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Indentation experiment and analysis on mold and resin material during the nanoimprint process †

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Abstract

Analysis and experiment on mold and resin materials for the nanoimprint process are conducted in this study. We developed FEM analysis of indentation stress induced in mold materials when they come in contact with 1MPa expanded uniaxial stress. Experimental analyses of viscosity, thermal expansion coefficient, and shrinkage rate of acrylate-based UV resin are likewise undertaken. Experimentally, Hertzian indentation and adhesion tests are used as model test systems for the micro/nanoimprint process. For the study, indentation test variables investigated are the contact load for various mold materials such as Si, glass, and PDMS(polydimethylsiloxane). The adhesion test is performed to measure the maximum uniaxial load required to separate the mold from the resin material. The results highlight that the adhesion stresses are not negligible during the demoulding process, while the indentation stresses are negligible during the imprint process.

Keywords: Nanoimprint; Mold; UV resin; Contact stress; Demoulding

1. Introduction

Nanoimprint lithography is a promising technology which could be utilized to obtain nanometer-scaled patterns, generally with sub-100nm features, into the substrate by transferring the pre-determined mold stamp patterns using electron beam, UV, or high pressure. Among these, UV nanoimprint lithography has an advantage of simplicity, low operational cost, and high productivity [1, 2].

Fig. 1(a) shows the schematic diagrams of the UV nanoimprint process. For this study, the stamp was pre-prepared by cast molding. Typically, the nanoimprint lithography mold is coated with an antisticking layer [3], while the fluorinated base material is mainly used as the anti-sticking layer. A self-assembled monolayer (SAM) is more commonly used, in which UV resins are pressed by mold and the patterns are transferred to resin through UV curing. Finally, the patterns are established on the substrate by etching the base layer and removing the resin layer.

In contact-based nanoscale lithography, such as UV nanoimprint, the contact and antisticking applications are indispensable. Therefore, contact stress during imprint and adhesion stress during the demoulding process both have an influence on the reliability and the lifetime of the mold and substrate.

This work aims to investigate contact and adhesion stress levels during the nanoimprint process. For the study on contact stress, FEM is performed using ABAQUS software to analyze the induced stress of mold materials when it comes in contact with 1MPa expanded uniaxial stress. Experimentally, Hertzian indentation stress-strain curves have been obtained for various mold materials [4]. The schematic diagram of Hertzian indentations is shown in Fig. 1(b).

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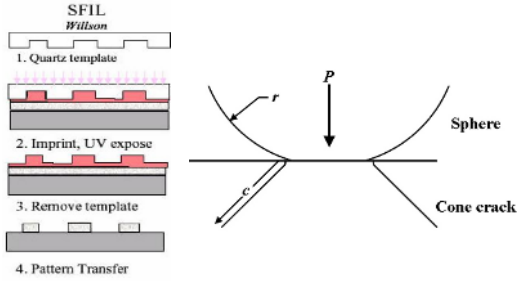


(a) (b)

Fig. 1. Schematic diagrams of (a) the nanoimprint process and (b) the Hertzian indentation test.

For the study on adhesion stress, the load to completely detach the mold from the resin layer is measured. Several characterizations on the acrylate-based UV resin are also undertaken.

2. Experimental procedure

2.1 Materials preparation

The materials chosen for the main focus of the present study were commercialized glass (FISHER, Germany), Si wafer (Namgang Hightech., Korea), PDMS, and acrylate-based UV curable Resin (OR-08, Minuta Technology Co., Korea). The PDMS was fabricated in the laboratory by pouring PDMS sol into the mold, which was then dried and detached.

The densities and some mechanical properties are shown in Table 1. The sintered densities of the solid materials were measured using the Archimedes method. The elastic modulus and Poisson's ratio were measured from the acoustic impulse excitation apparatus. A conventional Vickers indentation was performed to measure hardness at loads of P = 10N ~ 50N, on the polished surfaces. Hardness is calculated through the following Eq. (1):

$$\text{Hardness,} \quad Hv = \frac{P}{2\left(\frac{d}{2}\right)^2} \tag{1}$$

where d is the indentation diagonal length.

The prepared sample materials in the form of Si wafer, PDMS, and Glass, were cut into 20mm × 4mm × 2mm dimensions, after which the surfaces were polished to a 1μm finish and then coated with gold to observe contact damages. Three- layered samples were prepared for Si wafer (with ~ 500-550μm thick

Table 1. Material properties of the mold and resin materials.

