FRICTION PERFORMANCE OF COATINGS

D. M. Pai, B. Kailasshankar, M. S. Konchady, X. Wang
J. Mason, J. Sankar, and S. N. Yarmolenko

Center for Advanced Materials and Smart Structures
NC A&T University
Greensboro, NC 27411

Introduction

Coatings are thin layers of materials that are deposited onto a bulk material to achieve properties not easily attainable with substrate alone. They have attracted considerable research interest because of their numerous technical applications. Coatings are used widely in optical, microelectronic, packaging and decorative applications as they impart good mechanical, chemical, electrical, magnetic and optical properties[1]. Friction and wear properties are a critical issue for the manufacturing industry and hence characterization of friction and wear properties is important in assessing the potential of coating materials. Friction and wear properties of coatings are traditionally measured in industries using tribometer. The tribometer employs one of the standard configurations such as ball-on-disc, ring-on-disc, pin-on-disc and pin-on-block to reproduce the motion typical of many real world mechanisms. The technique is well suited for monolithic materials as well as thick coatings. However, friction and wear phenomena are essentially the outcome of surface interactions and these properties are affected by surface geometry, material and environmental conditions especially for thin coatings.

Generating mechanical property profiles as a function of depth and performing varying load tests provide additional insight in characterizing adhesion as well as wear resistance of thin coatings. The Nano Indenter is a specialized piece of mechanical characterization equipment well suited for this purpose. In the present experiment, thin and thick coatings produced on low carbon steel using dip-impregnation technique are characterized by both tribometer and Nano Indenter.

In our case, the friction test procedures are demonstrated on two types of materials listed below. The test materials are not a limiting factor, as any pair of untreated and treated specimens can be used.

1. SAE 1018 ground steel bars of 9.5 mm square cut into approximately 25 mm lengths. Hardening was achieved by zinc phosphating followed by impregnated with chrome oxide aqueous solution and fired at elevated temperature and furnace cooled. The impregnation and firing cycles were repeated for multiple cycles.
2. Silicon wafers coated with alumina thin films of ~600 nm thickness produced by pulsed laser deposition.

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Experimental procedure

Tribometer Testing

Calibration

Calibration can be defined as the procedure that establishes the correct output scale for the measuring system or instrument. Calibration procedures attempt to identify and eliminate errors by comparing with a known standard[3]. The undergraduate students were given basic training in calibration of load sensors used in the tribometer. Calibration procedure for the UMT-2 tribometer is given in Appendix 1 at the end of this paper. Linearly reciprocating ball-on-flat wear testing was done per ASTM Standard G133-02. Since tests are time-consuming, students in a 2-hour lab session can participate in the calibration procedure and watch the initiation of the tribometer testing and return the next lab session to analyze the results.

Procedure

1. Load the DFH-5 dual Friction/Load sensor – range 0.5 N to 50N
2. Load lower linear reciprocating motion drive
3. Switch on the machine
4. Adjust travel to 5 mm manually
5. Zero the F_x and F_z pots for zero traces
6. Connect the 5/32 in diameter ball specimen
7. Connect electrical contact resistance sensor
8. Set up a test sequence consisting of two sheets. Make sure the black box is checked to save the data
9. Sheet 1 is for settling time for carriage to establish the initial normal load
    a. Carriage – applies 25N constant force for a duration of 10 seconds
    b. Spindle – Velocity is 0 revs/min
    c. Slider – Idle
10. Sheet 2 is the test for wear for 16000 seconds
    a. Carriage – applies a 25N constant force for a duration of 16000 seconds
    b. Spindle – Velocity 60 revs/min, Move – continuous, Direction – clockwise
    c. Slider – Idle
11. View the results from the viewer software

Determination of coating weight [4]

- Phosphate a part of known surface area
- Thoroughly clean part to remove all oil
- Weigh part to nearest tenth of a milligram
- Strip phosphate coated part in a 2 ½ % chromic acid solution at 160°F immersing for 10 min for zinc phosphate coating, 15 min for manganese phosphate coating and 5 min for iron phosphate coating
- Rinse in clean water
- Dry
- Reweigh stripped part to nearest tenth of a milligram; difference in weight from the earlier step equals total coating weight

- Calculate weight of coating per unit of area; standard units are milligrams per square foot

Safety precautions while handling acids

1. Wear gloves and safety glasses or goggles.
2. If there is a spill on the body, flush out immediately with plenty of water for several minutes.
3. If the spill is all over you, use the safety shower in the lab.

