Temperature-controlled light diffusion in random media

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We have studied diffusion of light in a disordered medium of which the scattering strength could be tuned via temperature. Such a tunable disordered system was realized by infiltrating a macro porous glass with a liquid crystal. In particular, we have measured the diffusion constant in time-resolved transmission measurements on macroporous silica glass infiltrated with liquid crystal 8CB. The measured temperature dependence of the diffusion constant can be understood qualitatively from the behavior of liquid crystals in confined geometries, and provides an alternative method to resolve liquid crystal phase transitions in such structures. The observed effect allows for control of the scattering strength in multiple light scattering studies and can be regarded as the optical counterpart of negative temperature coefficient resistivity in electronics.

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I. INTRODUCTION

A. Complex dielectrics

The behavior of light waves in complex dielectric systems is complicated yet very interesting. Complex dielectrics are dielectric structures in which the dielectric constant varies on length scales that are roughly comparable to the wavelength of light. Such materials have an index of refraction that can be either periodic in space, thereby forming crystal-like structures, or an index of refraction that has random spatial variations (powders, colloidal suspensions). Also intermediate structures like quasicrystal are possible.

Ordered dielectric structures, with a lattice constant comparable to the wavelength, behave like a crystal for light waves and at high enough refractive index contrast a photonic band gap can exist.[1] Photonic band gap materials are of interest both from a fundamental research point of view and for applications in photonics.[2]

In disordered dielectric structures, on the other hand, light waves undergo a complicated multiple scattering process. Such materials are common in daily life and have an opaque and usually white appearance: they diffusively scatter light. Chalk, white paint, paper, and dense fog are all examples of disordered dielectrics. Random multiple scattering of light waves in disordered dielectrics shows many similarities with the propagation of electrons in (semi)conductors, and various phenomena that are common for electron transport have now also been found to exist for light waves in disordered dielectrics.[3] Important examples are the photonic Hall effect,[4] optical magnetoresistance,[5] universal conductance fluctuations,[6] weak localization,[7] and Anderson localization.[8] In addition, by combining multiple scattering with optical amplification it is possible to obtain random laser action.[9] Important applications of light diffusion include medical imaging[10] and diffusing wave spectroscopy.[11]

The two crucial parameters in all studies on multiple light scattering are the mean free path (defined as the average distance between two successive scattering events) and the diffusion constant. Anderson localization, for instance, manifests itself as a phase transition in the diffusion constant at a certain (small enough) value of the mean free path[8] and it is the diffusion constant that determines the laser threshold of a random laser.[9] The mean free path and diffusion constant are a measure for the scattering strength of a disordered system. For this reason one would like to be able to vary the mean free path and diffusion constant of a sample without changing its geometry and other properties. So far this was very difficult to realize, since the mean free path is an intrinsic material property that depends on the refractive index contrast and size and geometry of the scattering elements, and normally could not be influenced for a given sample.

Recently Busch and John proposed to infiltrate *ordered* dielectric structures (inverse Opals) to create tunable photonic band gap materials.[12] It was demonstrated experimentally that this allows us to shift the band gap of a photonic crystal with temperature,[13] and change the reflection and transmission properties of liquid crystal infiltrated direct Opals via an external electric field.[14] Inspired by the ideas of Busch and John we will explore in this article the possibilities of liquid crystal infiltration in *disordered* dielectric media to obtain external control over their scattering strength.

B. Liquid crystals

Various liquid crystal phases are opaque, which means that light waves inside are multiply scattered.[15] Of special interest is the nematic phase, which is characterized by a global alignment of the molecules along some common axis labeled by a unit vector (or ''nematic director'') $\mathbf{n}_0$. The nematic phase is strongly scattering due to fluctuations of the nematic director.[16] The partial ordering leads to both a birefringence in the refractive index and an anisotropy in the mean free path.[17] Anisotropic light diffusion in nematics was recently observed by Kao *et al.* in static experiments,[18] and later studied also by us in time-resolved experiments.[19]

