

**CHARACTERIZATION OF SURFACES and INTERFACES.
An investigation of the dynamics of confined fluid-like films under
shear-forces at the nanometer scale**

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A. ABSTRACT

Full understanding of the physics underlying the striking changes—in viscoelasticity, relaxation time, and phase transitions—that mesoscopic fluid-like systems undergo either when under confinement or at a solid-liquid interface remains a major scientific challenge. This proposal aims to investigate, in particular, the dynamics of mesoscopic fluids confined between two sliding solid boundaries.

To study such complex surface phenomena, the PI will exploit the versatile capabilities of the Shear-force/Ultrasonic Near-field Microscope (SUNM), a novel technique able to concurrently and independently monitor the effects that fluid-mediated interactions exert on *both* the microscope's probe and the sample to be analyzed. This project will further investigate preliminary findings that correlate the film's viscoelastic properties, changes in the probe's resonance frequency, and the film's ability to generate sound. In addition to the requested support, this proposal will benefit from the availability of samples with tailored surface characteristics fabricated at the Argonne National Laboratory and at Portland State University (PSU), as well as from high-precision analytical equipment provided by the Oregon Nanoscience And Microtechnologies Institute's matching fund program.

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B. THE FOCUS OF THIS PROPOSAL

B.1 Scientific context of this proposal.

Understanding the new dynamic behavior that emerges when fluids are constrained to small regions presents a continuing scientific challenge.

The physical properties of fluids confined to nanometer-sized regions differ greatly from those displayed by bulk fluids. For example, the fluids undergo layering adjacent to the solid boundaries, the effective shear viscosity is enhanced, and viscoelastic relaxation times are prolonged.^{1,2}

Understanding the behavior of such mesoscopic fluid films is central to studies of tribology,³ adhesion, wetting, and the microfluidity of biological membranes. Understanding the dynamics of fluid-like systems at fluid-solid interfaces also has implications for Near-field Scanning Optical Microscopy (NSOM)⁴—one of the PI's research areas—where shear-force interactions^{5,6}—between the NSOM's probe, the substrate, and the adsorbed fluid layer in between—are exploited to implement a feedback mechanism intended to keep the probe-substrate distance constant during the probe's lateral scanning process. (The nature of the so-called “shear-forces” between the probe and the sample, as used in NSOM, remains controversial.⁷ Some groups argued that, during the lateral scanning process, the probe's tip interact solely with the adsorbed layer, while others—based on the observed increased in the resonance frequency experienced by the probe when the tip is brought close to the sample—argue that the probe's tip actually taps onto the solid substrate. The possibility that the adsorbed layer itself can produce not only a damping affect on the probe but also provoke an elastic response constitutes a more recent argument; see Aim-2 in Section B.3.2 below in this

proposal.) Finally, insights into the properties of mesoscopic films are also important for understanding flow behavior in granular,⁸ ceramic⁹ and composite materials,¹⁰ in which deformation occurs by means of the shearing of thin interfacial layers at the grain boundaries.

In spite of long standing interest, however, the dynamics, thermodynamics, and the molecular structure of **fluids confined to nanometer-sized regions**, is **not yet well understood at the fundamental level**. What determines the molecular organization of a fluid film along the surface? What are the dynamics of liquids in intimate contact with a solid boundary? Which aspects of the behavior of thin fluid-like films can be attributed to confinement alone? **These questions identify the most unresolved aspects in understanding the physics of liquids**. Another clear example of this challenging situation is observed in the field of tribology. Indeed, although materials scientists have been able to improve material performance substantially through the development of new alloys, composites and surface techniques,^{11,12} their discoveries have usually been serendipitous. They have been less successful at *a priori* design of materials with improved performance, largely because tribology is not understood at the molecular level.¹³ In an even broader scope, studies of sliding friction can also lead to an understanding of the dynamics of earthquakes.³

Altogether, surface phenomena involving fluid-like films constrained between solid surfaces present an exquisite source of fundamental challenging questions that require answers from a nanometer scale perspective.

B.2 Current experimental strategy in the study of mesoscopic films and its connection with tribology.

Investigation of single-asperity contacts to understand fundamental micromechanical and tribological properties of surfaces and interfaces.

