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IN-SITU QUANTITATIVE INTEGRATED TRIBO-SPM NANO-MICRO-METROLOGY

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ABSTRACT

A novel quantitative nano/micro-tribometer with integrated SPM and optical microscope imaging has been developed to characterize numerous physical and mechanical properties of liquid and solid thin films and coatings, with in-situ monitoring their changes during micro and nano indentation, scratching, reciprocating, rotating and other tribology tests. Both the materials properties and surface topography can be assessed periodically during the tests.

INTRODUCTION

Quantitative nanometer resolution metrology tools have become a standard in semiconductor, data storage and other hi-tech industries where products have to be tested for thin-film properties. Though it is critical to characterize advanced thin films and coatings, today's off-line nanoscale metrology tools can capture only a limited number of parameters. There is an immediate need for process control instruments capable of in-situ nanometer scale quantitative characterization at different stages of manufacturing process.

Latest advances in nano/micro tribology forced the development of integrated instrumentation. An innovative optical in-situ surface characterization experimental system, combined with the Raman spectroscopy instrument, was reported by Singer et al [1, 2] for investigation of chemical/mechanical properties of debris during tribological testing. Recently, co-focal Raman spectroscopy was combined with Atomic Force Microscope/Scanning Probe Microscope (AFM/SPM) technology for nondestructive chemical characterization at nanometer scale [3]. A combinatorial approach in microscale tribology tests [4] indicated the need for in-situ characterization instruments

integrated into a tribometer. A recent nanoscale tribometer combination with AFM instrument has been reported [5]. Therefore, the number of novel tribometer applications is growing fast and covering fast changing industries including biomedical [6].

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.

EXPERIMENTAL SETUP

Schematics of the newly developed instrument are shown in Fig. 1. The integrated Universal Nano/Micro Tribotester (UNMT) system (Center for Tribology Inc., Campbell, USA) consists of a fully automated tribometer [4] capable of performing multi-axial tribological tests, 3 μ m resolution optics, closed loop XY SPM scanner and a nanoindentation instrument (Nanohead-1) installed into the SPM scanner. The SPM scanner is installed onto the motorized Z-stage. Nanohead, SPM controller, 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 closed-loop XY SPM scanner 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 400 μ m 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

closed-loop SPM scanner has a 100x100um (optional 200x200um) scanning area.

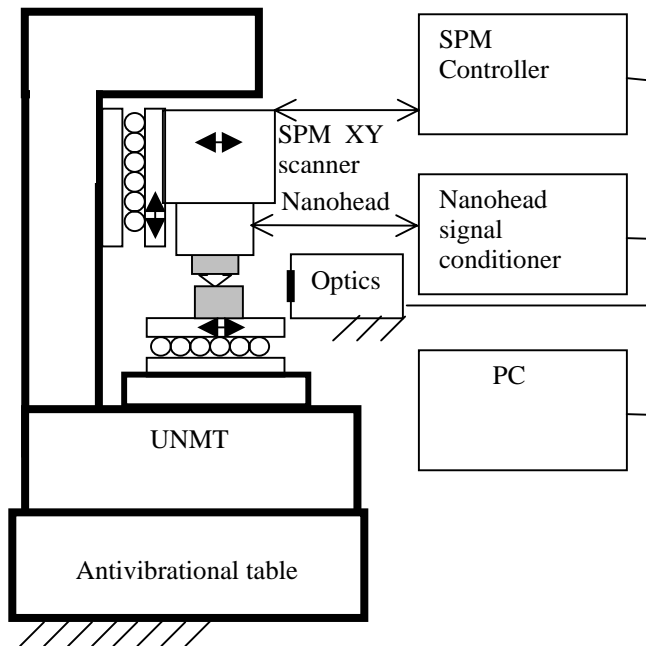


Figure 1. Schematics of UNMT with integrated Nanohead-1, XY SPM Scanner and precision optics.

EXPERIMENTAL PROCEDURE

A Chemical Mechanical Planarization (CMP) processed [7] 5um thick Cu coated Si wafer was placed into the UNMT system and compressed by 5N load. The CMP process was carried out by a commercially available CP-4 (Center for Tribology Inc., Campbell, USA) machine. A 5mm workhardened steel sphere used in Tribology tests (ASTM G99-90) was utilized for this experiment. 5mN load nanoindentation tests were performed before and after workhardening of the Cu layer. A cube corner diamond pyramid tool of 40nm radius was used for all 56 indents. The nanoindentation ramp loading profile consists of 10s loading, 3s holding and 10s unloading segments.

