# NanoWestern



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Dear Nanofab Users,

This past July, I have accepted to renew my position as the Western Nanofabrication Director. The challenges associated with your projects, as well as our industrial client projects, gave me the desire to push further our capabilities and maintain at the best level our facility. Several improvements have been realized in the past three years including better and cheaper access to our imaging facility, new instrumentation (electrochemical deposition, mask aligner, profilometer), new grey preparation room with smaller instrumentation (spin coater, ozone cleaner, laminar flowbench...) as well as several fabrication projects

partially supported by the facility. Presently several projects are under review including a large CFI grant to acquire a cross beam cryogenic imaging station allowing nanotomography imaging and an RTI to acquire a reactive ion etcher. Together with Dr Todd Simpson and Tim Goldhawk, we intend to continue in this direction in the next years together with promoting our facility in the different faculties at Western, in other universities and to potential industries.

The single entrance point of our open-access nanofabrication facility makes it particularly efficient and reactive and we are always striving on working on new challenges. Please do not hesitate to contact us and have your HQP trained to benefit from this exceptional on-campus facility.

Sincerely yours,

François Lagugné-Labarthet



Western Nanofabrication Facility nanofab.uwo.ca





## **Critical-Point-Dry Assisted Photolithographic Fabrication of High Aspect-Ratio SU-8 Pillars**

**Shaolin Zhou PDF**, Prof. Jun Yang Faculty of Engineering Western University, London ON Canada

High-aspect-ratio (HAR) micro pillar fabrication is an absorbing research of interest due to the great potentials in varieties of applications, e.g. 2D photonic crystal, sensitive biosensor, superhydrophobic surfaces, bioinspired surfaces such as the fibrillar structures for biomimetic dry adhesive etc. Meanwhile, dense and HAR micro pillar arrays with feature size close to diffraction limit can be readily fabricated on SU-8 by UV photolithography. On one hand, SU-8 proves to be the favourite material with great versatility in micro/ nanofabrication due to its excellent chemical, mechanical and thermal stability as well as high optical sensitivity to facilitate efficient polymerization of HAR structures with steep sidewall. On the other hand, SU-8 based UV photolithography is a facile and efficient way for HAR structures patterning compared to other routes including X-ray, e-beam and laser direct writing lithography.

However, except for many factors that still need intensive optimization including non uniformity caused by spin-coating, low cross-link level by insufficient UV dose and post baking time, internal stress etc., structure collapse or bending during the process of rinsing and drying becomes the most challenging problem especially when structure (pillar) density increases and feature size shrinks. Attempts to overcome this problem include using rinsing liquid with low surface tension and wettability to decrease the capillary effect acting on sidewall of SU-8 pillars as well as the way of freeze However, liquid with low surface tension drying. still exert appreciable capillary force to cause pillars collapse and the freeze drying process is always extremely time-consuming. The critical point drying which was typically used for biological tissue dehydration in SEM specimen preparation is employed here to assist the photolithographic process to finally release uprightly standing dense SU-8 pillars with high aspect ratio of above 10:1.







*Figure 1* Optical microscopic images of *(a)* SU-8 pillars collapse after blow-drying by air gun. *(b)-(c)* SU-8 pillars bend and stick to each other after drying by natural evaporation







*Figure 2 (a) SEM results of high aspect ratio SU-8 pillars fabricated by the critical-point-dry assisted photolithographic process. (b) aspect ratio of 9:1 (c) aspect ratio of 11:1* 



A

Diol

Amino acid side chain with -CO<sub>2</sub>H or -NH<sub>2</sub>

#### Poly(ester amide) Nanoparticles with Covalently-Conjugated Drugs for Drug Delivery

Amira Moustafa, Prof. Elizabeth Gillies

Department of Chemical and Biochemical Engineering Western University, London ON Canada

Poly(ester amide)s (PEAs) (Fig a) are promising materials for a wide range of biomedical applications including tissue engineering and drug delivery, as they combine the degradability of esters with the favourable mechanical properties of polyamides. The choice of dicarboxylic acid, diol, and  $\alpha$ -amino acid can be used to tune the polymer properties and degradability. The incorporation of  $\alpha$ -amino acids with pendant functional groups such as carboxylic acids or amines can provide sites for further PEA functionalization to conjugate cell signalling molecules, drugs, or targeting moieties for biomedical applications. Here we describe the development of PEA nanoparticles with a covalently immobilized anti-cancer drug floxuridine. Covalent attachment of the drug should provide a slow and controlled release in comparison to physical encapsulation strategies, which typically result in a rapid, burst release of a large fraction of the drug.

