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STRESS-STRAIN BEHAVIOR OF NANO/MICRO THIN FILM MATERIALS

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ABSTRACT

Nano/micro size thin films are being developed at present for various applications as stretchable electronic displays, flexible and foldable solar panels, body conformable smart electronic textiles and fabrics, and other embedded sensor-actuator surfaces. This paper describes the results from an experimental investigation of elastic-plastic deformation behavior of nano/micro thick conducting metal such as gold (Au) on a highly compliant, biocompatible polymer such as polydimethylsiloxane (PDMS). Two layer laminates were fabricated with various nano Au film on micron scale PDMS substrate and tested using a micro-tensile tester. Also, *in-situ* scanning electron microscope (SEM) tests captured the digital images of Au film under static load to explain the changes in grain structure of the gold film. Stress-strain data for the gold film under large deformation was extracted using a mechanics of material based model, and changes in stiffness values of the Au film were determined. The experimental results validated the Hall-Petch law indicating that the material strength properties have inverse relationship with grain size. Results indicated that the metallic Au films sustained large deformation without rupture and SEM studies indicated that at higher strain levels, grains in the Au film experienced intra-granular fractures.

Keywords: thin films, mechanics of layered media, metal on polymer.

Notations

F_c, F_{Au}, F_{PDMS}	Force in composite, gold film, and PDMS polymer
A_c, A_{Au}, A_{PDMS}	Cross-sectional area of composite, gold film, and PDMS polymer
σ_c, σ_{Au}, σ_{PDMS}	Stress in composite, gold film, and PDMS polymer
E	Elastic modulus
t_c, t_{Au}, t_{PDMS}	thickness of composite, gold film, and PDMS polymer
ε	Strain in composite

1. INTRODUCTION

The undergraduate courses on engineering materials and mechanics of materials deal with the mechanical properties of bulk materials. Students understand the basic stress-strain relationship in the elastic and plastic range of deformation, and use material strength properties- elastic modulus, yield and ultimate strength, and ductility for design purpose. However, it's not clear if undergraduate students comprehend the stress-strain behavior of multi-layered materials with vastly different elastic modulus and ductility values. This behavior becomes more difficult to explain if the layered materials are in nano/micro scales than in macroscale. The need to understand elastic-plastic deformation, modulus values in the elastic-plastic regime, appearance of shear bands (Luder lines) prior to rupture-failure, and the nature of failure mechanisms has become significantly important as engineering materials in multi-scale are beginning to find new applications in industry. The systematic treatment of thin film fabrication, and conventional as well as noninvasive SEM testing methods for stress and strain in this paper will benefit students and researchers in enhancing their understanding of mechanics of thin film materials under quasi-static uni-axial load.

2. STRESS-STRAIN BEHAVIOR OF LAYERED MATERIALS

Figure-1 illustrates the stress-strain behavior of ductile metals and polymers, each with its own stress and

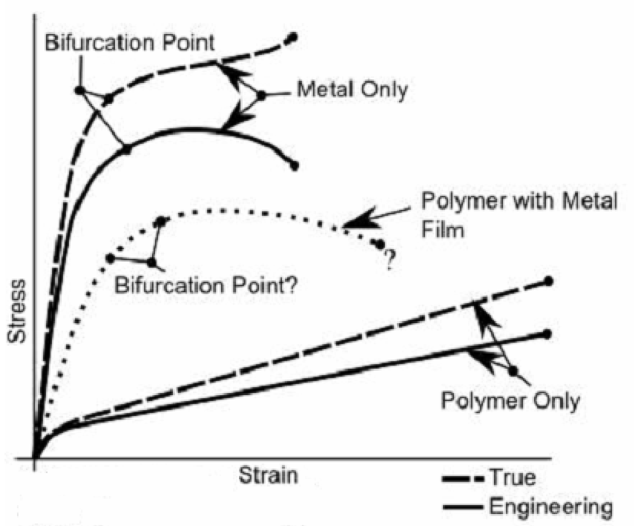

Figure-1. Stress-strain behavior of ductile metals and polymers.

strain characteristics: metals with high stiffness, easily identifiable yield and ultimate strength points, unstable plastic instability (bifurcation) point associated with large strains, whereas plastics have large strain and low stiffness values. For the combined metal and polymer test specimens with vastly different stiffness values, a speculated deformation behavior is indicated in the Figure with question marks. The range of strain, stiffness values, appearance of bifurcation point, and the grain size effects are not well understood if the test specimens are of multilayer metal on polymer. This paper describes the nature of elastic and plastic deformation of nano gold (Au) film on a highly compliant polymer, PDMS.

