The University of Western Ontario

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Volume 2, Issue 4 Fall 2006

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Additive-surface Interactions: A Combinatorial Approach to Studying Lubricant Additives

By Gavin Pereira
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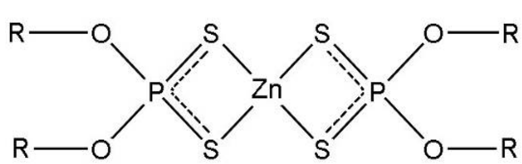
Tribology is a term which arose in the 1960s to describe the study of interacting moving surfaces involving friction and wear. Tribology encompasses aspects of physics, chemistry, applied mathematics, material science, mechanical and chemical engineering and applied mechanics. By applying the science of tribology to practical problems, such as reduction of friction and wear, innovative strides are being made towards molecular level knowledge and understanding in the transportation industry.

Due to continuous efforts in the automobile industry for reduced production costs and improved car design, not to mention increased fuel economy, there is a significant focus on efforts to reduce or replace the cast iron content in an engine in order to reduce engine weight and frictional losses due to wear. Aluminum alloys are a class of materials that are being used as replacements for cast iron in engine components. Aluminum itself is a poor alternative to replace steel due to its inadequate wear resistance; thus alloys of aluminum have been introduced that offer better wear protection, higher strength and fracture toughness, high specific rigidity, good thermal and electrical conductivity, and easy machining. Alloying aluminum with silicon can increase the strength of aluminum as silicon is known to form a separate hard phase in an aluminum matrix.

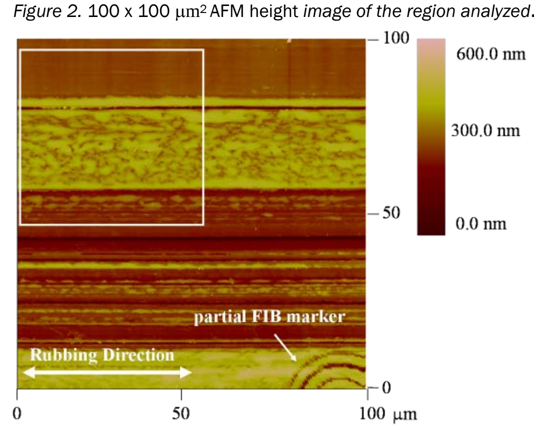
Figure 1. The structure of zinc dithiophosphates. The R group dictates the chain branching (ex. alkyl or aryl).



R = 1°,2°,3° alkyl or aryl group

Our research group (in collaboration with General Motors of Canada Ltd.) focuses on a group of molecules called zinc dialkyl-dithiophosphates (ZDDPs) (*see Figure 1*) which have been used as engine oil additives for over 50 years. They are important chemically-active additives, known for their anti-oxidant and antiwear characteristics. ZDDPs are known to form a protective film (tribofilm) at rubbed surfaces (*see Figure 2*), typically on the iron containing metals surfaces ubiquitous in the automotive industry. Neither the mechanism of this film formation nor the origins of its efficacy in preventing wear are well understood; this is due to the complex nature of the engine environment; The mechanical properties of these tribofilms formed from ZDDP and its breakdown products are believed to be the main determinants of the antiwear action of the films. We focus on how ZDDPs break down on

Figure 2. 100 x 100 μm^2 AFM height *image of the region analyzed*.



these aluminum alloys, which have already replaced steel as engine material in some automobiles, what chemical species have formed, and we determine and correlate the mechanical and chemical properties. However, ZDDPs and the products formed, have harmful environmental properties, and thus an initiative to replace ZDDPs is being vigorously encouraged by various levels of government.

The intent of my research is to elucidate the function and properties of ZDDPs at the

(Continued on page 3)

Growth & Characterization of Semiconductor Nanowire Devices

By Dr. Ray LaPierre
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Western's Nanofab has been collaborating with McMaster University's Centre for Emerging Device Technologies (CEDT) for the growth and characterization of semiconductor nanowire (NW) devices. The most dominant method for growing semiconductor NWs is the vapor-liquid-solid process whereby source material is transported to a substrate that is seeded with metal nanoparticles (usually Au). Preferential nucleation of the source material occurs at the Au-semiconductor interface, resulting in the growth of one-dimensional semiconductor NWs (*see Figure 1*). The electron beam lithography facilities at the Nanofab and the CEDT have been used to pattern Au into regular arrays of nanoparticles with diameters down to 100 nm (*see Figure 2*). The Au-templated samples are then used for the growth of NW arrays, using McMaster's molecular beam epitaxy facilities.

