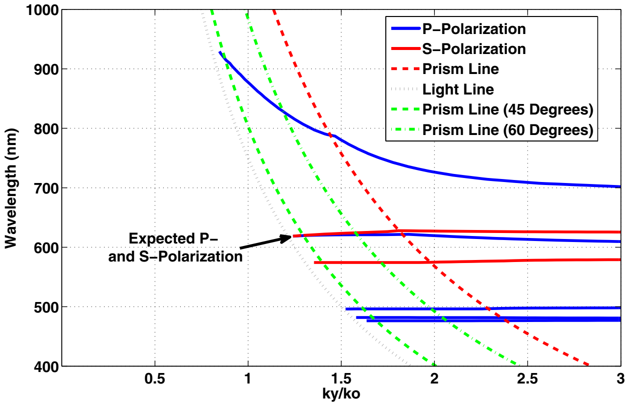

FIG. 2. (Color online) SP dispersion curves for p- and s-polarizations obtained using the theoretically predicted material parameters of our designed NIM. k_y denotes the tangential wavevector along the air/NIM interface, which is given by $k_y = k_o n \sin\theta$, where k_o is the free space wavevector, n is the medium refractive index, and θ is the incidence angle. The light line ($n=1$, $\theta=90°$), the prism line ($n=1.515$, $\theta=90°$), as well as the prism lines at $\theta=45°$ and $\theta=60°$ angles of incidence at the prism/air interface are shown. Excitation of a SP mode at a particular polarization is indicated by an intersection point of the corresponding dispersion curve with one of the lines.

are also apparent, which may arise from insufficient milling of the starting powders.

A test bed for the measurement of surface plasmon (SP) resonances in metamaterials was developed for validating the negative index of the MgB_2/SiC samples. This measures reflectivity as a function of angle where coupling of SPs can be observed, indicated by a "dip" above the critical angle, θ_c, of the prism/air interface ($\theta_c = 41.3°$ for a BK7 prism). For a typical conductor, only p-polarized light can excite a SP when the permittivity is negative. Conversely, for s-polarized light, a SP can be excited only when the permeability is negative. In a NIM where both ε and μ are negative, however, SPs can be excited for both s- and p-polarized light.[30,31] Advantages of this method include independent evaluation of ε and μ and the ability to measure negative parameters in thick or lossy materials. Our experiments were performed in the Otto configuration, where a small air gap exists between the prism and the material under test. The shape of the reflectivity curves depend on the width of the gap as well as on the material properties.

Using the effective material properties from Ref. 12 and $\omega_p = 2$ eV, $r_{SiC} = 120$ nm, $f = 0.3$, and Drude scattering frequency $\gamma = 0.002$ eV, the SP dispersion curves can be constructed for both p- and s-polarizations, as shown in Fig. 2. Here, k_y denotes the tangential wavevector along the air/NIM interface (i.e., $k_y = k_o n \sin\theta$, where k_o is the free space wavevector, n is the medium refractive index, and θ is the incidence angle). The light line, the slowed down light line through the BK7 prism (prism line), and the prism lines at 45° and 60° angles of incidence (at the prism/air interface) are also shown. The prism is used since it is impossible to excite SPs with light coming directly from free space (no intersection point between the dispersion curves and free-space light line exists). At 632 nm, SPs should be excited for both s- and p-polarizations (both dispersion curves coincide at this wavelength and intersect with the prism lines for approximately 45°–60° incidence).

Figure 3 shows reflectivity versus angle for a slab of pure MgB_2 while varying the air gap for both p- and s-polarizations. The light source is a 632 nm HeNe laser. In both cases, the data match the predicted curves well, and as expected, a dip occurs only in p-polarization since MgB_2 is an $\varepsilon < 0$, $\mu > 0$ material at this wavelength. For both sets of results, SP excitation angle shifts to smaller angles as the air gap increases.

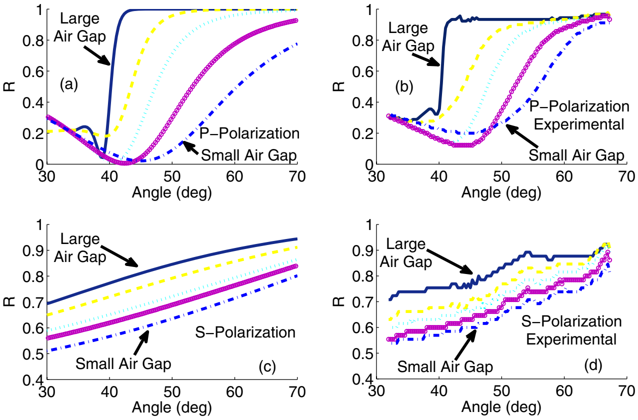

FIG. 3. (Color online) Theoretical [(a) and (c)] and experimental [(b) and (d)] results for the reflection of MgB_2 as a function of incident angle and width of the air gap for p-polarization and s-polarization, respectively.

