Final report on the ESF - Exchange Grant

"Towards surface force apparatus experiments with nanostructured mica surfaces"

by Renato Buzio

ESF Activity: nanotribology (NATRIBO)
Reference Number: 455
Title: "Towards surface force apparatus experiments with nanostructured mica surfaces"
by Renato Buzio and Carlos Drummond
Host institution: Centre de Research Paul Pascal CRPP - CNRS, Pessac Cedex, Bordeaux (France), Dr. Carlos Drummond

Purpose and duration of the visit

The purpose of Dr. Renato Buzio visit at Centre de Research Paul Pascal (CRPP) has been *to perform for the first time Surface Force Apparatus (SFA) experiments involving controlled nanoscale roughness on mica surfaces*, under the supervision of Dr. Carlos Drummond and coworkers. The nanostructured mica samples have been previously prepared at the Physics Department in Genoa (Italy) by Dr. Buzio and Dr. Drummond, using ions sputtering (for details see Dr. Drummond report to ESF).

The duration of Dr. Buzio visit at CRPP has been of 4 weeks, from Monday**,** February 21, 2005 (arrival in Bordeaux) to Saturday, March 19, 2005 (departure from Bordeaux).

Description of the activity

The work carried out by Dr. Buzio during his visit is reported in detail in the following.

1st week

During the first week of visit at the CRPP centre, Dr. Buzio has been introduced to the major technical features and measurements protocols of the SFA.

The device used at CRPP consists of a homemade instrument capable to perform force spectroscopy, sliding friction and rheological tests on nanometer thick lubricant films. The instrumental design resembles the SFA–3 model, described for example by Golan et al. (Y. Golan, C. Drummond, J. Israelachvili, R. Tenne, Wear 245, 190 (2000)). A scheme is reported below for clarity (Fig.1).

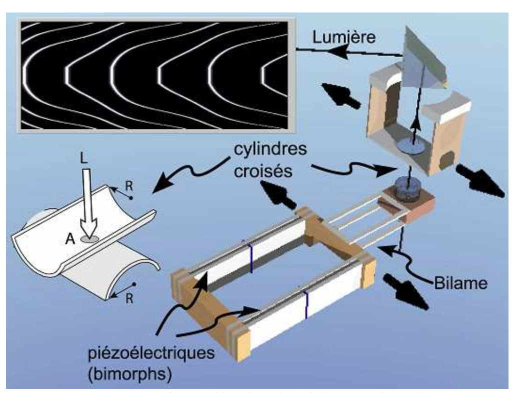


Figure 1. Design of the SFA instrument used at CRPP for tribological investigations.

Two mica sheets, usually of equal thickness, are back-coated with semi-reflective silver layers and glued onto curved, polished silica discs that are mounted in a cross-cylinder geometry. The two silver layers form an optical cavity in which constructive interference of white light generates a series of Fringes of Equal Chromatic Order (FECO). The emerging light is collected with a microscope objective and guided into a spectrometer where the beam is dispersed; the resulting

FECO are recorded with a video camera. The upper and lower surfaces are mounted on a friction-sensing device equipped with piezoelectric strain gauges and a piezoelectric bimorph slider. On applying normal load and relative shear motion to confining surfaces, their separation as well as contact area conditions can be monitored by studying FECO (see J.N. Israelachvili, J. Colloids. Interf. Sci. 44, 259 (1973)).
A short training period of three days has been spent to follow the basic steps of an SFA experiment: smooth mica surfaces preparation (gluing mica on silica discs, cleaning procedures to avoid particles contamination), acquisition of quasi-static force-distance curves for hexadecane confined between smooth mica surfaces, acquisition of friction data under different conditions of load and sliding velocity, observations of FECO to establish surfaces relative distance, normal applied load, presence of contaminants at the interface and appearance of wear effects.
Apart from the planned choice of using hexadecane as lubricant to test nanostructured samples, few nanostructured (ns-) mica samples has been also studied by Atomic Force Microscopy (AFM) in deionised water, in order to investigate surface stability in liquid environment as well as the efficiency of surfactant adsorption on it. Preliminary tests have proved stability of surface ripples in water for hours and efficient adsorption of surfactant molecules (tests performed with Didecyl Dimethyl Ammonium Bromide DDAB molecules).