At high temperatures the nematic phase goes over into the isotropic phase, which behaves as a normal liquid without

birefringence. The refractive index of a liquid crystal is therefore strongly temperature dependent. This creates interesting possibilities for manipulating the scattering properties of both ordered and disordered materials after liquid crystal infiltration, either by changing the temperature or by applying an external field to align the nematic director. This feature is apparent, for instance, in polymer dispersed liquid crystal films that can be switched from opaque to transparent by an external field or by temperature.[20] Liquid crystals in confined geometries like porous glasses have been studied extensively in different contexts, using various experimental techniques like high-resolution ac calorimetry, NMR, magnetically induced birefringence, dielectric spectroscopy, and x-ray scattering.[20,21] Light scattering from liquid crystals in confined geometries was studied, for instance, by Bellini *et al.*,[22] who measured, among other optical properties, the extinction of a laser beam through infiltrated silica aerogel at various temperatures. They observed a large increase of opacity below the nematic-isotropic transition temperature. Despite the great progress in the understanding of the physical properties of confined LC^s, the diffuse propagation of light in these materials has not been investigated so far.

C. Outline

We have measured the diffusion constant of light in random porous glasses with large pores impregnated with liquid crystal, by means of time-resolved transmission measurements. These experiments are discussed in Sec. II. We observe a strong dependence of the diffusion constant on temperature which we report in Sec. III. Here we also discuss how measurements of the diffusion constant can provide an alternative method to resolve phase transitions of liquid crystals in confined geometries. In Sec. IV we discuss the analog of our system with electron transport and show that the observed behavior can be regarded as the optical counterpart of negative temperature coefficient (NTC) resistance in electronics.

II. TIME-RESOLVED TRANSMISSION MEASUREMENTS

In order to characterize the scattering strength of a disordered medium one has to measure either the transport mean free path or the diffusion constant. The transport mean free path can be measured by various techniques amongst which coherent backscattering.[7,23] To measure the diffusion constant, a time-resolved technique has to be used. The most simple possibility is to use a sample with a slab geometry, have a short laser pulse incident on the front sample interface, and follow the time evolution of the transmitted diffuse light. By comparing the transmitted light to the solution of a diffusion equation for a slab geometry, one can find the value of the diffusion constant.

Our samples were prepared in the following way. As matrix we used macroporous silica glass with randomly oriented and interconnected pores. Macroporous silica glass is strongly scattering and exhibits multiple scattering.[24] Our macroporous silica glass had an average pore diameter of 100 nm and volume fraction of the pores of 38%. In order to obtain a tunable random medium, the macroporous glass was infiltrated with the liquid crystal octylcyanobiphenyl (8CB). The matrix was a solid two millimeter thick plate with flat (optically polished) and parallel surfaces. We impregnated this porous matrix with 8CB at temperatures corresponding to the isotropic phase. Bulk 8CB has a smectic-*A* phase in the temperature range of 21.1–33.5 °C, whereas a nematic phase is formed in the range of 33.5–40.8 °C. The sample was temperature controlled with a relative accuracy of 0.2 °C and an absolute accuracy of 0.5 °C.

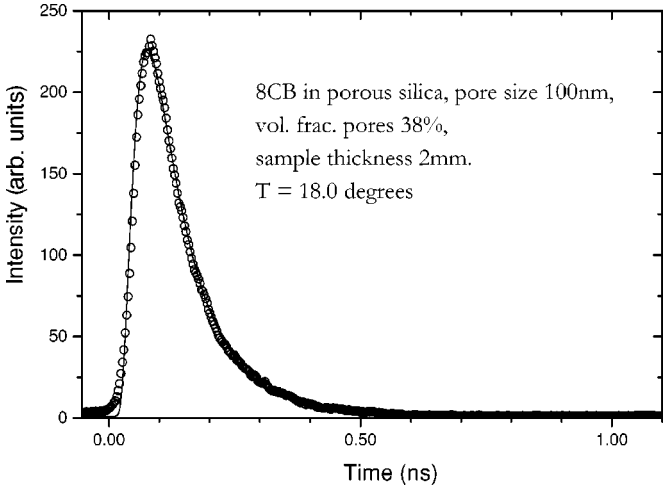

FIG. 1. Time-resolved transmission through a slab of porous silica, infiltrated with the liquid crystal 8CB. Pore size 100 nm, volume fraction of the pores 38%, sample thickness 2 mm, and temperature 18.0 °C. The solid line is a fit from diffusion theory using Eq. (2). One can see that the agreement with diffusion theory is very good. The minor discrepancy at the start of the rising edge is most likely due to the limited time resolution of the experiment. The resulting diffusion constant is $D=4.2\times10^3$ m^2/s ($\pm10\%$).