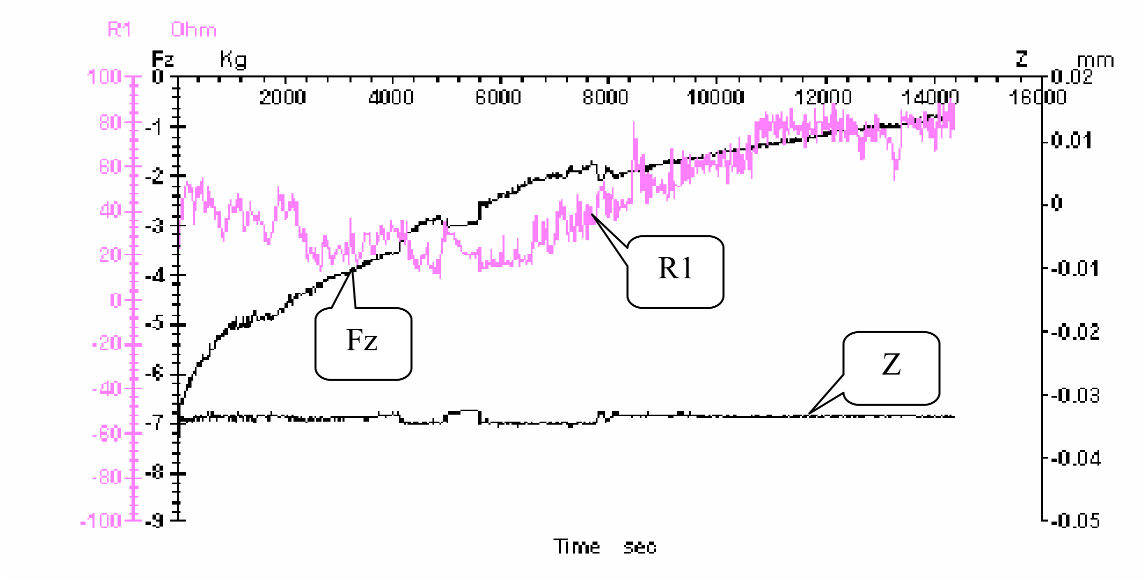

Figure 1. A typical graph of wear test from tribometer

The X-axis represents the test time. In the Y-axis, three parameters are plotted. Fz is the load applied on the sample during the test which remains constant. Z represents the wear with time. R1 represents the contact resistance of coating. The contact resistance starts decreasing as the coating wears out.

<u>Nano Indenter scratch Test</u>

1. Loading samples
   1) Mounting samples at the same height

   The sample mount system is made of the clamping mechanism and the sample tray. The exposed portion of the samples must be ser at proper height. The indenter will not be able to reach the sample surfaces and the experiment will abort if the samples are mounted too low, but no damage to the system will ensure. On the other hand, although the indenter is retracted into the indenter housing, care should be taken that the samples are not mounted too high in the sample tray. The maximum height of the samples must be lower than the bottom of the indenter housing. Not only must the samples be mounted at the proper height, but all of the samples' surfaces must be at the same height. A large deviation in height from one sample to the next will cause problems during indentation. The maximum allowable deviation between sample heights is 1 mm.

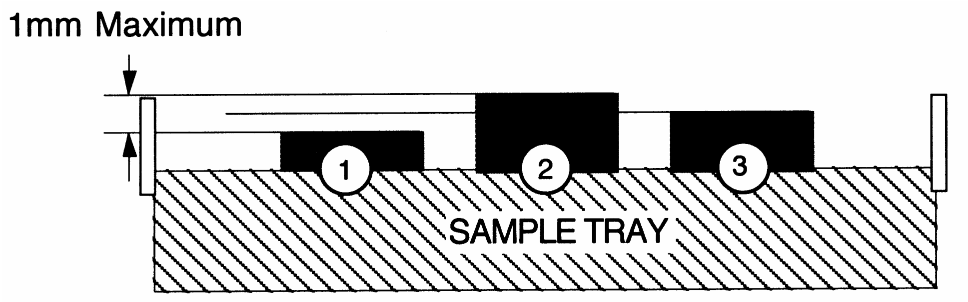

Figure 2. How to mount sample at proper heights within range of the indenter[4]