Figure 1. GaAs nanowires grown by molecular beam epitaxy.

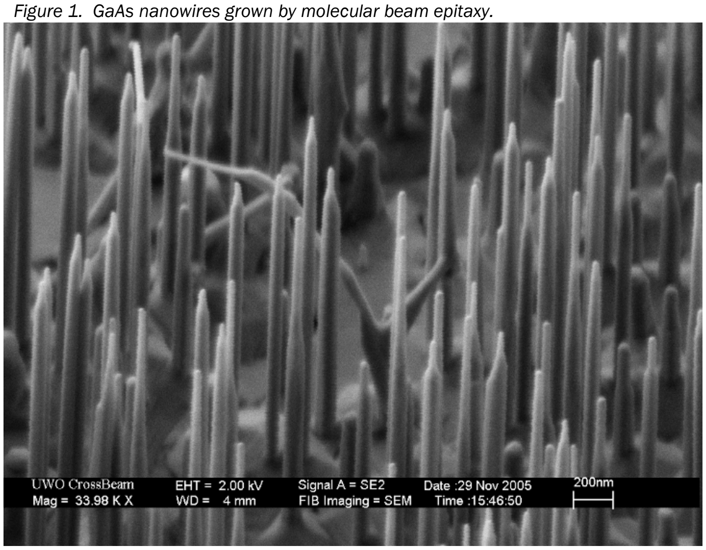
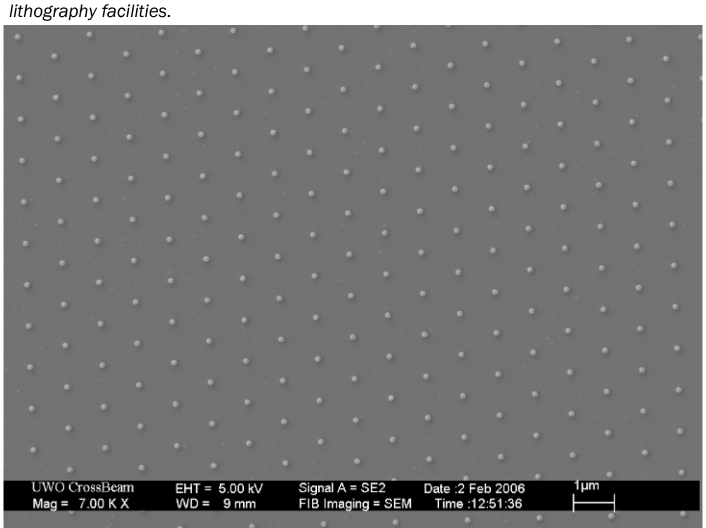
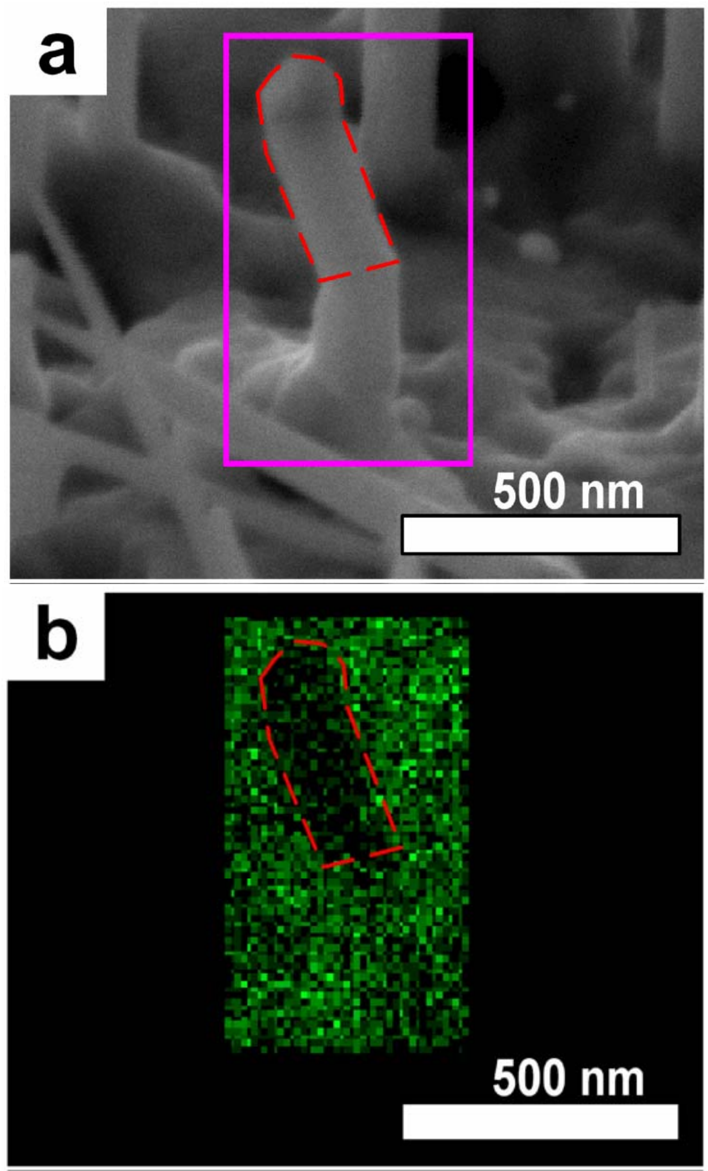


