Summary Descriptions of Selected Projects Being Investigated
(Sungho Jin and collaborators)

*--- Innovative approaches to solve some major problems*

1. Low cost Si slicing for photovoltaic cells [Drastically reduced kerf loss]
2. Thermoelectric materials [Easy scale-up manufacturing of nano-grained TE materials.]
3. Wear-resistant, self-cleaning, superhydrophobic coatings [Ceramic based, teflon-free, inexpensive and durable super-omniphobic coatings on glass, plastic and other surfaces]
4. Concentrating solar power (CSP) system, solar-thermal generator [Spectrally selective, sunlight-absorbing coatings]
5. Dye sensitized solar cells (DSSC) and perovskite sensitized solar cells (PSSC) [FTO glass free, transparent and high-electrical-conductivity electrodes]
6. Compliant thermal interface material (TIM) [To provide mechanical stress accommodation and high thermal conductivity simultaneously]
7. Ultra-high-density vertical solar cell array [DUV processed or nano-imprinted Si]
8. Sunlight reflective coatings [To keep buildings and automobiles cooler in the summer and warmer in the winter]
9. Universal solders [For easy integration of solar cells involving difficult-to-bond surfaces like Ag-free metallic electrode surface]

**Si Nano/Micro-Shaping for Reduced Cost Photovoltaic Solar Cells**

*Massively parallel, Si chemical slicing for low cost, low-loss thin wafers*
--- Current PV solar cells are too expensive for wide applications as renewable energy source.
--- One of the major cost factors is the Si material, which amounts to almost one-half of the single crystal solar cell cost.
--- Typical wire-saw slicing of Si wafer results in ~200 μm loss of Si per cut for ~200 μm thick wafer slicing.
--- We have developed a new slicing technique that reduces the cut loss to as little as ~10 μm, which also allows a fabrication of very thin Si wafers (~5-30 μm thick) for additional reduction in Si materials usage in PV solar cells.
--- This novel technique can also be utilized for shaping of Si into tall nano-micro wires, zigzag nanowire arrays, nano-tunnels, very thin flexible wafers for various applications such as energy, photonics, electronics, biomedical devices as well as flexible circuits.
Thermoelectric Materials
--- The thermoelectric figure of merit (ZT) can be expressed as $ZT = \frac{S^2 \sigma}{\kappa} T$ (where $S$ is the Seebeck Coefficient, $\sigma$ is the electrical conductivity, and $\kappa$ is the thermal conductivity. For higher ZT, these materials parameters need to be optimized.
--- Thermoelectric (TE) alloys such as Bi-Sb-Te and skutterudites are promising energy materials for waste heat recovery and solar energy generation. For enhanced TE properties such as the energy conversion efficiency, it is essential to increase the phonon scattering and reduce the thermal conductivity. Various nanoparticle synthesis techniques based on physical, chemical or mechanical approaches are utilized to produce variety of nanoparticles of thermoelectric alloys and sintered nanograined alloys having excellent thermoelectric properties. Further nanostructure controls are being investigated to increase the phonon scattering, Seebeck Coefficient, and electrical conductivity of various thermoelectric materials.
--- Wear resistant, superhydrophobic glass surface made of transparent ceramic nanostructure, rather than easily smearable polymer coatings. Such coatings are needed for many industrial and consumer market applications.
--- For anti-fingerprint surface (on cell phones or touch-sensitive screens), both superhydrophobic + superoleophobic properties are needed.
--- Also, for “maintenance-free, anti-reflective PV cell array and clean high-rise-building window glasses.
--- Such transparent ceramic coatings have been developed (past 5 yrs R&D effort), and scale-up manufacturability is being considered in the nanostructure design and processing.

Self-cleaning glass surface
--- Nanostructured glass or silica surfaces having superhydrophobic and omniphobic properties have been developed for maintenance-free solar panel surface (with minimal needs for washing/cleaning during solar cell use lifetime).

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Self-Cleaning Surface --- water nonwetting & oil nonwetting are necessary conditions.

--- Schematic illustration of contact angles for hydrophilic, hydrophobic, and superhydrophobic surfaces. A similar definition of oleophilic, oleophobic and superoleophobic, also applies for oil wetting instead of water wetting.
--- Super-omniphobic ceramic surface (having both superhydrophobic and superoleophobic) has been demonstrated.
Composite of nanopillars/nanowires (providing super-omniphobic nonwetting properties) and flat but transparent shoulder grid array (providing non-scratch wear resistance).

