

Characterization of Overcoat Durability for MEMS

Summary: The mechanical properties of thin overcoats were studied and differentiated utilizing the UMT Series Micro-Tribometer at micro-level. In this paper, we evaluated different silicon nitride overcoats typically used in MEMS. Three techniques were employed to study the materials, namely: Knoop hardness, nano-indentation and dynamic scratch resistance with the UMT. Using the scratch-resistance test method, it was determined that an U-passivated overcoat significantly outperformed both L-passivated and regularly-passivated overcoats, when the other two test methods showed no differences in the properties of L- and U-passivated coatings.

Experimental procedure: Seven silicon wafer samples were analyzed with Knoop hardness, nano-indentation and UMT scratch resistance tests. Each of the wafers was overcoated with the same silicon nitride, but then processed with different passivation technologies: regular (sample 1), L-passivation (samples 2,3), U-passivation (samples 4,5).

Prior to testing, the wafer samples were inspected with a Metallographic Microscope. They all had microstructures of 40X40 μ m with the height of about 400 nm and the depth of 600 to 700 nm.

Scratch-resistance tests were performed with a Micro-Tribometer, Model UMT. This precision instrument provides very accurate measurements of the tribological properties of materials, using such parameters as:

- 1 - Friction force and coefficient,
- 2 - Adhesion force,
- 3 - Wear and wear rate (including fatigue, abrasive and adhesive wear),
- 4 - Contact resistance or capacitance,
- 5 - Contact acoustic emission,
- 6 - Temperature in the friction zone.

The UMT can be used effectively for the tribological testing of metals, plastics, ceramics, paper, composites, thin and thick coatings and lubricants.

The UMT can accommodate both upper and lower samples of practically any shape. Wear measurements can be performed in-situ to an accuracy of 50 nm; finer measurements can be made with an atomic force microscope or a surface profiler. A precision spindle can rotate either the lower or upper specimen at speeds from 0.001 rpm up to 5,000 rpm. Ultra-accurate strain-gauge sensors perform simultaneous measurements of loads and torques. The forces can be measured precisely in the ranges from milligrams to kilograms, with a resolution of 0.1% of the full-scale and very high repeatability. A normal-load sensor provides feedback to the vertical motion controller, actively adjusting the sample position to ensure a constant load during testing. The tester has fully automated PC-based motor-control and data-acquisition.

Results and discussions:

1. Scratch/wear-resistance test: Scratch-resistance testing with the UMT was performed on the silicon wafer samples under the following test conditions:

- Diamond stylus of 2 μ m radius and 60° angle,
- Normal loads from 20 mN to 160 mN,
- Sliding velocity of 0.5 mm/s,
- Scratch length of 4 mm.

The depth of the scratches was measured by an AFM and reported as the average for 5 locations at each load level. The scratch resistance criteria was the critical load **L_c** at which the scratch depth becomes equal to the overcoat thickness.

Sample #1 was significantly different from the others. The very low critical load of 22 mN was enough to break through the overcoat of 430 nm. Samples #2 and #3 showed significant improvement in wear resistance properties. Their critical loads were 55 mN and 75 mN, respectively. Samples #4 & #5 showed the highest scratch resistance properties, when even a high load of 160 mN was insufficient to break the overcoats, and produced only scratches less than 300 nm thick. Figure 1 demonstrates these results.

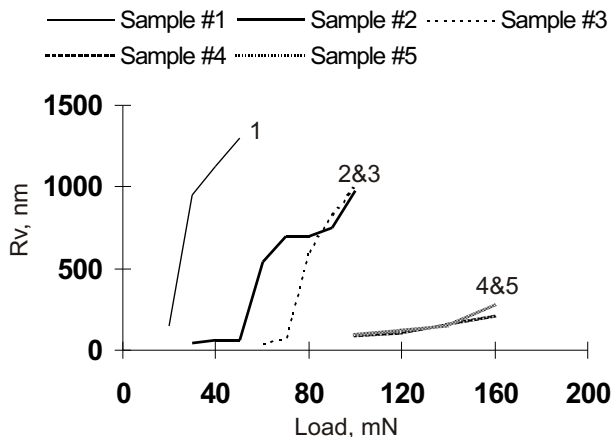


Fig. 1. Scratch resistance of different overcoats

2. Knoop hardness: Knoop hardness was performed per ASTM E384-89. The indenter was a rhombic diamond pyramid with longitudinal edge angles of 172.5° ND 130° ; diagonal or diameter - $10\mu\text{m}$ to 1mm ; depth - $0.3\text{-}30\mu\text{m}$. Loads of 25 g and 100 g were used for samples #1 and #2 - #5, respectively. Table 1 presents the data.

Table 1. Knoop Hardness Data

Indents No	Hardness, HK (kg mm^{-2}) of samples				
	#1	#2	#3	#4	#5
1	268	1204	961	1241	1103
2	240	1073	1015	1169	961
3	268	1136	1043	961	987
4	261	1073	1015	987	1015
5	261	1169	1043	1103	961
Average	260	1131	1015	1092	1005
Stdev	12	58	33	119	59

Sample #1 showed the lowest value of 260 kg mm^{-2} . Results on the other samples were very similar and indicate micro-hardness about 4 times higher than that of sample #1.

3. Nano-indentation: The samples were analyzed with Continuous Stiffness Measurement technique.

A vector of indents was performed on each sample to a peak load of about 35 mN. Afterwards, the positioning of indents was observed, and the indents located too close to the micro-structures were dismissed. A total of 9 to 12 indents per sample were deemed

appropriate for the analysis. The maximum depth into surface was 300 nm. Figure 2 shows the average for the indents on each sample.

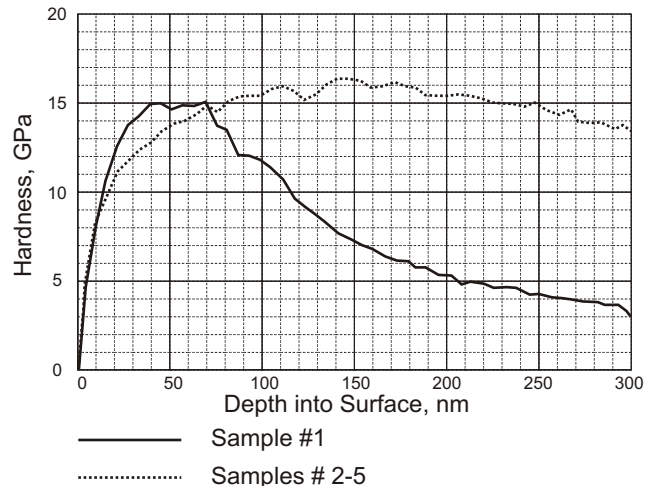


Figure 2. Nano-hardness vs. displacement for samples # 1-5

Sample #1 was significantly different from the other samples and showed nano-hardness of 4 GPa. The other samples showed 3.5 times higher hardness, but no difference between them.

Conclusions

1. The dynamic scratch-resistance measured with UMT Micro-Tribometer demonstrated substantial differences in the protective properties of the L- and U-passivated overcoats. These differences could not be observed with either the Knoop hardness or nano-indentation techniques.
2. Thin silicon nitride films may have very different wear-resistance properties depending on the technology of their passivation.
3. The best scratch-resistance properties were exhibited by U-passivated samples. Even at loads as high as 160 mN the scratch depth did not exceed 300 nm. The L-passivated samples showed good scratch-resistance properties, with the critical load of about 70 mN. The regularly-passivated samples showed the lowest scratch-resistance properties and were completely damaged at loads as low as 25 mN.

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