Nanotechnology E-Bulletin

Sculptured thin films with transverse architecture

Back in the 1880s, the German scientist August Kundt (1839-84) put an electrified metal wire in a chamber, lowered the pressure, and raised the temperature. Metal vapor created by an electric arc at the wire tip was collected on a flat substrate to form a thin film. Optical experiments showed that the film was birefringent, and that the birefringence varied with porosity. Much later, in the 1960s, scanning electron microscopy showed that such thin films, called columnar thin films (CTFs), are assemblies of parallel nanowires (see Figure 1(a)) oriented in the general direction of the incoming vapor flux. Macroscopically, these films are like biaxial crystals.

Suppose the substrate is being rotated while a directional vapor is condensing on it. Then, instead of straight nanowires, shaped nanowires will grow. This was realized during the mid-1990s, when the concept of sculptured thin films (STFs) emerged.[1] STFs are assemblies of parallel, shaped nanowires that can be fabricated by design using physical vapor deposition techniques, such as thermal and arc evaporation, sputtering, and pulsed laser ablation. As the nanostructure comprises multimolecular clusters of 3-5nm diameter, rapid changes in the average direction of the incident vapor flux (relative to the substrate) lead to the growth of parallel nanowires of curvilinear shapes. At infrared, visible, and lower-ultraviolet wavelengths, the assembly of nanowires can be effectively considered a continuous anisotropic medium whose electromagnetic response properties are nonhomogeneous in the growth direction, i.e., normal to the substrate. This unidirectional nonhomogeneity permits the exploitation of commonplace design techniques for STF-based devices such as optical polarizers, filters, and sensors.

Atomic self-shadowing due to oblique-angle deposition enables the nanowires to grow continuously, to change direction abruptly, and to maintain constant cross-sectional diameter. Figures 1(b) and 1(c) show STFs comprising nanowires of bent nematic (2D) and helicoidal (3D) shapes, respectively. The individual nanowires of a chiral STF stand out in 1(d). That the shape can be changed during growth is shown in 1(e), wherein helicoidal nanowires transition into chevronic nanowires. The change is virtually abrupt, as indicated by the
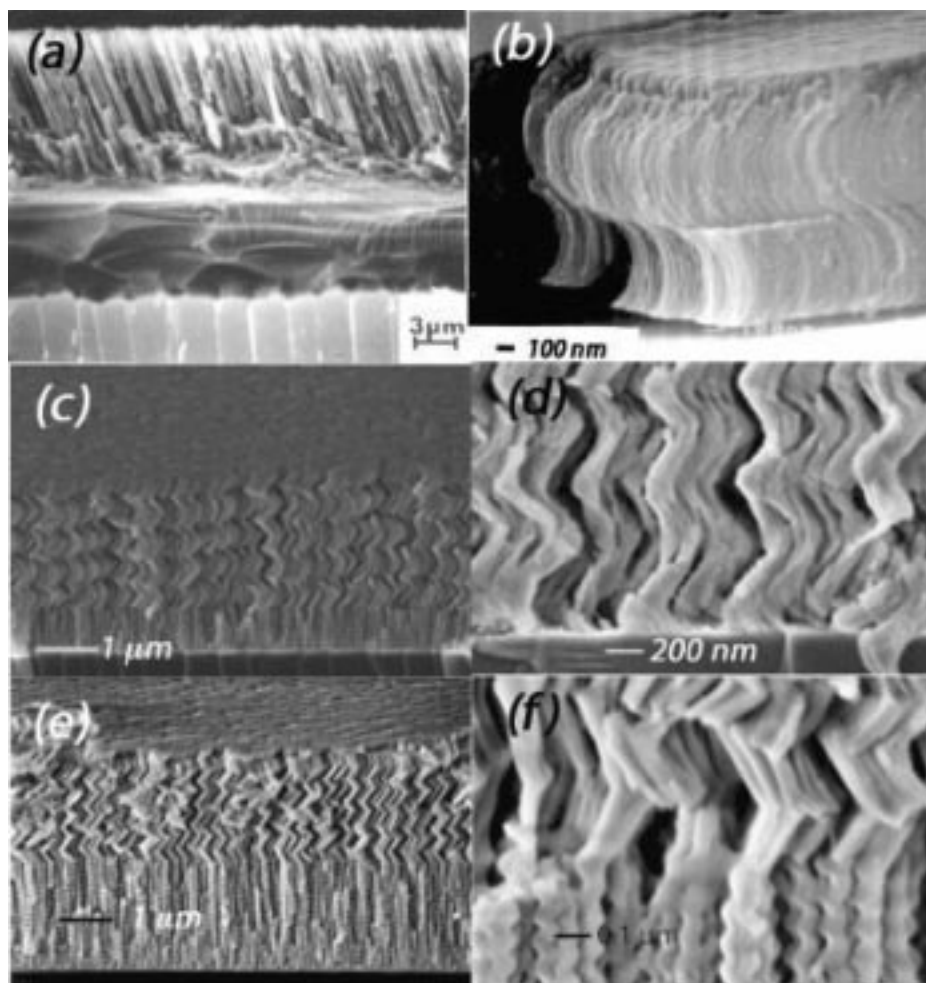

Figure 1. Sculptured thin films: (a) straight nanowires of amorphous germanium; (b) s-shaped nanowires of magnesium fluoride; (c) helicoidal nanowires of silicon oxide; (d) magnified view of helicoidal nanowires; (e) chevronic nanowires grown on top of helicoidal nanowires of silicon oxide; and, (f) magnified view of the transition of shape from helicoidal to chevronic.

magnified view of the transition region in Figure 1(f).