3. RELATED RESEARCH

The mechanical response of flexible, multilayered thin films is important because they are beginning to find applications as flat paper like displays [1, 2], sensitive skins [3], embedded stretchable electronic surfaces [4, 5], flexible solar cell panels [6], and sensitive membrane skins [7], to name a few interesting devices. These devices will be designed to withstand static and dynamic loads in daily use and hence the study of stress-strain behavior of thin films at nano/micro scale is appropriate and timely for mechanical, electrical, material science majors.

Many researchers are actively pursuing this research currently to characterize the mechanical response of flexible electronic thin film material structures. Majority of them deal with analysis and point out to the need for experiments. A few notable contributions include: Hill and Hutchinson defined the plastic instability (bifurcation) point [8], Hutchinson and others [9, 10, 11] attempted to explain the failure modes in stiff, thin films, Storen and Rice [12] explained local necking in single films, Nix [13], Tvergaard and Needleman [14] and Yu and Spanean [15] studied large strain localization in thin films, Pashely [16] used scanning electron microscope for observing grain structure, Xiang, Li, and Vlassak [17] studied Cu on stiff substrates, Cairnes, Crawford., *et al*., [18] analyzed TiO on PET for resistance changes under load, Li and Suo, and others [19, 20, 21] used analytical, finite element numerical simulations for large strain analysis, Swaminadham Midturi, *et al*., [22], and Onobu, A., *et al*., [23] conducted experiments to explain damage mechanisms of Au + PDMS films under quasi-static loads. R. A.C. Slater [24] offers an excellent description for the elastic and plastic deformation and stress and strain relationships for single layer bulk metallic materials under static loads in his textbook, and it can serve as a helpful tool in formulating analytical models for elastic-plastic response due to mechanical loads.

4. SCOPE OF THIS RESEARCH

The primary objective of this paper is to explain the stress and strain behavior of nano/micro scale Au + PDMS thin film materials under tensile load and to validate the Hall-Petch law, which states that the material strength varies inversely with grain size. The paper will explain, in brief, film preparation, micro tension method to establish stress-strain graphs, and a method to extract the stress and modulus values of gold film. The paper will also describe the changes in Au film grains due to mechanical load using scanning electron microscope (SEM).

The reason for choosing Au on PDMS is: PDMS is a biocompatible material and gold is an excellent conductor of electricity and together they can form a bio-sensor to detect body temperature and pressure. This and similar sensors can become integral part of smart fabrics and e-textiles with embedded sensor and receiver arrays. This research is a prelude to explain how a conducting metal on highly compliant polymer at nano/micro scale would behave under quasi-static tensile load.

5. EXPERIMENTS

Experiments were conducted to analyze the elastic-plastic deformation of 50, 75, and 100 nano-meter (nm) thick Au films (E, Young's modulus or stiffness constant, 79 GPa) on highly compliant 1000 micron (μm) polymer PDMS (E of 2 M Pa) substrate. Test specimens were tested up to 60%.