The difficulty in studying confined mesoscopic fluid-like films is that (i) they are buried between two sliding bulk phases, and (ii) there is relatively little material available for analysis (see schematic in Fig. 1a). Such confinement occurs because the profile of a common solid surface is very irregular at micrometer and nanometer scales. Since the physical properties of confined fluids differ greatly from their bulk ones,¹⁴ they are

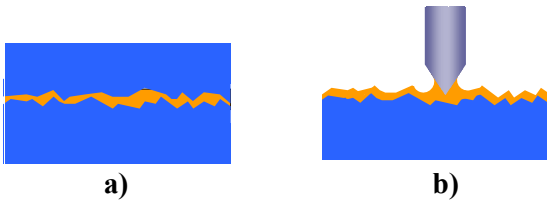


Fig.1 *a) Surfaces are uneven at the sub-micrometer level and, at ambient conditions, are typically covered by a layer of fluid-like moisture. Two surfaces in contact leave fluid-like films confined to nanometer-sized regions, whose enhanced physics properties appear to play a major role in sliding friction.¹⁵ b) A sharp probe, acting as a nanometer-sized asperity, allows the study of fundamental micromechanical and tribological properties of surfaces and interfaces at nanometer lateral resolution. This proposal adopts the strategy of using a single asperity to study surface phenomena.*

believed to play an important role in sliding friction.¹⁵ The many intervening interactions in such solid-liquid-solid systems (van der Waals, capillary, electrical, and viscous forces, to name a few) make their study challenging.

To simplify the problem, one approach consists of reducing the area of one of the surfaces in contact, thus resembling the interaction of one surface with another having just one or a few asperities, as schematically shown in Fig. 1b. (Other significant

approaches are: the surface force apparatus^{16,17} that measures both static¹⁸ and dynamic^{1,19} forces between atomically flat surfaces; the quartz microbalance technique, where an increased damping in the quartz resonator response is associated with the sliding of adsorbates relative to the quartz surface;²⁰ infrared spectroscopy, where friction determines the spectral line-width of vibrations;²¹ and computer simulations.²²) The importance of investigating **single asperity contacts** in studies of the fundamental micromechanical and tribological properties of surfaces and interfaces has long been recognized.^{2,23,24}

The scenario resulting from concentrating the interest on just a few asperities resembles the typical arrangement encountered in scanning probe microscopes, as seen, for example, in atomic force microscopy (AFM)²⁵ and near-field scanning optical microscopy (NSOM).⁴ As a matter of fact, AFM was the first technique to be used to study friction at the nanometer scale. In this technique, a sharp stylus is laterally dragged along the surface while the investigator monitors its lateral bending caused by the frictional force acting between the probe and the specimen. The smaller the bending experienced by the probe, the lower the frictional force.^{26,27}

B.3 Specific aims of this proposal.

B.3.1 AIM 1: To investigate the dynamics of mesoscopic fluid-like films under shear force using novel analytical instrumentation.

A limitation of the AFM technique is that it senses the effects that friction forces cause *only* on the probe, but information about effects on the substrate and its adsorbed layer is lost. **How is the energy transferred to the sample before being converted to**

heat? Is such transfer caused by electrical interactions²⁸ or by phonons²⁹? What is the role of (as well as the effects on) the adsorbed fluid-like layer¹⁵? Such issues are generally difficult to address experimentally, not only because of the delicate nature of the phenomena, but also because it is difficult to design experiments that admit unambiguous interpretation. Capturing simultaneously as much information as possible (i.e. multiple signals) resulting from the region of interaction would be highly desirable to improve our understanding of mesoscopic films, since many interaction forces may be acting at once.

