The research of nanoindentaion-ultrasonic measurement technique

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Abstract

In this paper, the nanoindentation and ultrasonic measurement systems are successfully integrated to characterize the mechanical properties for TFT-LCD glass. The results indicated that the measured hardness and reduced modulus are reasonably obtained while the penetration depths are deep enough. Furthermore, the Young's modulus and Poisson's ratio of TFT-LCD glass can be simultaneously obtained by the proposed methodology.

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1. Introduction

Since the thin film transistor liquid crystal display (TFT-LCD) has the attractive merits such as the light-weight, economized use of power as well as the less radiation, it has been more and more applicable in the notebook, PC monitor, TV, etc. Within the TFT-LCD, the back light unit (BLU) and the liquid crystal display module (LCM) are the major components. The function of BLU is to provide the uniform luminance and it composes of cold cathode fluorescent lamp (CCFL), diffuser, light guide plate, etc. The function of LCM is to control the display color and it makes from the glass, polarizer, liquid crystal, etc. Because TFT-LCD is frequently subjected to thermal and/or mechanical loads during either the manufactured or operation circumstances, the reliability of TFT-LCD is considerably dependent upon the mechanical properties of the constituent materials [1-2]. Generally specking, TFT-LCD are classified into different generations according to the dimensions of the glass used in TFT-LCD. Prior knowledge of mechanical properties of glass is an important issue as the dimensions of TFT-LCD become larger. For this reason, in this paper, a methodology was proposed to characterize the mechanical properties of TFT-LCD glass.

Traditionally, it is necessary to manufacture the tested specimen into the specific configuration when the experiment of determination of Young's modulus and Poisson's ratio for glass is performed [3]. On the other hand, owing to the conventional prepare of experiment, the nanoindentation method has been widely used to characterize the mechanical properties in various categories [3-6]. The experiment of nanoindentation method is only to employ an indenter with the known properties to penetrate into the surface the tested material. The indentation hardness and reduced modulus can be obtained through recording the experimental data as well as the contact theory. However, it should be emphasized that the reduced modulus is not the Young's modulus of the tested materials but a relationship between the Young's modulus and Poisson's ratio. If the Young's modulus would want to known form the nanoindentaion method, the Poisson's ratio has to guess in advance. As the assigned Poisson's ratio is considerably different from that of real, the Young's modulus will result in the significant error. To overcome this crucial drawback of nanoindentation method, in this paper, the nanoindentation-ultrasonic measurement system that integrates the nanoindentation and ultrasonic measurement techniques is proposed. The Young's modulus and Poisson's ration could be simultaneously estimated from the proposed methodology.

2. Theory of nanoindentation [3]

Regarding nanoindenation measurement, an indenter with the known mechanical properties is employed to penetrate into the tested material while the load as well as the penetrated depth are simultaneously recorded during the indentation process. Typical configuration of indentation and the curve of applied versus displacement for loading and unloading are illustrated in Fig. 1. Based on the distinct behaviors for different materials in conjugation with the theory of contact mehcanics, the hadrnss and reduced modulus can be obtained. The definition of indentation hardness is as follows:

$$H = \frac{P_{max}}{A} \tag{1}$$

where P_{max} is the maximum load and A is the projected area. The unloading behavior had been show to be described appropriately by the power-law relationship, i.e.

$$P = \alpha (h - h_f)^m \tag{2}$$

where P is the applied load, h is the total displacement, h_f is the residual depth after removing the applied load, α is the constant, and m is the order of the power-law equation. According to the theory of contact mechanics, it gives

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A}} \tag{3}$$

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S} \tag{4}$$

where $E_r = \dfrac{1-\nu_1{}^2}{E_1} + \dfrac{1-\nu_2{}^2}{E_2}$ is the reduced modulus, ν_1 and E_1 are the Poisson's ratio and Young's modulus of indenter, ν_2 and E_2 are the Poisson's ratio and Young's modulus of the tested material, and ε is the geometrical constant of indenter. The contact stiffness (S) is the slope of unloading curve while the maximum load is applied. Thus,

$$S = \frac{dP_{max}}{dh_{max}} = m\alpha \left(h_{max} - h_f\right)^{m-1} \tag{4}$$

Because the radius of indenter is rather small, the blunting appearance in the immediate vicinity of indenter tip shall be modified. To account for its influence on the contact area, the following equation can be used to describe the contact area.

$$A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16} \tag{5}$$

Substitute Eqns. (4)-(5) into Eqn. (3), the reduced modulus of tested materials can be obtained.