5.1. Preparing test specimens

The 1000 μm PDMS substrates were prepared by mixing a pre-polymer (Slygard 184) with a curing agent in a 10:1 ratio at room temperature, the mixture was cast into a dog bone shape mold, and was cured for 12 hours at a temperature of 60 ˚C. The polymer was then deposited with a 10 nm thick chromium film to make gold adhere well to the substrate. Films of Au with thickness- 50, 75, and 100 nm were electron beam deposited on PDMS using an E-Beam Evaporator. Full details of the film deposition and preparation are discussed in [23]

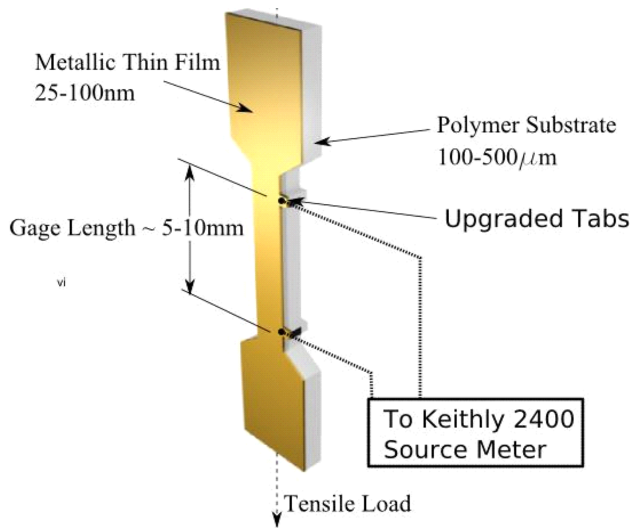

Figure-2. Thin film and substrate tensile specimen.

5.2. Testing specimens under tension

The dog bone shaped PDMS+ Au test samples shown in Figure-2 were tested in an Instron Micro tensile tester at a constant strain rate of 0.001 mm/s. Motion of the cross- head measured the change in gage length and hence the strain. A high precision load cell with a resolution of milli-Newton measured force on the test specimens and hence the stress.

In addition to Instron tension tests, in-situ SEM tests using an embedded nano-manipulator in Philips SEM were conducted to observe changes in the grain structure of gold film. Figure-3 explains the basic principle of

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operation. It's to be mentioned here that the test specimens' geometry and loads in SEM experiments are identical to Instron micro-tests. Digital images of Au film were captured in synchronism with load. Present grain change interpretations are considered qualitative, although the results indicated significantly useful information on the initiation of failure mechanisms of gold conducting film with increasing strain.

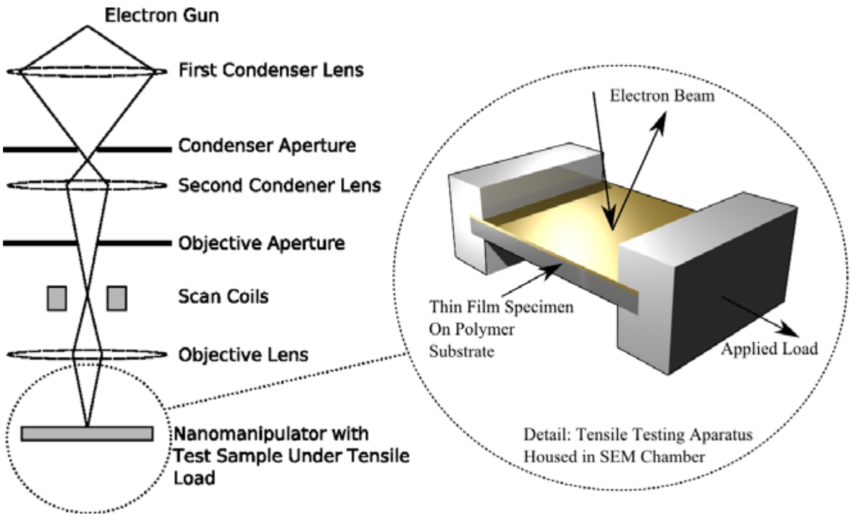

Figure-3. In-situ SEM setup to observe grain changes in Au film.

