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Application Note #1546

Rapidly Quantifying the Effects of Humidity on Nylon's Viscoelastic Properties

Moisture and temperature have a profound effect on the mechanical performance of polymers. However, due to the slow diffusion process that limits the uptake of moisture in polymers, humidity-dependent mechanical testing often requires conditioning times of months to achieve equilibrium. Near-surface mechanical testing allows for large surface-to-volume ratios in the test region, enabling equilibrium conditions within the test volume to be reached in less than an hour and expanding the range of test conditions that can be explored in a time-effective manner. Thus, nanoindentation-based humidity tests can be used to efficiently and quantitatively evaluate the mechanical and dynamic properties of polymeric materials. In this application note, the humidity-testing process for two nylon samples is performed, including the evaluation of time to equilibrium and several viscoelastic properties calculated from the lock-in enabled nanoDMA III transducer.

Method

A Hysitron® TI 980 TriboIndenter® equipped with a xSol® humidity control, nanoDMA® III, and XPM™ were used to characterize the mechanical and dynamic properties of two commercially available nylon samples (nylon 6 and a rigid glass-filled nylon 66) at 10%, 50% and 70% relative humidity (RH) at room temperature. Figure 1 shows the experimental setup and an exemplary sample area with topographic and property maps.

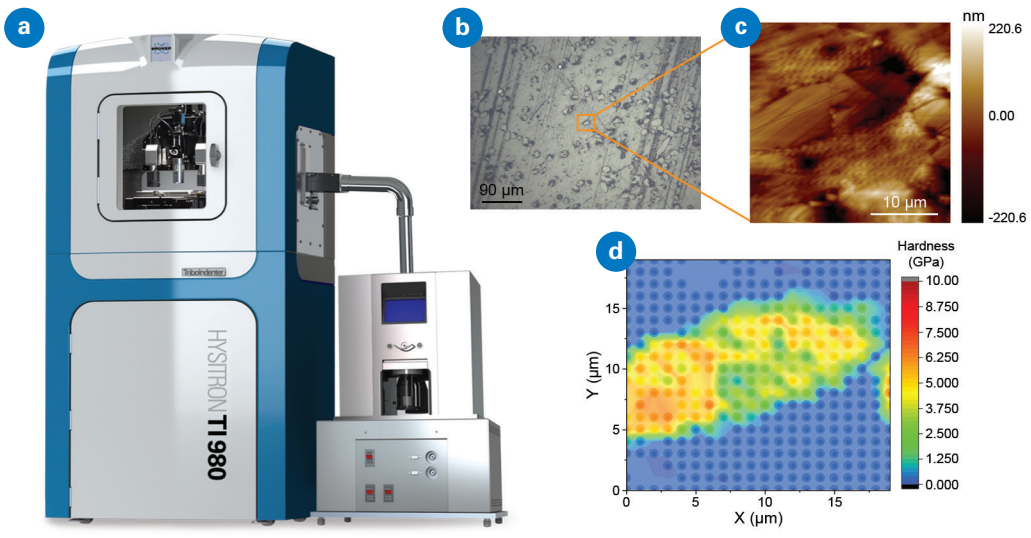

FIGURE 1

(a) Hysitron TI 980 with humidity setup, (b) optical image of glass-filled nylon at 10% RH, (c) SPM image after XPM test, showing both glass and nylon regions as well as the 400 indents from XPM, and (d) property map of hardness from the area shown in (b).

Each sample was prepared as a 1 cm diameter disk machined from 2.54 cm diameter rod. The sample surface was polished utilizing standard polishing techniques.

Grids of indents were performed with a Berkovich indenter while simultaneously ramping humidity (5 to 10, 10 to 50, and 50 to 70% RH) using PID feedback and a MEMS-based humidity sensor within the micro-environmental sample chamber. Measured properties were then compared to the measured humidity in the chamber to calculate time to equilibrium. These tests were performed using the nanoDMA III reference frequency sweep load function at different frequencies (1, 20, 100, and 200 Hz) with a 1200 µN peak load and 3 µm indent spacing.

The maps of the nylon 66 tests were performed using Bruker's accelerated property mapping (XPM) with a 20x20 grid of indents (500 µN peak load and 1 µm indent spacing). The maps were measured after equilibrium for each humidity level to generate property maps across both nylon and glass fiber regions. Each grid of 400 indents took only three minutes to complete.

Results

Using a step function to change the humidity, the response time of the storage modulus is less than 20 minutes (Figure 2a). The small overshoot in measured humidity does not appear to affect the mechanical properties.

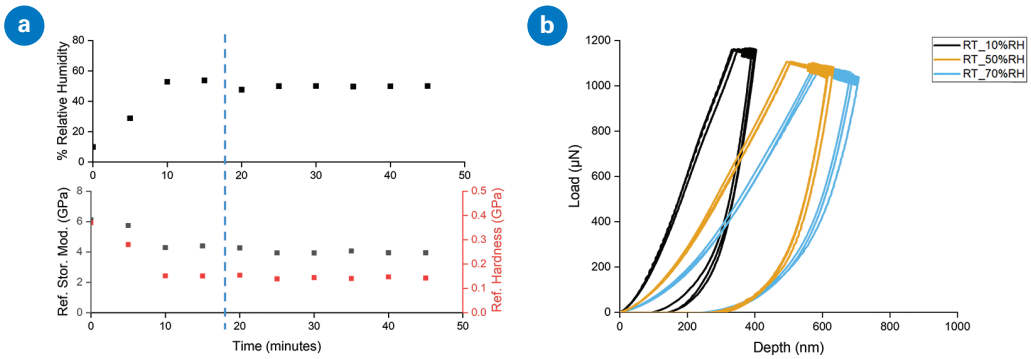

FIGURE 2

(a) Equilibrium time plot at 20 Hz while RH ramped from 10 to 50%, and (b) load-displacement curves at 10, 50, and 70% RH after 60 min of humidity equilibration.