From the aforementioned description, only the reduced modulus, i.e. the relationship between the Young's modulus and Poisson's ration, is provided from the nanoindentation experiment. If the Young's modulus and Poisson's ratio would want to be separated, another independent relationship must be provided. Hence, in this paper, the nanoindentation system is integrated with the ultrasonic technique such that the Young's modulus and Poisson's ratio can be individually obtained.

3. Theory of ultrasonic technique [7]

The experiment of ultrasonic technique is first to utilize a transducer to generate the ultrasonic waves within the medium. Generally, the ultrasonic waves can be classified into two categories, i.e. body wave and surface wave. The former wave includes the longitudinal wave and transverse wave and they respectively propagates across the thickness direction and along the surface of the tested material. Because the energy of surface wave easily decays, the longitudinal wave was generated in this paper to incorporate with the nanoindentation technique. According to the theory of wave motion, it gives

$$E = \frac{(1+\nu)(1-2\nu)}{(1-\nu)} \rho c_L^2 \tag{6}$$

where ρ is the density and c_L is the longitudinal wave velocity. This equation provides an independent relationship between the Young's modulus and Poisson's ration with respect to the nanoindentation result.

4.Experiment

4.1 Specimen preparation

The nanoindentation measurement system (TriboIndenter, Hysitron Inc., U.S.A.) was integrated with ultrasonic measurement technique (Fig. 2) to simultaneously measure Young's modulus and Poisson's ratio of the TFT-LCD glass. Prior to the experiment, a piece of glass flake was cut. A micrometer (CD-6"BS, Mitutoyo Co., Japan) was utilized to measure its dimensions and they are 30.06 (mm)×100.10 (mm)×0.7 (mm). An electronic balance (AT21, Mettler Toledo Inc., Switzerland) that can be traceable to SI unit was used to measure its mass and it is 5.24 (g). Therefore, the density of the glass is 2495.2 (kg/m^3). Then the glass was placed on the specimen platform of Fig. 2.

4.2 Nanoindentation experiment

After the preparation of specimen, the nanoindentation experiments are sequentially carried out. The Berkovich indenter was employed in the nanoindentation experiments and its constituent material is diamond and therefore its Young's modulus and Poisson's ratio are 1140 GPa and 0.07, respectively. Moreover, three dissimilar modes in nanoindentation experiments, i.e. open loop, loading control and displacement, are successively performed. The open loop mode means that the indentation experiments are progressed without feedback control function. The experimental models for loading and displacement controls are both proceeded with the aids of the feedback control function but the corresponding load and displacement in the experiments are controlled respectively in the former and latter modes. To investigate the results from shallow to depth indents, the applied load are successively selected from 200 μN to 7000 μN both for the open loop and loading control modes and the maximum penetration depth are varied from 40 nm to 270 nm for the displacement control mode. The experimental schemes are listed in Table 1 and four experiments are indented for each condition at the different positions to understand their reproduction.

4.3 Ultrasonic experiment

The experimental arrangement of ultrasonic measurement system is depicted in Fig. 2. The ultrasonic experiment is aimed to measure the longitudinal wave velocity in Eqn. (6) so that an independent relationship between Young's modulus and Poisson's ratio can be acquired. First, to make the ultrasonic wave excited by the transducer with 50 MHz (V215-BB, Panametrics Inc., U.S.A.) easily transmits into the glass flake, an appropriate amount of couplant (Propylene Glycol, Panametrics Inc., U.S.A.) was smeared on the surface of the glass flake. The transducer was placed and appropriately pressed against the couplant surface. The pulse signals are periodically provided by the pulser/ receiver (Model 5900 PR, Panametrics Inc., U.S.A.) and send to the transducer. Then the ultrasonic wave are generated by the transducer and propagated along the thickness direction of the tested specimen. The ultrasonic wave would reflect while the ultrasonic wave arrives at the bottom surface of the tested specimen. The reflectively wave can be also detected by the same transducer and the time domain signal can be observed on the oscilloscope (TDS-2002, Tektronix Co., U.S.A.). Since the thickness of the glass is known, the longitudinal wave velocity is ready to calculate through the time interval between the wave peaks displayed on the oscilloscope.

5. Results and discussions

Regarding the nanoindentation experiment, Fig. 3 is the topography of the residual imprint after the indenter is withdrawn. As can be seen from the figure, even though the pile-up phenomenon occurs in the immediate vicinity of residual imprint but this behavior is not obvious. Therefore, it demonstrates that the TFT-LCD glass is appropriate to measure its properties by means of nanoindentation method.

