TRIBOLOGICAL PROCESS CHARACTERIZATION WITH IN-SITU QUANTITATIVE NANO-SCALE METROLOGY

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ABSTRACT
A novel quantitative nano+micro-tribometer with integrated nanoindenter, SPM and optical microscope imaging has been used to characterize mechanical properties of Cu coated Si wafers at various test stages. A 2D Finite Element Model was developed to study changes on workhardened contacts assessed via nanoindentation experiments.

INTRODUCTION
Quantitative nanometer-scale metrology tools have become a standard in semiconductor and data storage industries where coatings and thin films have to be tested for materials properties at the design and quality control stages. A synergy of the nanoscale metrology and manufacturing processes can significantly help in understanding undergoing materials changes. A nano+micro scale tribometer with integrated nanoindenter, SPM and optical microscope imaging has been developed to monitor changes in nano-mechanical properties during micro-tribology tests. Materials properties measurements and surface topography can be assessed at various stages of the test [1].

Internal stress evaluation is a very important objective in thin coatings development area. Though analytical tools such as nanoindentation are versatile, they can not extract stress information from the loading-unloading data. Here, a combination of nanoindentation and advanced analytical/numerical methods is required [2, 3].

A 2-D Finite Element Method (FEM) analysis model was developed to simulate the work-hardening effects. A Lagrangian approach was used to model the elasto-plastic contact problem of nanoindentation, where the work-hardening was simulated by altering von Misses criteria related stiffness. A good agreement between experimental nanoindentation data and numerical results was achieved.

EXPERIMENTAL
The integrated Universal Nano/Micro Tribotester (UNMT) system [1] consists of a fully automated tribometer capable of performing multi-axial tribological tests, high resolution optics, SPM and a nanoindentation instrument (Nanohead-1). The SPM scanner is installed onto the motorized Z-stage. Nanohead, SPM, optics and UNMT instrument are controlled by a dedicated PC and sophisticated control software package. The whole system is placed on an active antivibrational table to ensure nanometer precision measurements. Both, the Nanohead and the SPM have sub-nanometer resolution in terms of displacement noise floor. Also, the Nanohead has a sub-micro Newton force noise floor. Maximum Z – displacement and maximum force ranges are 300um and 500mN, respectively. Instrument calibration routines and mechanical properties extraction for the Nanohead was performed according to the ISO 14577 standard for instrumented nanoindentation.

A number of copper coated silicon wafers were tested for changes in material properties during pin-on-disk tribological tests. Nanoindentation loads of 50µN to 5 mN were applied for the surface probing. Integrated SPM images and nanoindentation test results revealed substantial changes in elastic modulus, hardness and surface topography.
FEM MODELING

A finite-element analysis model was developed to simulate the work-hardening effects on compressed Cu samples. A 2D contact problem was chosen since intensive FEM contact modeling efforts concluded that there is very little difference in results between 2D and 3D contact models [3]. Therefore, 2D model is much less computation intensive. A FEM solid model for nanoindentation process simulation with Berkovich nanoindenter is shown in Fig. 1. Here, 5um length model was meshed using 4800 quadrilateral elements. Included angle used in the model was 70.1. Mechanical boundary conditions were chosen as follows: one translation sliding degree of freedom along the z-axis was constrained for the tip and vicinity of the contact. Static displacement response was derived on the selected DOFs representing tip of the diamond. Here, a non-linear Newton-Rhapson iteration problem was solved using the frontal solver of the commercially available ANSYS FEM software. Materials properties used in the model were obtained from the experimental data. A Lagrangian approach was used to model the elasto-plastic contact problem of nanoindentation, where the work-hardening was simulated with a von Misses criteria. A FEM solid model was loaded with 3000, 3500 and 4500mN equivalent load in the kinematic excitation manner where all selected DOFs representing the diamond tip were selected.

![Fig. 1. Meshed FEM solid model of the indentation contact with corresponding boundaries.](image)

RESULTS AND DISCUSSION

Typical loading-unloading curves from tribology test workhardened (1) and before the test (2) samples are shown in fig. 2. Here, after the pin-on disk test, curve (1) indicates more pronounced elastic response typical for the contact workhardening phenomenon. Greater contact stiffness yields the higher value of reduced elastic modulus and Martens hardness, and were found to be in the range of 135±10GPa and 1.45±0.17GPa, respectively. A load-displacement curve for the not compressed area (2) exhibits more plastic-ductile type response and has a lower contact stiffness value for reduced elastic modulus and hardness. Experimentally derived reduced elastic modulus and hardness values were 112±8GPa and 1.30±0.15GPa, respectively. A FEM simulated loading data is superimposed on the experimental loading-unloading curves indicating a good agreement. Corresponding equivalent stresses can be derived at any FEM node using this model.

In the present study, performance of a quantitative nano/micro-tribometer, integrated with SPM and high resolution optical microscope imaging, is demonstrated on Cu coated Si wafer samples where quantitative materials properties were derived at several intermediate characterization steps by means of the scanning nanoindenter.

![Fig. 2. Loading-unloading curves for treated (1) and not altered (2) Cu coated Si wafer sample. (*) - FEM modeling results.](image)

CONCLUSION

Performance of the newly developed instrument is demonstrated by the Cu layer workhardening effect that was observed on the nanoindentation loading-unloading curves for the Cu coated Si wafer sample. A 2D FEM model was developed to simulate workhardening process which correlates well with experimental results. A potential combination of the scanning nanoindenter, tribometer and FEM model can be a versatile nano/micro scale manufacturing/characterization workstation for a modern materials scientist.

REFERENCES