2) Loading mounting disks into the sample tray
   The standard sample tray can accept multiple samples on standard metallographic mounting disks (1.25'' diameter, 1'' height). The following steps have to be done to load the samples,
   a. Insert the sample disk into the bored sample hole;
   b. Tighten the set screw in place against the sample disk;
   c. Repeat this procedure for all the desired samples and mount the tray upside down so that the leveling arms are facing downward.
   d. Set the sample mount tray on a flat, smooth, and lint-free surface;
   e. Loosen all of the sample disks, so that the sample tray rests entirely on the sample leveling arms.
   f. Once the entire sample disks are loose and resting against the "flat surface", retighten the set screws until each sample disk is fixed firmly in place;
   g. Turn the sample tray back over, and sight across the leveling arms to ensure that all of the samples are even and level.
   h. The sample is ready to load into the rails.

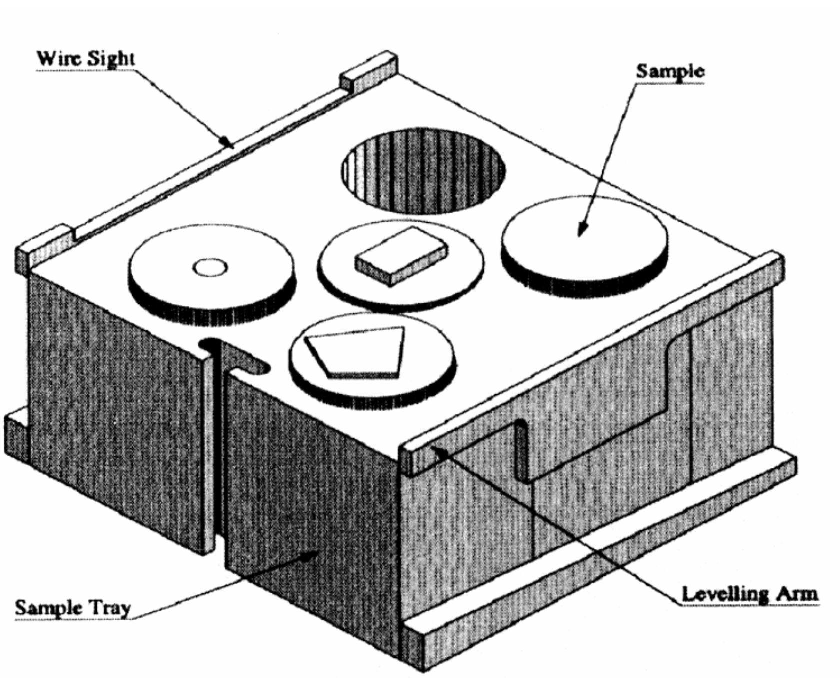

Figure 3. Sample tray[4]

3) Loading tray into rails
   The tray slides into the rails on the Y stage and the clamping mechanism slides into the vertical posts on those rails, and the slot on the sample tray. Turning the clamp screw forces the sample tray into the wedges on the trails, and tightly locks the sample tray in place.

2. Microscope to indenter calibration
   The microscope to indenter calibration is used to establish the relative positioning between the microscope and indenter so that when properly calibrated, the indentation will be performed at the site chosen while the sample was under the microscope, and after the indentation is performed (under the tip), the site of the indentation will be returned to a point directly under the microscope. The following steps have to be done to perform a microscope to indenter calibration
   a. Go to the "Tools" menu item and select "Microscope to Indenter Calibration";
   b. At the window, verify the site you have chosen for the calibration and click on "Yes";
   c. A series of five indentations will be made by the XP (one at each corner of a "square" with a single indentation in the center.) When the stage returns to the microscope, the cross hairs should be directly centered on the indentation in the center of the "square". A message will display for you to find the indents;
   d. Do NOT click "OK";
   e. Move the dialog to the side and use the video handset to locate the red cross hair over the center indentation;
   f. Now press "OK";
   g. Another dialog will be displayed indicating that the calibration is complete.