The experimental results of nanoindentation measurement system are shown in Fig. 4. As can be observed from Fig. 4(a), in spite of the indentation modes used in nanoindentation experiments, the measured magnitudes and their uncertainties of hardness are dramatically increased while the penetration depths are smaller than 50 nm. Notice that the function in Eqn. (5) is utilized to account for the influence of blunting phenomenon around the indenter tip on the projected area. In this equation, the orders of the first two terms are either equal to or greater than one and, on the other hand, otherwise are smaller than one. The property of this function expresses the magnitudes of projected area in Eqn. (5) are dominated by the first two terms under deeper penetration depth. When the penetration depth is shallow, the magnitude of Eqn. (5) may be fluctuated and the magnitude of projected area will be dominated by other terms. Consequently, it is easily to understand that nanoindentation results may be unreasonable under the shallow depth. This is the reason to cause the magnitudes and uncertainties of hardness significantly increase under the shallow depths. Furthermore, the measured magnitudes of hardness obtained from different indentation modes are in good agreement while the nanoindentaion experiments are performed under the deeper penetration depth. For the sake of comparison between different indentation modes, the measured magnitudes of hardness are averaged under the penetration depths greater than 125 nm to represent the obtained hardness from different indentation modes. The hardness indented from displacement control, loading control and open loop are 8.19 GPa, 9.21 GPa and 8.86 GPa, respectively.

Fig. 4 (b) is the experimental results of reduced moduli obtained from different indented modes. As similar to those of hardness measurements, the values of reduced moduli and uncertainties are dramatically increased when the penetration depths are shallow. This is also resulted from the identical reason as in the hardness discussion. The representative of reduced modului obtained from different indented modes can be attained through averaging the measured values of reduced moduli under the penetration depth deeper than 125 nm. The reduced moduli obtained from displacement control, load control and open loop are 79.62 GPa, 83.27 GPa and 81.50 GPa, respectively. The three values for reduced moduli represent the three distinct relationships between Young's modulus and Poisson's ratio from different indented modes. Three relationships will be successively combined later with the ultrasonic measurement results such that the Young's modulus and Poisson's ratio can be individually obtained.

The time domain signal detected from ultrasonic measurement system is in Fig. 5. The aim of ultrasonic experiment is to calculate the longitudinal wave velocity in Eqn. (6) so that another relationship between Young's modulus and Poisson's ratio of TFT-LCD glass could be provided. The time interval, Δt, between the peaks where is indicated in Fig. 5 is first measured and it is 0.232 μsec. The longitudinal wave travels along the thickness of glass there and back for one time and therefore the longitudinal wave velocity can be calculated to be 6034.4 (m/sec). Substitute this value as well as the magnitude of glass density into Eqn. (6) and another relationship between Young's and Poisson's ratio is obtained. Plot this relationship and the antecedently obtained relationships indented in different modes from nanoindentation experiments in Fig. 6. The intersection points in the figure indicate the results of Young's and Poisson's ratio measured by the proposed theory in this paper. After some calculation, the corresponding Young's modulus and Poisson's are 74.24 GPa and 0.26

for the displacement control, 79.09 GPa and 0.22 for the Loading Control and 76.69 GPa and 0.24, respectively.

6. Conclusions

In this paper, the nanoindentation and ultrasonic measurement systems are successfully integrated to characterize the mechanical properties of TFT-LCD glass. As can be observed from the experimental results, both the hardness and reduced modulus of TFT-LCD glass measured by nanoindentation system are unbelievable under the shallow depth regardless of displacement control, loading control or open loop is performed. Furthermore, the magnitudes of hardness indented from different modes are similar and dependable while the penetration depths are deep enough. On the other hand, the Young's modulus and Poisson's ratio of TFT-LCD glass are obtained by combining the nanoindentation and ultrasonic measurement results. The results from different indented modes are also similar when the penetration depths are enough.