The STFs fabricated until quite recently did not possess transverse architecture, as the constituent nanowires were randomly nucleated on planar substrates. The film growth rates were quite low, depending on the deposition method employed, due to the large oblique angles during deposition. Furthermore, the transverse area

rarely exceeded 1cm × 1cm without significant loss of transverse uniformity. These three factors stymied the economic exploitation of STFs, despite the theoretical design and experimental realization of many STF-based optical devices.[1]

The combination of large thickness (> 3µm), large-area uniformity (75mm diameter), and high growth rate (up to 0.4µm/minute) in as-

semblies of complex-shaped nanowires on lithographically-defined patterns has recently been achieved for the first time.[2] The nano- and the microscale have been blended together in STFs with transverse architecture.

Planar substrates are easily transformed into topographic substrates (with lines, checkerboards, and arrays in relief) using standard micro- and nanofabrication techniques. Selective growth of nanowire assemblies occurs thereafter on a topographic substrate during physical vapor deposition, subject to certain limitations. A variety of transverse architectures can thus be realized transverse to the nonhomogeneity that is parallel to the substrate normal due to substrate rotation. Figure 2 shows three different assemblies of helicoidal nanowires grown on topographic substrates.

The ability to sculpture the nanowires out of any material that can easily be deposited by a physical vapor deposition technique—coupled with micro- and macroscale 1D and 2D topographic substrates—opens a whole new realm of photonic, fluidic and sensor devices. Applications include nanowire assemblies for bionanotechnology, because nanopatterning of surfaces is known to influence protein adsorption and cell behavior. Photonic-bandgap engineering should also be impacted, because complex periodic features can be lithographically etched on large-area substrates, with lattice sizes >50nm.

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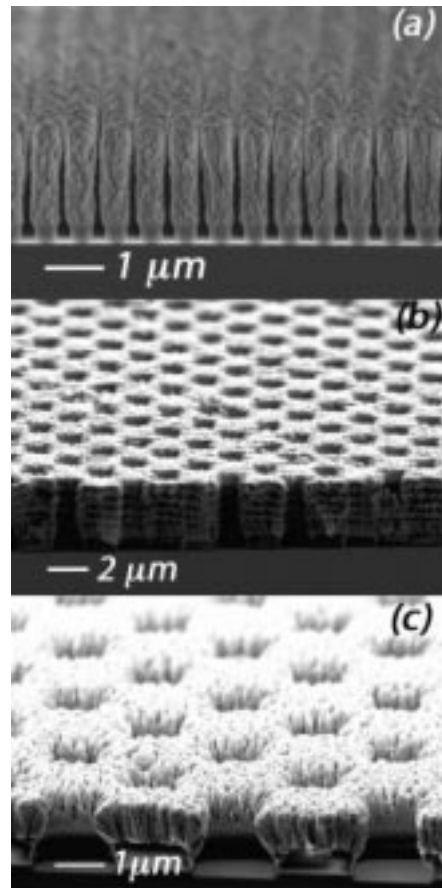
Figure 2. Sculptured thin films with transverse architecture grown on topgraphic substrates: (a) silicon oxide pillars comprising helicoidal nanowires grown selective atop a body-centred cubic array of disks; (b) helicoidal nanowires of silicon oxide deposited on a checkerboard; (c) helicoidal nanowires of chromium deposited on a checkerboard.

Multi-component active photonic structures via holographic photopolymerization

Increased demand for instantaneous information and the shift to network-centric information distribution has been driving the development of all-optical networks and circuits. The next generation of electro-optic components will have to enable networks, and this has motivated much research in the field of photonic-bandgap (PBG) materials, also known as photonic crystals. The way refractive-index modulations at periodicities on the order of the optical wavelengths suppress photon propagation is analogous to that of electrons in a semiconductor lattice. Depending on index contrast and structure, the PBG may deplete photonic states for propagation in limited or all directions (partial or complete PBGs). Challenges in directly fabricating inorganic PBGs with submicron periodicities required for the near-infrared and visible wavelengths have spurred exploration of organic options. Organics allow easy, large-area, and low-cost processing: features that outweigh the relatively low index contrast for applications where extremely high fidelity is unnecessary, such as tunable filters.

Numerous methods have been developed for fabricating organic PGBs including self-assembly of block co-polymers, two-photon microfabrication, and holography. Holographic patterning involves the interference of two or more laser beams and allows rapid fabrication (~seconds) and high design flexibility not limited by multi-phase equilibrium. Such patterning generally falls into two categories, lithography and photopolymerization. The former involves holographic exposure of a cross-linkable resin and, though face- and body-centered cubic photonic crystals have been fabricated this way, the removal of unreacted material can be difficult: especially with submicron features.

