Enhanced Magneto-optic Behavior at a Photonic Band Gap of Three-Dimensional Fe_3O_4/SiO_2 Magnetic Photonic Crystals

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***Abstract*-Three-dimensional magnetic photonic crystals were fabricated from silica spheres containing magnetite nano-particles, assembled in a magnetic field. Transmission studies showed significant, sharp features in the magnetic circular dichroism and Faraday rotation, centred on the edge of the third photonic band gap (PBG), but five times narrower than the transmission band gap in absence of a magnetic field. Narrow ellipticity and rotation peaks were observed exactly at the PBG due to the birefringence of the three-dimensional crystal.**

Keywords- Magnetic Photonic Crystals; Magnetic Circular Dichroism; Faraday Rotation; Photonic Band Gaps; Silica Spheres; Magnetite Nano-particles

I. INTRODUCTION

Magnetic photonic crystals (MPCs) are promising candidates for various new functional applications including optical sensors for magnetic fields, adjustable distributers for light and μm-sized polarization controllers for optical communication devices [1-7]. Like any photonic crystal [8-9], their optical transmission spectra exhibit photonic band gap bands (PBGs) that are caused by Bragg diffraction of light inside the structure. In these spectral regions, there are typically strong field enhancements within the crystal, which can magnify weak magneto-optic effects [10-11]. Compared to bulk materials, the magneto-optic Kerr and Faraday effects are reported to be enhanced by factors of 10 – 100 in nano-composite structures containing magnetic nano-particles [3, 11-15]. The enhancement of magneto-optic linear and circular anisotropy, and the possibility of steering the linear and non-linear optical properties by applying an external magnetic field, makes MPCs containing magnetic nano-particles interesting for various optical, electro-optics and magneto-optics applications [1, 11-15].

Three-dimensional (3D) MPCs and heterostructures are difficult to fabricate, hence their transmission and magneto-optic properties have been studied only briefly in a few papers [3, 11-14]. One method of introducing magnetic functionality into the opal structure is to fill the pores between the silica spheres with Fe_3O_4 [11-13]. In these cases, the relevant refractive index contrast is the difference between the silica (n = 1.46) and the Fe_3O_4 with n ~ 2.3 in the energy region of interest [13].

Another approach is to coat the spheres with a nanometer layer of metallic cobalt [15]. The paper reports enhanced Faraday rotation in the vicinity of the first band gap for 3D MPCs of silica spheres that were coated with a thin layer of Co-nanoparticles. Reflection bands were found shifted to slightly lower frequencies, compared with the PCs of the same spheres without the Co-coating. Faraday rotation (FR) and magnetic circular dichroism (MCD) peaks had similar widths to the transmission band gaps.

In this paper, optical and magneto-optic studies of 3d fcc-structured crystals of transparent SiO_2 spheres, which contain a small concentration of nano-sized Fe_3O_4 crystallites, are reported.

The samples were weakly magnetic and their transmission spectra show characteristic photonic band gaps [16]. The measured parameters include: the rotation, θ and the ellipticity, η, as a function of the wavelength λ, and the magnetic field strength, H. We report here measurements of the Faraday rotation, FR~[$\theta(\lambda,H) - \theta(\lambda,-H)$], the magnetic circular dichroism, MCD ~[$\eta(\lambda,H) - \eta(\lambda,-H)$], and also nonmagnetic components $\theta(\lambda,0)$ and $\eta(\lambda,0)$ as a function of wavelength around the photonic band gap. The MCD and FR showed peaks of comparable magnitude at the photonic band gap; however the changes in the magnitude of $\theta(\lambda,0)$ and $\eta(\lambda,0)$ with wavelength, near the PBG, were larger by a factor of about 20. This property of a silica photonic crystal has not been investigated previously.

A splitting of the absorbance peaks for p- and s-polarizations is obtained for light incident at small angles to the normal of the (111) plane surface in the absence of external magnetic field [16]. This behavior, which differs from that reported for similarly-structured, non-magnetic [17-21] or magnetic [10-13] photonic crystals, is shown to be due to a slight distortion of the SiO_2 spheres.