3. Instrumented Scratch Testing
1) Introduction
   The Nano Indenter XP indentation/ scratch head is load-controlled. The load is applied normal to the sample surface by the magnet/ coil system, allowing for precise and fast control. The maximum distance allowed for the indenter travel, normal to the sample surface, is about 1.5 mm. Over this entire range, displacement resolution is less than 0.1 nm. The maximum normal load capacity for the standard system is 500 mN with a precision of less than a μN. However, the maximum X-Y plane load that can be applied during a scratch is 200 mN.
   During a scratch test, the normal force on the sample is controlled and can be held constant, increased, or decreased at a linear rate. Scratch velocity and scratch path followed by the indenter on the specimen surface are defined by the operator. Scratch velocity is typically held constant through the scratch experiment, and can be set between 0.05 μm/s to 2.5 mm/s.

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2) Scratch procedure

A typical scratch experiment is performed in three stages: an original profile, a scratch segment and a residual profile.

- In the original profile, surface morphology is obtained by pre-profiling the surface under a very small load at a location where the scratch is to be performed.
- The indenter actual penetration depth under the sample surface is estimated by comparing the indenter displacement normal to the surface during the scratching, with the topography of the original surface at each position along the scratch length.
- For the scratch segment, roughness and slope of the surface are taken into account in the calculation of the indenter penetration.
- In a similar manner, a final profile helps establish residual scratch depth.

3) Scratch direction and normal load selection

At least two scratching directions are used for the scratch tests- face forward and edge forward (Figure 5). Two kinds of normal loads are usually applied: constant normal loads and ramping loads (Figure 6) which are increased, or decreased at a linear rate.

Figure 7 displays the nanoscratch results for Al_2O_3 thin films deposited with a substrate temperature of 600 °C and a laser energy of 700 mJ. In the figure, the coefficient of friction is plotted as a function of the load applied on the sample. The point associated with the critical load (corresponding to a sudden increase in the coefficient of friction) in the plot is marked with a circle. The optical micrograph on the left image represents the scratch tracks made on the film surface. The area associated with the initiation of crack or delamination of the film, where the critical load was reached, is also marked with a circle. It can be seen that the critical load is about 320mN and friction coefficients are about 0.25 before coating delamination and 0.45 after coating delamination. Figure 8 displays the cross profiles of a low carbon steel (SAE 1018) 9.5 mm square bar nanoscratch with a constant load of 80 mN and ramping loads from 0 to 80 mN.

Three main phases in the scratch experiment. Between each phase the indentor returns to the origin of the experiment

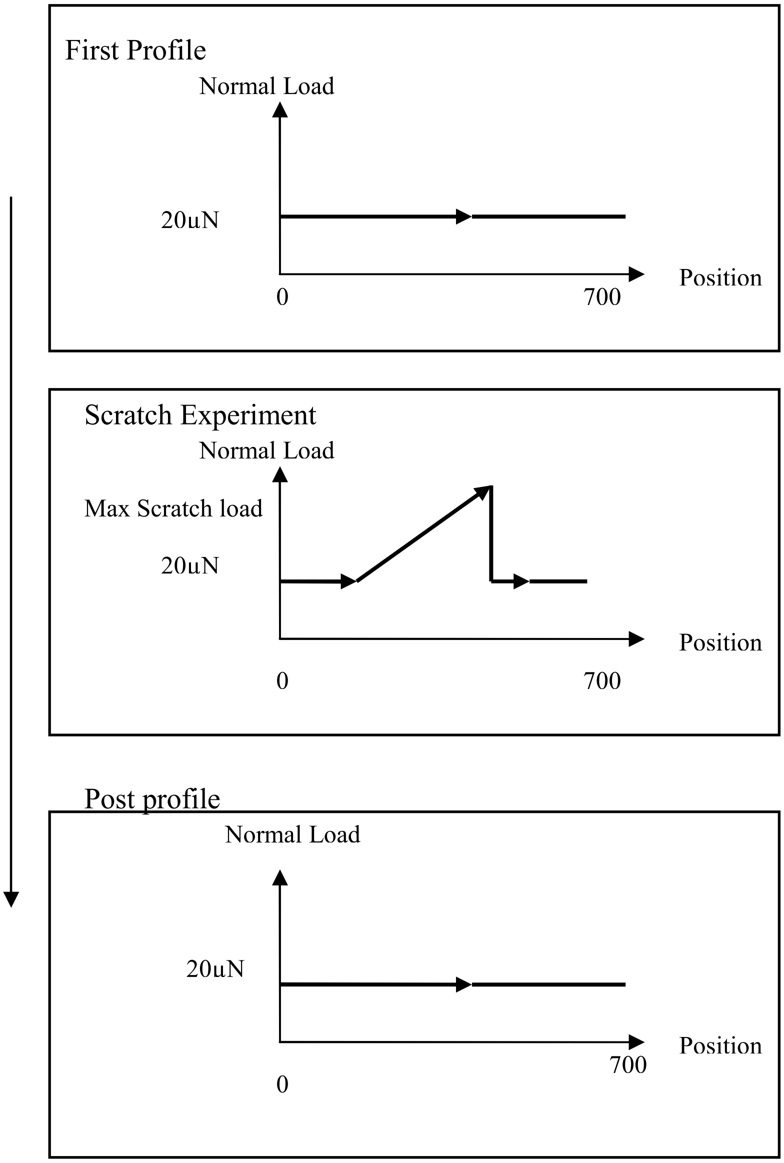
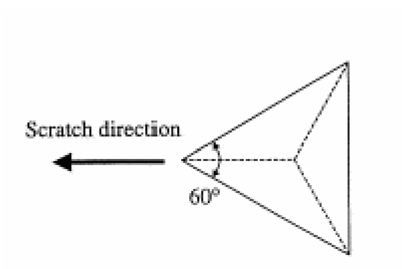
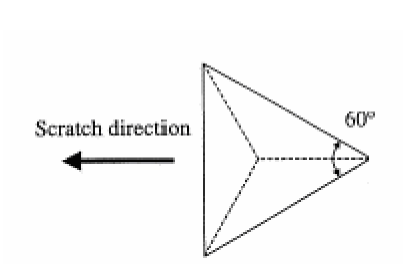