The photopolymerization process holographically initiates polymerization of a monomer within a syrup. Non-reactive components in the syrup phase—such as liquid crystals or nanoparticles—separate from the regions of rapid molecular weight increase. This enables one-step fabrication of electro-optic structures such as electric-field-switchable transmission and reflection structures (holographic polymer-dispersed liquid crystals or H-PDLCs). The inherent flexibility of the photopolymerization process allows fabrication of switchable multi-dimensional structures as well as reflective Bragg stacks that are not directly accessible with lithography. However, photopolymerized structures have more intrinsic interfacial roughness.

As for applications of organic photonic crys-tals, the photon localization exhibited by PGBs at the stop-band edges can allow low-threshold lasing. The ability to simultaneously spatially-segregate active elements and create robust integrated electrode interfaces make holographic photopolymerization well-suited for this application. Recently, switchable optically-pumped lasing from coumarin 485 was demonstrated using a 1D H-PDLC reflection grating as a distributed-feedback cavity. By increasing the dimensionality of the H-PDLC to a 2D columnar structure (Figure 2, foreground), the lasing threshold decreased by 40% compared to a 1D structure. Two things account for the improved performance: increased photon localization, and the ability to engineer the PBG in such a way that the absorption of the pump beam was maximized while the edge of the stop band was tuned to the fluorescence maximum of the pyrromethene 597 (Figure 2).

The rapid speed of formation, uniformity, good domain-size control, switchability, and addition of optically-active components make the H-PDLCs an attractive polymeric/organic platform for optically-active PBGs and organic nanophotonics. Through the controlled interference of multiple lasers at discrete angles, a seemingly endless possibility of photonic crystal structures can be made to accommodate a variety of excitation and emission wavelengths.

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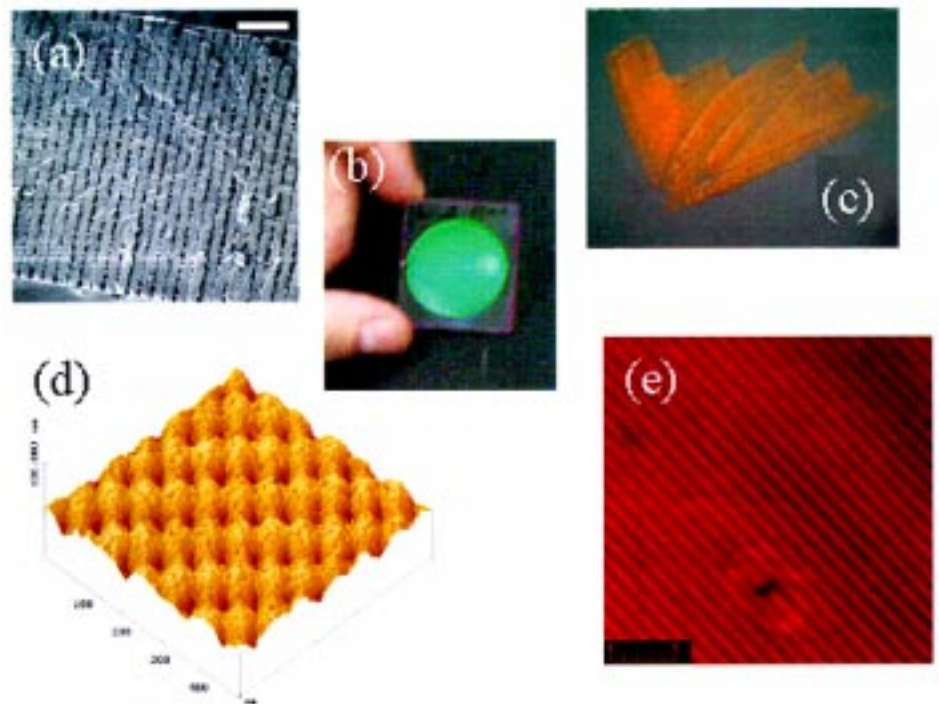E-mail: {Rachel.Jakubiak, Timothy.Bunning, Richard.Vaia}@wpafb.af.mil

Figure 1. Examples of multi-component photonic structures from holographic photopolymerization. (a) SEM micrograph of an H-PDLC transmission grating. The scale bar represents 1500nm. Light areas are cross-linked polymer and the dark cavities are liquid crystal domains. (b) Photograph of an H-PDLC tunable filter demonstrating large area fabrication.[8] (c) Photograph of an H-PDLC hologram: note the detail on the wings. (d) SEM micrograph of the top surface of a 3D H-PDLC photonic crystal.[7] (e) Confocal micrograph of a transmission grating from holographic photopolymerization of a syrup containing monomer and gold nanoparticles.[6] Scale bar represents 5μm.