5.3. Extracting stress-strain data from measurements

Since test specimens are nano/micro thick, attaching a clip on strain indicator was not practical, and so crosshead motion of Instron tester was used to find strain. From the measured force in mN by the load cell and measured distance in (μm) of the crosshead, stress and strain data of test specimens was extracted. Details of the procedure [22] for extracting the stress-strain data, in brief, are:

The applied force on the laminate can be written as

$$F_c = F_{Au} + F_{PDMS} \quad (1)$$

Where

$F_{Au} = \sigma_{Au} A_{Au}$
for the gold film, and
$F_{Pdms} = \sigma_{PDMS} A_{PDMS}$
for the polymer. Here, σ is the stress and A is the area of cross-section.
The tensile stress in the entire laminate is

$$F_c/A_c = \sigma_c = (\sigma_{Au}\, tAu / t_{total} + \sigma_{PDMS}\, t_{PDMS} / t_{total}) \quad (2)$$

In equation (2), σ_c is the stress in the two-layer film, σ_{Au} is the stress in the Au film, σ_{PDMS} is the stress in the PDMS substrate, t_{Au} is the thickness of the Au film, t_{PDMS} is the thickness of the PDMS substrate, and t_{total} is the total thickness = $tAu + t_{PDMS}$.
Eq. (2) yields the stress in Au as,

$$\sigma_{Au} = (\sigma_c - \sigma_{PDMS}\, t_{PDMS} / t_{total}) / (t_{total} / t_{Au}) \quad (3)$$

In equation (3), we use $\sigma_{PDMS} = (E \times \varepsilon)$ where E is the elastic modulus of the PDMS substrate and ε is the applied strain.

6. RESULTS AND DISCUSSIONS

Measured stress-strain data was stored by Instron computer in Excel format and the stress and strain information of the gold film was extracted using equation (3). Plots of stress on y-axis in MPa and strains on x-axis for 50, 75, and 100 nm thick Au films were plotted. Slope of the stress-strain curve in the entire strain range is derived from the curve fit. Stiffness or modulus values of 24.5, 6.1, and 1.97 M Pa for the Au film correspond to 50, 75, and 100 nm, respectively. This trend clearly establishes Hall-Petch law. Figures in 4a, 4b, and 4c show the stress-strain behavior of the Au + PDMS laminates. Although there is a considerable plastic deformation involved in the laminate, it's difficult to identify the bifurcation point from these plots.

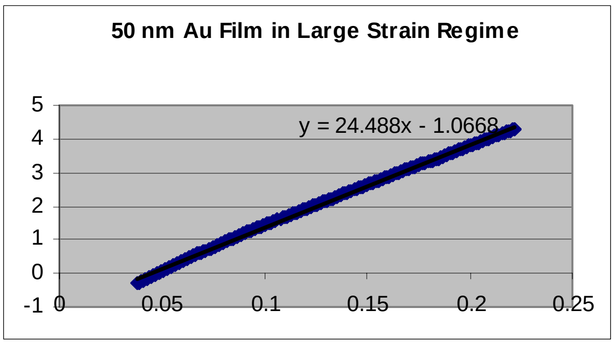

Figure-4a. Stress (MPa) and strain behavior of 50 nm Au film.

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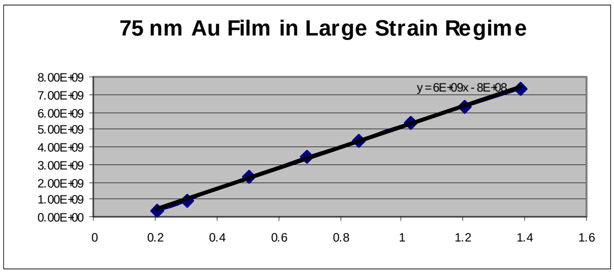

Figure-4b. Stress (MPa) and strain behavior of 75 nm Au film.

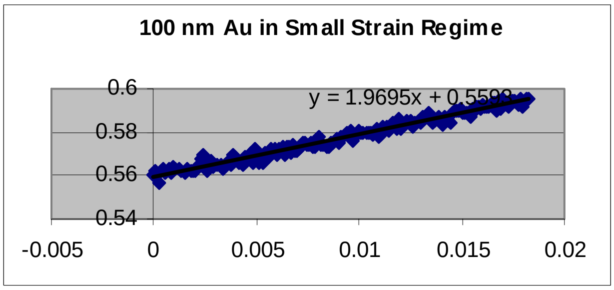

Figure-4c. Stress (MPa) and strain behavior of 100 nm Au film.

