

## Non-destructive High-Resolution Stiffness Mapping of Composite Engineered Surfaces

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### Introduction

A novel tool Nano-Analyzer on the Universal Nano+Micro Tester UNMT-1 was used to obtain non-destructive stiffness maps of composite materials, topographically-smooth surfaces and patterned sub-surfaces. Quantitative nano-mechanical characterization of a multi-component composite is a tedious chore. For example, nano-indentation involves indenting the material at many locations to obtain the mechanical property map, with the resolution limited by the need for sufficient spacing between the indents and the time of such mapping being un-affordably long.

### New Technique – Nano-Analyser

To overcome the above shortcomings, we have developed a Nano-Analyser (NA). The NA evaluates the surface by scanning it with a vibrating cantilever with a tiny (less than 100 nm) diamond nano-tip at very low loads (of micro-newtons). The amplitude of the tip vibrations is kept constant, while the vibrational frequency variations are monitored. The frequency shift depends on the tip-sample contact stiffness, which in turn, depends on the local elastic properties of the sample. The NA simultaneously and non-destructively generates AFM-like topographic images and stiffness maps of the scanned area.

### Stiffness Maps of Composite Material

PMAA – Poly-methyl-meth-acrylate is a versatile polymeric material used in such

industries as optics and electro-optics. TiO<sub>2</sub> addition to PMMA changes the linearity of its optical behavior, as well as of its mechanical properties.

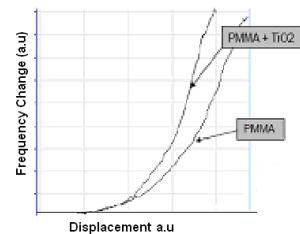


Figure 1. Material response curves for TiO<sub>2</sub> in PMMA

The study was done to optimize the TiO<sub>2</sub> concentration to get optimal optical and mechanical properties. Figure 1 shows the change in the response of the NA oscillating tip as it scans the pure PMAA and the one with TiO<sub>2</sub>. These response curves are correlated to in-situ generate a stiffness map as shown in Figure 2.

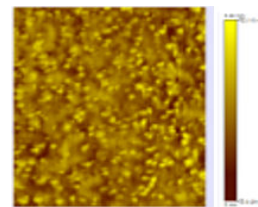


Figure 2. Stiffness map of PMMA impregnated with TiO<sub>2</sub>

The effects of particle concentration and agglomeration can be easily observed and evaluated with this map. Further tests were done with varying TiO<sub>2</sub> concentration.

### Stiffness Maps of Topographically Featureless Material

Ni is typically used for copper wire bonding in a semiconductor industry. Sometimes Ni diffuses in the copper wire-lines and thus increases electrical resistance considerably.

This study was undertaken to detect the Ni contamination in the copper surface. Using SEM for this purpose is laborious and requires cross-sectioning the specimen, while NA was used on the as-is sample. An AFM cannot distinguish between the matrix and the contaminants; the nano-indentation would require several days to locate Ni. Figure 3 below shows topographical and stiffness maps obtained with the NA. The stiffness map clearly showed the Ni contamination in copper due to a considerable difference in their mechanical properties.

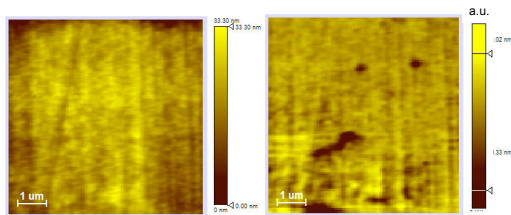


Figure 3. Maps of copper with Ni contamination: (a) Topographical map (b) Stiffness Map - dark areas are Ni

### Stiffness Maps of Topographically Rough Material

Lapping is a precision process used to provide flatness and surface finish to very demanding tolerances. It is widely used in a disk drive industry for head sliders. A lapping plate consists of a soft metal impregnated with hard diamond sub-micron particles. It is important to characterize the number of diamond particles remaining on the plate after lapping to estimate its lapping efficiency and life. AFM topographical maps cannot distinguish between the diamond

particles and rough metal matrix. Using SEM for this purpose is laborious and requires destructive sectioning of the large plates. We used the NA to plot a stiffness map of the as-is lapping plate (Figure 4), which clearly shows the diamond particles on the matrix.

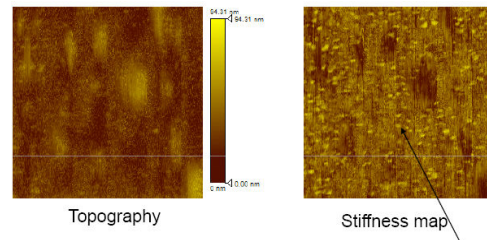


Figure 4. Maps of lapping plate: (a) topographical map (b) stiffness map

### Stiffness Maps of Material with Sub-surface Features

The NA was used to generate the stiffness maps for determination of the effect of underlying patterns on the top surface. Figure 5 shows a polymeric surface with underlying 3-D features. The underneath pattern imposes tensile stress on the top of the polished surface, which was revealed on the stiffness map.

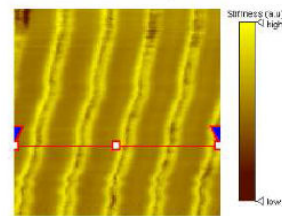


Figure 5. Stiffness map of a polymer surface with underlying pattern

### Conclusion

Non-destructive in-situ topography and stiffness mapping of various hard and soft materials with the novel Nano-Analyzer is effective for studies of multi-component composites and sub-micron particle distribution in the surface layers.