Material	Density (g/cm^3)	Poisson's ratio	Elastic modulus (GPa)	Hardness (GPa)
Si wafer	2.33	0.28	112.4	1.15
PDMS	1.01	0.499	8	0.8
Glass	2.33	0.29	63	4.1
Resin	1.2	0.164	7.2	0.16

ness for each layer), which was bonded by cyanide adhesive to exclude the substrate's effect during the indentation test. Two glass samples (75mm × 25mm × 1mm) were also bonded through the same method. For the adhesion force test, glass/PUA/resin/glass layered specimen was fabricated by UV curing, placing resins (UV resin and PUA resin) between slide glasses without using any anti-sticking layer.

2.2 Analysis and experiment

For the study on the physical properties, we measured viscosity, thermal expansion coefficient, and the shrinkage rate of UV resin. The viscosity measurement of liquid resin was performed using a universal viscometer machine (DV-11+Pro, Brookfield, USA). In order to understand the material's fluid property, we increased the spindle speed from 10 to 200rpm. Using a dilatometer (L76, LINSEIS, Germany), we then measured the thermal expansion coefficient of UV-cured resin in air, as the temperature of the specimen was changed from room temperature to 200°C, at a heating rate of 10°C/min and a cooling rate of 10°C/min. The resin shrinkage rate was calculated from the volume change of resin during UV curing.

The model and FEM analysis was performed in ABAQUS. This permits the pressure, through the use of specific elements, to determine the contact stress fields in each material. In this algorithm, the loads were pressed downward (U2 direction) incrementally (up to 1MPa) onto the top surface of the prescribed materials, the sizes of which were 3mm × 4mm. The mesh size of the substrate was 0.05. The properties of materials used in the analysis are shown in Table 1. We constrained U1 and U2 at the substrate bottom. Finally, the axi-symmetric stress contours were calculated.

In order to evaluate the materials, indentation stress-strain curves were obtained using WC spherical

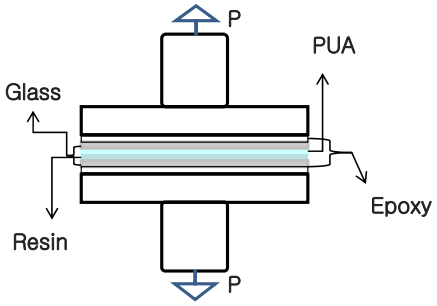
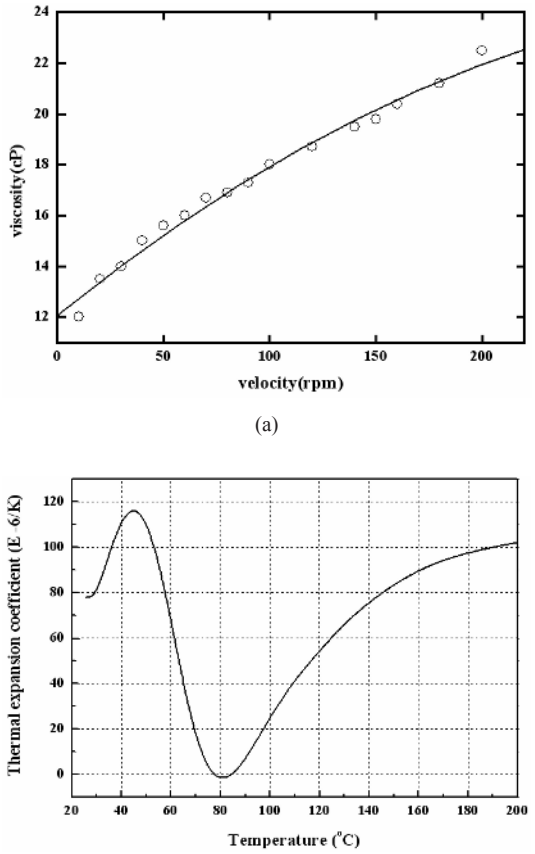


Fig. 2. Schematic diagram of adhesion force test.

indenters of radius r = 3.18 mm (J&L Industrial Supply Co., MI, U.S.A.) at loads of up to P = 600 N, and a constant cross head speed of 0.2mm/min in a universal testing machine (Model 5567, Instron, Canton, U.S.A.). The diameter and area of the indentation sites were also examined and measured. From measurements of contact radius a at each value of P and r, indentation stress ($p_0 = P/\pi a^2$) and indentation strain (a/r) could both be evaluated, enabling the construction of the stress-strain curves.

The adhesion force test was performed by arranging the prepared layered specimen onto the manufactured jigs and increasing loads by up to 4000N, at a constant cross head speed of 0.02mm/min in a universal testing machine (Model 5567, Instron, Canton, U.S.A). The schematic diagram of the adhesion test is shown Fig. 2.