II. EXPERIMENTAL METHODS

The studied MPCs consist of 3-dimensionally organized silica spheres, impregnated with nano-crystallites of Fe_3O_4, in an air matrix [16]. The Fe_3O_4 nano-crystallites, of 8 nm size, were synthesized through a co-precipitation process with oleic acid surfactant. Their magnetization followed a superparamagnetic S-shaped curve with a low coercivity, 10 Oe, and a saturation magnetisation of 70 emu/g. The silica spheres of 440 $\pm$20 nm size were fabricated and impregnated to a fraction of about 0.4 % weight giving a Fe_3O_4 filling factor of 0.15 % volume, in a sol-gel room-temperature process. The saturated magnetization of the impregnated spheres was about 0.25 emu/g at room temperature and they were also almost superparamagnetic, with a low coercivity of 10 Oe.

The MPCs were deposited from an ethanol-suspension of the impregnated spheres on thin glass substrates for samples A and B, and on sapphire for sample S. They were assembled under an external 3000 Oe magnetic field, normal to the substrate surface for A and S, and under an in-plane field for B. While the ethanol evaporated slowly the spheres self-assembled to a crystal nucleating from the surface. A 3D fcc-structured crystal with the (111)-planes parallel with the substrate surface was formed where each sphere had turned into its most favorable orientation in the applied magnetic field (as was seen in-situ in an optical microscope). The average thickness of these MPC samples was 8 layers of spheres, the thickness of sample B was the most uniform, verified with both optical and scanning electron microscopes. The optical quality of the photonic crystals depended on using silica spheres with a smallest size distribution. The results reported here were obtained using silica spheres with a 5% size variation. The best-quality films appeared uniform to the eye and had the deepest band gaps observed in the transmission spectrum.

The optical studies of the magnetic circular dichroism (MCD) and Faraday rotation (FR) were performed using a xenon lamp, a monochromator and a photoelastic modulator that allows for the simultaneous recording of the rotation and ellipticity as a function of wavelength [22, 23, 24]. Measurements were in transmission geometry, with a narrow (1.5mm width) beam incident on the substrate side, normal to the (111) planes of the MPC surface. For plane-polarized incoming light, the ellipticity η and rotation θ of the emerging light were derived. Since there were non-magnetic contributions from both the photonic crystal and the substrate, the MCD and FR were obtained from the difference in signals with magnetic fields of 14 kOe applied parallel and anti-parallel to the incident light.

The photonic band gaps (PBGs) of the samples were determined from the hemispherical transmittance spectra measured with a Perkin Elmer Lambda 900 spectrophotometer equipped with an integrating 150 mm sphere detector. For samples A and B, the specular transmission spectra were also studied using the absolute spectrophotometer described in [25], with p- and s-polarized light, and for incidence angles in the range 0 – 30°, to investigate the effect of the orientation of the magnetic field applied during fabrication on the optical transmission properties.