The diffusion constant was measured in a time-resolved transmission experiment in the following way. A short (10 ps) pulse was incident on the front sample interface and the diffuse light in transmission was monitored by a streak camera. For that purpose, the output surface of the sample was imaged onto the input slit of the streak camera. The light source was a cavity dumped, mode-locked dye laser operating at 613 nm wavelength. The incident beam diameter on the sample was 0.5 mm. The final time resolution in the measurements was better than 30 ps. The sample was oriented in the x-y plane and the laser was incident on the front sample surface along the z axis.

An example of a time-resolved transmission measurement at fixed temperature is shown in Fig. 1. Here we have plotted the time evolution of the diffuse transmission through the sample, as monitored at the back surface from a region of 0.5 mm diameter around $x=y=0$, which corresponds to the region of highest intensity. Due to the large optical thickness of the sample, the coherent beam is extinguished completely in transmission.

It can be shown, starting from Maxwell's equations, that in this regime the energy density of the light follows a diffusion equation of the form[3]

$$\frac{\partial W(\mathbf{r},t)}{\partial t}=\boldsymbol{\nabla}\cdot\mathbf{D}\boldsymbol{\nabla}W(\mathbf{r},t)-\tau_{\mathrm{i}}^{-1}W(\mathbf{r},t)+S(\mathbf{r},t), \qquad (1)$$

with $W(\mathbf{r},t)$ the energy density, $S(\mathbf{r},t)$ a source function, **D** the diffusion tensor, and τ_i the inelastic or absorption time, which is the characteristic time over which light is absorbed inside the sample. In our samples the absorption at 613 nm wavelength could be neglected. The diffusion tensor **D** is anisotropic in the nematic regime, if an external field is used to obtain a global anisotropy in the system.[18,19] Without external field the nematic anisotropy is only local and averages out over large length scales. In that case **D** simplifies as $D_{xx}=D_{yy}=D_{zz}=D$, and the diffusion constant can be related to the (transport) mean free path l and the transport velocity[25] v_e as $D=1/3v_e l$.

The boundary condition for the diffusion equation (1) for a slab geometry oriented in the x-y plane is $W(\mathbf{r},t)=0$ at $z=-z_0$ and $z=L+z_0$, with L the physical thickness of the slab. The distance z_0 is called the extrapolation length and accounts for the fact that the diffuse intensity necessarily is nonzero at the sample interfaces. The extrapolation length depends on the internal average reflectivity R of the sample surface due to index mismatch between the sample and its environment and is given by[26] $z_0=2/3l(1+R)/(1-R)$. We assume that the incident light pulse (laser beam) is fully scattered at depth l inside the sample and, for symmetry reasons, that the last scattering event takes place at $z=L-l$. The time evolution of the transmitted intensity is given by Fick's law[27] as $I_{\mathrm{tr}}=-D\nabla W(\mathbf{r},t)|_{z=L-l}$. Equation (1) is solved with source function $S((\mathbf{r}),\mathbf{t})=\delta(\mathbf{x})\delta(\mathbf{y})\delta(\mathbf{z}-l_{\mathbf{z}})\delta(\mathbf{t})$ and above boundary conditions and this gives us for the transmitted intensity[27,28]

$$I_{\mathrm{tr}}=\frac{I_0\exp(-t/\tau_i)\exp(-\Delta x^2/4Dt)\exp(-\Delta y^2/4Dt)}{\pi^{3/2}(4t)^{5/2}D^{3/2}} \times\sum_{n=-\infty}^{+\infty} A\exp(-A^2/4Dt)-B\exp(-B^2/4Dt), \quad (2)$$

with $A=(1-2n)(L+2z_0)-2(z_0+l)$ and $B=(2n+1)(L+2z_0)$. Note that in the limit of long t and in the absence of absorption, the transmitted intensity falls off as an exponential with time constant $\tau=(L+2z_0)^2/D\pi^2$.

The solid line in Fig. 1 is obtained from Eq. (2), using $\Delta x=\Delta y=0$. Since the boundary condition is a slab geometry, the value of the extrapolation length z_0 is needed in Eq. (2), which depends on the mean free path l and average reflectivity R at the sample interface. The value of l can be obtained via $l=3D/v_e$ with $v_e=c_0/n_{\mathrm{av}}$ and n_{av} the average refractive index of the sample. For simplicity we can take n_{av} constant and equal to the geometrical average $n_{\mathrm{av}}=1.52$.[29] From Ref. 26 we find for the internal reflectivity $R=0.57$. The final result for D will depend only weakly on the exact value of n_{av}. We found that typically less than 10 terms were needed to have good convergence of the series in Eq. (2). One can see that the experimental data correspond well with diffusion theory. From the fit to the data we find a diffusion constant of $D=4.2\times10^3$ m^2 s ($\pm10\%$) for 8CB in porous silica at $T=18.0\,^\circ$C.