2nd week

In the course of the 2nd week, Dr. Buzio has performed data analysis of AFM topographies acquired on ns-mica samples during their preparation in Genoa. Data analysis has been done to classify the morphological properties of nanostructured samples and define a set of candidate surfaces for SFA experiments.
In Fig.2 a graph is reported summarizing the morphological properties of available samples: for each sample (marked by a circle) the ripples wavelength is plotted against surface roughness.

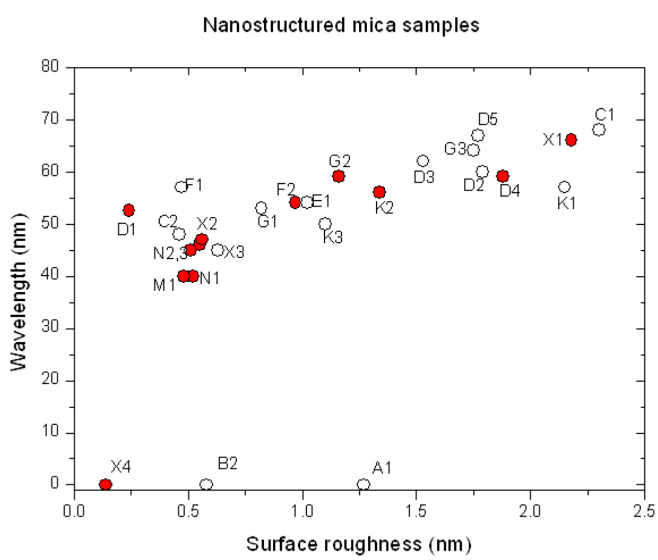


Figure 2. Graph showing the morphological properties of mica samples prepared by ions sputtering.

Close to any circle a letter and a number are reported, representing respectively mica thickness and sample number (that is, sample K2 is the second sample prepared in Genoa with thickness K, to be measured later by FECO). Samples X4, B2 and A1 have been prepared under normal-incidence ions sputtering to produce isotropic roughness instead of surface ripples.

Despite the preparation of 28 ns-mica samples in Genoa, at CRPP only 12 of them have been defined suitable for SFA experiments (marked by a red circle), displaying a good compromise of size, adhesion to mica backsheet and easy handling (related to their reduced or excessive thickness).
Based on the previous analysis, Dr. Buzio and Dr. Drummond have started the first tribological experiment on the sample named N2. The sample has been chosen due to reduced values of roughness (0.5nm) and ripples wavelength (45nm). Sample N2 has been prepared for SFA measurements in hexadecane, mounted to face an atomically smooth mica surface. The ripples wave vector has been arbitrarily fixed perpendicular to the sliding direction, in order to impose shearing along ripples channel.
The main purpose of the experiment has been to study hexadecane response under confinement and understand the role of surface roughness on lubricant dynamics.
Quasi-static force-distance curves have been acquired to define the nature of interacting forces between smooth and nanostructured mica. Two curves measured in 3 hours at the same location have however revealed *an unexpected evolution of surface contact during successive approaches*. In Fig.3 the normal force against surfaces separation is reported. It clearly appears that the curve of the second approach differs from the first one; the latter shows a step, at about 30nm of relative distance, which has been attributed to local surface failure, as confirmed also by FECO. Problems of contact stability and measurements reproducibility have been observed on different locations during shear measurements at the lightest available loads, indicating an intrinsic fragility of contact interface and the appearance of unavoidable wear effects.