Figure 2. Au dots patterned by lift-off process using Western's e-beam lithography facilities.



After growth, the morphology of the resulting NWs has been examined using the Nanofab's LEO/Zeiss 1540XB scanning electron microscope (SEM). In situ cross-sectioning of the NWs in the SEM can be achieved by milling with a Ga focused ion beam (FIB). Energy dispersive x-ray spectrometry (EDS) with a probe size less than 3 nm can provide compositional analysis of the NWs (*see Figure 3*).

Figure 3. GaAsP/GaP heterostructure nanowires viewed by (a) SEM, and (b) energy dispersive spectrometry (EDS) showing As compositional map.



The FIB-SEM has also been used to fabricate "dimples" with diameters below 100 nm in GaAs (*see Figure 4*). We are developing a process to seed the holes with Au nanoparticles for NW growth as an alternative to the electron beam lithography process.

For the fabrication of nanoscale electrical contacts, the Nanofab's FIB-SEM is also proving to be extremely useful. Figure 5 shows a tungsten line formed by the Nanofab's FIB-SEM. The W line joins two interdigitated Au fingers created by photolithography at McMaster's cleanroom facilities. We hope to use this process to create contacts for single NW electronic devices such as biosensors and nanolasers.

These joint efforts between McMaster's CEDT and Western's Nanofab demonstrates the type of collaboration that is essential in achieving tomorrow's next generation of nanoscale components. We would like to thank the staff of the Western Nanofab, particularly Todd Simpson, for a very fruitful collaboration.

This work has been supported by the Ontario Photonics Consortium, OCE, and NSERC

Figure 4. FIB holes fabricated in GaAs substrate.

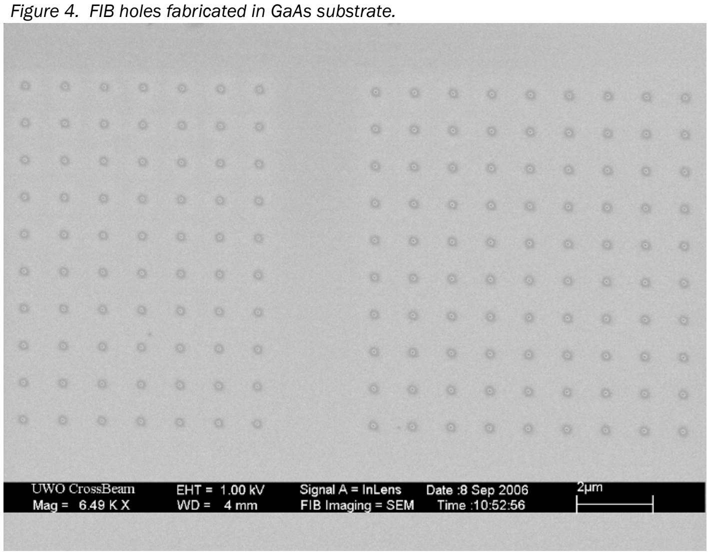
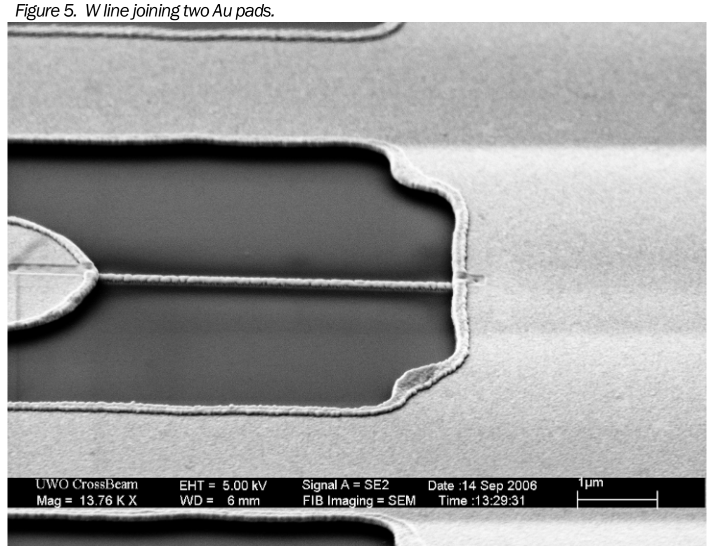


