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Neutron diffraction from gratings in holographic polymer dispersed liquid crystals

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Date(s) of Experiment:	4th – 9th April 2008

Scientific Objectives

This experiment was part of a series to gain insight into the photopolymerization induced phase separation process in (holographic) polymer dispersed liquid crystals (H-PDLC). The samples contained a homogeneous mixture of liquid crystal, photosensitive monomers and a photo initiator. Exposed to a holographic interference pattern photopolymerization occurs. The reaction rate is higher in bright areas, leading to diffusion of monomers into the illuminated regions and subsequent polymerization, while the liquid crystal molecules congregate in the dark areas [1]. To contribute to the understanding of these complex phenomena and to obtain detailed information on the formation of H-PDLCs and the process of phase seperation (especially the LC droplet size and shape), neutron diffraction was employed. It offers an appropiate tool for this purpose and has already been used in earlier experiments [2, 3, 4, 5].

For this series of measurements samples of HPDLC with different grating spacings Λ were investigated, thus exploring the role of diffusion. Furthermore, we performed first experiments on holographically patterned nanoparticle dispersed polymers (ZrO_2 or SiO_2), the nanoparticles thereby replacing the LC component. Finally, a practical purpose was to take another step in the optimisation of H-PDLCs and nanoparticle dispersed polymers for use as neutron optical elements, in particular as beamsplitters and mirrors in interferometry.

Experimental Technique

Small-angle neutron diffraction experiments were performed at SANS-2. The used neutron wavelengths were 11.58 Å and 19.6 Å with a spread of $\Delta\lambda/\lambda$ = 10 %. We employed the full available collimation length of 16m, using diaphragms of 20 x 20 mm² or 10 x 40 mm² at the entrance and a 5 mm pinhole in front of the sample. Because of the small diffraction angles $\Theta_B = \lambda/2\Lambda \sim 0.5$ mrad the maximum sample to detector distance (21 m) was employed.

The investigated samples of H-PDLC were part of a series from a single batch. To create a sample the volume between two glass plates, separated by spacers of 50 µm thickness, was filled with a mixture of 55 wt.-% LC (TL203, Merck), 33 wt.-% prepolymer (PN393, Nematel) which is photosensitve in the uv [6] and 12 wt.-% 1,1,1,3,3,3-Hexafluoroisopropyl acrylate (HFIPA, Sigma-Aldrich). An Argon ion laser operating at 351 nm wavelength was used in a two wave mixing setup to record sinusoidal gratings in the samples. By changing the angle between the two beams, samples with four different grating spacing were produced

(Λ = 1200 nm, 1000 nm, 560 nm, 433 nm). A broadband uv-lamp was used for postcuring. The partial phase separation and the highly polarizable LC components yield efficient anisotropic gratings for light [6, 7], the modulation of the scattering length density on the other hand, constitute contrast for neutron diffraction [8]. The samples containing nanoparticles have been provided by Y. Tomita [9].

Achievements and Main Results

Complete rocking curves were conducted on H-PDLCs and nanoparticle samples, that is the angular dependence of the diffraction efficiency η (diffracted intensity over incoming intensity). We were able to demonstrate profound diffraction efficiencies for **nanoparticle dispersed polymer** samples for the first time. Fig. 1 shows the detected intensity (zero, $\pm 1^{st}$, $\pm 2^{nd}$ orders) of neutron diffraction (19.6 Å wavelength) from a sample containing SiO2 nanoparticles in a polymer matrix. The reason for the detection of higher orders is twofold: 1) the diffraction process involves multiple beam coupling (in the rigorous coupled wave regime) and 2) the grating is slightly nonsinusoidal. Fig. 2 depicts the angular dependence of the diffraction efficiency for the first orders for a grating made of a ZrO_2 particle dispersed polymer.

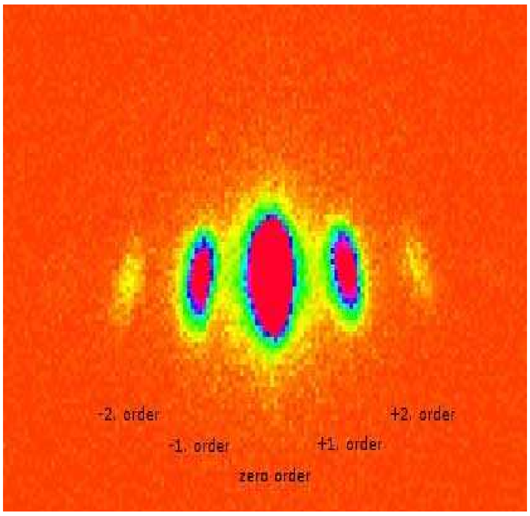

Figure 1: Detector image (neutron counts in lin scale) of a SiO2 nanoparticle dispersed polymer sample, displaying the 0th, ±1st and ±2nd orders.
Wavelength: 1.96 nm, particle size ~13 nm, grating spacing Λ = 1000 nm, grating thickness 50 micron.

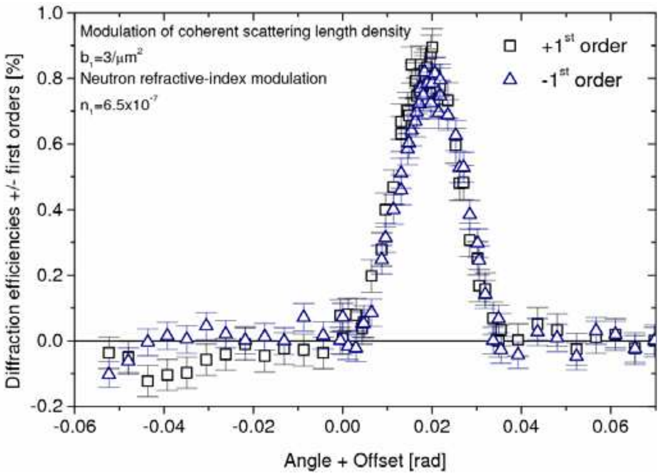

Figure 2: Rocking curve of a grating built from a ZrO2 nanoparticle dispersed polymer sample.
Wavelength: 1.16 nm, particle size ~2 nm, grating spacing Λ = 1000 nm, grating thickness 51 micron.

A systematic study of the diffraction efficiency of 11 **HPDLC samples** with various grating spacings and thicknesses was done during our experiments. Samples with lower grating spacing (433 nm, 560 nm) and larger thickness (100 micron) show less diffraction, probably related to the fact, that the phase separation process is far from being complete. Best results were achieved with the Λ = 1 μm samples. The results together with a reasoning for optimal diffraction gratings were accepted for publication recently [10]. The diffraction efficiency of the H-PDLCs were around 0,9 % for 1.16 nm wavelength, the nanoparticle dispersed polymers using SiO_2 (20 wt.-%) resulted in a η of 3 % while ZrO_2 (12 wt.-%) showed an efficiency of 0,7 %. The samples and thus the gratings are rather thin (d = 50–100 μm) and can't be much higher for HPDLCs because of strong optical scattering during the holographic preparation process.

H-PDLCs are interesting for the fabrication of neutron optical devices (gratings, lenses, mirrors,..) for cold neutrons. Nanoparticles dispersed in a polymer matrix are even more promising candidates for neutron optical gratings, e.g. in a LLL-neutron interferometer. Still the photopolymerization process needs further investigations to optimize the materials for light optical and neutron optical purposes.

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