NANOINDENTATION STUDIES OF PAPER

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INTRODUCTION

Paper materials consist of a porous web of cellulose polymeric fibers held together by entanglement and fiber-to-fiber bonding. These materials usually contain lignin and hemi-cellulose carbohydrates remaining from the pulping process. Pulped fibers are a flattened ribbon shape on the order of 30 microns wide, 10 microns thick and from one to four mm long. Paper web porosity typically varies between 30 and 60% by volume. All of these features increase the anisotropy and non-homogeneity of paper materials and complicate the characterization of mechanical properties. The z-direction modulus of paper is an important property as it can affect calendaring and printing processes as well as performance of laminated paper products, such as postage stamps. Acknowledging the above, the present paper discusses the use of nanoindentation to measure the z-direction stiffness of paper.

There is relatively little prior literature on measuring of z-direction moduli in paper. Pawlak and Keller [1] measured the compressive characteristics of paper sheets. Associated studies on paper are contained in Refs. 2-8. References 9 and 10 apply nano-indentation techniques to wood, whereas a relevant paper by Hasan *et al.* [11] uses nano-indentation to measure the mechanical properties of cell walls in aluminum foam.

NANOINDENTAION: BACKGROUND AND TECHNIQUE

Indentation techniques such as the Brinell, Rockwell and Vickers hardness tests have been employed since the early 1900's. More recent indentation studies utilize highly instrumented equipment to monitor the applied load and associated displacement

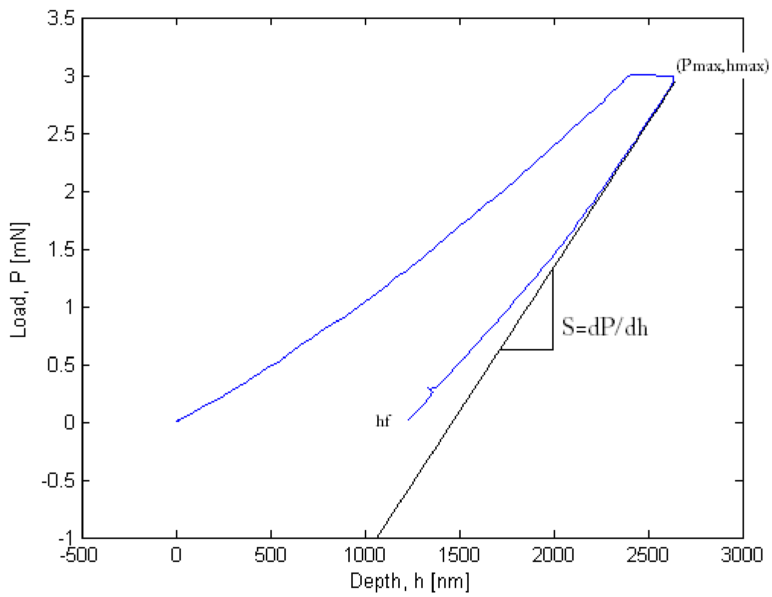

***Fig. 1** – Typical load-depth (P-h) curve.*

to characterize micro- and nano-scale mechanical properties (e.g., hardness, strength, residual stresses, fracture and elastic modulus/stiffness). While various analyses have been developed to extract mechanical properties from measured nanoindentation load-depth (P-h) data, the Oliver-Pharr method [12-14] is widely accepted. This approach assumes (i) the unloading deformation is purely elastic; (ii) the radius of the contacted surface is very large compared with that of the indenter; and (iii) the compliances of the indenter tip and the sample combine to form an effective modulus.

Knowing the Young modulus, ***E*** and Poisson ratio, ***ν*,** for the indenter material, and assuming $\boldsymbol{\nu}^2$ for the specimen is small compared with one, the modulus of the specimen**,** $\boldsymbol{E_s}$**,** can be determined from the initial slope, ***S***, of the unloading P-h curve, Fig. 1. Jakes *et al* have demonstrated the possible need to account for structural compliance and/or significant local changes in in-plane compliance (e.g., when one approaches the edge of a specimen or when indenting between wood fibers) [10].