7. References

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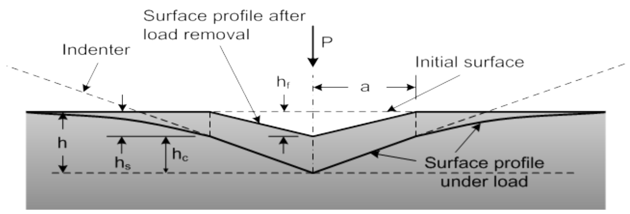
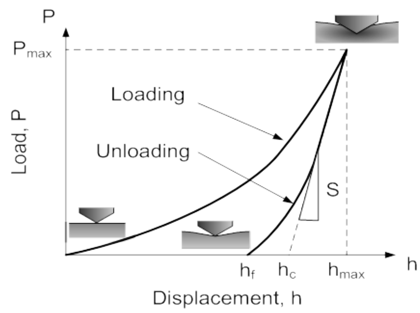


(a) Typical configuration of indentation



(b) Typical loading and unloading curve

Fig. 1 Typical configuration of indentation and the loading and unloading curve

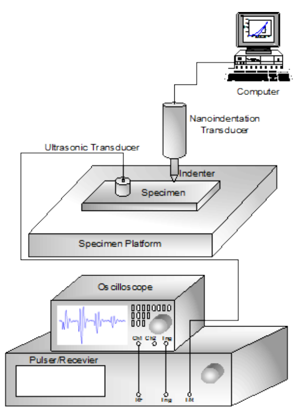


Fig. 2 The schematic of the experimental configuration

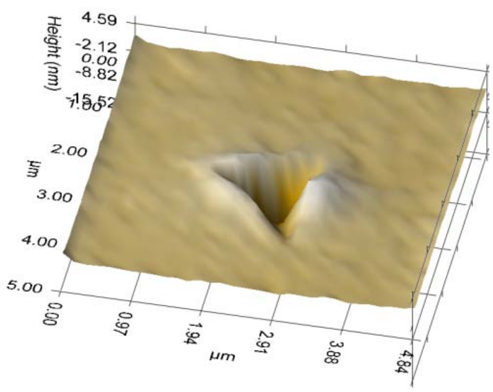
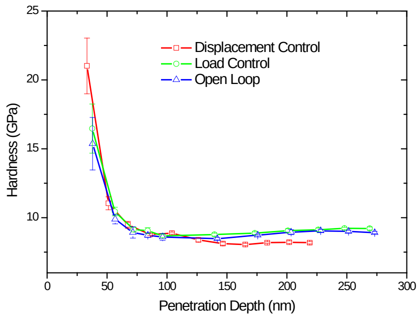
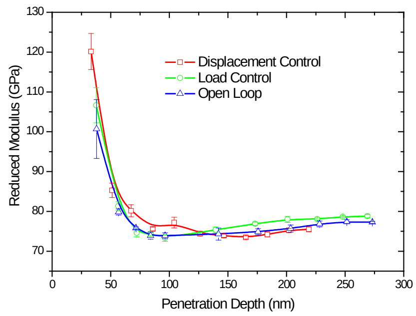


Fig.3 Topography



(a) Hardness



(b) Reduced modulus

Fig. 4 The experimental results of nanoindentation measurement system

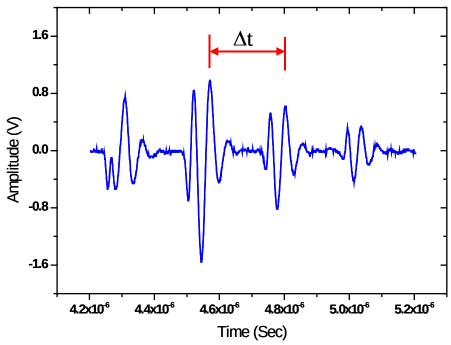


Fig. 5 The experimental results of ultrasonic method

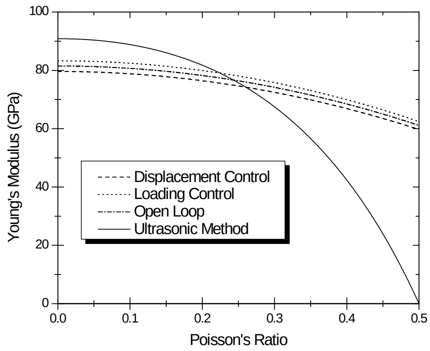


Fig. 6 The relation between Young's modulus and Poisson's ratio from different approaches

Table 1 The experimental schemes for nanoindentation experiments

Conditional no. / Modes	1	2	3	4	5	6	7	8	9	10	11
Open Loop (μN)	200	400	600	800	1000	2000	3000	4000	5000	6000	7000
Loading Control (μN)	200	400	600	800	1000	2000	3000	4000	5000	6000	7000
Displacement Control (nm)	40	60	80	100	120	145	170	195	220	245	270