Figure 4. Standard scratch procedure

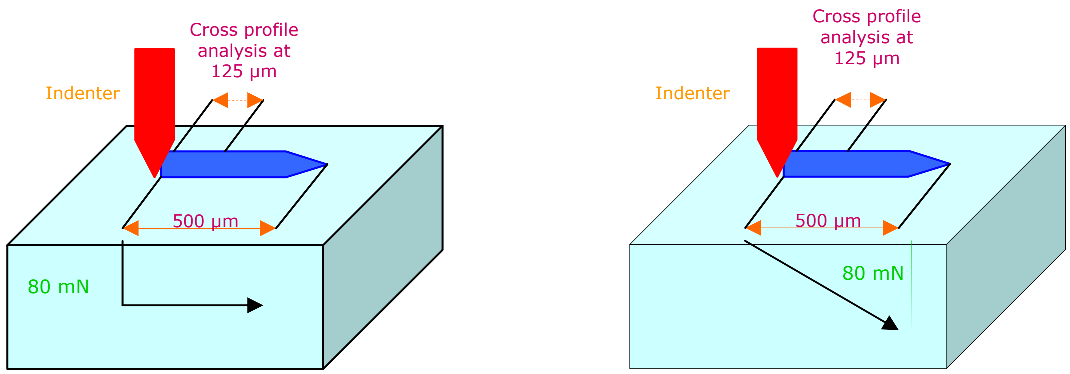


Face forward scratch [5]



Edge forward scratches

Figure 5. Scratch direction



Constant load Ramping load

Figure 6. Nanoscratch tests with constant load and ramping load[6]