Figure-5 shows the effect of grain size on the stiffness of Au. As the grain size increases, stiffness value decreases, validating the Hall-Petch law. This investigation demonstrates that the Hall-Petch law is also valid for two-layer thin film materials.

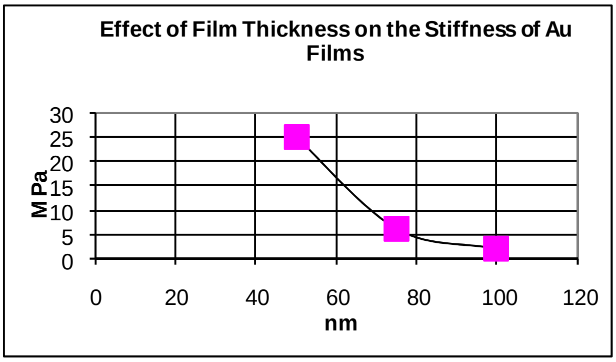

Figure-5. Hall-Petch law validation: stiffness value vs. grain size in Au film.

Figure-6 shows the SEM images of grain structure of the gold film. These images clearly show that the grains at near zero strain are larger and as the load or stress is increased, the grains begin to fracture, and at large strains (~ 60 %), the grains have become much smaller indicating that the intra-granular fracture is the dominant failure mechanism. It is interesting to note that although the metal film strained up to 60%, it never completely broke. The polymeric substrate provided this extra ductility for the laminate.

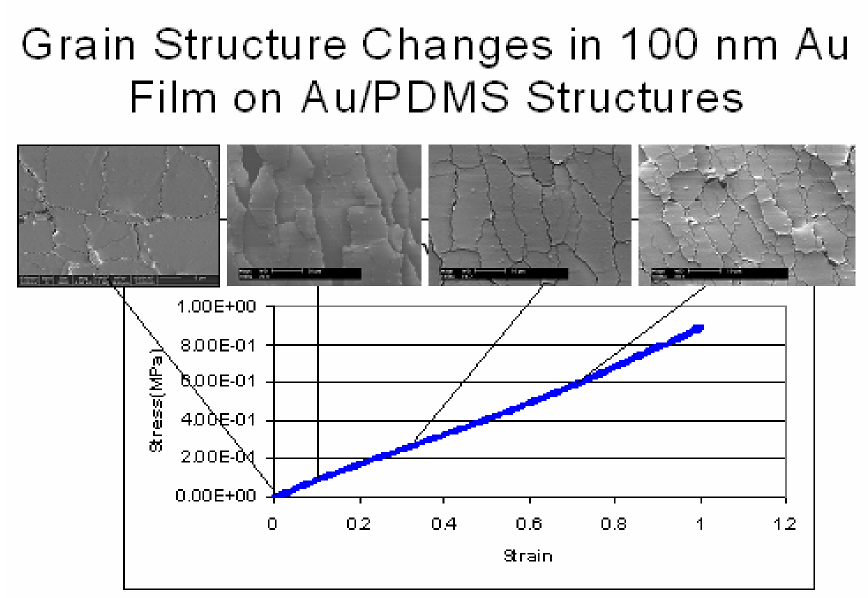

Figure-6. Scanning electron microscope in-situ images of changes in Au film grain structure.

7. CONCLUSIONS

This paper examined the stress and strain behavior of nano size Au films on highly compliant thick PDMS substrates. The data indicated that the Au+PDMS could sustain large deformations without rupture in metallic (Au) films. The stiffness of the Au film was modified and it decreased compared to its bulk material modulus value when the Au-PDMS laminate was stretched to large strains. Stiffness (modulus) of Au films was dependent on the thickness of the film and the experimental results indicted that the stiffness of Au films was higher for 50 nm Au film compared to 100 nm film.

These tests validated the Hall-Petch theory for two layer media. Changes in grain structure observed through *in-situ* SEM tests in Au films exhibited intra-granular fractures with increasing strains. The future research will construct theoretical model(s) to determine the plastic instability, and determine modified stiffness and hardening index for the laminate and metallic Au films.

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