Studying Mechanical Properties of Nanocrystalline Cellulose with Nanoindentation

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Cellulose Mechanical Applications

Polymers have become increasingly used in a variety of electronic or biomedical applications. A type of polymer, cellulose, has a source biomass production, where the material is a green by-product. However, ensuring strength of the polymers is crucial to use in these thin film applications. Nanoindentation has been previously used for studying metallic films on substrate, but it is shown to follow similar patterns when using a polymeric material including cellulose.

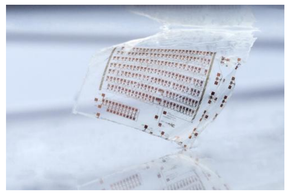

Fig. 1. Example of an electronic application for a polymer, such as cellulose (from phys.org).

Testing Methods

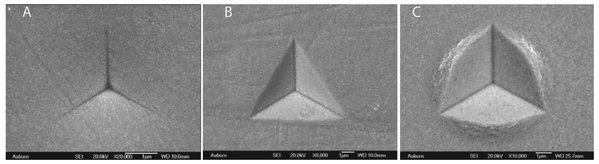

Fig. 2. An indent exhibiting a) sink-in, b) normal response, or c) pile-up.

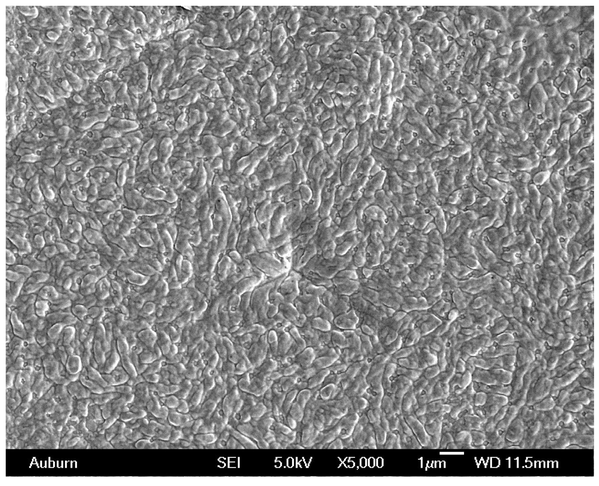

Fig. 3. An indent of gold film on cellulose substrate, exhibiting sink-in.

Nanoindentation is used to measure elastic modulus and hardness of films on substrates. The indents are imaged using scanning electron microscopy to discern more information from the residual indent impression. Depending on the interactions, Fig. 2 shows an indent can have a) sink-in, or c) pile-up. When studying cellulose, the indent impression was shown to exhibit sink-in. For cellulose preparation, nanocrystalline cellulose, NCC, was obtained from Auburn University Department of Forestry. The films were created on glass slides with increasing amounts of cellulose in either a solution of sulfuric acid, along with a sodium perchlorate base.

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Reference

[1] Zhou, B. and B.C. Prorok, *J. Mater. Res.* **25(9), pp. 1671-8, 2010.**

Nanoindentation Results Varying Cellulose Content

By testing increasing amounts of NCC in the polymeric solution, a clear trend was found (Fig. 4). Both elastic modulus and hardness increase with increasing NCC components. Also, when coated with a gold film, the composite properties are greatly improved (Fig 5.). An indentation model can separate these material properties and understand their interactions.

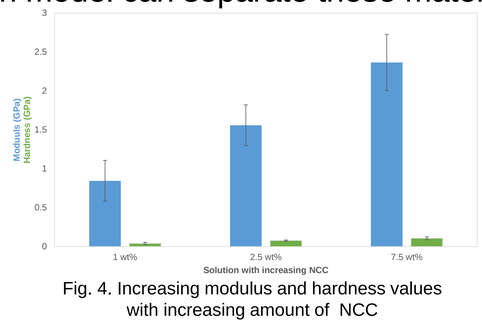

Fig. 4. Increasing modulus and hardness values with increasing amount of NCC

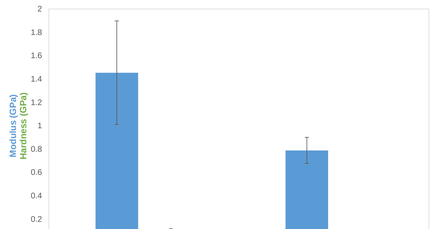

Fig. 5. Increased composite properties with a thin gold film coating.

Implications from Mechanical Testing

The Zhou-Prorok model [1] was shown to predict the behavior until about 700nm in Fig. 6. The increase can then be due to the substrate contributions underneath, a glass slide. Previously, only metallic films have been studied, but this now opens more opportunities for understanding the polymeric system and how the cellulose may be contributing to the overall material response.

$$\frac{1}{E'} = \frac{1}{E_f'}(1-\Phi_s)\cdot\left(\frac{E_f'}{E_s'}\right)^{0.1} + \frac{1}{E_s'}\Phi_f$$

E', E_f', E_s' are elastic moduli
$\Phi_s = e^{-\alpha_s(t/h)}$ and $\Phi_f = e^{-\alpha_f(t/h)}$
α_s and α_f are Poisson's ratios
h is displacement into sample
t is film thickness

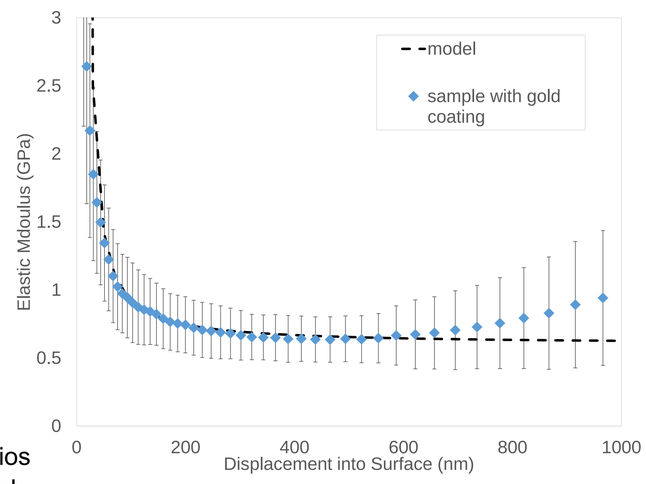

Fig. 6. Composite elastic modulus data of gold on cellulose (diamonds) following an indentation model (dotted line).

Conclusions

- Increasing cellulose content increased modulus and hardness.
- Metallic film coatings on the NCC polymers increased modulus and hardness.
- There is more to be done to dissect how the nanoindentation response can show what components of the film/substrate system are contributing to overall material properties.