A Review on Nano - Indentation of Thin Polymeric Films

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Abstract - Nano-indentation is a versatile technique used for studying the mechanical properties of thin films on substrate. Depth-sensing Nano indentation technique provides a continuous record of variation of indentation load with penetration depth into the specimen and this technique has high resolution even at low load scale. Currently, the Nano indentation technique is being applied to determine hardness and Young's modulus. This paper characterize the mechanical properties of ultrathin polymer films and its application.

Keywords – Nano-Indentation, Polymeric Films, Mechanical Properties, Application.

I. INTRODUCTION

Nano-Indentation has been increasingly popular technique for material characterization in nano-scale. Developed in 1970's commercial nano-indenters have since been developed and this technology is widely available for researchers interested in thin films.

In a traditional indentation test (macro or micro indentation), a hard tip whose mechanical properties are known (frequently made of a very hard material like diamond) is pressed into a sample whose properties are unknown. The load placed on the indenter tip is increased as the tip penetrates further into the specimen and soon reaches a user-defined value. At this point, the load may be held constant for a period or removed. The area of the residual indentation in the sample is measured and the hardness, H, is defined as the maximum load, P_{max}, divided by the residual indentation area, A_r:

$$H = \frac{P_{\max}}{A_r}$$

For most techniques, the projected area may be measured directly using light microscopy. As can be seen from this equation, a given load will make a smaller indent in a "hard" material than a "soft" one.

Young's Modulus - The slope of the curve $\frac{dp}{dh}$, upon unloading is indicative of the stiffness 'S' of the contact. This value generally includes a contribution from both the material being tested and the response of the test device itself.

The stiffness of the contact can be used to calculate the reduced Young's modulus E_r:

$$E_r = \frac{1\sqrt{\pi}S}{\beta 2\sqrt{A_p(h_c)}}$$

$A_p(h_c)$ is often approximated by a fitting polynomial as shown below for a Berkovich tip:

$$A_p(h_c) = C_0 h_c^{\,2} + C_1 h_c^{\,1} + C_2 h_c^{\,\frac{1}{2}} + ... + C_8 h_c^{\,\frac{1}{128}}$$

Where C_0 for a Berkovich tip is 24.5 while for a cube corner (90°) tip is 2.598. The reduced modulus is related to Young's modulus of the test specimen through the following relationship from contact mechanics:

$$\frac{1}{E_r} = \frac{(1-\nu_i^{\,2})}{\frac{E_i + (1-\nu_s^{\,2})}{E_s}}$$

For a diamond indenter tip, E_i is 1140 GPa and ν_i is 0.07. Poisson's ratio of the specimen ν_s , generally varies between 0 and 0.5 for most materials (though it can be negative) and is typically around 0.3.

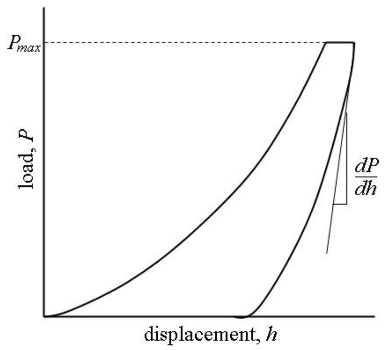

Fig 1. The schematic of load-displacement curve for an instrumented nano-indentation test for a viscoelastic plastic material.

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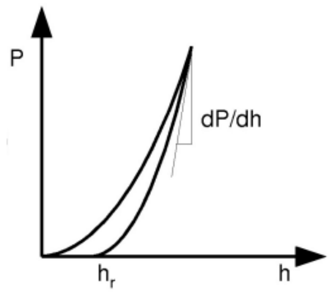

Fig 2. The schematic of load-displacement curve for an instrumented nano-indentation test for an elastic-plastic solid.

