Micro Materials

Excellence in Nanomechanics

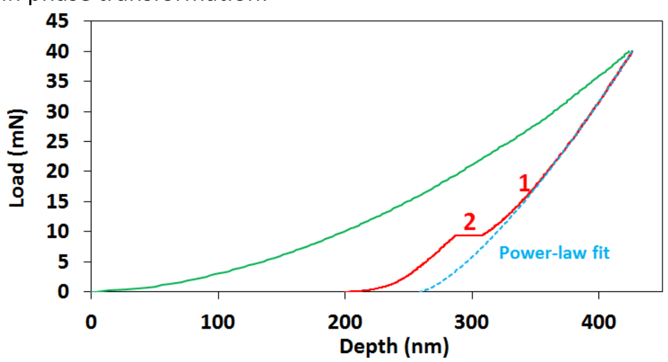
Electrical Contact Resistance Measurement

Introduction

The multi-sensing capability of the multifunctional NanoTest Vantage nanomechanical test system has been extended by the development of the Electrical Contact Resistance (ECR) measurement module. The ECR module measures the electrical current and voltage between an electrically conductive indenter and the sample during any of the nano/micro mechanical measurements (nanoindentation, nano-scratch, nano-impact, nano-fretting, reciprocating nano-wear) possible in the NanoTest Vantage. The additional insight by combining tests with ECR measurements are illustrated in the two case studies below: (1) phase transformations during nanoindentation of silicon (2) reciprocating nano-wear of metallic materials.

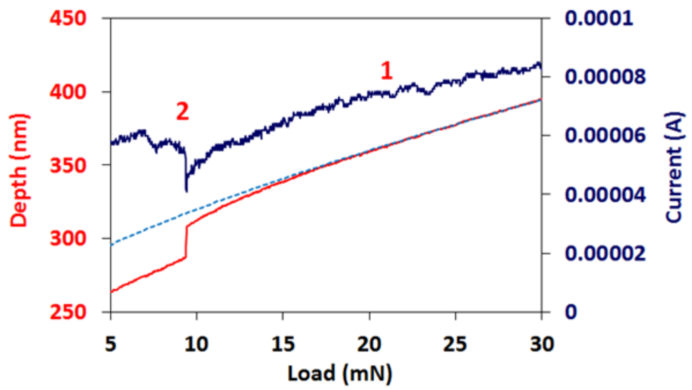
Phase transformation in nanoindentation of silicon

Silicon undergoes complex behavior during indentation, with phase transformations and brittle fracture at higher load. To investigate these in greater detail *in situ* electrical contact resistance measurements using a Boron-doped diamond Berkovich indenter as the electrically conductive probe can provide additional information about the phase transformations that occur in loading and unloading. In this case study mirror polished Si(100) was indented with Boron doped diamond Berkovich to 40 mN at 2 mN/s, with different unload rates = 0.267-20 mN/s to investigate rate dependence in phase transformation.



The standard method used to measure hardness and modulus involves power-law fitting to elastic unloading curves, so the deviation from this can be used to pin-point the start of non-elastic deformation e.g. due to phase transformation (point 1 above, at 0.267 mN/s unload rate).

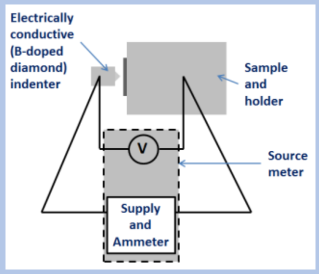
Measurements on Si(100) show the initial deviation from elastic unloading associated with phase transformation begins (point 1 in figures below and bottom left) before the well-known "pop-out" event (point 2) that occurs with further unloading.



ECR and depth data show changes during unloading due to phase transformation which begin before the main pop-out event. Initial deviation from elastic behavior was accompanied by a more rapid *decrease* in electrical current. The subsequent pop-out was accompanied by an abrupt *increase* in electrical current. This is consistent with Si-II initially transforming to a-Si with a later transition to more electrically conductive high pressure phases such as Si-III/Si-XII at the pop-out.

How it works

Electrically conductive probes such as metallic materials or Boron-doped diamond are required for ECR measurement. The conductive indenter creates an electrical circuit during a nano-scale contact (indentation, scratch, impact, fretting or nano-wear). Tests can be run in constant current or constant voltage modes.