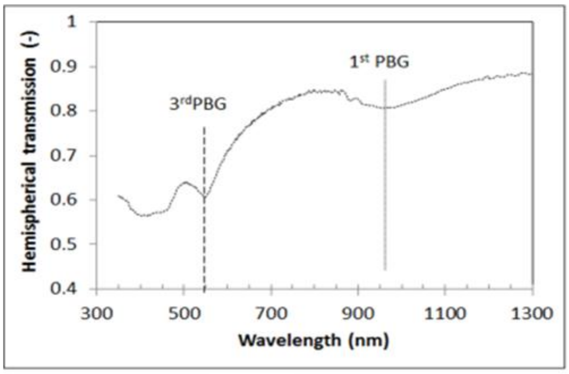
III. RESULTS AND DISCUSSION

The hemispherical transmittance spectrum for sample A (Fig. 1a), measured in the absence of magnetic field, showed clearly two band gaps at wavelengths 970 nm and 550 nm. The hemispherical transmission spectrum, as well as specular transmission and hemispherical and specular reflectance spectra were measured for a square area with side of 1 cm of Sample A. The reference spectrum for the transmission spectra was measured through the same limited square-shaped entrance hole of the integrating sphere detector, as the spectrum of the sample, but without the sample. The analysis of the optical properties was performed as before [16]. The hemispherical absorbance spectrum is as shown in Fig. 1b. The absorbance $A(\lambda)$ is defined by $A(\lambda) = \log_e(I_{incident}/I_{transmitted}(\lambda))$. The spectrum was fitted to a combination of five Gaussian peaks and a Rayleigh scattering background, as shown in Fig. 1b. Bragg diffraction is allowed from planes [*h, k, l*] where *h*, *k*, *l* are either all even or all odd for the f.c.c. structure; however due to the finite lattice, stacking faults and other imperfections, contributions from all the other planes [*h, k, l*] are also allowed. For the geometry used here where the light is incident along (1,1,1) and perpendicular to the film, the wavelengths of the absorption features are given in terms of the lattice constant of the opal lattice a_0 and the appropriate refractive index n_{hkl} by $\lambda_{hkl} = \frac{2n_{hkl}a_0}{\sqrt{3}}\left[\frac{h+k+l}{h^2+k^2+l^2}\right]$. The first peak can be assigned to [1,1,1] and the third peak to a combination of Bragg reflections, [200], [220] and [222]; the broad second peak is due to contributions from [211], [210] and [221] and the higher peaks most probably arose from Bragg reflections from planes [131] and [422] and [130], [21-1]. An unambiguous interpretation of the absorption requires an evaluation of n_{hkl} for each plane and the effects of the distortion induced in the samples by the ***B*** field.

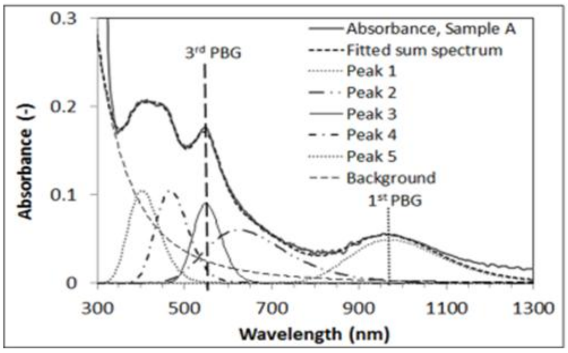
The MCD and FR spectra, shown in Fig. 1c, were measured for sample S over the wavelength range 350 – 600 nm. The background signal from the sapphire substrate for sample S is included in the MCD and FR curves. The MCD is the ratio of the difference between the transmitted light in left and right circular polarization to their average [23, 24]. The MCD spectrum showed a much narrower MCD peak (with FWHM- width less than 10 nm) than the corresponding 3rd transmission PBG of sample A (FWHM width 50 nm), as seen from Fig. 1a and b; the wavelength resolution had to be reduced to 5 nm in order to resolve this narrow peak. The wavelength at which the maximum MCD occurred was slightly less than that for the maximum of the FR as was reported earlier [12, 13]. Measurements were made of the field dependence of the ellipticity and rotation at selected wavelengths.

The magnetism of the small Fe_3O_4 nano-particles located inside the spheres introduces a complex term $\tilde{\varepsilon}_{xy}$ in the dielectric tensor in the photonic lattice. It is very small, but its effect is enhanced due to the photonic structure at the PBG [1, 3, 9-11], leading to the narrow magnetic-optic MCD, η, and FR, θ, peaks observed in Fig. 1c. For lattices with low absorption, η and θ are related to the real and imaginary parts of $\tilde{\varepsilon}_{xy}$; $\eta = \frac{4\pi\varepsilon''_{xy}}{\lambda_0 n}$ and $\theta = -\frac{\pi\varepsilon'_{xy}}{\lambda_0 n}$, where λ_0 and n are the light wavelength and the refractive index of the medium respectively. These observations at the 3rd PBG are in contrast to the work on inverse opals, where a sharp feature was seen in the Kerr reflectivity data at the 1st PBG but the MCD measured in transmission showed only the smooth features of bulk Fe_3O_4 [12]. The results differ also from the broad FR and MCD peaks, of similar width to the corresponding PBG, reported for the three-dimensional MPCs consisting of thin-Co-layer-coated silica spheres [15]. Furthermore, to understand the reason for the shift between the PBG and the MCD and FR peaks requires further analysis. It is not caused by the substrate background that is constant around the PBG, as shown in Fig. 1c.

