exposed to 0.1 Å/s Si flux for 30 s. The final structure consisted of a 2-µm-thick  $Si_{0.89}Ge_{0.10}C_{0.01}/Si$  superlattice (100 periods with each sublayer 10 nm in thickness) grown at  $500^{\circ}C$  and lattice matched to the Si substrate. This structure was doped with Sb to approximately  $2 \times 10^{19}$  cm<sup>-3</sup>, and the sample was capped with a 100-nm Si layer.

The cross-plane thermal conductivity of the superlattice was found to be 0.085 W/(cmK), or over one order lower than that of Si (1.5 W/[cmK]). The researchers grew SiGeC/Si superlattice microcoolers with dimensions ranging from  $40 \times 40 \ \mu\text{m}^2$  to  $100 \times 100 \ \mu\text{m}^2$ . The coolers were tested on a constant-temperature heatsink by measuring the device cooling as a function of electrical current. Maximum cooling temperatures of 2.8 K and 6.9 K were obtained at heatsink temperatures of 25°C and 100°C, respectively, indicating better microcooler performance at higher temperatures. The maximum cooling temperature was also found to increase with decreasing device size. These cooling temperature data correspond to maximum cooling power densities of the order of 1000 W/cm<sup>2</sup>. The SiGeC/Si microcoolers were compared with Si microcoolers, and a threefold improvement in maximum cooling was observed for the SiGeC/Si coolers.

The addition of carbon to SiGe enables the design of a SiGeC layer which is lattice matched to silicon. This allows the SiGeC layer to be grown directly on a Si substrate without strain which reduces the cost of material growth and simplifies integration of the cooler with Si-based devices. Furthermore, the addition of C also enables the use of thermionic emission to enhance TE cooling for SiGeC/Si materials due to the large conduction band offset between SiGeC and Si. According to Fan, "Solid-state coolers that are monolithically integrated with microelectronic and optoelectronic devices are an attractive way to achieve compact and efficient cooling. Our results indicate that efficient refrigeration is possible with coolers made of conventional semiconductor materials."

STEFFEN K. KALDOR

## Self-Assembled Monolayers of BPS Control the Molecular Aggregation of Polymer-Dispersed Liquid-Crystal Films of 5CB

S. Kato and C. Pac of the Kawamura Institute of Chemical Research have controlled the molecular aggregation of polymer-dispersed liquid-crystal (PDLC) films by varying the hydroxyl densities of selfassembled monolayers (SAMs) of BPS, a boronate-terminated silane compound, on the substrates. Previously the researchers demonstrated that in PDLC films of 4cyano-4'-pentylbiphenyl (5CB), a typical nematic LC, the LC molecules and the polymers form an interface layer at the substrate with different molecular alignment than the interior. In the current work, fluorescence analysis shows that the presence of the SAM on the substrate affects the properties of the interface layer and allows control of the molecular alignment.

As reported in the March issue of *Chemistry of Materials*, PDLC films were made by photopolymerization-induced phase separation (PIPS) of a mixture of 30 wt% 5CB and diacrylate monomers between two quartz substrates modified with SAMs in five ways. Some substrates were immersed in a water/ethanol mixture for different lengths of time, creating samples that were 38%, 57%, or fully hydrolyzed. Some of the fully hydrolyzed samples were reacted with dodecyltrichlorosilane to obtain a long hydrocarbon chain. The fifth substrate type was unhydrolyzed.

The fluorescence spectra of the films were taken by excitation at 290, 310, and 320 nm. At 320 nm, the absorbance of 5CB is low so the signal is from the entire sample. These spectra were nearly identical for each film and show they are excimer-rich. Since the excimer is formed more easily in the nematic phase than in others, this implies that the interior of all types of samples is nematic. At 290 nm, however, the absorbance is higher and the signal is from a layer less than 60 nm thick. The fluorescence results show that as the SAMs become more hydrolyzed, the interface layer becomes less nematic.

The research group also studied the electro-optic effects of the SAMs on PDLC films of 77 wt% 5CB made on indiumtin-oxide-coated glass substrates modified as described with the other sample. The transmittance-voltage response to a 1-kHz sinusoidal voltage at room temperature suggests that the high concentration of 5CB allows the molecular alignment in the interface to affect the molecular alignment in the interior. The group is continuing to study the electro-optic response of PDLC devices.

ELIZABETH A. SHACK

## Infrared Liquid Immersion Microscopy Reveals Details of Green Bodies and Compaction Process

Fabrication of a green body, which is an intermediate form of the final ceramic piece or sample, is a key step in the ceramics manufacturing process. Flaws in the green body will determine the mechanical properties of the final material.

Liquid immersion microscopy is an innovative technique that allows observation of transparent ceramic green bodies in the optical microscope and this allows observation and evaluation of internal defects introduced by the fabrication process. The transparency is achieved by immersing the particle in a liquid with similar refractive index. In this manner, suppression of the reflected light at the interface allows the particle to become optically transparent. This procedure is currently under development by a group of scientists from the Nagaoka University of Technology and the Japan Fine Ceramics Center.

The basic requirements for this technique are thin specimens and an adequate immersion liquid. The refractive index of this liquid has to be within 5% of that of the particle to achieve transparency in visible light. Sometimes such liquids are difficult to find since the refractive indices of many ceramics are very large. In the case of Si<sub>3</sub>N<sub>4</sub>, the liquid found with appropriate refractive index is unstable and toxic. In the January issue of the Journal of the American Ceramics Society, the researchers demonstrate how IR light can be used to expand the range of potential liquids that will achieve transparency. Due to the larger wavelength of IR light, the difference between the refractive indices of the liquid and the particle can be 3.6 times larger than that allowed for visible light.