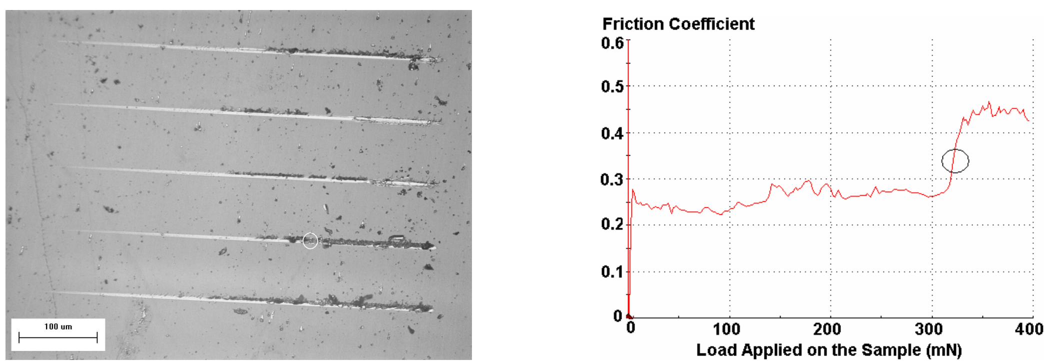
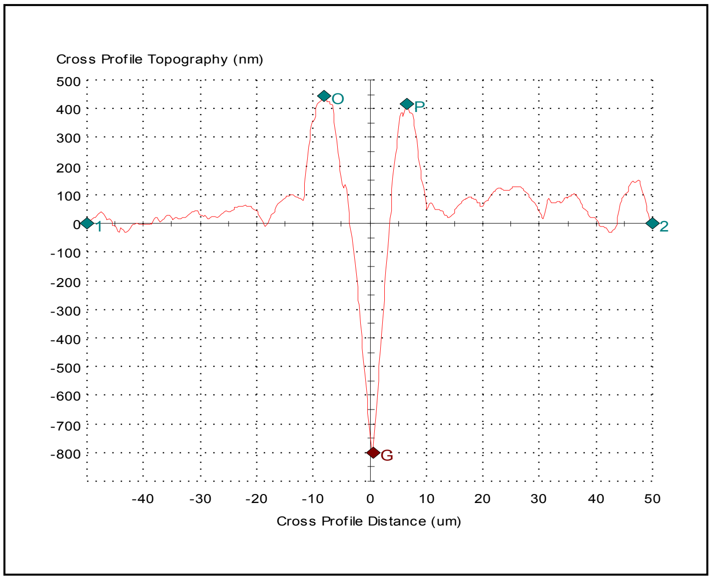


Figure 7. Optical micrograph of a set of ramping load nanoscratch tests (left) and coefficient of friction profile for one of these tests (right) for an Al_2O_3 thin film deposited with a substrate temperature of 600 °C and laser energy of 700 mJ using Pulse Laser Deposition method



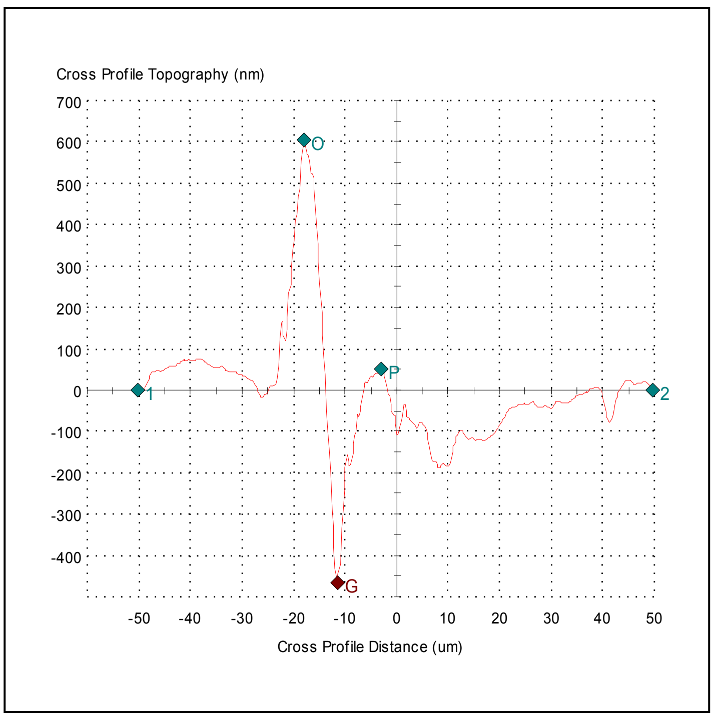


Figure 8. Cross profiles of a low carbon steel (SAE 1018) 9.5 mm square bar nanoscratch with a constant load of 80 mN (top) and ramping loads from 0 to 80 mN (bottom).[7]

Comments

The paper discusses the procedures involved in tribometer testing and Nanoscratch tests. The procedure helps in demonstrating characterization of thin and thick film coatings.