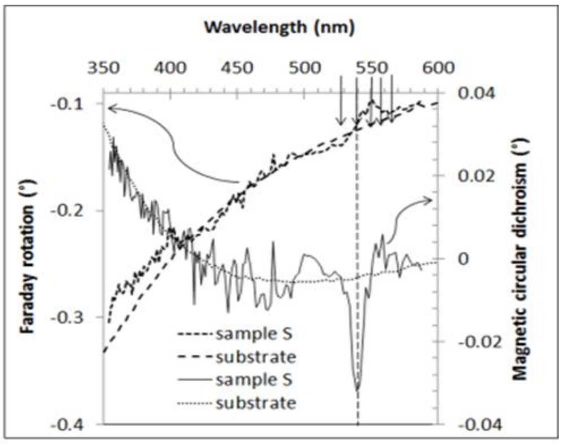
An MCD peak should lie at a wavelength that is between the positive and negative peaks of the FR as shown by Scott [23] and observed in [12]. This is demonstrated in Fig. 1c, where the FR extrema are at 551nm (up) and 528 nm (down), and the MCD maximum is at 539 nm. The shift of the MCD peak from the PBG means that the feature at 528 nm is less visible than the one at 551 nm. If the MCD absorption is a single Lorenzian function of frequency [27] then the magnitude of the positive and negative peaks in the FR should each be half that of the MCD, which is also observed.



(a)



(b)



(c)

Fig. 1 a) The hemispherical transmittance spectrum of sample A is shown in the absence of a magnetic field showing the 1st and 3rd PBGs. b) The measured absorbance spectrum calculated from the transmission spectrum (a) and the mathematical fitted function of it are shown as a sum of Gaussian peaks representing the PBG and Rayleigh scattering. c) The MCD and FR spectra were measured for sample S and a bare 0.4 mm sapphire substrate in 14 kOe magnetic field. The arrows in (c) indicate the wavelengths at which the ellipticity and rotation curves, shown in Fig. 2, were measured

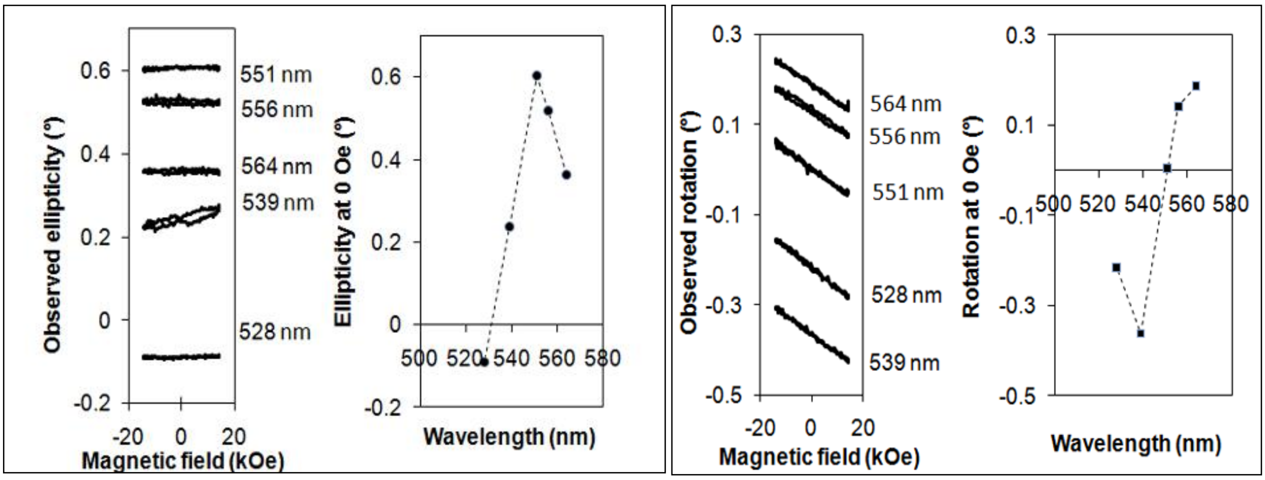
To obtain more information about the magnetic properties, the total ellipticity and rotation were studied as a function of magnetic field at wavelengths around the PBG to compare the magneto-optic response to a magnetic field to the magnetization measurements taken at RT; the results are shown in Fig. 2. The sizes of the MCD and Faraday signals from the MPC near the photonic band gap are small, ~0.03, as is shown in Fig. 2, and consistent with Fig. 1c, due to the very low concentration of Fe_3O_4 particles. An S-shaped response to the applied magnetic field, hysteresis, is seen only where there is a measurable MCD or Faraday signal from the MPC; elsewhere the response is linear. (Fig. 2) The open hysteresis loops are seen for the MCD at