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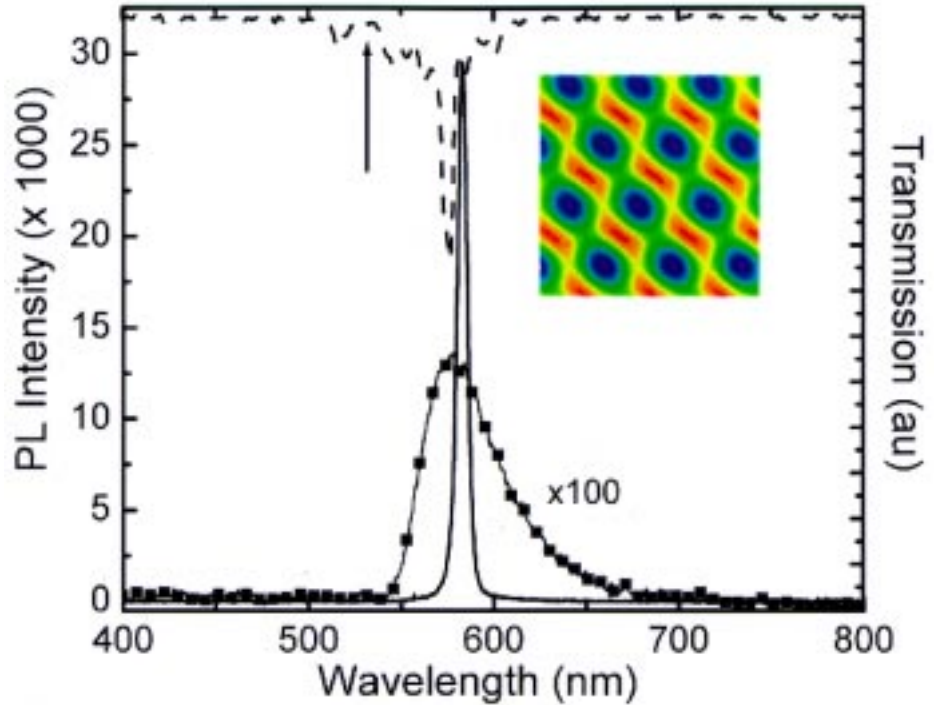
Figure 2. Performance of a 2D columnar H-PDLC laser resonator. White-light transmission through a switchable 2D organic laser (upper dashed line). The light intensity distribution from three intersecting laser beams used to make the H-PDLC is given in the foreground where the dark regions represent the low-light-intensity LC-rich regions. Above the excitation threshold ($9\mu J/cm^2$) the photoluminescence of pyrromethene 597 incorporated in the 2D H-PDLC narrows from 35nm to 4nm full width at half maximum.

Nanophotonics

Paras N. Prasad, State Univ. of New York/Buffalo

This introductory course examines the challenging opportunities for studying the interaction between light and matter on a scale much smaller than the wavelength of radiation. The use of such a confined interaction to spatially localize photochemical processes offers exciting opportunities for nanofabrication. Nanophotonics also has important biomedical applications in bioimaging, optical diagnostics, and photodynamic therapy.
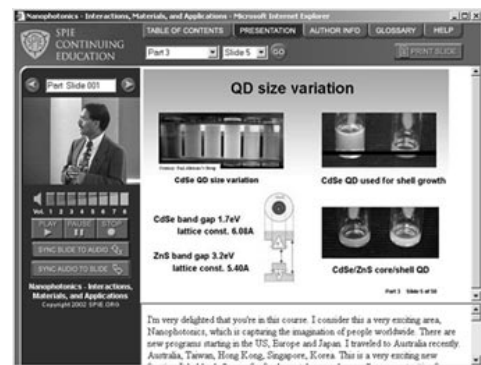Course level: Introductory.

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Meso-architectures from nanotubes

Nanotubes are the wonder materials of nanotechnology: since their discovery in 1991, all kinds of high-tech applications have been demonstrated for this material. Such applications span from use as single electron devices to their exploitation as structural reinforcements. The two major varieties of nanotubes—muti-walled (2-20nm diameter) and single-walled (~1nm diameter)—essentially differ in the number of layers that constitute the structure. Both have found this broad utility. The materials differ from their parents—graphite precursors—in their geometry, dimensionality, and structural organization: the helical ordering of the honeycomb lattice is an example. Thus they behave completely differently. In terms of their mechanical, electrical, and thermal properties, there are few other materials that can match their excellence.

Nanotubes are made by a variety of techniques including electric-arc discharge, laser ablation, and chemical-vapor deposition. The last few years of research in this field have proved that the latter technique is the best in terms of yield, scale-up, and the ability to organize nanotubes into well-defined architectures. This assembly of nanotubes into organized meso-structures is an important goal: it will allow us to tailor the basic infrastructures as aligned arrays and networks, ideal for electronic applications[1] (e.g. interconnects). We have made considerable progress in this direction and are now able to configure nanotubes into various complex structures, both free-standing and over patterned substrates.