As expected, the load-displacement curves in Figure 2b show an increase in contact depth and a decrease in the unloading stiffness, indicating a decrease in hardness and modulus with increasing humidity levels. The viscoelastic properties measured using the nanoDMA III reference frequency and plotted in Figure 3—storage modulus, tan delta, and hardness—corroborate the trend of decreasing structural stiffness as humidity increases. This was also the case for the glass-filled nylon sample in regions distant from the glass fibers, which were found using scanning probe imaging.

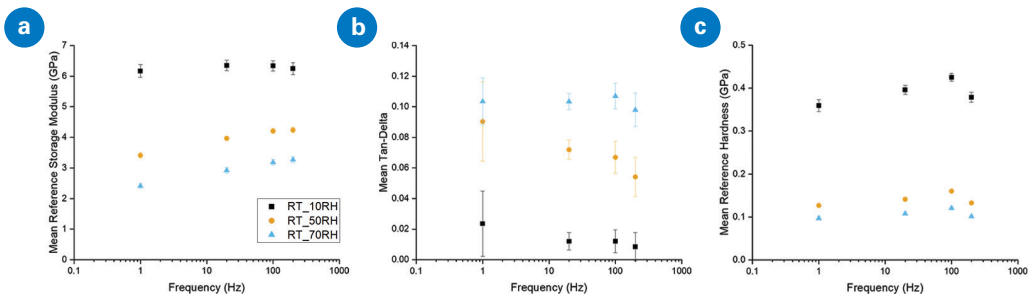

FIGURE 3

Dynamic (a) storage modulus, (b) tan delta, and (c) hardness from 10 to 70% RH and at reference frequency 220 Hz.

Property maps in Figure 4a-c indicated that there is an interphase region between glass fibers and the surrounding nylon matrix. The measured data was separated into three regions using a K-means clustering algorithm (included in the Tribo iQ™ analysis package): matrix, interphase, and fiber. Interestingly, the modulus measured by XPM of the interphase region showed increasing modulus with increasing humidity (Figure 4d), a trend opposite that of the surrounding matrix.

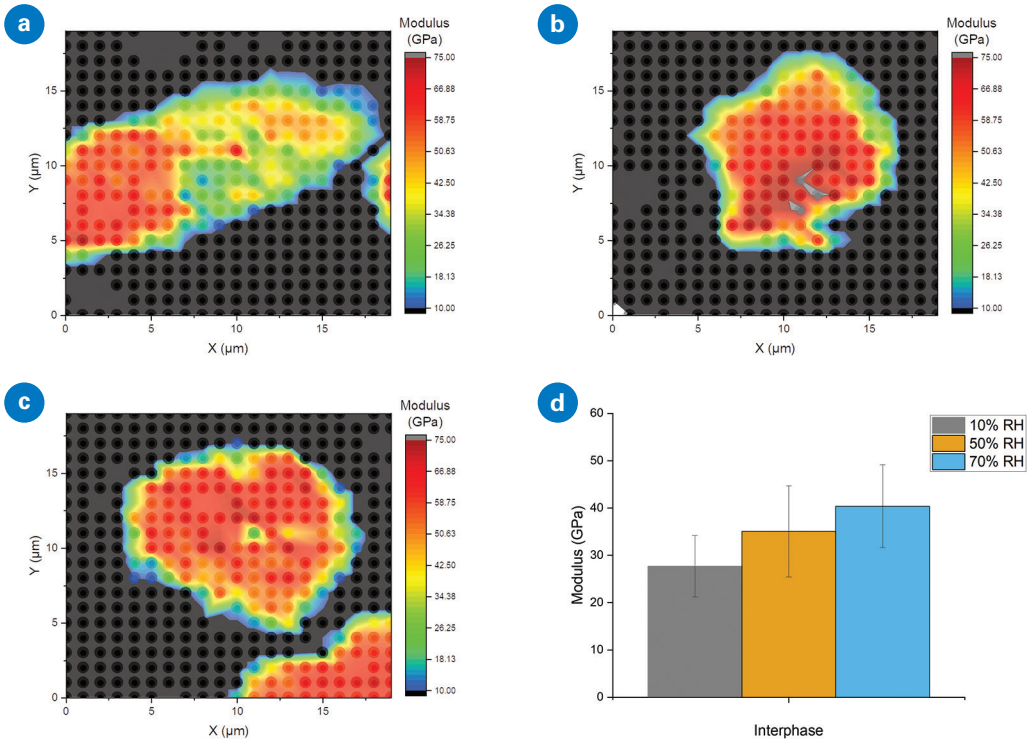

FIGURE 4

Reduced modulus of glass fibers and the interphase regions surrounding them for different RH: (a) 10%, (b) 50%, and (c) 70%. Increase in (d) storage modulus with increaseing RH was seen in the interface region.

Conclusions

Humidity control capability of this system allows the user to reach desired equilibrium level in even polymeric materials within a reasonable time period, shown here to be less than 20 minutes. The hardness and storage modulus decreased with increasing humidity levels, as nylon is hygroscopic. Clustered data however, showed that the properties in the interphase region coupled to the glass fibers did not change significantly at different levels of humidity. The combination of Bruker's humidity control system with nanoDMA III and XPM test methods provide the ideal platform for investigating the effects of moisture on the mechanical and dynamic properties of various materials.

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