(a) Nanopillar array (transparent SiO₂)
(b) Shoulder grid array (e.g., transparent SiO₂)
(c) Substrate (e.g., glass)

Super-hydrophobic
More
Super-hydrophobic
Super-omniphobic

JIS K 5600 Standard Hardness Tester using 45° Tilted Pencil for scratch testing for evaluation of wear-resistance

Anti-fingerprint tested (promising results)

 Wear resistance tested on micropillar version surface hardness (strong enough with H=9) with some graphite, now switching to nanopillar version to prevent pencil debris trapping, for AR

SEM of easily manufacturable nanopillar version surface with improved ~98% light transmission and already having only ~2% reflectivity (Self-cleaning + AR coating)

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SEM of easily manufacturable nanopillar version surface with improved ~98% light transmission and already having only ~2% reflectivity (Self-cleaning + AR coating)

Anti-fingerprint tested (promising results)

(a) SiO₂ w/o anti-fingerprint patterns
Obvious fingerprint mark

(b) SiO₂ with anti-fingerprint patterns
Less obvious fingerprint mark

Panel Width/Spacing=10 µm/1,000 µm

Panel Width/Spacing=50 µm/200 µm

Original glass
Nanopattern etched on glass
Nanopattern etched on SiO₂ buffer layer

Transmittance (%)
Wavelength (nm)
Concentrating Solar Power --- High Performance Nanostructured Spectrally Selective Coating

- **Concentrating solar power (CSP) ---** A viable, commercialized technology that competes effectively with the photovoltaic solar energy. This Carnot cycle-based energy conversion is based on focusing sunlight onto the Spectrally Selective Coating (SSC) on a steel pipe that contains a molten salt heated by the absorbed thermal energy. The molten salt is sent to the power plant where steam is generated to operate steam turbines and generate electricity. The solar thermal Carnot cycle efficiency is high, ~40% at the current operating temperature of ~450°C. The goal of this DOE-funded project is to further increase the efficiency toward ~60% regime by developing new, more efficient, sunlight absorbing SSC layer that will enable a 700-750°C CSP operation. These efficiency values are much higher than the typical photovoltaic energy conversion efficiency (~25%).

- **Spectrally selective coating (SSC) ---** A critical component that enables high-temperature and high-efficiency operation of concentrated solar power (CSP) systems. SSC has a profound impact on the performance and cost of CSP systems. The optical properties of the SSC, namely, absorption in the solar spectrum range (UV/Vis) is maximized by materials design as we pursue a bandgap-adjusted, nanoparticle semiconductor materials while the reflectance, while the undesirable black body emission loss in IR (infrared) regime is minimized.

- For higher temperature operation to achieve higher Carnot efficiency, the semiconductor material nanoparticles need to be protected from oxidation, e.g., with the synthesis of a variety of nano core-shell structures. configuration surface layer as investigated in this project.
**Rare-Earth-Free Permanent Magnet Alloys**

--- The high price of rare earth metals, especially Dy utilized in Nd-Fe-B magnets has instigated active R&D toward new, rare-earth-free permanent magnet materials.

--- We employ a spark erosion technique to easily produce nanoparticles of Mn-Bi magnet alloys so that the high magnetocrystalline anisotropy of the material is fully utilized with minimal domain wall motion. Soft-magnet / hard-magnet exchange coupled spring magnets with higher coercive force are also being developed.

--- **Hc vs Temp. for spark eroded MnBi nanoparticles**

- Hc approaching 30 KOe
- The stability of Hc well beyond 200°C, up to ~300°C (573K) demonstrated.
- Exchange coupled core-shell magnets being designed.

--- **Smell-O-Vision Devices Using X-Y Matrix Controlled Odor Release**

--- Virtual reality can be made more realistic with a three-dimensional or other sensory input. Out of the five senses humans have (i.e., vision, sound, smell, taste and touching), we have already incorporated the first two senses in modern communications and entertainment systems such as TVs, mobile phones, computers, and movies.

--- To enhance the quality of entertainment and communications, it would be nice to incorporate another sense, a sense of smell. Synchronization of odor release to the corresponding image on the screen can be accomplished conveniently by electronic signals using a reliable, inexpensive, and not cumbersome device.

--- Odor releasing devices that allow easy on-off switching of odor flux could have a significant impact on the effectiveness of virtual reality. We have developed a fast, repeatable new odor/gas releasing system having a novel X-Y matrix addressable
The device lasts for a long time as the scent release on-off takes only millisecond and the battery use is minimal.