In the present work, the initial slope, ***dS/dh*** at $\boldsymbol{P_{max}}$, is evaluated from the analytical derivative of a 6^{th}-order polynomial that is fit to the unloading data.

Nanoindentation studies utilize spherical, flat/ cylindrical or pyramid (Berkovich) shaped indentor tips. A spherical indenter tip is preferred for materials with high surface roughness, such as paper. Most chemically pulped papers have local topographic features with roughness variations on the order of the fiber height, about 10μm. The contact area of a flat, punch-type, indentor tip is unknown for materials with these features, especially at penetration depths of less than the surface roughness. A spherical indentor tip provides in-creasing contact area with penetration, tending to minimize the effect of local roughness variations.

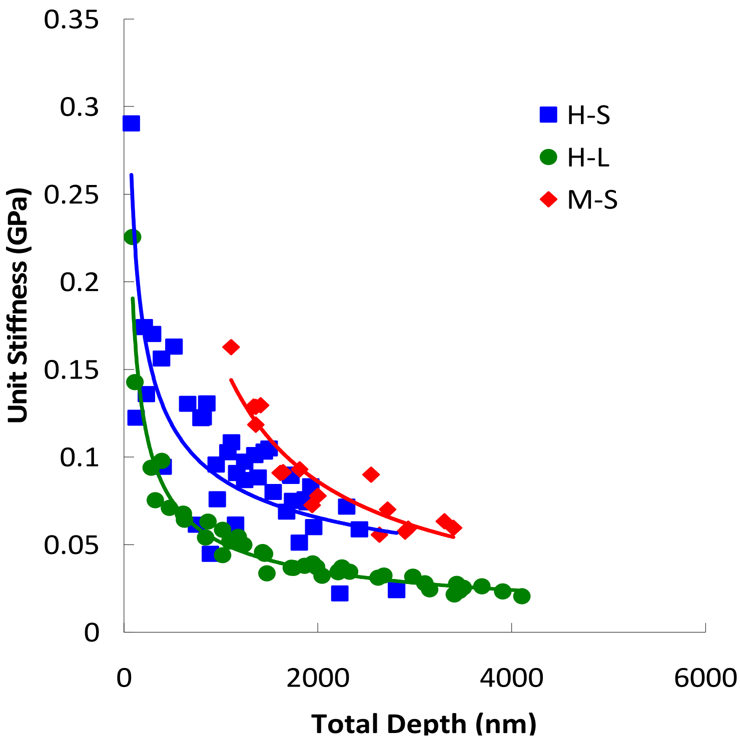

Fig. 2 *- Measured z-direction stiffness of envelope paper vs total penetration depth. Three data sets that were obtained from two different systems (H-Hysitron and M-MTS) and using two different size spherical indenters (L-260 μm and S-127 μm) are shown. The line included for each data set is a power law fit as is included simply too guide the eye.*

Classical nanoindentation analyses assume that the material being assessed is homogeneous, isotropic, elastic, and occupying a half space. The present paper samples are non-homogeneous, their constitutive response is anisotropic, nonlinear and inelastic, and they are relatively thin (~80 µm thick).

RESULTS AND SUMMARY

The z-direction stiffness of envelope paper has been measured using two different commercial nanoindentation systems: a Hysitron Triboindenter and an MTS Nano Indenter XP. Spherical indenter tips, with radii of either 127 µm or 260 µm, were employed. Figure 1 shows a typical P-h curve obtained on paper, consisting of loading, hold and unloading segments. Note that during the hold period, creep of the paper is observed.

Figure 2 summarizes data from a series of indentation tests completed to examine variation in z-direction stiffness with depth. Each data set was obtained by conducting multiple indentation tests of varying depths and extracting the z-direction stiffness for each measurement. Results of Fig. 2 show a significant decrease in effective stiffness with indentation depth. An expanded set of measurements and a description of the underlying mechanics contributing to the observed decrease in stiffness with increasing depth will be presented.

ACKNOWLEDGEMENTS

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