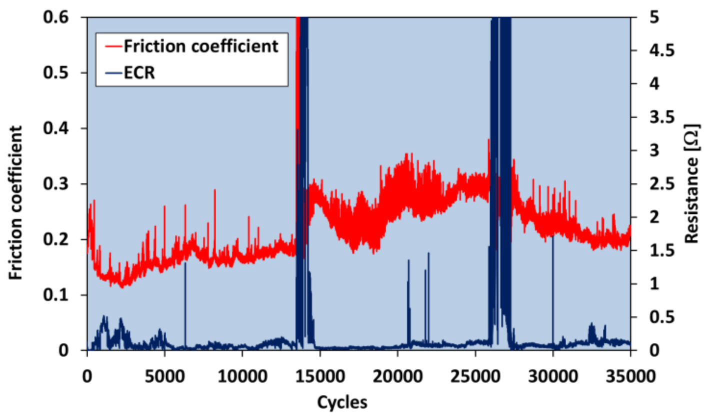


Reciprocating nano-wear of metallic materials

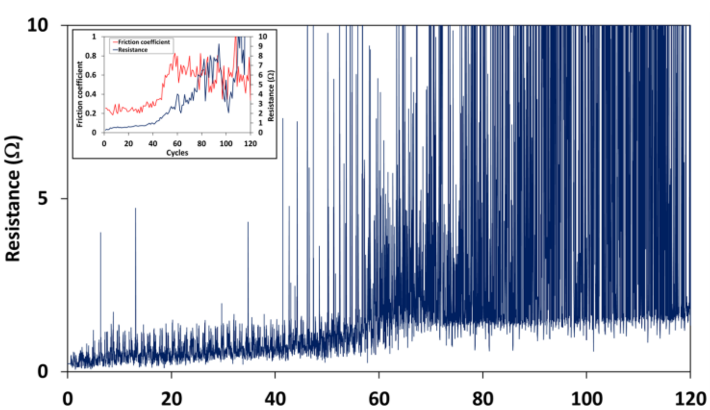
As single-asperity contacts, the nanotribological tests in the NanoTest provide unique opportunities to study the interplay between the severity of the test, surface topography and mechanical properties on the friction and wear that develop. ECR measurements can be combined with other NanoTest modules such as in reciprocating nano-wear tests with the NanoTriboTest. The combination of the NanoTriboTest module with *in situ* ECR measurements has been used to investigate wear of (1) multi-layer electroplated sliding connectors and (2) bulk noble metal alloys in longer tests.

Sample	Probe	Load (mN)	Track length (mm)	Set I (A)	Cycles	Time (s)	Sliding distance (mm)
Au multilayer	Steel	10	5	0.1	200	1000	2000
Ag alloy	Ag alloy	50	10	0.5	1221	5746	24420
Au alloy	Au alloy	200	5	0.5	11532	54908	115320
Ag alloy	Ag alloy	200	5	0.1	35000	166482	350000

The NanoTest Vantage has the stability to perform long duration tests. Experimental conditions are summarised in the Table. Noble metal-noble metal contacts (Au-Au and Ag-Ag) showed much longer endurance than gold vs. steel contacts in the reciprocating tests. Ag-Ag showed only isolated failures after 10,000 cycles in the 35,000 cycle, 46 hr, test (see figure below).



On the multi-layer sliding metallic connector failures were much more rapid (see below). The mean friction and contact resistance over each cycle were determined in the NanoTest software, confirming the clear correlation between friction and contact resistance in the failure mechanism of the multi-layer coating (see inset).



Summary

ECR and depth data on Si(100) show changes during unloading due to phase transformation consistent with (i) with slow unloading - Si-II initially transforming to a-Si starting before the abrupt pop-out and then to more electrically conductive high pressure phases such as Si-III/Si-XII at the pop-out (ii) with fast unloading - Si-II transforming to a-Si.

ECR measurement during reciprocating nano-wear tests provides unique insights into how surface topography and mechanical properties influence the friction, wear and endurance of sliding metallic connectors and multilayer coating systems. Improved detection of the onset of wear and the subsequent failure mechanisms was possible by simultaneously monitoring friction and ECR.

Specifications

- ECR module for measurement of electrical current and voltage between an electrically conductive indenter and sample during any of the nano/micro mechanical measurements (indentation, scratch, impact, fretting, wear) possible in the NanoTest Vantage.
- Conductive Boron-doped diamond Berkovich indenter is included for nanoindentation.
- Custom metallic probes can be used for tribological tests.
- User defined voltage or current applied to the sample using a computer controlled high accuracy National Instruments Dual-Output Power Source-Measure Unit. This unit covers the ranges: (I) from 200 µA to 2 A with resolution up to 10 nA, and (V) +/- 6 V at a resolution of 0.1 mV and +/- 20 V at a resolution of 0.33 mV.
- Data displayed in the NanoTest software as V or I vs time, displacement or load.

References

Correlation between electrical contact resistance, deviation from elastic unloading and phase transformation in silicon, BD Beake and T Jochum, TMS 2022.

Friction and electrical contact resistance in reciprocating nano-scale wear testing of metallic materials, BD Beake, AJ Harris, TW Liskiewicz, J Wagner, SJ McMaster, SR Goodes, A Neville, L Zhang, Wear, 474-475 (2021) 203886.

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