Our recently introduced Shear force/Ultrasonic Near-field Microscope (SUNM)³⁰

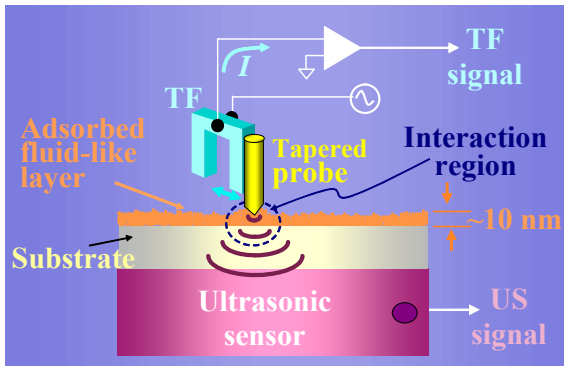


Fig.2 Schematic of the SUNM. The ultrasonic waves generated at the adsorbed mesoscopic film by the laterally oscillating tip are detected by a sensor located underneath the substrate (US signal). A piezoelectric tuning fork monitors simultaneously the effects on the probe (TF signal). The ability to simultaneously monitor the effects that frictional forces exert on the probe and on the adsorbed layer constitutes one of the attractive features of the SUNM.

helps to address these demands. As shown in Fig. 2, the SUNM integrates two sensory modules: a piezoelectric tuning fork (TF, to which the microscope's probe is attached) and an ultrasonic transducer (in intimate contact with the sample). As the probe's tip laterally oscillates and while immersed in the adsorbed layer, both the ultrasonic waves generated at the fluid-like layer and the damping effect on the probe's motion are simultaneously detected by the SUNM's sensors as a function of the

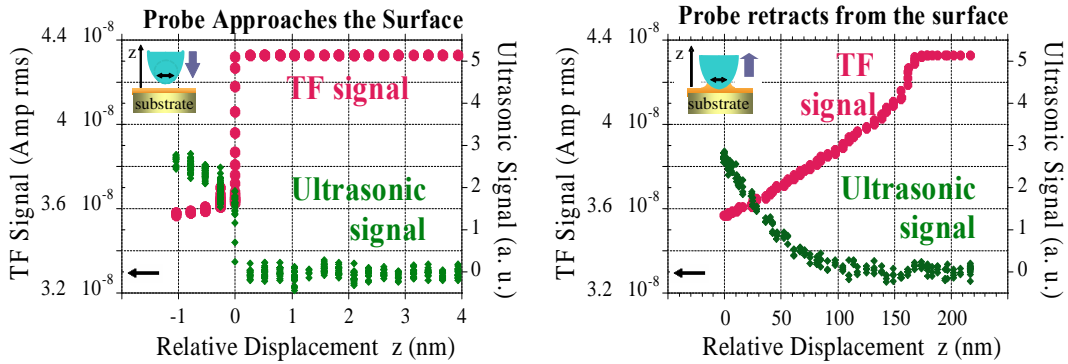


Fig. 3 *The sound of shear forces. Simultaneous detection of the effects that sliding interactions have **on the probe** (its lateral oscillation amplitude is monitored by the TF electrical signal) and **on the adsorbed fluid-like layer** (where acoustic waves are generated and subsequently detected by the ultrasonic transducer located underneath the substrate). The probe's max amplitude of oscillation is 4nm and the equivalent external lateral force driving the probe is 60 nN.³⁰ Notice the different horizontal scale in each graph. The $z=0$ position is defined (arbitrarily) at the point where the tip encounters the fluid-layer boundary during the approach.*

probe-tip distance (Fig.3).

The direct detection of the “**sound of the shear forces**” (i.e. the detailed monitoring of surface-interactions mediated by mesoscopic fluids between two sliding solid boundaries) *via* ultrasonic means by the SUNM is unprecedented.^{30,31} (In effect, the SUNM differs from other ultrasonic techniques that are based on direct solid-solid contact between the probe and the substrate,³² or from others where the probe dithers perpendicular to the sample's surface³³ or is kept stationary while an ultrasonic wave is applied to the sample.³⁴) **The richer and potentially correlated information obtained from the two interacting bodies (the probe and the sample's adsorbed film) constitutes a unique advantage of the SUNM** over other metrology instruments that monitor the response from the probe only.