(a)

(b)

Fig. 3. The measurement results of UV resin on (a) viscosity and (b) thermal expansion coefficient.

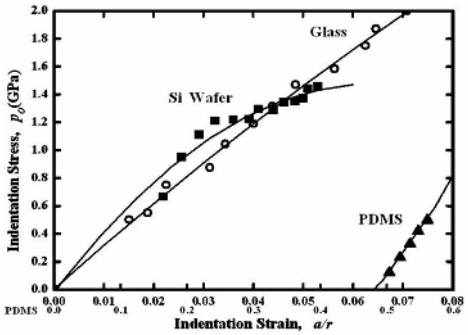
3. Results and discussion

The viscosity of UV curable liquid resin is plotted as a function of spindle velocity during the tests as shown in Fig. 3(a). The nonlinear behavior indicates that the resin is a type of non-Newtonian fluid.

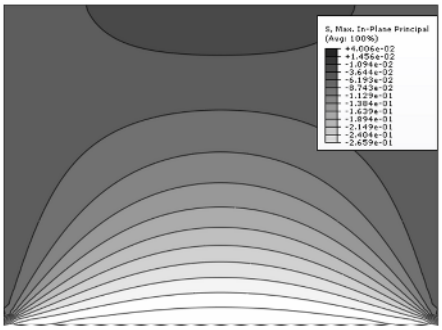
Fig. 3(b) shows the measurement of resin's thermal expansion coefficient at temperatures ranging from room temperature to 200°C. As can be seen, although positive thermal expansion occurs as the temperature increases, the extent of thermal expansion increases at the first stage, decreases up to 80°C, and finally increases to 200°C. This difference can be attributed to the weight change due to solvent evaporation from 45 to 80°C. As a larger thermal expansion of the resin material suggests a larger thermal residual stress built up during cooling, the UV curing should be controlled to avoid the residual tensile stress.

The volume shrinkage rate of the resin materials in this study was measured as ~ 10% during UV curing.

Indentation stress-strain curves of mold materials for the nanoimprint process are plotted in Fig. 4(a). Given that all indentations performed without any failure, the results suggest all mold materials have minimum and maximum contact resistance rates of up to 500MPa (for PDMS) and ≈ 2GPa (for glass), respectively. Fig. 4(b) shows the typical stress contours of the glass mold materials for the nanoimprint process coming in contact with 1MPa expanded uniaxial stress as calculated by ABAQUS. The results show that at ≈ 0.04MPa, the maximum in-plane stress levels of materials produced under 1MPa of uniform pressure are not too high. By comparing Fig. 4(a) with Fig. 4(b), we can see that all mold materials of this study can endure the contact stresses.



(a)



(b)

Fig. 4. (a) Indentation stress-strain curves of Si wafer, PDMS and glass material, and (b) diagram showing typical stress contour.

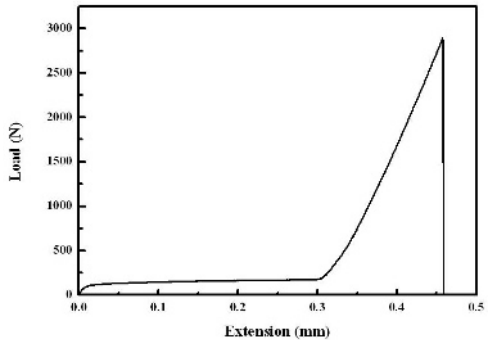


Fig. 5. The load-extension curve of the mold/resin layered system during adhesion test.

On the other hand, the load-extension curve of the glass/PUA/UV resin/glass layered specimen as shown in Fig. 5 indicates that the adhesion stresses are not negligible in the demoulding process. he maximum load is approximately 3000N. The adhesion test result highlight the fact that the maximum stress is 1.6MPa, which is not small when we consider repetitive nanoimprint process.

4. Conclusions

In this study, analysis and experiment on mold and resin materials for the nanoimprint process were conducted. The characterizations on the acrylate-based UV resin show non-Newtonian behavior when it is in liquid state, temperature-dependent thermal expansion when it is solid, and 10% shrinkage during UV curing. Indentation stress-strain curves of Si, glass, and PDMS materials using the Hertzian indentation test demonstrate the suitability of glass as a good material for molds. The results of FEM analysis using ABAQUS software indicate that the calculated maximum in-plane principal stress in the glass mold materials are 0.04MPa when it comes in contact with 1MPa expanded uniaxial stress. On the other hand, adhesion test results show that the maximum stress is 1.6MPa, which should not be considered negligible in the demoulding process.

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