The results obtained for the MgB_2/SiC sample are shown in Fig. 4 for varying air gap values. The experimental and theoretical reflectivity plots show good correlation for both p- and s-polarizations. For a large air gap, the reflectivity reaches nearly unity above the critical angle, as expected. For smaller air gaps, a reflectivity dip occurs above the critical angle for both polarizations at angles of incidence within the 45°–60° window, as predicted by the dispersion curves. As this occurs for both polarizations, it indicates both $\varepsilon < 0$ and $\mu < 0$ at this wavelength.

We have used HIPing to fabricate a three-dimensional isotropic NIM in the visible regime consisting of MgB_2 as a host material with SiC nanoparticle inclusions.[12] Pure MgB_2 made by the same technique displayed SP excitation for only the p-polarized incident light as expected whereas SP was observed for both the p- and s-polarized incident light for MgB_2/SiC, indicating negative permittivity and negative permeability behavior at 632 nm. The measurements demonstrate that the material is seen by the incoming light as a bulk

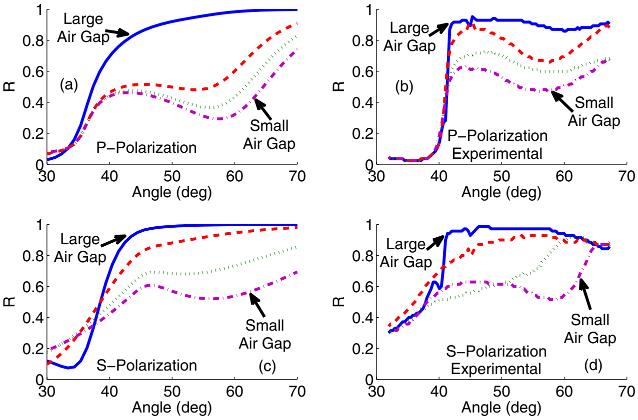

FIG. 4. (Color online) Theoretical [(a) and (c)] results from the material parameters of Fig. 2 and experimental [(b) and (d)] results for the reflection of MgB_2/SiC as a function of incident angle and width of the air gap for p-polarization and s-polarization, respectively.

medium with the appropriate properties. This approach for NIMs utilizing random distributions of inclusions provides an alternative to current designs.

This research was supported by the Air Force Office of Scientific Research Grant No. FA9550-05-01-0314 and Defense Advanced Research Project Agency SBIR Contract No. W31P4Q-08-C-0153. The authors would like to acknowledge the Air Force Research Laboratory at Hanscom AFB and Dr. Al Drehmann for providing UV-VIS spectrometer results; Professor Aram Karakashian and Professor William Goodhue for their assistance in the theoretical and experimental development of the testbed; as well as Scott Morrison, Arthur Gavrin, Douglas Freitag, and John Lock for helpful discussions. HIPing was performed by Stephen DiPietro of Exothermics, Inc in Nashua, NH. Micrographs were obtained by John Knowles of Microvision Laboratories.