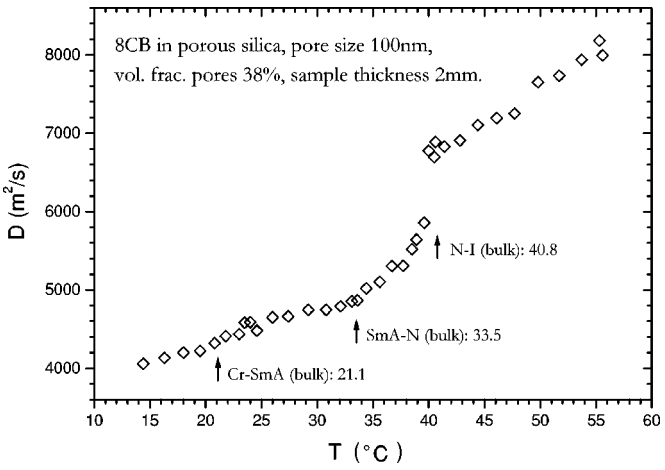

FIG. 2. Temperature dependence of the diffusion constant of porous silica infiltrated with the liquid crystal 8CB. Pore size 100 nm, volume fraction of the pores 38%, and sample thickness 2 mm. The arrows indicate the phase transition temperatures of a bulk (free) sample of 8CB. The phase sequence of 8CB is crystalline (Cr)–Smectic A (SmA)–Nematic (N)–Isotropic (I). Note that even the nematic–isotropic phase transition is not sharp due to the geometrical confinement of the liquid crystal. We see that a considerable variation of the diffusion constant can be obtained by changing the temperature of the system.

III. TEMPERATURE-DEPENDENT DIFFUSION

We have measured the diffusion constant in a temperature range from 14.4 °C to 55.6 °C, which amply covers all the phases of bulk liquid crystal 8CB. The results are plotted in Fig. 2. With arrows we have indicated the phase transition temperatures of nonconfined bulk 8CB. We can clearly see a strong dependence of the diffusion coefficient on temperature, especially in the temperature range of the nematic and isotropic phases. In the nematic phase the liquid crystal is birefringent, with ordinary and extraordinary refractive indices respectively n_o and n_e. Without an external field, the orientation of the nematic director will be random and different in every pore, so that a disordered birefringent medium is formed. The birefringence averages out on a macroscopic scale. In the isotropic phase also the local birefringence disappears, so that the refractive index contrast between liquid crystal and porous matrix changes considerably. This explains the strong decrease of D, when lowering the temperature into the nematic region.

The observed dependence of the diffusion constant is very different from the typical phase behavior of bulk LC, due to the confinement in the porous silica. The porous silica host will force the liquid crystal molecules close to the pore walls in a certain direction. The surface boundary condition for liquid crystal molecules on the pore walls is expected to be perpendicular due to the formation of hydrogen bonds between the CN group and the OH groups at the silica surface. Due to this confinement both the smectic-A to nematic and the nematic to isotropic phase transitions are smeared out, which is in agreement with recent NMR, calorimetric, and spectroscopic studies of 8CB confined in random pores.[30–32] The smectic-A to nematic phase transition is so strongly

smeared out that the diffusion constant increases smoothly with increasing temperature. The notable change in the temperature derivative around 33.5 °C indicates however that the smectic-*A* phase is still formed inside the pores. The decrease of the diffusion constant below $T<21.9$ °C is probably due to supercooling of LC in pores below the crystallization temperature.