539 nm (Fig. 2a) and, very weakly, for the FR as shown in Fig. 2b at 556 nm but not at 539 nm where the "negative" FR from MPC reduced the total FR.

The total ellipticity η and rotation θ, observed as functions of energy E, were found to vary as $\eta(H, \lambda) = a(\lambda) + a'(\lambda)H^2 \pm$ MCD(H, λ) and $\theta(H, \lambda) = b(\lambda) + b'(\lambda)H^2 \pm$ FR(H, λ) because only the MCD and FR switched sign as the field was reversed (Figs. 2a-d). Earlier work had seen evidence for quadratic field effects from terms $a'(\lambda)H^2$ and $b'(\lambda)H^2$ [12] but was not understood. The origin for offsets at zero field, i.e. the terms $a(\lambda)$ and $b(\lambda)$, can be understood by considering their behavior as a function of the wavelength. They both were strong functions of the energy in the region of the PBG showing sharp peaks (Fig. 2b and d). The ellipticity peak is centered exactly at the PBG wavelength 550 nm, and the rotation peak is zero at the same wavelength. Their magnitudes are some 20-times larger (0.7° peak-to-peak) than the narrow MCD peak 0.03°. The modeling shows that this behavior is exactly what is expected from the birefringent properties of a perfect three-dimensional photonic crystal structure opal [11]. It is not a magnetic effect, but is seen when using a modulated input beam as suggested by Sato [23], and as used in the measurement of the magneto-optic spectra in this study. This is a strong effect and is present in a perfect non-magnetic crystal. Anomalies in the $a(\lambda)$) and $b(\lambda)$) coefficients should be expected at each PBG. It was noted that these coefficients do obey the expected relations [23] as the maximum in $a(\lambda)$ occurs exactly at the energy where $b(\lambda)=0$.

The narrow MCD peak is centered at a 10 nm shorter wavelength than the PBG and ellipticity peak, lying thus on the high energy edge of the band gap, as can be seen in Figs. 1a-b and 2a-d. This is the region, at the edge of the band gap, where there is slow light whose intensity peaks in silica spheres.

It was concluded that the narrow offsets of the ellipticity and rotation relative to the PBG are caused by the crystal structure of the MPC, and the narrow features of the MCD and FR at the high energy edge of the PBG are related to the magnetism of the impregnated magnetite nano-particles. These fundamental properties are very useful for various functional applications of MPCs.



(a) (b) (c) (d)

Fig. 2 a) The total observed ellipticity and c) rotation curves including the MCD and FR hysteresis, measured in varied magnetic field for sample S at the wavelengths 528 – 564 nm, around the obtained MCD and FR peaks seen in Fig. 1. The graphs b) and d) show the birefringence offsets: a(E) of the ellipticity and b(E) of the rotation at the zero field for the data of graphs a) and c) respectively

In an earlier study, the authors have reported that the optical transmission spectrum, e.g. the PBG energies, is dependent on the concentration of the magnetite nano-particles in the spheres [16]. In this part of work it was studied, what kind of impact the direction of the magnetic field applied during the assembling of these MPCs would have in particular on the 3rd PBG. The specular transmission spectra were measured for a 4 × 4 mm^2 area of the crystals with the absolute spectrophotometer described by Nostell [25]. The transmittance spectra, taken in zero-magnetic field, were measured using linearly s- and p-polarized light at incidence angles of 0° - 15^o away from the normal to (111) plane for two samples made of the same spheres but assembled under different direction of the magnetic field. The results are shown in Fig. 3. For the samples that were assembled under a magnetic field normal to surface, the band gaps were found to shift more for p-polarization than for s-polarization, as shown in Fig. 3; and the differential shift increased with the concentration of Fe_3O_4 [16]. No such polarization dependent shift was found for sample B that had been assembled in an in-plane field. This difference in transmission between s- and p-polarizations has not been observed for non-magnetic PCs at small incidence angles in the tests or in literature [15-19].

