HYSITRON®

APPLICATION NOTE

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Continuous Nanoscale Dynamic Mechanical Analysis of PMMA

Utilizing the Power of nanoDMA® III with *CMX* and *in-situ* Drift Correction

As advanced materials for all different application types are continually developed with decreasingly small constituents, thinner films/coatings, and increased complexities, the techniques used to characterize such materials must also continue to advance accordingly. For advanced nanoscale mechanical characterization of all material types, Hysitron has developed **nanoDMA III*** with *CMX* and *in-situ* drift correction to provide the scientific community with the leading edge in ultra-small scale materials research and development. To exhibit the powerful capabilities provided by **nanoDMA III**, PMMA, a commonly-used polymer material that exhibits time-dependent mechanical properties, was characterized at the nanoscale using several of the available advanced testing modes offered by **nanoDMA III**.

A Hysitron **TI 950 TriboIndenter®** equipped with a diamond Berkovich probe was used to perform dynamic nanoindentation tests on a standard PMMA sample. A frequency sweep was performed using the newly developed reference frequency technique by superimposing a small sinusoidal force oscillation on top of a constant quasi-static force. The resulting displacement amplitude was used to calculate the contact stiffness, and within a single test, the frequency was varied from 0.1 to 200 Hz. Between each test frequency, the probe was oscillated at the reference frequency of 220 Hz in order to measure

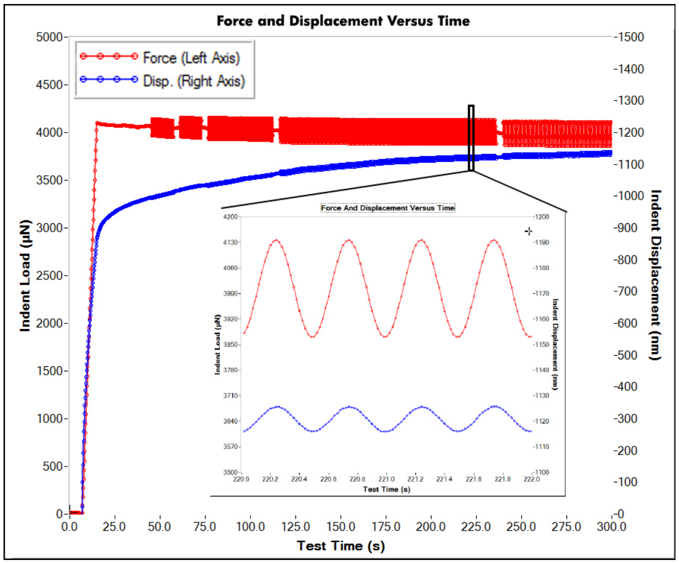

Figure 1: Force and displacement versus time from a portion of the test (inset), showing the force and displacement oscillations.

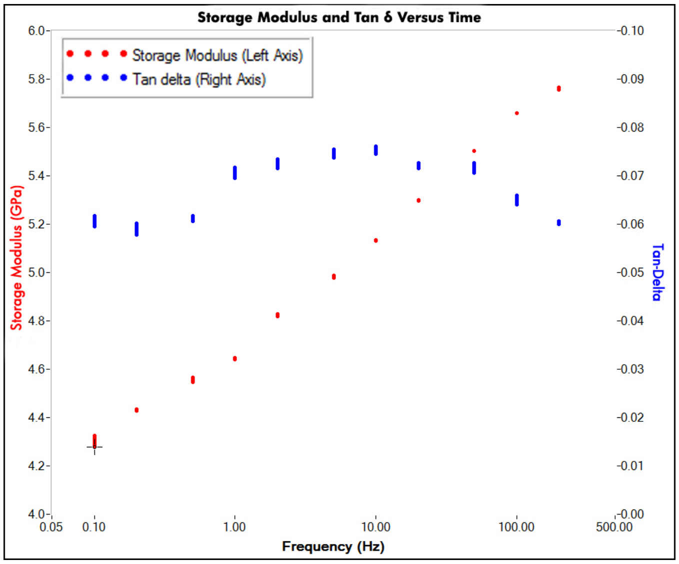

Figure 2: Storage modulus and tan δ versus frequency from a single 0.1 - 200 Hz frequency sweep test. Note the tan δ peak around 10 Hz.

* Patent Pending

the contact area, allowing for accurate calculation of the modulus despite the relatively long test times required to measure at low frequencies.

The modulus of PMMA was found to vary significantly with frequency. Over the frequency range tested, the storage modulus increased proportionally with log frequency from 4.25 GPa to 5.70 GPa. A peak in tan δ at ~10 Hz is due to a beta relaxation in the material.