Reduced elastic modulus and hardness values were derived by following the Oliver-Pharr methodology [8, 9]. In-situ pre and post indentation SPM images were taken and analyzed at the selected locations. Optical images were taken before indentations were made in order to find surface areas modified by compression.

RESULTS AND DISCUSSION

Typical loading-unloading curves for compressed (1) and not compressed (2) samples are shown in fig. 2. Here, compressed area 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 similar Cu workhardening phenomena can be observed by testing nanomechanical properties of Cu coated wafers during CMP process or by tribological tests and is reported in the literature [7].

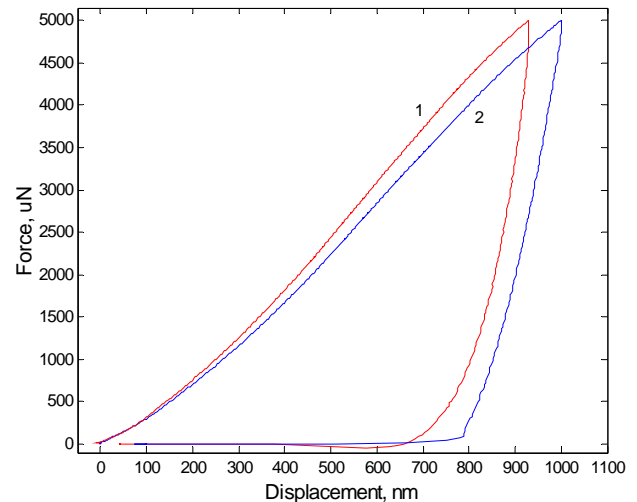


Figure 2. Loading-unloading curves for treated (1) and not altered Cu coated Si wafer sample.

The experimental results were repeatable and consistent with in-situ SPM images. An in-situ SPM image of a typical indent is shown in Figure 3. Here, a triangle shaped indent and small amount of associated material pile-up is seen in the center of the scan. Vertical scale of the SPM scan is 100nm.

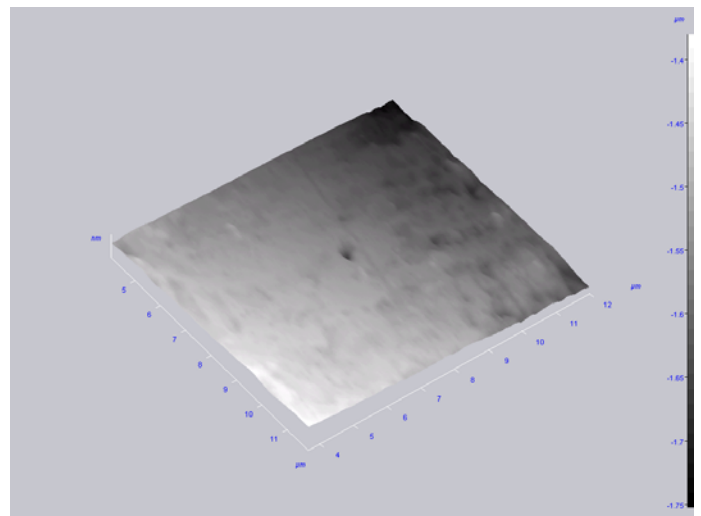


Figure 3. In-situ SPM image of indent.

An optical images of the sample indicated boundary where a compressed surface can be observed.

CONCLUSION

The newly developed instrument consists of a universal tribometer, powerful optics and a scanning nanoindenter with sub-nanometer spatial resolution. The load capability of the scanning nanoindenter is 500mN. The instrument can extract mechanical properties and surface topography at intermediate steps of tribological testing. A powerful combination of tribometer and scanning nanoindenter is filling the gap between microscale tribology test and nanoscale characterization metrology.

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 potential combination of the scanning nanoindenter, tribometer and CMP instrument can provide a mobile and versatile nano/micro scale manufacturing/characterization workstation for a modern materials scientist.

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