[1] J. B. Pendry, Phys. Rev. Lett. **85**, 3966 (2000).
[2] J. B. Pendry, A. J. Holden, D. J. Robbins, and W. J. Stewart, IEEE Trans. Microwave Theory Tech. **47**, 2075 (1999).
[3] V. G. Veselago, Sov. Phys. Usp. **10**, 509 (1968).
[4] T. J. Yen, W. J. Padilla, N. Fang, D. H. Vier, D. R. Smith, J. B. Pendry, D. N. Basov, and X. Zhang, Science **303**, 1494 (2004).
[5] V. Yannopapas and A. Moroz, J. Phys.: Condens. Matter **17**, 3717 (2005).
[6] M. S. Wheeler, J. S. Aitchison, and M. Mojahedi, Phys. Rev. B **73**, 045105 (2006).
[7] S. Zhang, W. Fan, K. J. Malloy, S. Brueck, N. C. Panoiu, and R. M. Osgood, J. Opt. Soc. Am. B **23**, 434 (2006).
[8] A. V. Kildishev, W. Cai, U. K. Chettiar, H. Yuan, A. K. Sarychev, V. P. Drachev, and V. M. Shalaev, J. Opt. Soc. Am. B **23**, 423 (2006).
[9] G. Dolling, C. Enkrich, M. Wegener, C. M. Soukoulis, and S. Linden, Science **312**, 892 (2006).
[10] A. N. Grigorenko, A. K. Geim, H. F. Gleeson, Y. Zhang, A. A. Firsov, I. Y. Khrushchev, and J. Petrovic, Nature (London) **438**, 335 (2005).
[11] G. Dolling, M. Wegener, C. M. Soukoulis, and S. Linden, Opt. Lett. **32**, 53 (2007).
[12] A. G. Kussow, A. Akyurtlu, A. Semichaevsky, and N. Angkawisittpan, Phys. Rev. B **76**, 195123 (2007).
[13] J. Valentine, S. Zhang, T. Zentgraf, E. Ulin-Avila, D. A. Genov, G. Bartal, and X. Zhang, Nature (London) **455**, 376 (2008).
[14] H. Yuan, U. K. Chettiar, W. Cai, A. V. Kildishev, A. Boltasseva, V. P. Drachev, and V. M. Shalaev, Opt. Express **15**, 1076 (2007).
[15] W. Cai, U. K. Chettiar, H. Yuan, V. C. de Silva, A. V. Kildishev, V. P. Drachev, and V. M. Shalaev, Opt. Express **15**, 3333 (2007).
[16] W. Wu, E. Kim, E. Ponizovskaya, Y. Liu, Z. Yu, N. Fang, Y. R. Shen, A. M. Bratkovsky, W. Tong, C. Sun, X. Zhang, S.-Y. Wang, and R. S. Williams, Appl. Phys. A: Mater. Sci. Process. **87**, 143 (2007).
[17] T. F. Gundogdu, N. Katsarakis, M. Kafesaki, R. S. Penciu, G. Konstantinidis, A. Kostopoulos, E. N. Economou, and C. M. Soukoulis, Opt. Express **16**, 9173 (2008).
[18] J. A. Gordon, R. W. Ziolkowski, Opt. Express **16**, 6692 (2008).
[19] W. Ku, W. E. Pickett, R. T. Scalettar, and A. G. Eguiluz, Phys. Rev. Lett. **88**, 057001 (2002).
[20] J. J. Tu, G. L. Carr, V. Perebeinos, C. C. Homes, M. Strongin, P. B. Allen, W. N. Kang, E.-M. Choi, H.-J. Kim, and S.-I. Lee, Phys. Rev. Lett. **87**, 277001 (2001).
[21] A. B. Kuz'menko, F. P. Mena, H. J. A. Molegraaf, D. Van der Marel, B. Gorshunov, M. Dressel, I. I. Mazin, J. Kortus, O. V. Dolgov, T. Muranaka, and J. Akimitsu, Solid State Commun. **121**, 479 (2002).
[22] Y. Fudamoto and S. Lee, Phys. Rev. B **68**, 184514 (2003).
[23] A. Balassis, E. V. Chulkov, P. M. Echenique, and V. M. Silkin, Phys. Rev. B **78**, 224502 (2008).
[24] C. U. Jung, M.-S. Park, W. N. Kang, M.-S. Kim, K. H. P. Kim, S. Y. Lee, and S.-I. Lee, Appl. Phys. Lett. **78**, 4157 (2001).
[25] H. Fujii, K. Togano, and K. Ozawa, Supercond. Sci. Technol. **21**, 015002 (2008).
[26] N. N. Kolenikov and M. P. Kulakov, Physica C **363**, 166 (2001).
[27] M. E. Jones and R. E. Marsh, J. Am. Chem. Soc. **76**, 1434 (1954).
[28] D. Chvostova, V. Zelezny, L. Pajasova, A. Tarasenko, A. Plecenik, P. Kus, and L. Satrapinsky, Thin Solid Films **455**, 213 (2004).
[29] V. Guritanu, A. B. Kuz'menko, D. Marel, S. M. Kazakov, N. D. Zhigadlo, and J. Karapinski, Phys. Rev. B **73**, 104509 (2006).
[30] R. Ruppin, Phys. Lett. A **277**, 61 (2000).
[31] R. Ruppin, J. Phys.: Condens. Matter **13**, 1811 (2001).