The tines of the TF used in the SUNM have an effective spring constant of $\sim 25,000$ N/m, however, due to its low energy dissipation (with mechanical quality factor Q ranging from 10^4 to 10^5 after mounting the tip) makes them very sensitive to sub-pN

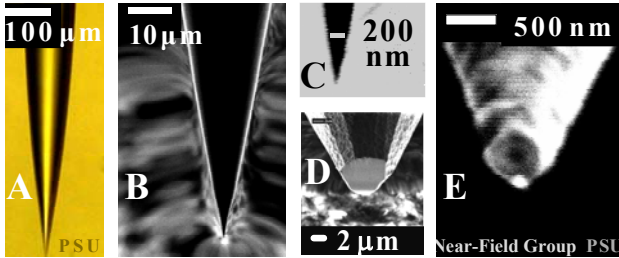


Fig.4 Probes fabricated at the PI's laboratory. **A,B,C:** Uncoated glass fibers **D:** tapered probe with a truncated flat apex fabricated with a focused ion beam.³⁶ **E:** Aluminum coated fiber with an aperture and flat apex³⁷ (convenient for additional optical and electrical characterization of materials).

shear forces.^{7,35} Another advantage in the SUNM setup is **the stiffness of the probe's position along the vertical axis** (simply because the probe is attached to a TF that is held by the microscope frame stage). Hence, the probe is not subjected to uncontrollable sudden changes in

the tip's vertical position towards the sample (as occurs in AFM) when the tip is gradually brought close to the substrate. By being able to hold the tip still at any desired probe-tip distance, the dynamics time response of the adsorbed layer can be monitored better in the SUNM. In this PRF proposal, the PI will implement a series of further systematic tests using a variety of probes (*i.e.* metallic and glass probes of tailored morphologies, and of different degrees of roughness; see Fig.4,) and samples of tailored characteristics (*i.e.* hydro-phobic, hydrophilic; see also Section B.3.3 below.) These additional tests are necessary to investigate also the role played by the nature of the solid boundaries in the dynamics of the confined mesoscopic fluid. The results will be contrasted with existent theoretical models of interfacial shear force microscopy,⁷ including the "shaking potential" model³¹ introduced by the PI's research group.

B.3.2 AIM 2: To study the correlation between variations in the probe’s shifted resonance frequency and the emission of sound.

A shift in the probe’s resonance frequency that depends on the probe-substrate distance is also observed through the response of both the TF and the US sensors (see Fig. 5). An important observation from these measurements is that the ultrasonic signal (see the purple “filled squares” curve in Fig. 5b for example) is clearly distinguishable from the background ultrasonic signal level (red “open triangles” curve in the same figure) only when a distinguishable change in the resonance frequency is produced (approx 5 Hz, as indicated by the corresponding purple “filled square” symbol in the inset of Fig. 5a). That is, the greater the resonance frequency shift, the greater the ultrasonic signal.

It is worthwhile to further pursue a systematic study (with different samples and probes) of the correlation between the **frequency shift** experienced by the probe **and the**

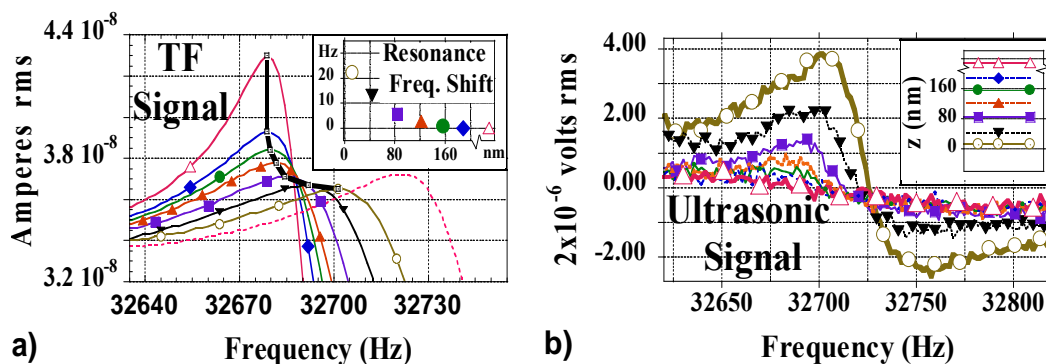


Fig. 5. Tuning fork and ultrasonic spectra taken at different probe-substrate distances as the probe retracts. For clarity, in a) the thick black line joins the peaks of each frequency trace; the inset shows the shift in the resonance frequency as a function of the relative probe-substrate distance. The $z=0$ reference is chosen (arbitrarily) at the point where the thick line reaches a minimum, which applies also to the inset scale in b). The dashed pink trace in a) corresponds to $z < 0$.³⁰