Granules of  $Si_3N_4$  were obtained after spray drying a slurry prepared by mixing powders with sintering aids, a binder, and a dispersant for 24 h. A green body was obtained by pressing the granules at 19.6 MPa and heating at 1300°C for 2 h. A thin section of about 150  $\mu$ m was prepared for microscopy observations, and the immersion liquid used was a saturated solution of sulfur in methylene iodide with a refractive index of n = 1.79. An infrared camera attached to the microscope operated at a maximum wavelength of 1.8  $\mu$ m. Radiation of less than 1.3  $\mu$ m was excluded with a filter.

Inspection of the spray-dried granules in the scanning electron microscope (SEM) revealed that some of them had a small dimple. On the other hand, the IR microscopy observations showed that all of the granules had not only a dimple, but also an internal pore. The thickness of the granules was about one-fourth their diameter.

A  $\rm Si_3N_4$  compact was formed by powder compaction of  $\rm Si_3N_4$  granules under high pressure. SEM observations of the powder compact showed that the boundaries of the granules deformed by the compaction pressure. The IR microscopy

276 MRS BULLETIN/APRIL 2001

observations also revealed that pores are still present in the center of many granules. No features were revealed when operating in visible light.

These results demonstrated that the liquid immersion method using IR exposure leads to significant improvement in detecting defects, although the minimum size of detectable features is 1 µm. However, the researchers indicate that this is not a problem because the size of fabrication defects in this type of ceramic is usually larger.

ŠIARI S. SOSA

## Photopolymerizable Glass Outperforms Traditional Holographic Materials

Studies of organically modified silica glass have produced a material with high refractive index modulation, high optical quality, and photosensitivity. Plus, the material can be cast as a monolith or a film. In the March 12 issue of *Applied Physics Letters*, Pavel Cheben of the National Research Council of Canada and Maria L. Calvo of the Complutense University of Madrid have reported that by combining a titanocene photoinitiator and an acrylic monomer with a sol-gel precursor, a material is produced which is ideal for volume holographic recording.

Photoinitiation of ethylene glycol phenyl ether acrylate was accomplished with bis(μ<sup>5</sup>-2,4-cyclopentadien-1-yl)-bis-[2,6difluoro-3-(1H-pyrrol-1-yl)phenyl]titanium. Both photoinitiator and monomer were added to the silica sol prior to the gelation point in concentrations of 0.4–0.8 wt% and 40 wt%, respectively. The solution was stirred for 20 min at 40°C until homogeneous, at which point it was cast into Teflon vials and allowed to gel and dry for 10 days at 60°C. Thin films of ~260 µm thickness were prepared by casting 0.1 mL of the solution onto a borosilicate glass plate and dried for 5 days at 60°C.

Characterization of the holographic performance of the glass was achieved by writing a grating pattern into the monolith or thin film, followed by reading the pattern with another beam. The glass refractive index was measured as n = 1.47by a prism-coupling technique. The diffraction efficiency dependence on the angle of incidence was measured by detuning the readout beam from the Bragg condition, yielding the grating angular selectivity. The refractive index modulation ( $\Delta n$ ) amplitude and effective thickness of the grating were calculated using Kogelnik's coupled-wave theory. The gratings in the monoliths produced  $\Delta n = 1.35 E^{-3}$  for  $E \sim 1.5 \text{ J/cm}^2$ , and thus a sensitivity of  $S = 8.6 E^{-4} \text{ cm}^2/\text{J}$  (E is expositivity of  $S = 8.6 E^{-4} \text{ cm}^2/\text{J}$ ). sure). A dynamic sensitivity  $S^* = d(\Delta n)/dE$ 

=  $1.8 E^{-3} \text{ cm}^2/\text{J}$  was achieved in the region of exposure 0.1– $0.35 \text{ J/cm}^2$ . The gratings in the 260-µm-thick films produced  $\Delta n = 4.5 E^{-3}$  for  $E = 0.72 \text{ J/cm}^2$  and thus a sensitivity of  $S = 6.2 E^{-3} \text{ cm}^2/\text{J}$ .

The researchers discussed the highly efficient formation of gratings in this material in terms of photopolymerization in the light regions. As the monomer is depleted in the light regions, mechanisms involving diffusion and capillary forces move monomers from the dark regions into the light regions where they participate in the photopolymerization process.

The hybrid organic-inorganic glass studied here exceeds the performance of traditional holographic materials. Furthermore, the ability to be cast as a monolith or as a thin film, the porosity of the final glass, the rigid silicon oxide backbone, and no requirement for solvent processing overcomes some problems associated with traditional holographic materials. These combined advantages demonstrate excellent prospects for practical holographic storage devices using this material.

DONALD F. CARTER

## Alumina With 50% Porosity Obtained After Freeze-Drying a Water-Based Slurry

Ceramic materials with a large surface area are commonly used as catalysts, filters, chemical sensors, and for other similar applications. Freeze-drying is a versatile fabrication method for this type of material and several variants of its use are found in the literature. A group of researchers from the Synergy Ceramics Laboratory and the National Industrial Research Institute of Nagoya in Japan are developing a freeze-dry process starting from a water-based slurry. They maintain that their method is more environmentfriendly since it eliminates the necessity for the use of a binder. Furthermore, the resultant material has good mechanical strength and the pore distribution is easily controlled. In the January issue of the Journal of the American Ceramics Society, these researchers explain how they obtained such a porous structure. They started by mixing alumina powders and a small amount of a dispersant into slurry with distilled water; ball milling occurred



Circle No. 23 on Inside Back Cover

MRS BULLETIN/APRIL 2001 277