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Biographies

DEVDAS M. PAI is a Professor of Mechanical Engineering at NC A&T State University and Associate Director (Operations) of the Center for Advanced Materials and Smart Structures. He teaches manufacturing processes and tribology related courses. A registered Professional Engineer in North Carolina, he serves on the Mechanical PE Exam Committee of the National Council of Examiners for Engineers and Surveyors and is active in several divisions of ASEE and in ASME.

BALA KAILASSHANKAR is a PhD student in the Department of Mechanical Engineering. He received the B. Tech. Degree in Metallurgy from the Indian Institute of Technology and the MS in Mechanical Engineering from NC A&T State University. He has over 22 years of research, development and manufacturing experience in the tribology and coatings industry.

MANOHAR KONCHADY is an MSME student and Graduate Research Assistant in the Department of Mechanical Engineering. He received the B. E. Degree in Mechanical Engineering from the University of Madras is working on the tribological characterization of ionic fluids.

XINYU WANG obtained his PhD in Mechanical Engineering from NC A&T State University in 2004. He holds BS and MS degrees in Mechanical Engineering in 1992 and 1995 from China. He is conducting postdoctoral research in tribological characterization of coatings and advanced materials.

JOSHUA MASON is a Mechanical Engineering senior undergraduate student and has worked with the tribology research group over the summer of 2004.

SERGEY N. YARMOLENKO is a Senior Research Scientist of the Center for Advanced Materials and Smart Structures at NC A&T State University. He received his Ph.D. from Institute of Organic Chemistry, Ukrainian Academy of Sciences. He conducts research and teaches courses related to advanced materials.

JAG SANKAR is a Professor of Mechanical Engineering at NC A&T State University and Director of the University's Center for Advanced Materials and Smart Structures. He received his Ph.D. from Lehigh University. He conducts research and teaches courses related to advanced materials.

Appendix 1

Load Sensor Calibration for UMT-2 tribometer

Materials:

Standard weights, Hanger, Screw driver, Angle plate, C- clamps, String and Shims.

Procedure:

1. Disconnect the sensor cable and remove the sensor from the machine and reconnect it using of extension cable.
2. Place the sensor on level flat surface.
3. Open the **UMT-2** software program and click on the **graph panel** button to open the automatic panel. Automatic panel should display default Fx and Fz values.
4. Open the Semi-Automatic panel by clicking **Semi-Automatic** panel button.
5. Select the **Data-1** tab and un-bias the sensor by clicking on **1111** button.

Fz Calibration

6. Adjust the Fz pot through the opening in the panel on the carriage until the **Fz** value displayed on the right of the automatic panel reads zero.
7. Place the standard weights on top of the sensor and note down the **Fz** value displayed on the right of the automatic panel.
8. Plot a graph with actual value of load along X-axis and the measured Fz value along the Y-axis. Slope of the graph gives the measured value (MV).
9. Calculate the calibration factor using the formula

   $$CF = SL/MV$$

   Where SL= Sensor Limit, MV = Measured Value and CF = Calibration Factor.
10. Open the **Options** menu in the program and go to **Edit** and select **data Acquisition Channels**, **Fz** and replace value in the **Scale** box with new calculated value. Go back to **Options** and click **Save** to save the new value.
11. Repeat Step 6 to Step 10 until Calibration Factor (CF) values converges.

Fx Calibration

12. Mount the sensor vertically against the angle plate with help of C-clamps and shims.
13. Attach the hanger/loading platform to sensor using a string.
14. Adjust the **Fx** pot to read zero in the automatic panel.
15. Place the standard weights on the hanger and note down the **Fx** value displayed on the right of the automatic panel.
16. Plot a graph with actual value of load along X-axis and the measured Fz value along the Y-axis. Slope of the graph gives the measured value (MV).
17. Calculate the calibration factor using the formula

    $$CF = SL/MV$$

    Where SL= Sensor Limit, MV = Measured Value and CF = Calibration Factor.
18. Open the **Options** menu in the program and go to **Edit** and select **data Acquisition Channels**, **Fx** and replace value in the **Scale** box with new calculated value. Go back to **Options** and click **Save** to save the new value.
19. Repeat Step 14 to Step 18 until Calibration Factor (CF) values converges.
20. Open the Semi-Automatic panel by clicking **Semi-Automatic** panel button.
21. Select the **Data-1** tab and click on **0000** button to bias the sensor.
22. Remove the extension cable, install the sensor back in the UMT-2 machine and reconnect the sensor cable.