Supplementary Materials

Accurate measurement of elastic modulus and hardness via a rate-jump method

In this method, the elastic modulus and hardness are evaluated at the onset of an unloading stage. Such an onset point for unloading is a rate-jump point, the true elastic modulus *Se* can be calculated from the data just before and after the unloading point using equation (1) [1],

$$\frac{1}{S_e-K}=\left(\frac{1}{S}-\frac{\dot{h_h}}{\dot{P_u}}\right)\frac{1}{\left(1-\frac{\dot{P_h}}{\dot{P_u}}\right)} \quad (1)$$

$$S_e=2aE_r=\frac{2Ea}{1-v^2}=\frac{2Ea}{1-0.3^2}=2.2Ea \quad (2)$$

where S is the apparent tip–sample contact stiffness at the onset of unload and equals to dP/dh, P is force and h is displacement. $\dot{h_h}$ is the displacement rate just before the unload, $\dot{P}_h$ and $\dot{P}_u$ are the loading and unloading rate, respectively. *K* is the fitting parameter and equals to zero when the indentation is performed in air condition. Following the Sneddon relation [2,3] we can have equation (2), where E_r and E are the reduce modulus and the elastic modulus of the tested materials, v is the poission ratio, ($v=0.3$ for glass-ionomer cement, [4]). a is the radius of the contact area. The contact size a can be estimated from a pre-calibrated shape function f(h_c) = πa^2 of the tip. The contact depth h_c is obtainable using the Oliver–Pharr relation [5] with the true contact stiffness *Se*, as shown in equation (3).

$$h_c=h_{max}-\varepsilon\frac{P_{max}}{S_e}=h_{max}-0.75\frac{P_{max}}{S_e} \quad (3)$$

Where h_{max} and P_{max} are the maximum indenter displacement at the onset of unloading and the load before unloading, respectively. ε is a constant (ε =0.75 for Berkovich tip). And the hardness can be measured by equation (4),

$$H=\frac{P_{max}}{A_c} \quad (4)$$

All the raw data collected from the nanoindentor were read and calculated via Matlab R2014a software. Here we show how the results of one set data were calculated.

Sample 1:

Since samples are tested in air testing condition, K=0,

$$\frac{1}{S_e}=\left(\frac{1}{S}-\frac{\dot{h_h}}{\dot{P_u}}\right)\frac{1}{\left(1-\frac{\dot{P_h}}{\dot{P_u}}\right)}=\left(\frac{1}{17543}-\frac{257.6\times10^{-9}}{-9.993\times10^{-3}}\right)\frac{1}{\left(1-\frac{7.142\times10^{-3}}{-9.993\times10^{-3}}\right)},$$

The true elastic stiffness S_e= 2.1×10^4 N/m;

The contact depth $h_c=h_{max}-0.75\frac{P_{max}}{S_e}=5700\times10^{-9}-0.75\times\frac{100\times10^{-3}}{2.1\times10^4}=2.13\times10^{-6}$m

The contact size A_c =1.11× 10^{-10} m²

The contact radius a = 5.95× 10^{-6} m

The true elastic modulus $E=\frac{S_e}{2.2a}$ =1.16 × 10^9 Pa=1.16 GPa

The hardness $H=\frac{P_{max}}{A_c}=\frac{100\times10^{-3}}{1.11\times10^{-10}}=9.01\times10^8$ Pa=0.9 GPa

References

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