The combination of lithography (to prepare the substrate template) and catalyst-assisted CVD can produce organized, oriented nanotubes over macroscopic areas.[2] Controlled networks of single-walled nanotubes (SWNT) of differing density and orientation can be built for electronic application on patterned silicon/silicon oxide structures. A simple thermal CVD technique is used, using methane (CH_4) as a carbon source. Extended networks of suspended nanotube cables are grown between predefined catalyst locations on substrates. If these cables can be connected to metallic contacts, the structure would constitute an amazing interconnected and highly-conductive nanowire network. Indeed, a great number of separated, suspended, individual SWNTs can be created, bridging pre-machined silicon/silica trenches (Figure 1). The use of such structures as parallel connected elements in molecular-sensing applications could result in extremely high sensitivity. This is due to the molecular
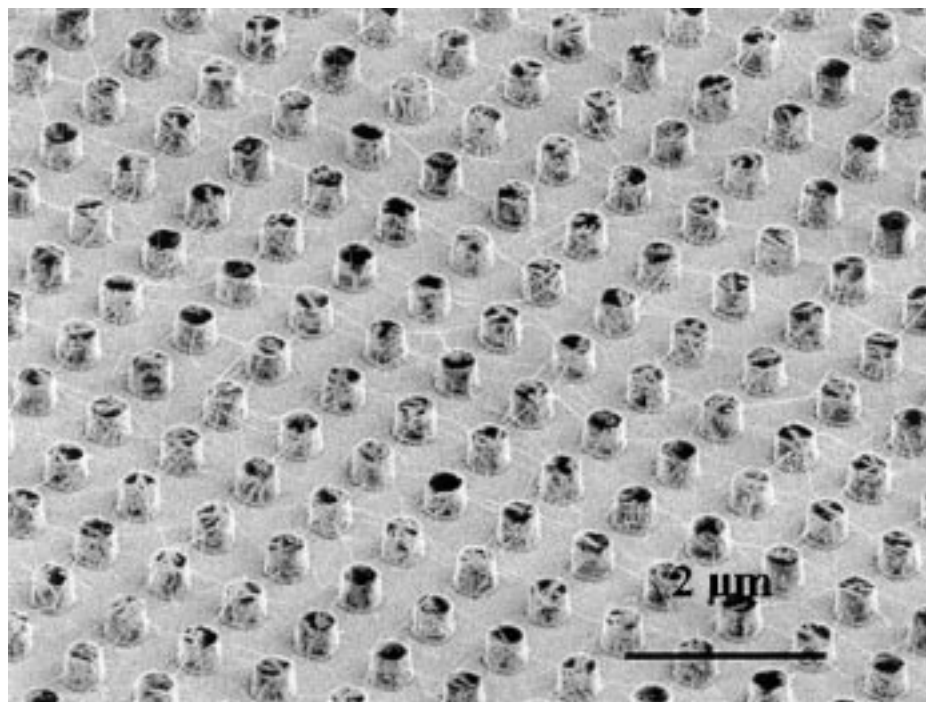

Figure 1. Large area of single-walled nanotube networks bridging predefined silicon pillars. A thin layer of catalyst metal (Fe) is deposited on the pillars and nanotubes grow on these during a chemical-vapor-deposition process.

dimensions and sensitivity of individual elements. Further chemical functionalization of the nanotube elements provides pathways for selective discrimination of molecular and biomolecular species.

Three dimensional nanotube architectures are now also a reality.[3] Multi-walled nanotubes (MWNT) have been assembled in predetermined orientations on silicon/silicon-dioxide substrates to build 1-to-3D architectures by harnessing the substrate selectivity of lithographically-machined non-planar templates. Simultaneous growth of highly-organized nanotubes in multiple directions is achieved in a single process using the preference of nanotubes to grow normal to, and selectively on, silica surfaces (Figure 2). These structures could be ideal for nanoelectro mechanical applications if the individual mesostructures can be addressed electrically. Such structures may also be considered for MEMS and may be selectively coated to produce integrated architectures for damping, high-thermal-conductivity vias for on-chip thermal management, and meso-scale electrical-brush contacts.

In addition to organized nanotube arrays built on rigid substrates, large macroscopically-long cables of aligned nanotubes could allow further progress. Such cables have been fabricated under optimized CVD conditions: we were able to produce SWNT strands[4] as long as ~20cm. These strands are built of well-aligned nanotubes and possess excellent electrical and mechanical properties (although inferior to individual nanotubes). Further, these aggregates provide the first examples of nanotube-based structures that could be evaluated and manipulated using methods that are readily available today. These highly-ordered mesoscale architectures, built from the most fascinating of building blocks (nanotubes), should become part of our daily life in the near future.

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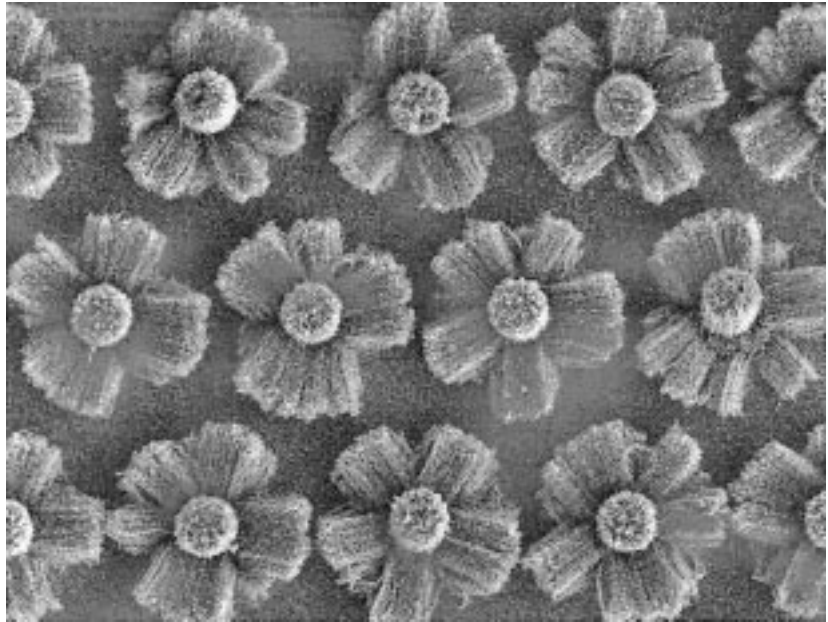
Figure 2. Highly-ordered three-dimensional complex patterns, created from organized multi-walled nanotubes on predefined silica surfaces. The tubes self-assemble in a substrate-selective growth process from the vapor phase, which contains hydrocarbon precursor and metal catalyst vapor.