Figure 5. W line joining two Au pads.



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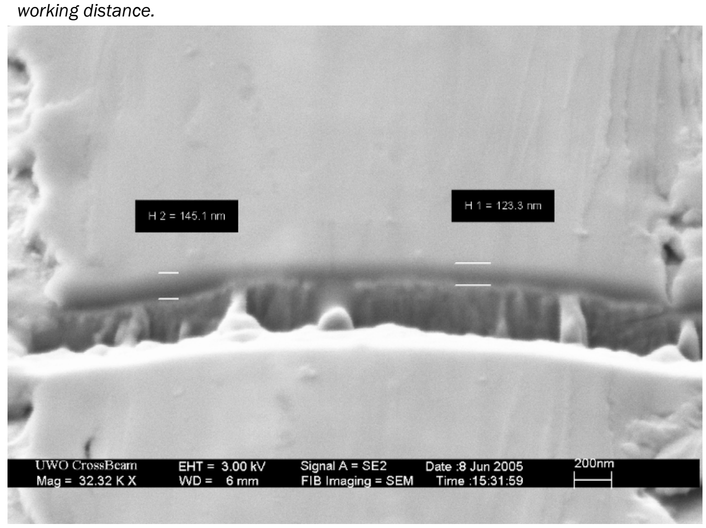
molecular level on aluminum-silicon alloys. Through a strong collaborative effort with many group members and industry, and using both theoretical and experimental approaches, we are studying these additives which are ubiquitous components of conventional engine oils that have been found to offer substantial protection to engine surfaces against wear, while at the same time, their use is accompanied by serious environmental drawbacks.

Antiwear films were prepared and the chemical nature of the film and substrates was analyzed using X-ray absorption near edge structure (XANES) spectroscopy, X-ray photoemission electron microscopy (X-PEEM), X-ray photoelectron spectroscopy (XPS), and energy dispersive X-ray analysis (EDX). Using fiducial markings (from the Nanofabrication Laboratory) on a sample (seen partially in Figure 2), the areas analyzed chemically on a micro-scale (X-PEEM) can be relocated and studied by atomic force microscopy (AFM), scanning electron microscopy (SEM), as well as permitting nanomechanical measurements to be performed on the same area. The boxed region in Figure 2 is the region analyzed by X-PEEM. The results can be found in the literature [1].

Methods developed in our group have permitted estimates to be made of the average P film thickness of the tribofilms by utilizing the high sampling depth of the K-edge energetic photons in the phosphorus edge region, in conjunction with Particle Induced X-ray Emission (PIXE). The thickness of the tribofilms from this method was found to be ~ 105 ± 5nm.

Accordingly, we have also made direct measurements of localized film thickness using focused ion beam (FIB) milling (also from the Nanofabrication Laboratory) selecting large pads to provide cross sectional cuts to be further studied with SEM. An example of a tilt corrected FIB milling cross section observed with the SEM, is shown in Figure 3. The thickness was shown to average about 180 nm with a variation of ~ 60 nm (averaged over 10 pads that were milled) [2].

Figure 3. Tilt-corrected SEM (electron yield mode) observation of the FIB cross section of the tribofilm formed on steel. SEM acquired at 3.0 kV, and 6mm working distance.