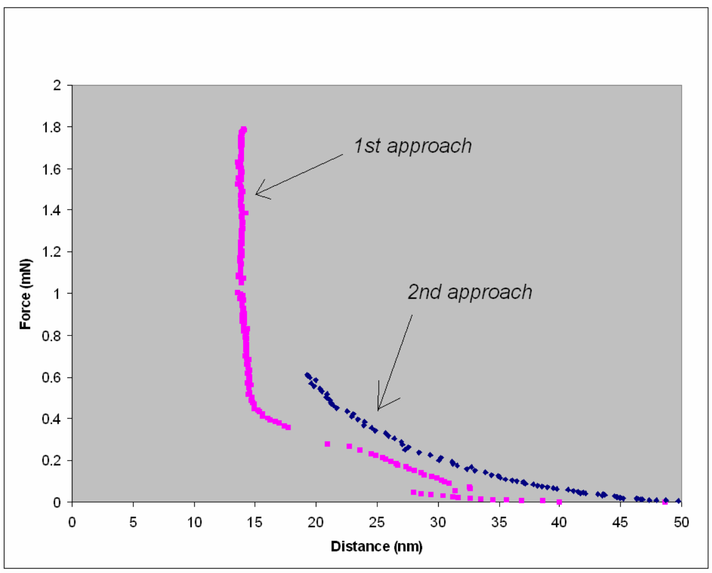


Figure 3. Quasi-static force-distance curves acquired successively on the same location by pressing smooth mica against nanostructured mica. No solvation forces are observed but a single step is present in the first curve, at about 30nm of relative distance, attributed to morphological surface failure.

3rd week

In the course of the 3rd week Dr. Buzio devoted few days to the morphological inspection of sample N2 by optical microscopy and AFM. The surface has been cleaned from hexadecane by ethanol and exposed to air in the course of morphological analysis; particular attention has been paid to reveal surface damage (compatible with the performed SFA experiment) and the supposed wear effects.
In Fig. 4(a) a typical AFM topography is reported showing the sample N2. We can clearly recognize the presence of surface ripples forming a uniform background. A straightforward comparison of such image with a similar one acquired in Genoa on the same sample (Fig. 4(b)) definitely confirms that surface morphology has not significantly changed during the successive

stages of surfaces preparation and SFA experiment, revealing an overall good stability to mechanical stress and temperature heating (at around 150°C during mica gluing to silica discs). Unfortunately no evidences of ripples wear have emerged by AFM imaging.