Smart windows and intelligent glass façades: nanomaterials for indoor comfort and energy efficiency

Increased use of air conditioning is clear evidence of our growing demand for high living standards and comfort. The use of air conditioning has risen generally, but is most noticeable in the USA, Europe and particularly South-East Asia where, in some regions, half the electricity consumption goes towards creating a good indoor climate!

We currently need all this air conditioning because windows allow in a lot of solar energy. About half of this arrives as visible light, and the other half is invisible infrared radiation. But the remedy to the energy problem is not to remove the windows or lower their transparency excessively: they create visual contact between indoors and outdoors, a feature that is absolutely necessary for our well-being, and they admit daylight. Generally speaking, installed areas of windows and glass façades have increased in new buildings over many years. Though this may be an architectural fad, it satisfies genuine human needs and is therefore sound.[1]

The challenge
Thus we have defined a problem—or created a challenge: to keep the windows without letting in too much solar energy. How can these demands be reconciled? A complete solution is not possible, but the situation can be improved by first making sure that the visible light is transmitted while the rest—as far as possible—is reflected. Window glass of this type, known as 'solar-control glass', has been on the market for several years.[2,3]

However with new technology for 'smart windows'[4,5] and 'intelligent glass façades',[6] it should be possible to have windows with variable transparency so that they let in light only when someone is present in the room. Simple estimates of the energy savings that result from this controllability show that smart windows would save as much electricity as could be generated by replacing it with a panel using today's most efficient solar cells. If, instead, we consider a room with a predominant heating load, the window obviously should be in a state of maximum transmittance when nobody is present.

The issue of energy efficiency is only part of the picture. The smart window is also a means of providing human comfort: allowing the occupant to see out, and to turn up and down the natural light and heat, as desired. The real benefit is that these two features are combined in the same device.

Nanomaterials and smart windows
How can one build smart windows with variable transparency? One solution is to use a thin nanomaterial-based surface coating, either on the glass itself or on a suitable foil laminated onto or between panes. Figure 1 illustrates the latter option. Specifically shown is a construction with two polyester foils, each with a transparent, electrically-conducting surface coating (ITO). Each of these has a second coating: one of nanoporous tungsten oxide, the other of nanoporous nickel oxide. The facing surfaces of the two oxides are joined by a polymer layer serving as a conductor for ions. The four surface coatings together can be less than a micron thick, while the polymer laminate can be thicker (100µm in a practical device).

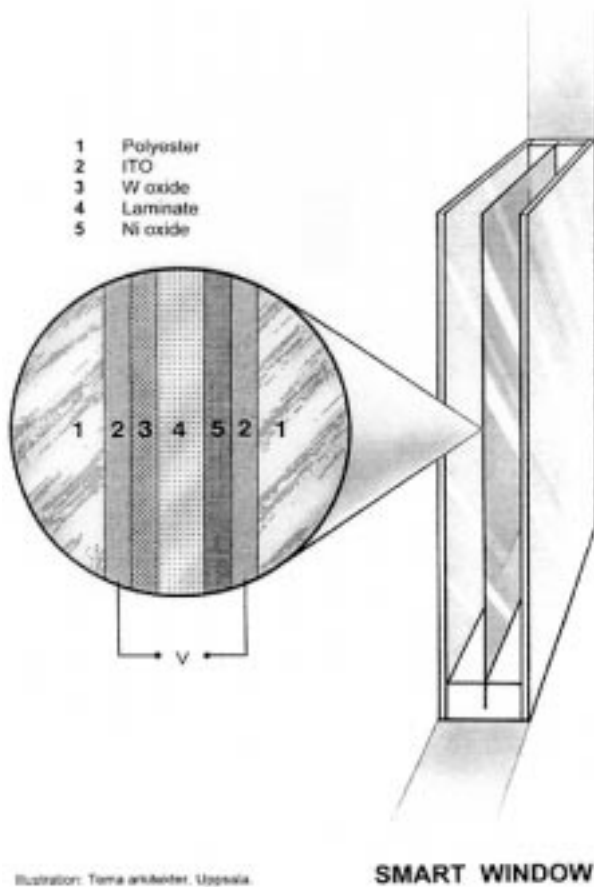The device shown in Figure 1 is similar to a

1 Polyester
2 ITO
3 W oxide
4 Laminate
5 Ni oxide

SMART WINDOW

Illustration: Torra arkitekter, Uppsala.