Measuring the temperature dependence of the diffusion constant provides an alternative method to resolve phase transitions of liquid crystals in confined geometries. The determination of the phase transition temperatures in these systems is generally quite complicated because transitions are smeared out and the transition region occupies a finite temperature interval. The advantage of our method compared to conventional transmission techniques becomes apparent at large optical thicknesses and very opaque samples where the coherent beam in transmission is below the detection limit and conventional optical experiments fail. For measurements of the diffusion constant, a large optical thickness of the sample is not a limiting factor but is even an advantage. The specific macroporous glass that we study in this paper has strongly polydisperse and irregular shaped pores. For light diffusion experiments this has no influence but for studies on liquid crystal phase transitions in confined geometries this complicates the interpretation of results. This problem can be solved by using a random matrix with monodisperse holes, obtained with the technology for making inverse Opals.[33,34]

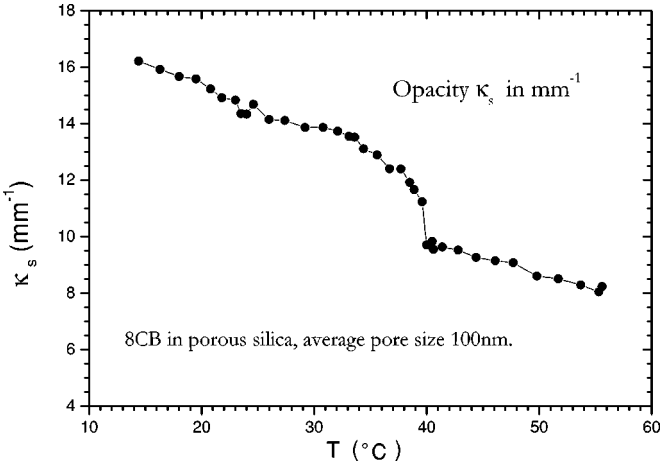

FIG. 3. Opacity of liquid crystal infiltrated porous silica versus temperature. (Liquid crystal 8CB, average pore size 100 nm, vol. frac. pores 38%, and sample thickness 2 mm.) The opacity is the optical analog of the electrical resistivity of a material. A decreasing opacity with temperature can be regarded as the optical counterpart of negative temperature coefficient (NTC) resistance in electronics.

IV. THERMALLY SENSITIVE RESISTANCE

A temperature dependent diffusion constant for light can be regarded as the optical counterpart of thermally sensitive resistance in electron transport. An important example of the many similarities between electron transport and multiple light scattering is the Ohmic behavior for light transmission through disordered dielectrics.[3] That is, the transmittance T of light through a disordered medium is inversely proportional to its thickness L:

$$T=\frac{l}{L}=\frac{1}{\kappa_s L}, \tag{3}$$

where $\kappa_s \equiv 1/l$ is the opacity or scattering coefficient of the disordered dielectric. This relation is the optical equivalent of Ohm's law for the conductance through a wire with length L and area A:

$$g'=\frac{1}{\rho L}, \tag{4}$$

with $g'=g/A$ the conductance per unit of surface area and ρ the resistivity of the material. This similarity is not surprising if one realizes that the transport mechanism in both cases is a diffusion process. In Fig. 3 we have plotted the opacity κ_s for our system of infiltrated porous glass, versus temperature, as calculated via $l=3D/v_e$. For simplicity we use again a constant transport velocity: $v_e=c_0/n_{\mathrm{av}}$ with $n_{\mathrm{av}}=1.52$. The opacity of the system decreases with temperature. This is the optical equivalent of a resistor with a NTC thermally sensitive resistivity. Thermally sensitive resistivity is often encountered in electron transport, as the mobility of charge carriers depends on temperature.[35]

V. DISCUSSION

We have shown that liquid crystal infiltration in random porous media can be used to obtain control over the diffusion constant via temperature. The maximum change in the diffusion coefficient that we obtained via temperature tuning was about a factor of two. The tuning range can be largely increased, however, using a porous glass / liquid crystal combination that is (almost) refractive index matched in the isotropic phase. In the case of perfect index matching, the diffusion constant would diverge at high temperatures. A diverging diffusion constant means that the material will become almost transparent at high temperatures and very opaque at temperatures below the isotropic-nematic transition.

Temperature tuning of the scattering strength via liquid crystal infiltration could proof to be a useful tool in various multiple light scattering studies amongst which the research on Anderson localization and the work on random lasers. Anderson localization requires very high refractive index contrasts which obviously are not obtained with infiltrated porous glasses, but which could be obtained in macroporous semiconductors. A good candidate for this purpose is the air-sphere structure of silicon used for photonic band gap materials.[34] By introducing a large amount of randomness in this material, one may obtain the required scattering strength needed for Anderson localization, which then could be tuned by liquid crystal infiltration. This would allow investigation of the phase diagram of the localization transition by temperature tuning of the scattering strength.

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