An explanation is as follows. The Fe_3O_4 nano-crystallites were fabricated and coated with a surfactant in the same process, and then they were embedded into the spheres. The morphology of the MPCs A and B showed defect structures aligning with the direction of the magnetic field during assembly [26]. Sample A was modelled by assuming a small prolate distortion of the crystal structure along the normal to the plane. It was found that this distortion of the crystal lattice does indeed account for a difference between the transmissions in p- and s-polarizations that is absent for the undistorted non-magnetic PC structure. In sample B prepared with an in-plane field there was no significant difference between transmittance for the *p*- and *s*-polarizations. This effect, which is here considered for the transmission off-axis, is different from the birefringence that is present for a perfect opal structure in normal transmission, which gives the variation of the parameters $a(\lambda)$ and $b(\lambda)$ as discussed earlier. The observation that the structure of the film may be affected by a magnetic field applied normal to the plane may be responsible for the observed dependence of the rotation and ellipticity on the square of the field strength [12].

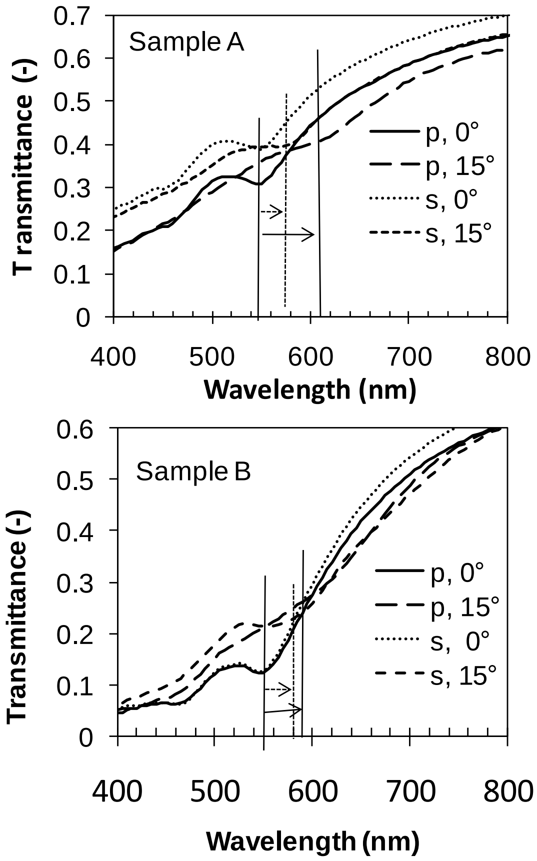


Fig. 3 Specular transmittance spectra measured, for sample A assembled in normal field and sample B in-plane field. The PBG shifts for sample A at 15° are 50 nm for p-pol. and 30 nm for s-pol. The PBG shifts for sample B at 15° are 30 nm for pol. and 25 nm for s-polarized light. In both cases the PBGs for the both polarizations at 0° are at 550 nm

IV. CONCLUSIONS

The three dimensional MPCs assembled in a magnetic field from silica glass spheres that contained Fe_3O_4 nano-particles showed significant, remarkably narrow peaks in the MCD and FR at the third PBG. The peaks were narrower than the PBG as seen in transmission, and also narrower than the peaks reported for MPCs with magnetic nano-particles coating the spheres or filling the space between the spheres. The total ellipticity measured at zero magnetic field showed a narrow peak exactly at the PBG, and in addition both the ellipticity and rotation showed sharp variation in the region of the PBG due to the enhanced birefringence of the photonic structure. The MPCs also showed an unusual polarization dependence of the PBG at small incidence angles which was shown to be related to the direction of the magnetic field applied in the MPC under self-assembling process. Due to the significant and narrow MCD and FR peaks at the high energy edge of the PBG and the high birefringence causing magnified ellipticity and rotation in the band gap region, these kinds of MPC structures have potential in functional micrometer-sized optical sensors of magnetic field and for polarization controller components.

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