Figure 1. Principle of a 'smart window' embodying a polyester-based laminated double-foil with coatings of nanoporous electrochromic oxides. ITO indicates transparent and electrically-conducting coatings of indium tin oxide.[7] The application of a voltage (denoted V) changes the transparency.
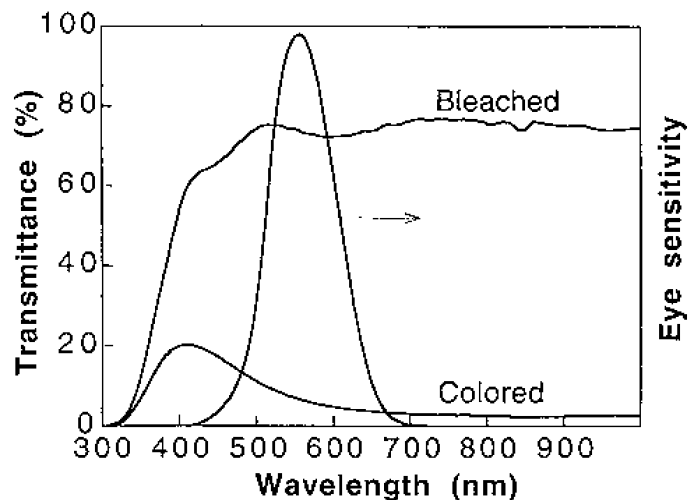

Figure 2. Wavelength-dependent transmittance of a variable-transparency foil of the type illustrated in Figure 1. The visible light (bell-shaped curve shows the eye's sensitivity) can be modulated between 75% and 7%.

battery, with the nanoporous oxide coatings being anode and cathode and the intervening polymer layer serving as electrolyte. This can be charged by the application of a voltage of the order of one volt—conveniently obtained from solar cells. The tungsten oxide is dark in the charged state and transparent when discharged, while the opposite is true of nickel oxide.[4,5] The transparency of the laminated double-foil in Figure 1 can, therefore, be varied by shuttling charge between the nanoporous oxide coatings. In particular, structural control of the materials on the nanometer level is needed for successful operation.

Figure 2 shows approximate boundaries of the transmittance. It is possible to stop at any intermediate transmittance level, i.e., the foils have a 'memory' so the voltage needs to be applied only for altering the optical properties. The time for going between clear and dark states depends on the size of the window, and anything between ten seconds and a few minutes can be regarded as typical. These times are comparable to the time it takes for the eye to adjust to different lighting levels.

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Nanoscopic engineering of dramatically improved electro-optic materials: ferroelectric lattices by design

We have used nanoscopic engineering of supramolecular, chromophore-containing bundles to achieve a dramatic improvement in macroscopic electro-optic activity, e.g., to values of 180pm/V at telecommunication wavelengths. Such values are a factor of six times greater than the electro-optic activity exhibited by the best commercially-available electro-optic material, namely lithium niobate. Enhanced electro-optic performance has also been achieved by incorporating new electro-optic materials into nano- and mesoscopic ring-resonator device structures for applications such as active (voltage-controlled) wavelength division multiplexing, voltage-controlled wavelength filtering, voltage-controlled optical-interconnect reconfiguration, and voltage-controlled wavelength tuning of light sources.

Electro-optic activity depends upon chromophores (characterized by large molecular hyper-polarizability) assuming non-centrosymmetric (ferroelectric) order. Such order is typically opposed by the inter-molecular electronic electrostatic interactions among high-dipole-moment chromophores. However, recent theoretical calculations[1-4] have shown that there are two competing components to the relevant potential describing these interactions. One favors centrosymmetric chromophore organization while the other favors ferroelectric. Theory further predicts that the relative weights of these two contributions will be defined by intermolecular and nuclear-repulsive (steric) interactions (i.e., by overall molecular shape).[1-5] For organic electro-optic materials, the electronic interactions will be dominated by π electrons while the steric will be influenced by the presence of all atoms (those involved in σ as well as π bonding).

Thus, a new paradigm for production of improved electro-optic materials has arisen based on nanoscopic control of chromophore shape. This paradigm has recently been extended to include incorporation of chromophores into nanoscopic dendrimers and dendronized polymers (see Figure 1) with factors of two enhancement in electro-optic activity over the best values that can be achieved for the same chromophore in a non-nanostructured environment.[6] These dendronized polymers can assemble into columnar, discotic arrays with enhanced ferroelectric ordering of incorporated chromophores. Theoretically-inspired structures exhibiting electro-optic activity on the order of 300pm/V can be anticipated in the near
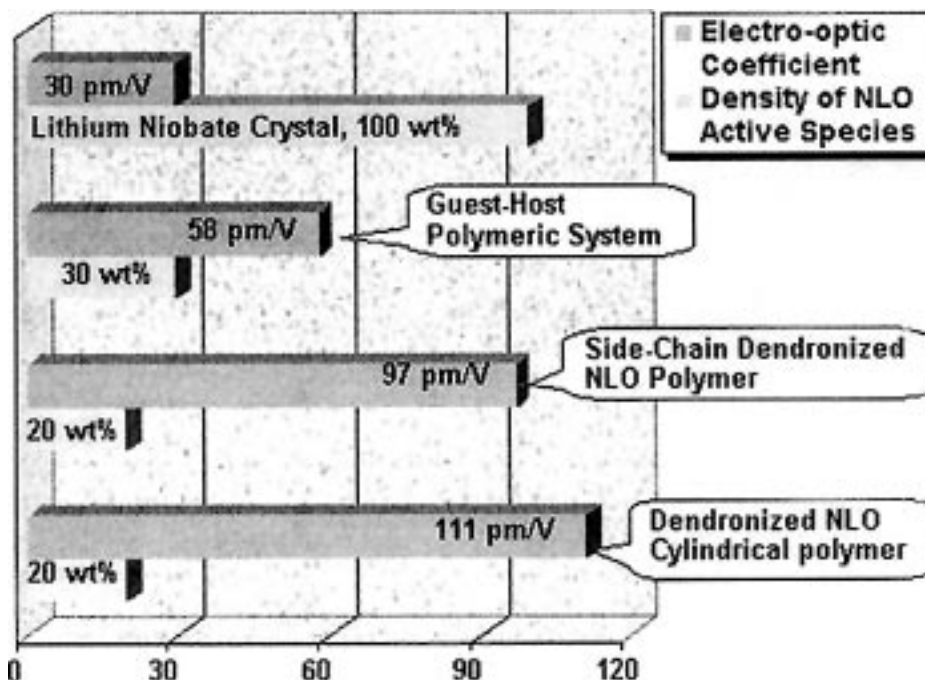