--- Odor generating system with improved kinetics and reliability, test controllability with a system embedded in TV, and demonstrate programmable odor release in a synchronized manner together with the visual images on screen are being investigated.

--- Odor releasing TV (a) Jennifer Lopez TV scene with synchronized release of Jennifer Lopez perfume (“Live by Jennifer Lopez”) smell. (b) Diagram of communication for the operation of hardware.

Laboratory demo of X-Y matrix odor release device

Four-scent release demo video (for Computer Monitor, TV or Cell Phone) --- Coffee, pizza, perfume, and fresh-cut grass, featuring Dr. Calvin Gardner

Wearable headset for Virtual Reality (VR) or Augmented Reality (AR) --- Add various selective, on-demand scents to headset and other computer or communication devices
**Graphene Processing and Properties**

- Graphene is a very exciting new material with many potential applications. For semiconductor use with graphene’s high carrier mobility and other unique properties, the band-gap has to be opened.
- New Anodized Aluminum Oxide (AAO) template with smaller 40-50 nm diameters, 200-300nm thickness developed.
- Such AAO templates were utilized to pattern graphene layer (CVD grown on Cu substrate followed by removal of Cu). Honeycomb-geometry graphene was obtained so as to produce enhanced edge effect and band-gap opening. Magnetic nano-island arrays are also being fabricated for enhanced magneto-transport properties.
- Electronic and magnetic properties of nano-modified graphene layers are being evaluated.
Sheet resistance ($R_s$) increase caused by nano-patterning of graphene (green arrows) using AAO template etch mask, and near-complete recovery of the electrical conductivity by HNO$_3$ chemical doping (red arrows), especially for the 40 sec etched graphene sample. The $R_s$ values are plotted for three different etching times (30, 40 and 50 sec).
UV–VIS spectra of pristine and nano patterned (NP) graphene films on quartz substrates, showing a significant increase of optical transmission by nano-patterning.

**Carbon Nanotube Geometry Control**

- Sharply pointed carbon nanotubes can have even smaller tip diameter because of elimination of catalyst particle radius of curvature. Such sharp tips are advantageous for enhanced field emission, high-resolution metrology, bio-insertion of molecules and functionalities, etc.
- While straight carbon nanotubes are relatively easy to grow, curved or bent nanotubes are difficult to synthesize — For technical applications, sharply bent or zig-zag carbon nanotubes are important for nano spring applications, sidewall tracing scanning probes, routing of nanoelectronics interconnects, and possible introduction of defects to form hetero-junction nanotube semiconductor devices.

*Bending and orienting of Carbon Nanotubes: Experimental Setup and Electric-Field-Direction Modeling*

![Bending and orienting of Carbon Nanotubes](image)

*E vector modeling done using Maxwell SV*
Periodic and Aligned Carbon Nanotube Array by Electric-Field-Guided CVD Growth

CVD growth chamber for bent or zig-zag nanotube growth


**Nanoelectronics**

**Carbon nanotubes for nanoelectronics --- Electrical switching behavior and logic in CNT Y-Junction transistors**

- Sharp transistor switching behavior enabled by 3rd branch as a gate.
- Natural CNT gate --- No external gate fabrication necessary.
- Three-way gating operation demonstrated.
**Nanoprobe Design and Fabrication**
- For creation of extremely fine AFM probe tips which have desirable high-resolution and mechanical durability.
- For field emission, nano lithography applications, etc.
- Nanoscale conductance probes for biological ionic conductivity measurements near ion channels (e.g., study of Alzheimer’s disease).
- For bio engineering modification of cells with nano-needles or nano-pipettes (by insertion of genes, growth factors, drug molecules, etc.) for cell behavior study and therapeutic applications.

**Carbon Nano Cone AFM Probe on Si Cantilever**

On AFM cantilever (By patterning of a single Ni island by lithography + Electric field guided chemical vapor deposition of carbon nanocones)

**AFM Tips Being Made by Jin Group**
- Using Sharp Carbon Nanocones.
- Tip radius of curvature ~ 1 nm regime.
- Electrically conductive (for bio imaging or conductance imaging).
- Mechanically durable.
- High aspect ratio for deep trench or via hole imaging.

