TECHNIQUE

Reference Frequency Testing Technique

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The reference frequency technique is a key component of **nanoDMA® III***. It allows for determination of the contact area within a test independent of the measured displacement. This stems from the modulus equation relating, in this case, the reduced storage modulus (E_r'), storage contact stiffness (k_s), and contact area (A_c).

Stiffness measured from oscillation

First reference frequency segment

$$E_r' = \frac{k_s\sqrt{\pi}}{2\sqrt{A(h_c)}}$$

Contact depth measured from displacement signal

Typically, the modulus is determined by measuring the contact stiffness and using the contact area calculated from the probe area function and the displacement signal. This method is still used to measure the modulus, but only at the start of the test after the quasi static load has been applied and at one specific frequency, referred to as the reference frequency.

Each subsequent reference frequency segment

Stiffness measured from oscillation

$$A_c = \left(\frac{k_s\sqrt{\pi}}{2E_r'}\right)^2$$

Storage modulus measured from beginning of test

Once the material's storage modulus at this specific frequency is known we can return to this frequency at any point during the test and use the modulus equation to evaluate the contact area independent of any creep or thermal drift. This is useful for tests with long time durations such as creep tests and low frequency tests.

* Patent Pending

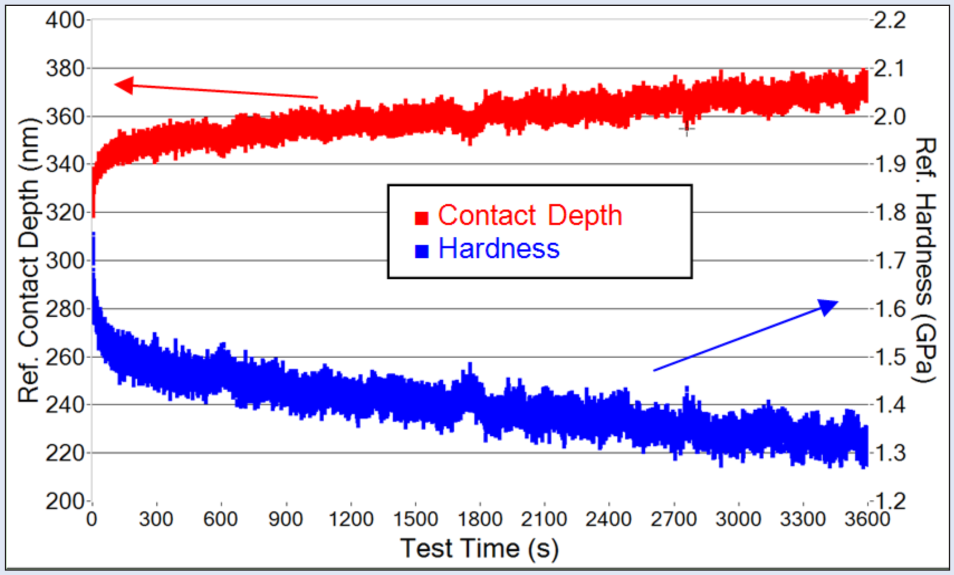

Figure 1. Contact depth and hardness as a function of time on single crystal (100) Cu.

Reference Creep Testing

In a reference creep test, the quasi static force is held constant in closed-loop feedback control while a small oscillation is superimposed for continuous measurement of contact stiffness. The modulus of the material is measured at the beginning of the hold segment when thermal drift error is negligible. That modulus is then used to calculate the contact area continuously for the remainder of the test, allowing for continuous measurement of hardness and penetration depth. This technique is insensitive to changes in drift rates so tests lasting one hour or more can routinely be performed.

Here a one hour long reference creep test was performed on a single crystal (100) Cu sample. A feedback controlled quasi static load of 5000 µN was applied to the sample with a superimposed dynamic load of 340 µN at 220 Hz. The creep rate is significantly faster at the start of the test but there is a significant long term component that is clear over the long test cycle.

Reference Frequency Sweeps

During a frequency sweep, the modulus is measured at a user specified reference frequency near the start of a test. As the test progresses from one frequency to the next, the system returns to the reference frequency. By measuring the stiffness at the reference frequency the contact area can be determined without relying on the displacement measurement. This allows for relatively long tests that are insensitive to changes in drift rate and also tracks creep during the test. As a result, large ranges of frequencies can be covered reliably in a single test that can span large time scales.

