

NANO AND MICRO INDENTATION AND SCRATCH TESTS OF MECHANICAL PROPERTIES OF METALS

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ABSTRACT

Experimental comparison of common nano and micro scales for hardness evaluation has been performed on metal samples and discussed in details. Instrumented indentation was found advantageous in both repeatability and a number of measured parameters over classical hardness methods for metals, while sclerometry was advantageous for ultra-thin films.

INTRODUCTION

Traditional macro-hardness tests of bulk materials are conducted at high loads of the order of kilo-Newtons with post-test indent analysis. Micro-hardness measurements of relatively-thick coatings and bulk materials are performed under the medium loads of the order of deca-to-hecto-Newtons. As the industrial technology advanced, the mechanical characterization went to nano levels, with the loads used for nano-indentation tests of thin films down to micro-to-milli-Newtons. A big step forward has been in-situ continuous force-depth monitoring during loading and unloading parts of an indentation cycle [1], which allowed for both instrumented-hardness and Young's modulus evaluation using equations like that of Oliver-Pharr.

In all the indentation tests, pressure distribution is concentrated under (in the front of) the indenter and thus makes it vulnerable to substrate effects for ultra-thin films. Thus, advantageous for thin films may be scratch-hardness tests with the pressure distribution (still in the front of the indenter) in the same surface layer where the indenter is sliding. As post-scratch detection of the shallow nano-scratches is challenging, utilization of the same tip for both scratching and nano-imaging may be the best way for nano-scratch-hardness testing of ultra-thin films.

This paper compares different techniques for micro and nano indentation and scratch tests on bulk and coated samples, touches on the fundamental constraints of the current techniques, and throws light on new technologies. It has been

done mostly on metals, but its techniques and conclusions are applicable to various materials.

EXPERIMENTAL

The indentation and scratch (sclerometry) techniques were compared on the same samples on the same instrument.

A novel Universal Nano+Micro Tester UNMT-1 by CETR has been utilized. It has several easily interchangeable modules for precision hardness and Young's modulus tests, including:

- traditional micro-indentation (loads up to 1 kN), with Rockwell (per the ASTM E18-05), Vickers (per the ASTM E92-82), and Knoop (per the ASTM E384-99) indenters, with post-test measurements of indent diagonals with an optical microscope (Vickers and Knoop) and of indent depth with a capacitance sensor (Rockwell),
- instrumented micro-hardness tests per the ISO 14577-1/02 (loads up to 1 kN) with the same Rockwell, Vickers, or Knoop indenters, but in-situ monitoring of load and displacement and automatic calculations of both instrumented hardness and Young's modulus,
- instrumented static nano-indentation tests per the ISO 14577-1/02 (loads from 0.1 micro-N to 0.5 N) with Berkovich, conical, cube-corner and other indenters, in-situ monitoring of load and displacement and automatic calculations of both instrumented hardness and Young's modulus,
- instrumented dynamic Young's modulus tests with spherical, Berkovich, and other indenters, in-situ monitoring of the frequency change during surface contacts, and both calculations and maps of Young's modulus,
- micro-scratch-tests per the ASTM 448-82 (loads from centi-N to hecto-N, sliding distances from a few microns to many millimeters) with numerous indenters (spherical, conical, micro-blades, etc.),
- nano-scratch-tests per the ASTM 448-82 (loads from 0.1 micro-N to hecto-N, sliding distances from 1 to 100 microns) with numerous indenters (Berkovich, spherical, etc.) and AFM-like imaging of scratches with the same tip.

The UNMT-1 allows for multi-scale measurements of the same sample without its removal, just with an easy exchange of the indenter modules.

RESULTS AND DISCUSSION

The case study involved characterizing multiple samples in ambient conditions (next stage of tests will be done at both high and low temperatures, available on the UNMT-1). Table 1 summarizes the samples and analysis done. The “+” sign shows that the technique was sufficient to measure the property of the sample, the “-” sign indicates that the technique failed to measure the film properties without a substrate effect.