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**Carbon nanocone tip on AFM probe**

![Carbon nanocone tip on AFM probe](image1)

**Carbon nanocone AFM probe deposited on pedestal cantilever**

![Carbon nanocone AFM probe deposited on pedestal cantilever](image2)

**Deep Trench Imaging Capability**

- 500 nm deep PMMA resist pattern on Si substrate, with a 300 nm line/space pattern

**Standard Si AFM Tip** (erroneous image)  **Carbon Nanocone Tip** (more accurate image)

![Standard Si AFM Tip](image3)  ![Carbon Nanocone Tip](image4)

**High resolution AFM image of Cu film by carbon nanocone probe**

![High resolution AFM image of Cu film by carbon nanocone probe](image5)
**Focused Ion Beam Modification of Nanostructures**
--- CNT with subtractive line defects + four-point lead wires for electronic transport measurements.

**Si nanophotonics --- For future ultra-high-density Semiconductor circuits**
- Focused-ion-beam carved to introduce delay lines for light propagation and to locally slow down light movement through Si waveguide --- by fabricating smaller, dimensionally optimized paths.

**FIB-induced geometry manipulations for thinning and necking of Si nanowire paths --- To create wavy Si, necked Si, narrowed wall Si**

Controlled location and uniform depth of milling into Si wall arrays using Focused Ion beam rastering
**Nanofabrication of 10-15 nm features**

E-beam patterned HSQ resist island array with 1.6 TB/in² density on Si

**Magnetic Nanostructure and Patterned Recording Media**

- There is a need to substantially increase the density of magnetic recording media.
- Patterned media with periodic array of ~10-20 nm regime magnetic nanoislands or nanowire magnets are highly desirable.
- 0-20 nm nanomagnet dimension is well below the available lithography limit.
- New, innovative synthesis/fabrication approaches are desirable.

### Upper Limit Recording Density vs Bit Size

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<th>Bit Size (nm)</th>
<th>2</th>
<th>3</th>
<th>5</th>
<th>10</th>
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<td>6</td>
<td>10</td>
<td>20</td>
<td>25</td>
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<tr>
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<td>18</td>
<td>6.5</td>
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Successfully Fabricated ~20 nm Diameter Vertically aligned CoPt Nanomagnets in Anodized Aluminum Oxide Membranes

CoPt nanomagnets (aluminum oxide matrix dissolved away to show the nanowires)
Advanced Patterned Recording Media by Nanofabrication

- One bit per each ‘island’ approach -- To go beyond the current hard disk memory limit of ~200 GB/in² memory.
- Areal memory densities of more than 1 TB/in² desired.
- Difficulty in 10 nm regime bit fabrications, large area, in circular periodic pattern --- very challenging.
- For eventual manufacturing for large-area patterning, Nano-imprint Lithography is the most viable technology.

Energy-Related Materials

Electrochemical Modification of Vertically Aligned Carbon Nanotube Arrays

--- The presence of metal particles (e.g., Ni) may interfere with the intended chemical or electrochemical reactions.
--- Tip-Opening (oxidation) + electrodeposition of Pt nano particles (reduction) onto vertically-aligned CNT arrays.

Carbon Nanotube Array for Fuel Cell Applications

--- On conductive substrate (carbon microfiber, carbon paper)
--- As electrode material to carry Pt catalyst particles

Bio Materials
Examples of Synthesis and Applications of Magnetic Nanoparticles

Potential Bio Applications – Cancer treatment, gene delivery, neural regeneration, drug delivery, magnetic cell sorting, MRI.

(a) Superparamagnetic Fe$_3$O$_4$  (b) Silica-Coated Fe$_3$O$_4$  (c) Fe$_3$O$_4$ Magnetic Nanoparticle Array

Temperature rise induced by remote magnetic field (100 KHz)
--- In a liquid containing various volume of magnetic nanoparticles.
--- ~10 nm diameter Fe$_3$O$_4$ particles.
--- Live cell magnetic hyperthermia experiments to be carried out.

PC-12 Neural Cancer Cells with Endocytosed Magnetic Nanoparticles -- Can be stimulated into Neurite growth by NGF growth factor

Nanotoxicity Study --- Response of PC12 neural cells to magnetic nanoparticles

Fe$_3$O$_4$ conc. = 15 mM (dramatic reduction of cell ability to generate neurites with increased concentration of magnetic nanoparticles --- spherical cell shape with much decreased surface area)

Immunofluorescence of typical PC-12 cells 4 days after endocytosis. Cytoskeletal structure shown with tubulin (fluorescein, green) and actin (rhodamine, TRITC labelled phallloidin, red).
Magnetically Guidable, Remote-Controlled Drug Delivery Nanocapsules