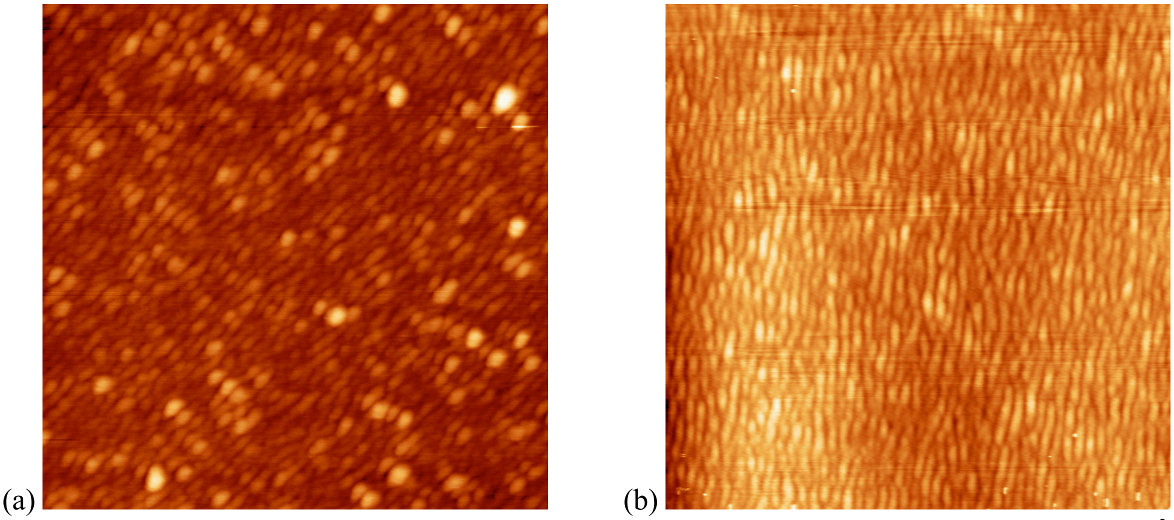


Figure 4. (a) AFM topography acquired at CRPP on sample N2 after the first SFA experiment: scan size 2×2μm^2. (b) AFM topography acquired at Phys. Dept. in Genoa on sample N2 after ions sputtering: scan size 2×2μm^2. The comparison reveals stability of nanostructured mica surface for standard SFA testing.

A second SFA experiment has been started, involving sample D4 facing a smooth mica surface. Quasi-static force-distance curves and squeezing experiments have been performed on the same location and they have confirmed contact instability and poor reproducibility of data at fixed experimental conditions. Sliding friction experiments have been performed on different locations at loads higher than those applied during the first experiment. Interestingly it has been possible to record stable friction data for hours, at fixed surfaces separation, and with reduced amount of wear. Friction data have been recorded sliding parallel and perpendicular to ripples channels, indicating a slight difference in friction coefficients according to the chosen geometry (Fig. 5).

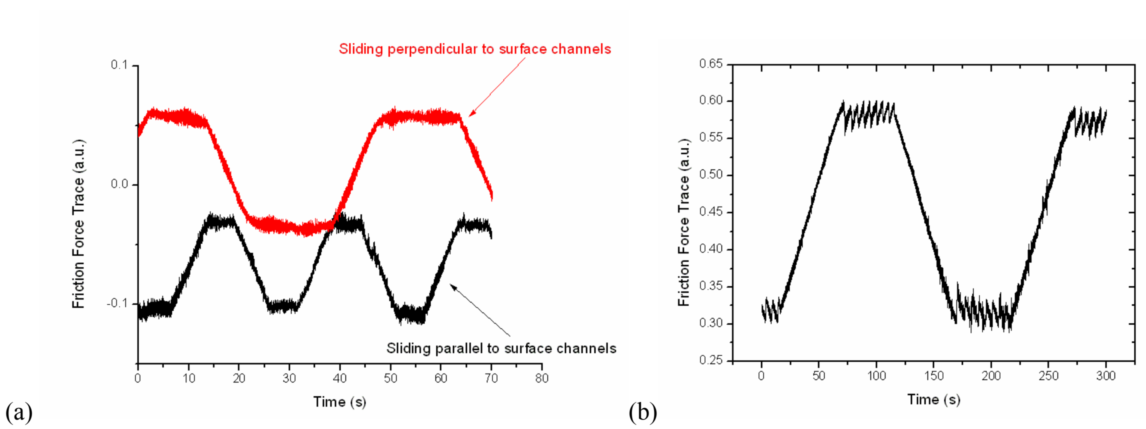


Figure 5. Typical friction traces recorded by SFA on sample D4 under low-wear conditions: the curves show the friction sensor output due to excitation of contact junction by a triangular wave applied to the bimorphs (not shown): static friction (ramps) and kinetic friction (plateau) appear during relative sliding of mica surfaces. (a) Friction traces obtained running parallel and perpendicular to channels directions under the same compressive load. (b) A typical friction trace showing the appearance of stick-slip motion.

4th week

In the course of the 4th week, AFM analysis has been first performed to show the presence of ripples on sample D4. In Fig. 6 typical AFM images are reported confirming expectations; nanometric particles are also visible, being attributed to the cleaning procedure of mica sample by ethanol and exposure to air, after the conclusion of the SFA experiment.