Diacid

First, a functionalized PEA containing 10% of a L-aspartic acid-based monomer was successfully synthesized, and protecting groups were removed from the amino acid to provide pendant carboxylic acid groups. Floxuridine was then conjugated via an ester linkage to afford the PEA-drug conjugate. Next, the conditions were optimized to prepare nanometersized particles based on this drug conjugate. This was achieved using an emulsification-diffusion method with poly(vinyl alcohol) (PVA) as a surfactant. Freeze-dried particles were characterized by dynamic light scattering (DLS) and scanning electron microscopy (SEM). These results suggested that the particles had an average diameter of ~200 nm. The release of floxuridine from the nanoparticles was studied in vitro. It was found that ~6% of the drug was release rapidly during the first several hours, but the remainder of the drug exhibited a slow release, likely due to the requirement for ester hydrolysis to occur within the hydrophobic core of the particles. This nanoparticle-based prodrug approach may therefore reduce the toxicity of this chemotherapeutic and reduce the frequency required for its administration.



*Figure: A)* Chemical structure of PEA; *B)* SEM images of PEA nanoparticles; *C)* DLS trace for PEA nanoparticles; *D)* Release rate of the covalently immobilized anti-cancer drug floxuridine from PEA



### Fabrication and Characterization of Bored Nanopores in Graphene Films

Jaewoo Park, Prof. Gioviani Fanchini Physics & Astronomy Western University, London ON, Canada

Porous graphene is a two-dimensional material that is potentially interesting for a large variety of applications in water purification and microfiltration. Porous graphene filters can be used as a selective filter because it is not only the pore size, but also the chemical functional groups attached to the pore edges that control the water filtration characteristics. We developed a proprietary method for fabricating nano-sized holes in few-layer graphene. Differently from the most commonly used techniques for achieving pores in graphene, bored holes through an entire multilayer are obtained with our technique.



High filtration rates at relatively low differential pressure are a possible through our hollow graphene flakes, as opposed to impractically low filtration rates through "traditional porous graphene". By controlling the size of the pores, we are able to selectively filter water through graphene while retaining larger and undesired molecules of contaminants. Scanning Electron Microscopy (SEM) and Electron Dispersive X-ray (EDX) spectroscopy at the Western Nanofabrication Faculty were used to verify the opening of holes in graphene films which are seen as white hollow dots in the SEM images (see Figure below). EDX measurements are useful to determine the composition of the functional groups attached at the edge of the pores, as well as estimating the presence of contaminants from the pore fabrication methods. We are now in the process of developing a scalable method to prepare largediameter filters based on collections of graphene platelets in which the entire amount of filtrated liquid passes through the pores, with negligible contributions from leaks from the interconnection between the flakes. This step requires filling our graphene flakes with specific resins and will open up the possibility to commercially utilize our filters. This project will bring important benefit to solving a large number of environmental issues and to understanding the interaction between nano-sized molecules and graphene.



Figure (A) Sem inlens image of nano-pores (white and hollow dots) on graphene film on silicon substrate and (B) its magnified image. (C) EDX spectrum on the pores.

# **Western Nanofabrication Facility**

nanofab.uwo.ca

Western University Physics and Astronomy Building Room 14 London, Ontario N6A 3K7

Prof. François Lagugné-Labarthet Facility Director flagugne@uwo.ca Todd Simpson Ph.D. Senior Research Scientist tsimpson@uwo.ca Tim Goldhawk Laboratory Supervisor tim.goldhawk@uwo.ca