Figure 1. Electro-optic activity and the concentration of electro-active species are shown for crystalline lithium niobate and the same organic electro-optic chromophore in three types of lattices (a chromophore/polymer composite and two nanoscopically-engineered dendronized polymer lattices). Note the factor of two improvement afforded by nanoscopic control.

future with the potential of values on the order of 1000pm/V if more complete nanoscopic structures can be implemented. The consequence of such an advance in materials would be the ability to fabricate a wide range of devices that could operate on less than one Volt and have bandwidths of several hundred giga-Hertz.

Another advance based on exploiting nanoscience/nanotechnology has been the incorporation of electro-optic materials into nano- and mesoscopic ring microresonator device structures.[3,4,7,8] Such devices (fabricated either by soft lithography, e-beam etching, or reactive ion etching) permit dramatic reduction in device dimensions (to much smaller than the size of a human hair) and permit realization of added functionality (e.g., wavelength selectivity). Quality factor values on the order of 100,000 are straightforwardly achieved permitting wavelength selectivity of 0.01nm at telecommunication wavelengths.[7] This can be used for active wavelength division multiplexing (see Figure 2), where information can be color

coded at the rate of 10Gb/sec (per color or per wavelength) at 1V. High-density integration of ring-resonator structures can permit data handling at rates of hundreds of gigabits per second. Organic materials have the advantage that they can be made into both temperature dependent and independent device structures. With thermal structures, both temperature and voltage can be used for device control. Nano- and mesoscopic ring resonators have been fabricated both out of organic electro-optic materials[3,4,7] and from silicon.[8]

Electro-optic materials and devices have many applications critical to defense, computing, telecommunications, transportation, health care, and homeland security. Common devices include phased-array radar, optical-routing switches, electrical-to-optical signal transducers, optical gyroscopes, spectrum analyzers, spatial light modulators, reconfigurable optical interconnects, sensors, radio-frequency signal generators, analog-to-digital converters and frequency-agile light sources.

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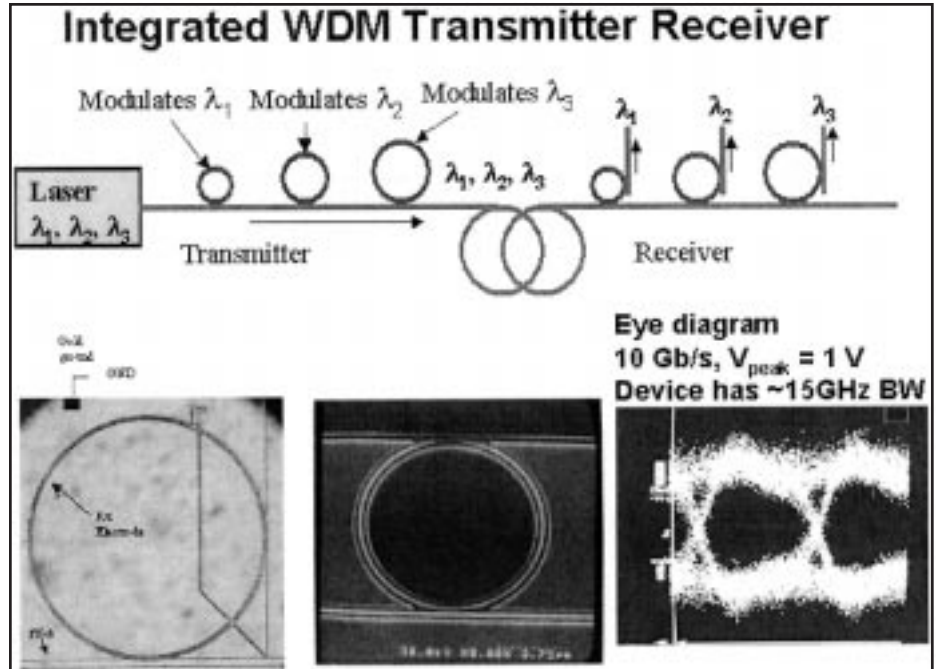   Dalton, **Appl. Phys. Lett. 83,** pp. 4689-4691,
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Figure 2. A schematic diagram of an active wavelength-division-multiplexing (WDM) transmitter/receiver is shown together with micrographs of ring microresonators. Also shown is an eye diagram demonstrating the performance of one of these ring resonators.

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