simultaneous increase in the sound signal, since it offers an opportunity to tackle the not well understood working principles of the so-called “shear force” mechanism (referred in Section B1 above.) widely used in high resolution optical imaging by NSOM.^{4,7} From the SUNM’s results,³⁰ it is very appealing to attribute the increased value of the probe’s resonance frequency to the relatively slow response of the viscoelastic film (i.e. as the probe moves to the right the fluid on the left does not fill the gap fast enough, thus creating a cavity and, hence, gives rise to an additional restoring force and, hence, an increase in the resonance frequency. This hypothesis has also been suggested in Ref. 32 although in a different experimental setup where much higher frequencies—MHz as opposed to KHz in the PI’s experiment—are involved. Alternatively, recent theoretical work suggests that cavitation formation is also possible even at moderate shear-rates, where the density dependence of the viscosity (and no viscoelasticity) constitutes the basic working mechanism.³⁸ The SUNM’s preliminary results³⁰ appears to align better with this latter hypothesis. This proposal aims to provide evidence that the nanometer-thick adsorbed fluid-like layer itself can produce not only a damping affect on the SUNM’probe but also provoke an elastic response.

Only additional SUNM experiments (to be performed in this proposal) with different samples, different types of adsorbed layers, and performing tests at different ranges of ultrasonic frequencies will put the above mentioned hypothesis on more solid ground. To that effect, samples with tailored surface characteristics fabricated at the Argonne National Lab will be used in this project³⁹ Also, a XYZ scanner (to be provided by the Oregon Nanoscience And Microtechnologies Institute matching fund program) equipped with capacitance sensors to overcome the scanner’s piezoelectric hysteresis

effects when controlling the probe's vertical position, will allow us to correlate the results obtained from the approaching and retraction process (see also Fig.3). Overcoming the hysteresis effect of the piezoelectric nanopositioning system will allow the PI to study better the separate but important inherent hysteresis effect presented by the adhesive fluid-film properties. It is believed that friction correlates not with the adhesive bonding itself, but rather with the adhesive irreversibility, that is, how differently surfaces behave when they stick together as compared with when they become separated.⁴⁰

B.3.3 AIM 3: To study the dynamics of films adsorbed on hydrophilic and hydrophobic polymer samples. Implications for natural (biological) water-based lubricants.

As current research provides evidence that the origins of nanoconfinement effects are related to the surrounding surfaces and interfaces that modify the relevant fluid dynamics,⁴¹ the PI will perform additional experiments on systematically selected intervening substrates in order to provide additional compelling supportive evidence for such origins. **Hydrophilic and hydrophobic polymer surfaces** constitute an attractive choice for study because of their wide potential technological applications and because they provide an interesting source of scientifically challenging problems related to nanotribology.

How do hydrophobic/hydrophilic surfaces affect the dynamics of mesoscopic fluids? This is one of the central questions motivating this proposals' aim. Natural (biological) lubricants are water-based, and cellular membranes control cellular functions by intricately managing hydrophobic and hydrophilic interactions. But exactly how does

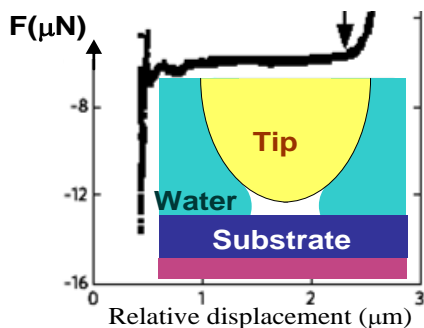


Fig. 6 Cavitation effects occurring upon hydrophobic interaction. The trace shows the distance dependence of the tip-substrate attraction. **Inset:** schematic representation of the sudden cavitation formation (at the separation-distance indicated by the arrow). Adapted from Ref 43. The added purple-colored block underneath the substrate is to suggest (in this proposal) that these phenomena also be monitored with an ultrasonic sensor.

this work? In addition to the currently accepted view that hydrophilic surface charges provide an electrostatic ‘double-layer’ repulsion,⁴² very recently it has been reported that micrometer-sized cavitation occurs suddenly as two hydrophobic surfaces approach (Fig.6), shedding light on the long-range hydrophobic interactions operating through water.⁴³ While this latter experiment was done with a micrometer-sized probe, we will perform similar experiments with nanoprobe in the SUNM to explore scenarios more relevant to the biological arena.