Table 1: Summary of Tests done on the UNMT-1

Samples	Traditional micro-indentation	Instrumented micro-indentation	Instrumented micro-scratch	Instrumented nano-indentation	Instrumented nano-scratch
Bulk steel, brass and aluminum	+	+	+	+	+
20-micron metal film	-	+	+	+	+
2-micron metal film	-	-	+	+	+
4-nm DLC thin film	-	-	-	-	+

Examples of actual test results are presented below.

Table 2 shows average hardness and its deviation for the same bulk metal samples with the same Vickers indenter, obtained in 20 instrumented versus 20 traditional standard Vickers tests. The instrumented indentation produced the same hardness as the traditional Vickers method, but with much less data fluctuation between the individual tests.

Table 2: Instrumented and Traditional Vickers Hardness

Material (bulk)	Vickers Hardness, HV		Instrumented Hardness, MPa	
	Average	St. Deviation, %	Average	St. Deviation, %
Aluminum	97 +-11	11	968 +-23	2
Brass	201 +-9	4	2004 +-19	1
Steel	519 +-20	4	5202 +-47	1

Table 3 shows average Young’s modulus and its deviation for the same coatings with the same Berkovich indenter, obtained in 20 static tests with a nano-indenter versus 20 dynamic tests with a nano-analyzer. The nano-indenter showed good data for bulk materials and micron-thick coatings, but a substrate effect for nano-coatings; it’s results for a 4-nm film reflected more the silicon-100 substrate than the film. The dynamic Young’s modulus characterization with a Nano-analyzer module, based on a frequency response in a tapping mode, showed much less of the substrate effect, with the data for the nano and micro films being almost the same.

Table 3: Static (Nano-Indenter) and Dynamic (Nano-Analyzer) Young’s Modulus Data

Coating on Silicon	Young’s Modulus Data, GPa		
	Nano-Indenter	Nano-Analyzer	Comment
Silicon 100	164+-18	164+-14	equal static and dynamic
2-micron Ti	98 +-13	98 +-10	equal static and dynamic
2-micron DLC	381 +-24	382 +-19	equal static and dynamic
1-micron DLC	372 +-29	379 +-21	equal static and dynamic
100-nm DLC	314 +-32	370 +-25	static shows substrate effect
4-nm DLC	195 +-39	361 +-27	static shows substrate effect

Table 4 shows hardness data for the same coatings with the same Berkovich indenter, obtained in 20 nano-indentation tests with a nano-indenter versus 20 nano-scratch tests with a nano-analyzer. Again, the nano-indenter showed good data for micro-coatings, but a substrate effect for nano-coatings. Indeed, a stress distribution in the front of a moving indenter, penetrating in the tested material hundreds of times more than the indenter itself, goes into the substrate in the nano-indentation, but stay within the nano-coating in the nano-scratch.

Table 4: Nano-indentation and Nano-scratch Hardness

Coating on Silicon	Hardness Data, GPa		
	Nano-Indenter	Nano-Analyzer	Comment
Silicon 100	11.4+-1.2	11.6+-1.0	equal indent and scratch
1-micron DLC	30.5 +-2.9	31.2 +-1.7	equal indent and scratch
100-nm DLC	24.6 +-3.6	30.8 +-1.7	indent shows substrate effect
4-nm DLC	15.7 +-3.2	29.1 +-1.9	indent shows substrate effect

CONCLUSIONS

1. In the micro-indentation and nano-indentation tests, the indents under 5-10% of the film thickness have produced repeatable and apparently substrate-independent results.

2. In the micro-scratch and nano-scratch, the scratches under 30-35% of the film thickness have produced repeatable substrate-independent results.

3. In the micro-indentation tests, traditional Rockwell and Vickers hardness tests produced more data variability than the instrumented-hardness tests with the same indenters in the same test setup, though the statistics requires more data.

4. The UNMT-1 provides a platform for comparative nano and micro indentation and scratch testing of thin films, thick coatings and bulk materials.

REFERENCES

1. D. Tabor (1996). Indentation hardness: Fifty years on. - Philosophical Magazine A, V. 74, No 5, pp. 1207-1212.