II. LITERATURE SURVEY

A brief review of contemporary research supporting this review paper is presented below:

Ch Srinivasa Rao et.al. [1] had applied finite element technique to study the loading-unloading characteristics, stress and strain fields of the bulk materials such as titanium, iron, copper and thin films of titanium and copper subjected to Berkovich Nano indentation process. The loading and unloading curves obtained from numerical simulation results are compared with the curves obtained earlier through the experimental results and a good agreement has been found. The substrate effect is ignored and only thin film behavior under indentation is considered. The thin films are indented within 5% thickness, rather than 10% rule of thumb.

Z. Chen et.al. [2] focusing on the characterization of polymers using Nano indentation, which is dealt with by means of numerical computation, experiments and parameter identification. An analysis procedure is developed using the FEM based inverse method to evaluate the hyper elasticity and time-dependent properties. This procedure is firstly verified with a parameter re-identification concept. An important issue in this publication is to take into account the error contributions in real Nano indentation experiments. Therefore, the effects of surface roughness, adhesion force and the real shape of the tip are involved in the numerical model to minimize the systematic error between the experimental responses and the numerical predictions. The effects are quantified as functions or models with corresponding parameters to be identified.

M. R. VanLandingham et.al. [3] studies the application of instrumented indentation devices to the measurement of the elastic modulus of polymeric materials is reviewed. This review includes a summary of traditional analyses of load-penetration data and a discussion of associated uncertainties. Also, the use of scanning probe microscopes to measure the nanoscale mechanical response of polymers is discussed, particularly with regard to the associated limitations. The application of these methods to polymers often leads to measurements of elastic modulus that are somewhat high relative to bulk measurements with potentially artificial trends in modulus as a function of penetration depth. Also, power law fits to indentation unloading curves are often a poor representation of the actual data, and the power law exponents tend to fall outside the theoretical range. These problems are likely caused by viscoelasticity, the effects of which have only been studied recently. Advancement of nanoindentation testing toward quantitative characterization of polymer properties will require material independent calibration procedures, polymer reference materials, advances in instrumentation, and new testing and analysis procedures that account for viscoelastic and viscoplastic polymer behavior.

III. DISCUSSIONS

A. Load vs Displacement

An indenter usually a Berkovich type is placed in contact with the flat surface of the specimen with a steadily increasing load. Both load and depth of penetration are recorded at each load.

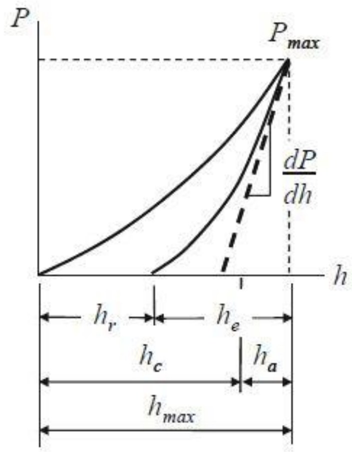

Fig 3. Load vs Displacement

Following the attainment of the maximum load, the load is steadily removed and the penetration depth recorded. The loading part of the indentation cycle may consist of an initial elastic contact, followed by plastic flow, or yield within the specimen at higher loads.

Upon unloading, if yield has occurred, the load-displacement data follow a different path until zero applied loads are reached and residual impression is left in the specimen surface. The maximum depth of penetration for a particular load, together with the slope of the unloading curve measured at the tangent to the data point at maximum load, lead to a measure of both hardness and elastic modulus of the specimen material. In some cases, it is possible to measure elastic modulus from not only the unloading portion, but also the loading portion of the curve.

B. Reduced Shear Modulus

The relaxed shear modulus is given by,

$$G_1(t) = \frac{3}{4}(1-\nu)P_0\frac{1}{h^{3/2}R^{1/2}}$$

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Diagram shows the shear modulus obtained from equation, together with the theoretic shear modulus. It can be seen that the difference between the inputted relaxed shear modulus and the shear modulus calculated is less than 10 percent. Thus, it would be safe to extract the shear modulus using equation at a certain range.