This PRF proposal will benefit from the support of Dr. M. Yan (Associate Professor, Chemistry Department, PSU) who will prepare polymeric samples having tailored hydrophilic and hydrophobic properties. This in-depth study of hydrophobic and hydrophilic interactions may lead to better understanding the principles underlying cellular bio-molecular dynamics.

C. BROADER IMPACT

To promote teaching, training, and learning, while enhancing student education in advanced technologies complemented with community service activities.

The PI, a recipient of the **PSU 2005-Civic Engagement Award “Excellence in Faculty/Community Partnership,”** and the **PSU 2006 John Eliot Allen “Outstanding Teaching Award”** will implement specific activities in three important areas:

a) Promote PSU student participation in educational community service activities.

The PI will exploit his experience in building bridges between PSU and local schools. Indeed, working along PSU’s institutional goal of becoming an Urban University⁴⁴ and working in coordination with the Center for Academic Excellence at PSU,⁴⁵ Dr. La Rosa and his enthusiastic students have made annual presentations on high technology topics at local schools during the last five

years: Skyview (Vancouver, WA), Wilsonville, Molalla;⁴⁶ and Sabin Elementary (a school that groups profoundly gifted kids) in Oregon. The PI will continue making these presentations, which serve the purpose of developing a spirit of community service in PSU students and, at the same time, help to attract the best high-school students into nanotechnology programs at PSU.



Fig. 6 Snapshots of the 2005 Civic Engagement Award ceremony, attended by the Portland, OR, Mayor Tom Potter (top right) and the PSU Provost Roy Koch (top left). The PI and Ms. Briley (Science Teacher at Molalla High School), represented in the ceremony by her student Jesús Ramírez (bottom left in the picture), received the award.



Fig.7 Promoting an interest for science in high school students. A talented and ethnically diverse group of PSU students, accompanied by the PI (far left), presenting their projects on Optical Telecommunication at the "Science Day", an event attended by students from local high schools. Oaks Park, Portland, Oregon (May 2000).

b) In the technical aspects: First, both the **graduate and the undergraduate students** participating in this project will have supervised direct **hands-on experience** working with state-of-the-art metrology instrumentation alongside the PI, who has a history of building sophisticated equipment from simpler components. Second, the required background knowledge that this project demands in the areas of **Acoustics, Optics and Electronics**

(the backbone disciplines involved in the instrumentation used in the PI's laboratory) will greatly enhance the horizons of PSU students' technical education.. Third: The PI will install a **student electronics shop** that will be important when training students to build their own electronic components. Keith Parker, the undergraduate PSU student participating in this proposal, has been chosen for his outstanding aptitude in electronics and superb abilities in building computer-hardware interfaces. The PI's research group will greatly benefit from Keith's participation.

Expand the instructional and educational services provided by the PSU Nano Science and Technology Group (NSTG), an initiative launched by 18 PSU researchers from four academic departments.

The PI is one of the main organizers of the multidisciplinary Nano Science and Technology Group (NSTG) www.nstg.groups.pdx.edu (currently composed of 18 PSU researchers from five academic departments; Chemistry, Geology, Electrical Engineering, Mechanical Engineering and Physics) that has launched an initiative to create the PSU Nanotechnology Center (NTC).⁴⁷ In addition to building interfaces between the traditional disciplines of science and engineering, the NSTG group aims to provide a forum for fostering partnerships with National Laboratories³⁹ and the industrial community in the Portland metropolitan area. The atmosphere of collaboration created under the umbrella of the NSTG benefit this proposal since samples with tailored characteristics (polymer films,⁴⁸ silicon nanowires,⁴⁹ and quantum dots⁵⁰) will be prepared by researchers of the NSTG group.⁵⁰ **The NSTG benefits PSU students** by providing broad exposure to modern problems and issues in nanotechnology, and by training future researchers in the methods, instrumentation, and theory underlying the broad field of nanotechnology. In fact, in the Summer of 2006 the NSTG offered the multidisciplinary hand-on course “Fabrication and Characterization of Nanomaterial,” which provided training in a variety of disciplines; the first of its kind to be offered at the PSU campus. The PI was one of the main organizers of this course.

