Optical Materials 85 (2018) 153–161

Contents lists available at ScienceDirect

Optical Materials

journal homepage: www.elsevier.com/locate/optmat

Investigating the mechanical and optical properties of thin PDMS film by flat-punched indentation

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ARTICLE INFO

Keywords:
Flat-punched indentation
PDMS thin film
Stress-strain
Total internal reflection
Microscopic imaging
Refractive index
Finite element analysis

ABSTRACT

We investigated mechanical and optical properties of a thin polydimethylsiloxane (PDMS) film through a flat-punched indentation experiment and a finite element simulation. A microscopic imaging method was used to measure the compressive strain of thin PDMS films, and its corresponding bulk refractive index (RI) was estimated using the relation between density and refractive index. A total internal reflection (TIR) experiment was conducted to estimate the local RI values near the bottom surface of PDMS film. We obtained the correlations between force and indentation displacement in thin PDMS films (10–70 micron). Stress-strain curves present the non-linear deformation of PDMS film with the instantaneous modulus depending on the load and the thickness. Poisson's ratio of PDMS film is estimated by fitting with Yang's asymptotic solution and is inversely proportional to the aspect ratio between the radius of the flat punch probe and the film thickness. RI of PDMS film increases with the decreasing film thickness as well as the increasing load. The bulk RI was increased up to 1.476 by the compressing load while the bottom-surface RI was increased only up to 1.435. This difference is explained qualitatively through the localized stress-strain distribution from finite element analysis. An inhomogeneous stress-strain distribution is observed in the simulation with lower strain at the bottom of the film, corresponding to experiment. A lower strain leads to lower local density resulting in a lower refractive index. Our research provides fundamental insights into the correlation between mechanical and optical properties of thin polymer films.

1. Introduction

Understanding the stress distribution in a microstructure is very significant for the development of microelectromechanical system (MEMS) devices such as flexible displays, Li-ion battery, tunable optics, or micro actuators [1–4]. Recently, MEMS devices are becoming smaller, thinner, and more complex with multi-layer structure or a shortened gap between elements to achieve high performance within the limited volume. This compact design can cause the malfunction in operation, provided a mechanical stress is exerted on the system. The mechanical stress can come from external causes, e.g., the bending of flexible organic light-emitting diode (OLED) display [5] or the swelling due to the heat generation of Li-ion battery [6–8]. In more severe cases, the constraint stress can result in a crack at the substrate layer of system [9,10]. From these reasons, it is necessary to investigate the internal stress distribution of microscale thin film structure for the development of high performance MEMS device.

The mechanical stress induces the deformation of physical dimension of film layer and it thus can affect the internal density field of the film [11]. In general, the density change of material results in the alteration of refractive index, which can be elucidated by using molar refractivity such as Lorentz-Lorenz equation [12–14]. Based on this correlation, the internal mechanical properties can be estimated by experimentally measuring optical properties. Researchers tried to develop optical method to measure mechanical stress distributed in material [15,16], and someone studied about the influence of mechanical stress on optical properties of materials [17–19]. A large number of studies about the stress-induced optical properties have been devoted mostly to macro-sized samples [11,20,21] and by a simulation approach [22,23]. In addition, most of the mechanical stresses in the previous studies are with tension force [24–26]. Only a few works introduced optical and mechanical properties for micro-scale specimen [27]. It is very rare to find experimental studies for microscale specimen subjected to compressive stress. In this paper, we investigate the difference between bulk and near-surface values in the optical, physical properties of micro thin film subjected to compressive indentation load

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https://doi.org/10.1016/j.optmat.2018.08.051
Received 26 May 2018; Received in revised form 18 August 2018; Accepted 19 August 2018
0925-3467/ Published by Elsevier B.V.

by using optical manners.

We apply two optical measurements to estimate bulk and local values of refractive index of thin film. First, the near-field optical method based on total internal reflection (TIR) is employed to measure critical TIR angles, which are related with the local refractive index around the bottom surface of thin film. Second, the far-field imaging with a regular bright field microscope is employed to measure the film's bulk deformation, and then the bulk refractive index is estimated using molar refractivity. Through the comparison between bulk and local values of refractive indices, we estimate the variation of density field inside film subjected to compressive stress. To verify experimental data, we conduct finite element simulations showing the distributions of internal stress and density in thin film.

2. Theoretical background

2.1. Flat punch indentation

The flat punch indentation of a compressible elastic film bonded to a rigid substrate was previously studied using asymptotic analysis by Yang [28]. When the contact radius (a) of the indenter is much larger than the film thickness (h), the indentation load (F) is proportional to the indentation depth (δ), contact area (A), and elastic properties of film while it is inversely proportional to the film thickness as follows:

$$F = \frac{\pi\delta a^2(\lambda + 2G)}{h} = \frac{(1-\nu)\delta AE}{(1+\nu)(1-2\nu)h}. \tag{1}$$

Young's modulus (E) of sample is determined with Poisson's ratio (ν) and effective modulus (E_{eff}) which as follows:

$$E = [1-\nu^2]E_{\mathrm{eff}}. \tag{2}$$

This simple relation is employed to indentation modeling when the Young's modulus of the rigid substrate is much greater than that of sample. For the indentation involving large deformation, the effective modulus is defined with the stiffness (S) and the non-linear factor (κ) of sample [29] as follows:

$$E_{\mathrm{eff}} = \mathrm{S}/2a\kappa. \tag{3}$$

Instantaneous stiffness is defined as the derivative of force with respect to displacement ($S = \mathrm{d}F/\mathrm{d}\delta$). The value of κ is estimated using the formula introduced in Zhang et al.'s paper [30].