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[2] G. Pereira, D. Munoz-Paniagua, A. Lachenwitzer, M. Kasrai, P.R. Norton, T.W. Capehart, T.A. Perry, Y.-T. Cheng. Wear (In press) (2006)

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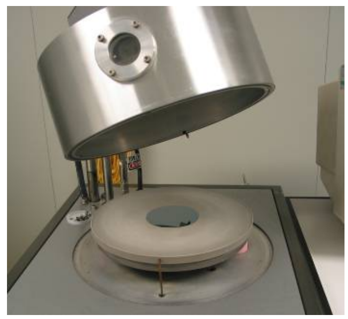
Nanofab Profile: STS310 PECVD

By Dr. Richard Glew
Nanofab Laboratory Manager
University of Western Ontario

The STS 310 is a Plasma Enhanced Chemical Vapour Deposition tool for depositing dielectric coatings of silicon dioxide (SiO_2) and silicon nitride (Si_xN_y).

In the PECVD process, gases are injected into a reaction chamber and are excited by a high electric field to produces a plasma. This results in a layer being deposited on the substrate. For silicon dioxide layer, silane (SiH_4) and nitrous oxide (N_2O) gases are used. For silicon nitride layers, silane and ammonia (NH_3) gases are used to create the plasma. The composition and quality of the layer is determined by the process gas flow, chamber pressure, substrate temperature and RF (radio frequency) power. The STS 310 is easy to use. All the processes are computer controlled and good run reproducibility has been demonstrated.



The STS 310 reaction chamber contains a 30cm diameter horizontal platen on which several wafers can be placed and heated to approximately 300ºC. The best results have been obtained with a single wafer placed at the centre of the platen. The refractive index and thickness of the deposited layer are routinely measured with the Gaertner L116C Ellipsometer and the thickness uniformity measured with the Nanospec 4150. To date, we have successfully deposited silicon dioxide and silicon nitride layers on silicon, glass and gallium arsenide substrates.

Thickness uniformity measurements of silicon dioxide (refractive index of 1.46) deposited on a 100mm silicon wafer, with an average thickness of 3585Å, had a sigma of 43Å (2.14%), measured with a 121 point map with 5mm edge exclusion. Thickness uniformity measurements on silicon nitride (refractive index of 2.02) deposited on a 100mm silicon wafer, with an average thickness of 1690Å, had a sigma of 33Å (3.7%), also measured with a 121 point map with a 5mm edge exclusion.

A useful feature of the STS 310 is that it is equipped with two RF power supplies, one for high frequency (13.56MHz) and for low frequency (187kHz) operation. Unlike silicon dioxide films which are always compressively strained, silicon nitride is compressively strained when deposited at high frequency and tensile strained at low frequency. So "net zero strain" silicon nitride can be deposited by cycling between high and low frequencies.

Testing Clean Room Equipment For Use In Tropical Rainforests

This past August the Nanofab faced its first severe environmental challenge: is our clean room equipment rainforest proven?

During the night of Wednesday August 2nd, thunderstorms rolled through southwestern Ontario. One of the many lightning strikes found its way to an electronic panel controlling the Nanofab climate in the Physics and Astronomy Building. Physical plant was monitoring the system remotely and no failure was observable. Everything seemed to be ok, including the climate control readouts for the Nanofab.

Unfortunately this was not true. Despite sending ok-data about temperature and humidity into the main university control system, the climate control had gone berserk. The humidifier had sent pure steam into the Nanofab and the adjoining corridors of the Physics and Astronomy building. Eventually, the solder links on all seven of the fire dampers in the ductwork melted, blocking air flow into the lab.

When Todd Simpson entered the building on early Thursday morning he was immersed in fog and was not able to see the far end the corridor in the basement of P&A. The room temperature in the Nanofab is normally kept at 21°C, so the water from the saturated air was condensing everywhere! Everywhere the air could reach: all the walls, in the electronic boards, inside all machinery, in the clean room filter system, on control panels, in our supply stocks, the gowning, computers, printer… Everything was soaking wet, like after a thunderstorm rain. The water was standing in puddles on the floor of the Nanofab collecting thousands of drops running and dropping downwards from everything. The only dry places were inside the chambers of the various vacuum systems.

What a shock! After an immediate call to physical plant, the air handler was stopped and the system repaired and re-set. The temperature was raised to 35°C and the humidity value set as low as possible to dry off the room and its equipment. The room was closed for access for nearly 3 weeks. After the drying period the instrumentation was tested piece by piece for damage and failure. Our robust instrumentation survived that rainforest test surprisingly well. The most delicate pieces of equipment, the two electron microscopes showed severe damage mainly in the electronic boards which haven been replaced in the past weeks from the Zeiss technician.

We are back to normal operation and have two additional monitoring systems installed; one of them located directly in the air in-stream system to the Nanofab. We hope that such a mishap does never occur again.