• Existing drug therapeutic techniques --- Inefficient in deep tumor drug delivery and lack on-demand drug release.
• A new technique that allows better drug penetration into cancer cell aggregates within tumors and subsequent switchable release is highly desirable.
• Developed hollow-sphere nanocapsules containing intentionally trapped magnetic nanoparticles and defined anticancer drugs --- To provide a powerful magnetic vector under moderate gradient magnetic fields.
• These drug-loaded nanocapsules can penetrate into the interior of tumors and allow a controlled on-off switchable release of the anticancer drug cargo via remote RF field.
• This imageable smart drug delivery system is compact (80~150 nm capsules).
• *In vitro* as well as *in vivo* results --- indicate that these nanocapsule-mediated, on-demand drug release is effective in reducing tumor cell growth.
• This magnetic vector nanotechnology may also be utilized to move the nanocapsules through BBB so as to release CNS drugs at selected locations.

(a) Drug-carrier nanocapsule fabrication and drug insertion

(Step 1) Porous silica or gold shell

Magnetic nanoparticles (Fe₃O₄)

Remove PS

(Step 2) Drug + liquid loading

Drugs + liquid

(Step 3)

Drug molecules

(b) FTIR confirmed PS removal

Silica capsule with polystyrene

Polystyrene peaks

PS-removed capsule

Relative Transmittance

Wave Number (cm⁻¹)

(c) TEM of nanocapsules (~100 nm) with trapped Fe₃O₄ magn. nanoparticles (10 nm dia, 45 vol. %)
Confocal microscopy images of the magnetic nanocapsule penetration into MT2 breast cancer cell colony using magnet gradient pulling force (vertical scale bar=50 µm) applied for 2 hrs by a Nd-Fe-B magnet (H=1,200 Oe near the cancer colony location). The Y-Z vertical section image, and X-Y horizontal section image near the bottom demonstrate a successful penetration of cancer colony by magnetic nanocapsules.
**Effect of Nanostructure of Bio Materials on Cell Growth**

**Confocal microscopy for BBB crossing** [Polystyrene surface + fluorescent dye attached + tail vein injection on mouse + confocal microscopy to trace magnetic nanocapsules (green). – BBB crossing is observed.]

**Blood vessel (TRITC-dextran, red)**

**Anthracene-Nanoparticle (green)**

**Red and green merged image**

Scale bar = 50μm

**Top view TEM micrograph of aligned TiO₂ nanotubes**
Control of Stem Cell Differentiation Dictated Solv by Nanotube Dimension

**Enhanced Osteoblast Cell Adhesion and Lock-In on Anatase TiO$_2$ Nanotubes**

Cell Growth into TiO$_2$ Nanotubes

**Effect of TiO$_2$ Nanotube Layer on Osteoblast Cell Growth --- Significantly Accelerated Growth by ~ 300 – 400%**

![Graph showing the effect of TiO$_2$ nanotube layer on osteoblast cell growth.](image)

**Control of Stem Cell Differentiation Dictated Solv by Nanotube Dimension**

- **(a) d=30nm**
- **(b) d=50nm**
- **(c) d=70nm**
- **(d) d=100nm**
- **(e) Oblique View, 100nm nanotubes**
- **(f) TEM section, 100nm nanotubes**

SEM micrographs of self-aligned TiO$_2$ nanotubes with significantly different diameters. The self-assembly layers were generated by anodizing Ti sheets. The images show highly ordered, vertically aligned nanotubes with four different nanotube pore diameters between approximately 30-100nm, created by controlling potentials ranging from 5 to 20 V. (e) Right-Top image is the oblique view of the 100 nm diameter TiO$_2$ nanotube, and (f) right-bottom image is the cross-sectional transmission electron microscopy (TEM) of the 100 nm dia. TiO$_2$ nanotubes. (All scale bars: 200 nm)
**Effect of Nanostructured Substrate on Growth of Mesenchymal Stem Cells**

Immunofluorescent images of reacted stem cells on 100nm diameter TiO$_2$ nanotubes after 3 weeks of culture.

FDA images of hMSCs on flat Ti and various diameter TiO$_2$ nanotubes (24 hr culture, Scale bar=100 μm).

- TiO$_2$ nanotube dimension significantly influences the hMSC (human mesenchymal stem cell) differentiation behavior
- Smaller diameter nanotubes (~30 nm dia) enhance cell adhesion and proliferation without differentiation, while larger diameter nanotubes (~100 nm dia) cause the stem cells to substantially elongate, stressed and preferentially differentiate into bone cells (osteoblasts), which can be useful for orthopaedic and dental applications.


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