A frequency sweep test was performed on an LDPE sample. The frequency ranged from 0.1 to 200 Hz. The test took

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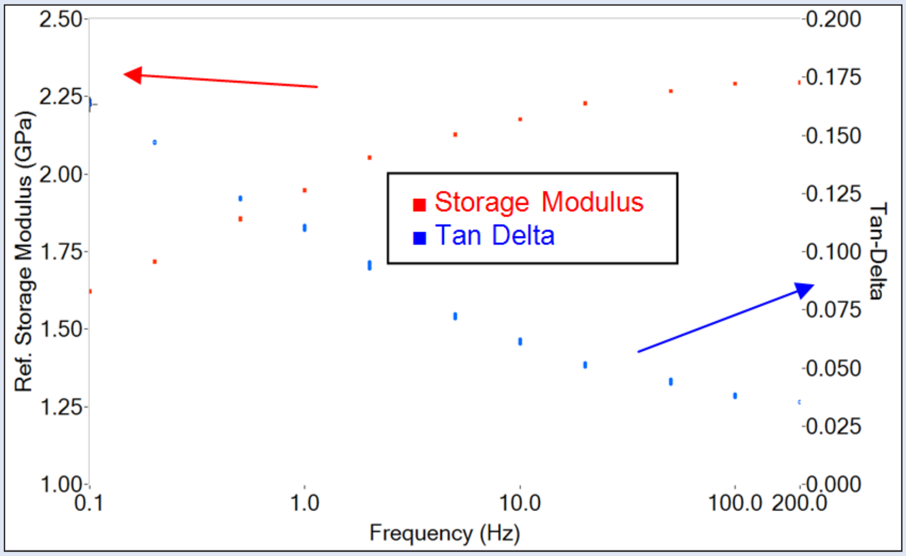

Figure 2. Storage modulus and tan delta as a function of frequency from a frequency sweep test on LDPE.

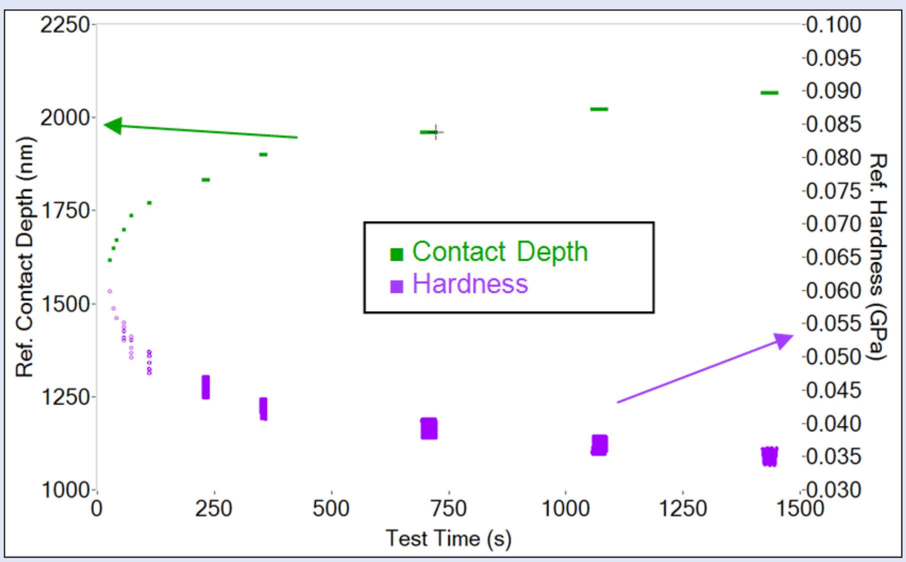

Figure 3. Hardness and contact depth as a function of time measured from the reference frequency segments during the frequency sweep test on LDPE.

approximately 1500 seconds to complete. Since this sample, like most polymers, exhibits creep behavior, the contact depth will change during the test even if thermal drift is not present. At the start of the test, the modulus was measured at the reference frequency of 220 Hz. The frequency then alternated between the test frequencies and the reference frequency so that the contact area could be updated throughout the test.

The modulus of the material is a function of the frequency of the applied load and is independent of the creep behavior. The storage modulus and tan delta are shown as a function of frequency in Figure 2. The measured hardness (mean contact pressure) however, will change with creep so it will be a function of time. The corresponding hardness and contact depth data from the reference segments of the same test are shown as a function of time in Figure 3.