2.2. Relation between density and refractive index

The relation between the refractive index and the mass density of a substance was developed from the correlation of two polarization equations induced with molecular polarizability and dielectric constant as follows [12,13]:

$$\frac{(n^2-1)}{4\pi + b(n^2-1)} = \frac{N_A\cdot\alpha}{M}\rho, \tag{4}$$

where n is refractive index, N_A is the universal Avogadro's number, α is volume molecular polarizability, ρ is density, and M is the molecular weight of the given material. By setting $b = 4\pi/3$, the most often used formula, Lorentz-Lorenz (L-L) relation is given [12,13] as follows:

$$\frac{n^2-1}{n^2+2} = \frac{4\pi}{3}\frac{N_A\alpha}{M}\rho. \tag{5}$$

Based on the assumption that the polarizability of material is independent of its density, the fractional term of '$4\pi N_A\alpha/3M$' can be replaced by a constant 'k', and the L-L relation is simplified as:

$$\frac{(n^2-1)}{(n^2+2)}\frac{1}{\rho} = k. \tag{6}$$

From the above simple relation, we presume that the refractive index is a function of density as follows:

$$n = \sqrt{\frac{2k\rho+1}{1-k\rho}}, \tag{7}$$

where the constant k is obtained from Eq. (6) with the referred values for the refractive index and density at the unloaded, normal condition. From the above equation (7), the final value of refractive index after compression can be determined with the final density of material that is estimated using strain data. More details will be described in the result section 4.4.

Dislike the direct estimation of final density using Eq. (7), the refractive index change can be applied to determine the final value of refractive index. By differentiating the refractive index n in Eq. (4) with respect to density ρ, the relation between refractive index change (Δn) and density change ($\Delta\rho$) can be expressed as follows:

$$\frac{\Delta n}{\Delta\rho} \approx \frac{dn}{d\rho} = \frac{(n^2-1)\cdot(4\pi + bn^2 - b)}{8\pi n\rho}, \tag{8}$$

where this differentiation is based on the assumption that the polarizability in Eq. (4) is independent of density. The density change rate ($\Delta\rho/\rho$) of the sample can be obtained in terms of the original and final volumes as follows:

$$\frac{\Delta\rho}{\rho_o} = \frac{\rho_f}{\rho_o} - 1 = \frac{V_o}{V_f} - 1, \tag{9}$$

where ρ_o and V_o are the original density and volume of the sample before compression, and ρ_f and V_f are the final density and volume at the end of compression, respectively. Then the refractive index change is calculated by the following equations:

$$\Delta n = \frac{(n^2-1)\cdot(4\pi + bn^2 - b)}{8\pi n}\left(\frac{\rho_f}{\rho_o} - 1\right), \tag{10}$$

$$\Delta n = \frac{(n^2-1)\cdot(4\pi + bn^2 - b)}{8\pi n}\left(\frac{V_o}{V_f} - 1\right), \tag{11}$$

where n is the original refractive index of the given material before compression. The index change Δn is dependent on the value of constant 'b', e.g. $b = 0$ according to Drude formula, $b = 0.5$ according to the empirical data of Ritland [12], $b = 2.1$ according to Gladstone-Dale (G-D) formula, and $b = 4\pi/3$ according to Lorentz-Lorenz (L-L) formula.

3. Experimental

3.1. Chemical, mechanical properties of PDMS material

We used a commercial PDMS elastomer named *Sylgard* 184 (Dow Corning) which consists of two components, a base (Part A) and a curing agent (Part B). The base contains poly-dimethylsiloxane ($-[\mathrm{OSi(CH_3)_2}]_x-$) polymer having a silicon vinyl group ($\mathrm{SiCH{=}CH_2}$), and the curing agent contains a different PDMS polymer having silicon hydride groups (SiH). Through the mixing of both components, two functional groups are bonded by hydrosilation reaction that is catalyzed by platinum and form $\mathrm{Si{-}CH_2{-}CH_2{-}Si}$ linkage [31]. The multiple reaction allows three-dimensional cross-linking structure. The typical chemical formula of base is $\mathrm{CH_2{=}(CH)\ (CH_3)_2Si-[OSi(CH_3)_2]_x-\ OSi(CH_3)_2(CH) = CH_2}$ with the average number of $x = 60$ [32]. The curing agent has a chemical formula $\mathrm{(CH_3)_3Si-[OSiH(CH_3)]_m-[OSi(CH_3)_2]_n-OSi(CH_3)_3}$, where $m = 3$ and $n = 7$ in average [32].

The value of b in Eq. (4) can be specified by estimating a molecular weight M and a molecular polarizability α. The molecular weight M is approximately $\{348.82 + 74.16\ (x + n) + 60.13\ \mathrm{m}\}$ g/mol based on the chemical formulas of base and curing agent. With $x = 60$, $m = 3$ and $n = 7$, M of the PDMS cross-linking is estimated as 5498 g/mol. The molecular polarizability α is obtained from the sum of polarizabilities of all atoms composing of the cross-linked compound. The polarizability

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