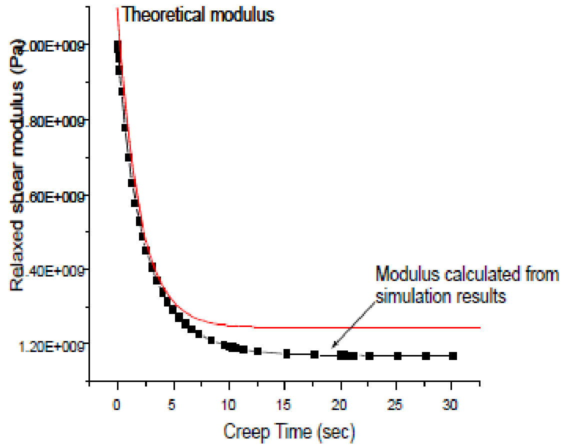

Fig 4. Reduced Shear Modulus

C. Effect of Poisson's Ratio

The changing of Poisson's ratio makes indentation problems more complicated to handle with. The results of two situations are presented in Fig.5. One situation is Poisson's ratio is constant with the value of 0.3, the other is the results of bulk modulus is constant with the initial Poisson's ratio value of 0.3.

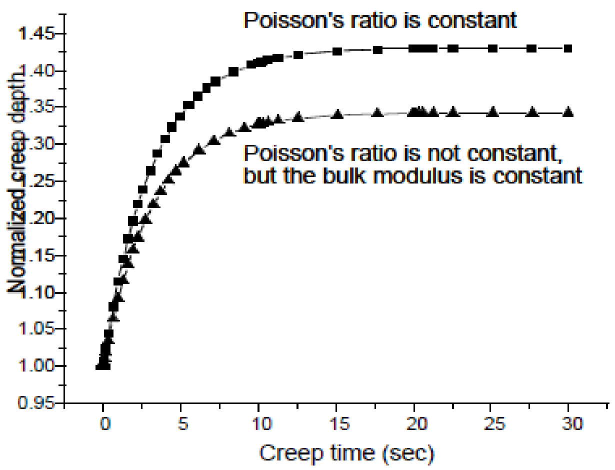

Fig 5. The difference between the normalized creep depths of constant Poisson's ratio and the constant bulk modulus

IV. CONCLUSION

The use of depth-sensing indentation to measure the elastic modulus of polymeric materials was reviewed. Included in this review were discussions of traditional analyses of load-penetration data, the use of depth-sensing indenters and scanning probe microscopes to measure the nanoscale mechanical response of polymers, and the associated uncertainties and limitations of the various nanoscale indentation measurements. The application of these methods to polymers often leads to inaccurate measurements of elastic modulus. For quasi-static indentation, viscoelastic behavior affects the shape of the unloading curve, resulting in modulus values that are high relative to bulk measurements. Attempts to characterize creep behavior during indentation experiments suffer from system limitations (e.g., relatively slow rates of loading compared to the necessary step loading) and potentially inappropriate assumptions regarding the associated stress and strain distributions. While additional complications might arise, dynamic indentation testing has the potential to alleviate many of the problems associated with quasi-static indentation testing. However, a rigorous analysis of dynamic indentation behavior of polymers, particularly with regard to whether linear viscoelasticity holds, has not been reported.

V. FUTURE SCOPE

It is meaningful to quantify the influence of surface roughness on the force displacement data in a more explicit way, which is practical to apply into the experiment or numerical computation as a calibration source.

Comparing work has to be done in order to verify the ability to characterize polymers from nano-indentation. One comparison will be made between the characterization of polymers from indentation performed on different scales, i. e. macro- and nanoindentation, leading to a quantification of the effects related to adhesion and surface roughness, which are sensitive in nanoscale but unimportant in macro-scale.

Nanoindentation experiments are typically carried out on multiple spatial scales, i. e. atomic-scale, nanoscale, microscale and continuum scale. In this case, multiscale simulations combining the greatest advantage of both atomistic and FEM simulations have to be developed in the field of nanoindentation of polymers. Therefore, multiscale simulation from atomistic simulation to finite element computation is our next key task in the research field on nanoindentation of polymers.

VI. REFERENCES

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