CMX tests were also performed on the PMMA sample by superimposing a sinsuoidal oscillation onto a constant strain rate quasi-static loading function. Hardness was measured continuously as a function of depth, and by performing tests covering a range of strain rates, the hardness was characterized as a function of both depth and strain rate. The hardness was higher near the surface but was fairly constant at depths greater than ~300 nm. The measured hardness was significantly strain rate-dependent, ranging from 0.31 GPa at a strain rate of $0.01s^{-1}$ to 0.38 GPa at a strain rate of $0.20s^{-1}$.

A one hour reference creep test was also performed on the PMMA sample to characterize the change in properties over longer time periods, quantified as a decaying hardness in Figure 4. In a reference creep test, the quasi-static load is held constant while a small oscillation is continuously superimposed, allowing for continuous measurement of stiffness and contact area over long periods of time. The measured hardness of the PMMA decayed to roughly half of its initially measured value by the end of the hold, with most of this hardness reduction occuring within the first 10 minutes of the one-hour test.

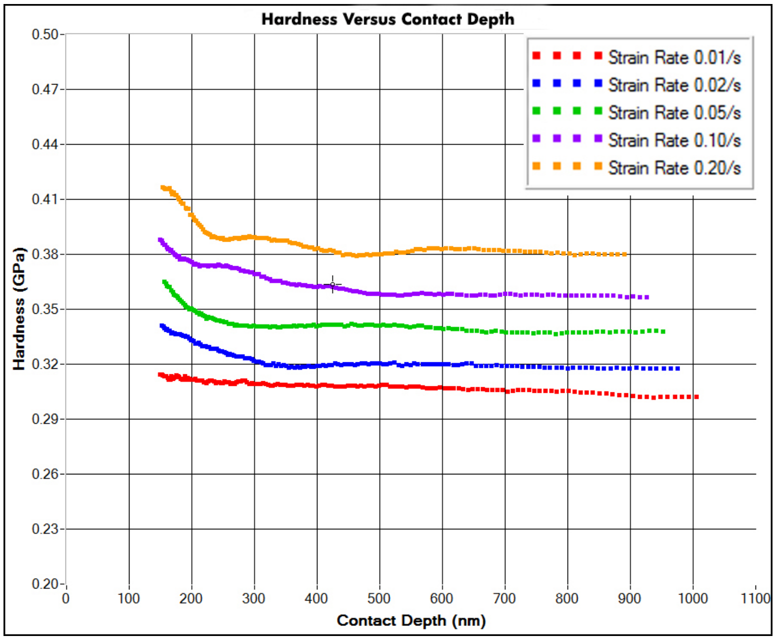

Figure 3: Hardness versus contact depth from *CMX* tests performed at different strain rates.

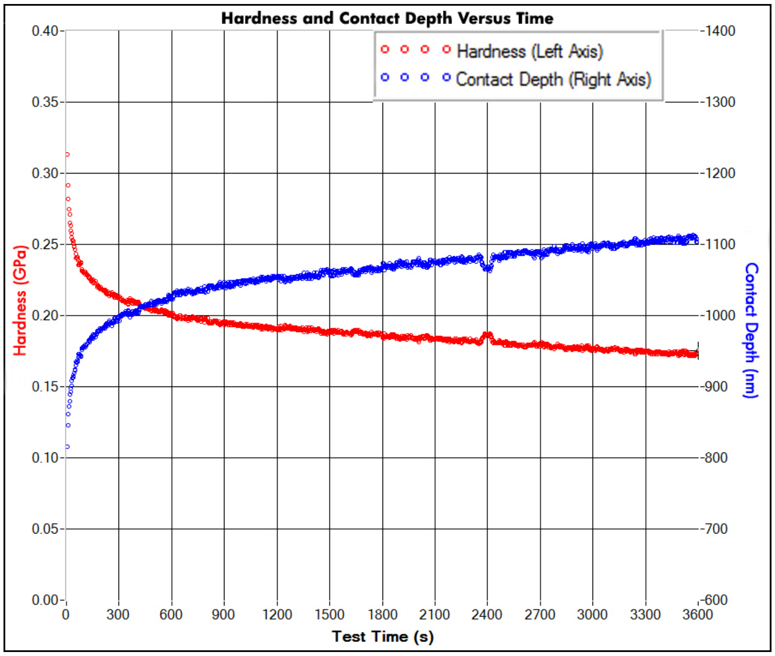

Figure 4: Hardness and contact depth versus time measured during a one-hour reference creep test utilizing quasi-static force held constant at 5 mN.