<http://www.nstg.groups.pdx.edu/Summer->

[2006 Fabrication and Characterization of Nanomaterials/Fabric Charc Nanomaterials.html](http://www.nstg.groups.pdx.edu/Summer-2006-Fabrication-and-Characterization-of-Nanomaterials/Fabric-Charc-Nanomaterials.html)

D. TIMETABLE OF PROJECT DEVELOPMENT AND EVALUATION

Year	1	2	3
O B J E	PI trains undergraduate student in Atomic Force Microscopy . <i>Project: AFM images of the samples prepared at the Argonne National Laboratory.</i>	Graduate and undergraduate students works on: Project-1: Correlation between shear forces and the generation of ultrasound. Project 2: Correlation between variations in the probe's shifted resonance frequency and the emission of sound. Project-3: To study the dynamics of films adsorbed on hydrophilic and hydrophobic polymer samples. Implications for natural (biological) water-based lubricants.	
	Undergraduate student integrates and test the new XYZ scanner (obtained from ONAMI's matching fund) into the Shear-force/Ultrasonic Microscope.		
C T I	Graduate student supported by the PSU-Physics Department (whose support is not included in this PRF budget proposal) completes his Master degree	Improve understanding of the working principle governing the so called " shear-force mechanism " (as used in NSOM).	Improve understanding on why the dynamics of confined water, oil, and polymer films differ from their bulk properties.
		Graduate student works toward his/her graduation.	
V	Undergraduate student works toward his/her graduation.		
E S	Undergraduate student in charge of implementing the Student Electronic Shop at the PI's laboratory.	PI trains undergraduate student in Near-Field Scanning Optical Microscopy and Shear-force Ultrasonic Near-field Microscopy	
	To train graduate, undergraduate and high school students in NSOM probe fabrication.		
	Summer internship awards offered to high school students each year.		
	Annual presentations on nanotechnologies at local elementary and high schools.		
	To develop partnership with researchers within the PSU Nano Science and Technology Group		
E V A	Graduate and undergraduate students publish their findings in a peer-reviewed journal	Graduate and undergraduate students publish their findings in a peer-reviewed journal.	Graduate student finishes his Master degree and advances towards his/her Ph. D.
			Undergraduate student Keith Parker graduates and advances to graduate school..

L U A	<p style="text-align: center;">Ultrasonic image of Crosslink Spin on Glass</p> <p>Sample to be prepared at The Argonne Center for Nanoscale Materials (CNM). HSQ (material that contains silicon) cross-links upon the incidence of an electron beam and becomes SiO₂. The attractive feature of this sample lies in its varying material composition without large topographic features.</p>
T I	<p>Near-field ultrasonic and optical characterization of polymer samples (collaboration with Dr. Yan, PSU-Chemistry), self-assembled InSb islands on GaSb and embedded InSb/GaSb (collaboration with Dr. Moeck (PSU-Physics)).</p>
O N	<p>Summer Internship award for a high school students in the PI's laboratory is institutionalized by PSU. The PI coordinated the award with the PSU Center for Academic Excellence.</p>
	<p>Three manuscripts published in refereed scientific journals.</p>

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⁴⁸ Dr. Mingdi Yan; Associate Professor, PSU-Chemistry Department. Her research area includes synthesis and microfabrication of organic and polymeric materials capable of molecular recognition <http://www.chem.pdx.edu/%7Eyanm/>

⁴⁹ Dr. Raj Solanki (PSU, Physics Department) will provide Si and Si/Ge. nanowires grown *via* atomic layer deposition. Dr. Solanki's group is involved in nanowire growth and characterization for the possible next generation of nanoscale optoelectronic and microelectronic devices.

⁵⁰ Dr. Peter Moeck (PSU, Physics Department) also supports this project by providing self-assembled InSb islands on GaSb and embedded InSb/GaSb nanoparticles. Dr. Moeck's research includes nano and micrometer scale materials science and engineering, self assembled semiconductor quantum dots and size effects on semiconductor and metal phase diagrams. pmoeck@pdx.edu <http://www.physics.pdx.edu>