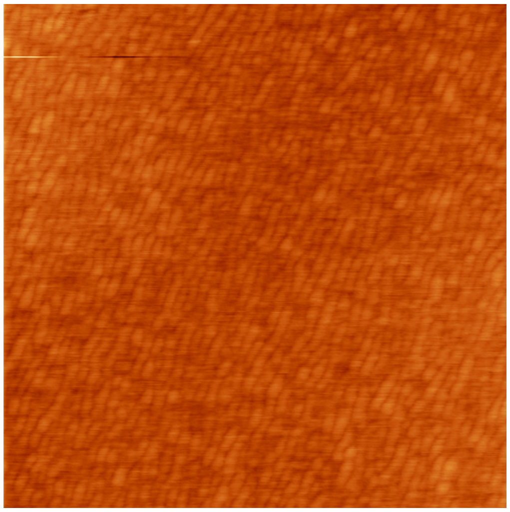
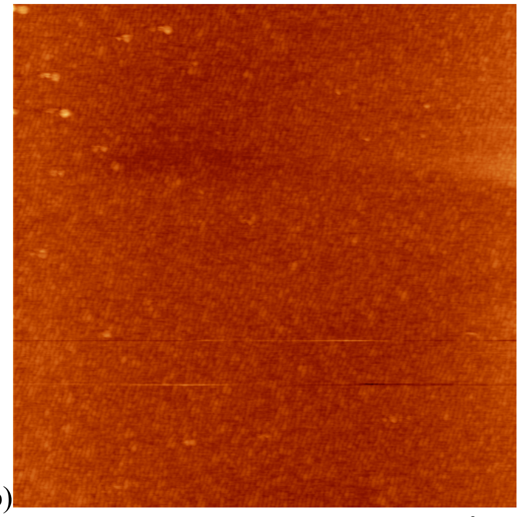
(a)  (b) 

Figure 6. AFM topographies acquired on sample D4 after the SFA experiment: (a) scan area of $2\times2\mu m^2$ and (b) $5\times5\mu m^2$.

A third SFA experiment has been started, involving sample X4 facing a smooth mica surface. It has been chosen due to the fact that it presents the smallest available roughness, considered the main cause of wear effects. Quasi-static force-distance curves have shown again poor reproducibility, comparable to evidences reported in the previous experimental attempts on ns-mica surfaces N2 and D4. However, a significant adhesion force has been recorded for the first time between ns-mica and smooth mica, as expected due to the reduced roughness on the nanostructured sample. The overall stability of friction signal during shearing at different loads and the small amounts of wear have indeed indicated sample X4 to provide the most robust nanostructured interface.

Description of the main results

The main results obtained within the project are reported below:

1. *Definition of a novel protocol, based on ions sputtering, for the introduction of controlled surface roughness on mica samples for SFA experiments*.
   The nanostructured surfaces are comparable to smooth crystalline mica for overall structural stability during the stages of samples preparation; they are also stable in deionised water and can be routinely used for standard SFA investigations on simple liquids and surfactants.

2. *Preliminary investigation of the system nanostructured mica/smooth mica in hexadecane environment by means of quasi-static force-distance curves and sliding friction curves*.
   The obtained results suggest that the introduction of nanoscale roughness in the SFA contact considerably increases local pressures, presumably causing squeeze-out of the lubricant and wear of contacting surfaces. This picture derives by investigating ripples of 40-60nm wavelength and surface roughness in the range 0.5nm-2nm and it actually reflects the typical behaviour of lubricated nanoscale asperities formed in AFM experiments.
   It is expected that reducing ripples amplitude to less than 1nm should allow observing lubricant response induced by surface roughness.

Future collaboration with host institution

Dr. Carlos Drummond and coworkers will perform, in the following months, SFA measurements on the nanostructured mica samples still available. Measurements will be performed in hexadecane or in aqueous solution with surfactants adsorbed on mica. New nanostructured mica samples will be prepared at the Physics Dept. in Genoa by Dr. Buzio, having surface ondulations of amplitude smaller than 1nm.

Projected publications/articles resulting or to result from grant

Based on the novelty of the research just started and on the planned SFA measurements on the remaining samples, at least one comprehensive article will result from the present ESF activity, describing the protocol for SFA measurements on ns-mica surfaces and the first results for lubricated contacts.