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# Fabrication of micro mold for hot-embossing of polyimide microfluidic platform by using electron beam lithography combined with inductively coupled plasma

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**Abstract**

This study describes the fabrication of Si molds by using electron beam lithography (EBL) combined with inductively coupled plasma (ICP) etching and its application to the hot-emboss of polyimide (PI) microfluidic platform. The dynamic mechanical thermal properties and the formability of a polyimide film were investigated as a function of temperature by dynamic mechanical thermal analysis (DMTA) and hot-embossing tests. Based on the results, the microfluidic channel could be replicated on PI surface with good fidelity by hot-embossing with the Si mold structured by using EBL combined with ICP etching.


*Keywords:* Polyimide; Hot-embossing; Dynamic mechanical thermal analysis; Micro mold; Electron beam lithography; Inductively coupled plasma

## 1. Introduction

Polyimides (PIs) are a class of high heat-resistant polymers with the unique combination of mechanical, thermal, and electrical properties, so they have been widely used in a variety of applications such as the flexible printed circuits (FPC), the specialty fabricated products (SFP), the pressure sensitive tape (PST) and others [1]. Most recently, PIs have also applied to microfluidic and biomedical applications (e.g., a flexible and implantable intracortical electrode array, and a microstructured substrate for contact guidance of osteoblast cell growth) due to its biocompatibility [2]. As the application fields of PI-based micro devices are expanded, the need for a high-throughput, a low-cost and a flexible lithographic technique has increased. Hot-embossing is one of promising techniques that can fulfill those requirements [3], but its application has been generally limited to thermoplastic polymers with thermal stability lower than 200 °C. Hence the hot-emboss of PIs is still challenging because most of the PIs have glass transition temperature in excess of 300 °C and melt temperature ($T_m$) above 400 °C due to their very bulky and rigid backbone structures.

In this study, a PI microfluidic platform was fabricated by hot-embossing. The deformation characteristics of polymer during thermal processing depend strongly on temperature, frequency and strain rate as well as the composition of the material, and accurate accounts for those behaviors are highly desirable to predict deformation processes during hot-embossing [4,5]. Thus, the changes of the storage elastic modulus ($E'$) and the loss tangent ($\tan\delta$) of PI sample were measured as a function of the temperature for three frequencies. Silicon micro molds for the replication of microfluidic channels were prepared by the combination of EBL [6] and ICP etching. The ICP etching conditions were tuned to obtain microfluidic channels with nearly vertical sidewalls. Based on the results regarding DMTA and preliminary hot-embossing tests, the microfluidic channels were replicated in PI by hot-embossing.

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## 2. Experimental procedure

Commercially available PI film (Kapton JP, DuPont) with 360 μm in thickness was used for both dynamic visco-elasticity measurement (dynamic thermal tension test) and hot-embossing tests. The results of surface topological analysis using an interferometric surface profiler revealed that the $R_a$ roughness was 164 nm. The dynamic mechanical thermal properties of the PI samples were measured with a Viscoanalyzer VA2000 by 01dBMetravib. Hot-embossing tests were conducted using hot-emboss equipment (the max. applied force: 200 kgf, the max. heating temp.: 700 °C and vacuum pressure: <1 Pa) with cross-head velocity at 50 μm/min and a heating rate at 0.5 °C/s. Silicon molds were made by EBL combined with ICP etching. The process started with spin-coating of an adhesion enhancer (HMDS) on Si substrate for 5 s at 300 rpm, then 25 s at 1500 rpm. After adhesion enhancing treatment, a negative-type EB resist (TGMR) was coated to the wafer and spun for 10 s at 1000 rpm, then 90 s at 2000 rpm, resulting in an approximately 350 nm thick layer. The wafer was then soft-baked on a hot plate at 90 °C for 90 s. E-beam writing was performed with an EBL system from Elionix (Japan) at the energy of 100 keV. After EB writing, the sample was heated on a hot-plate at 110 °C for 90 s. Development was carried out using NMD-3 solution at 23 °C for 4 min. After development, the sample surface was etched by ICP. The etching depth was controlled by varying ICP etching time. Finally, remained EB resist was removed by ashing for 10 min.

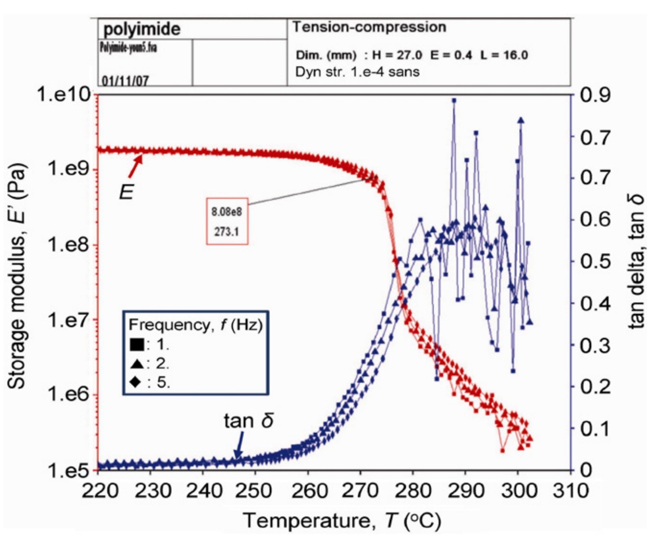


Fig. 1. Storage modulus and tan $\delta$ for polyimide obtained from DMTA testing in tension mode at three frequencies.

## 3. Results and discussion

The DMTA of a polyimide sample was performed in tension mode at the frequencies of 1, 2 and 5 Hz, and the DMTA spectra (i.e., $E'$ and the tan $\delta$) obtained are reported in Fig. 1 as a function of temperature. The tan $\delta$ is the ratio of the dynamic loss modulus ($E''$) to the storage modulus ($E'$), and provides information on the relative contributions of the viscous and elastic components of a viscoelastic material, i.e., materials with a tan $\delta$ less than 1 exhibit more elastic behavior [7]. With the elevated temperature, the $E'$ decreased generally due to an increase in free volumes and chain movements of PI sample, and the $E'$–$T$ curve shows three distinct regions for the dynamic behavior; glassy state region, viscoelastic region and rubbery state region. In the glassy zone, PI with rigid structures shows high $E'$ and low tan $\delta$ values due to the limited movements of macro-molecular chain. In the viscoelastic zone, the transition, which is characterized by the peak of tan $\delta$ and a drop in the $E'$, started at about 273 °C due to the beginning of the chain segment movements. In the elastic zone, the value of the $E'$ decreased and reached to the elastic plateau that is related to the degree of cross-linking. The applied frequency had an obvious influence on $T_g$ as well as $E'$ and tan $\delta$; the transition shifted to higher temperature with higher frequency.

Silicon molds, which have the reverse image of micro fluidic channels on its surface, were prepared by EBL, followed by ICP etching. The ICP etching conditions are listed in Table 1. Fig. 2 displays SEM images of sample Si molds. The surfaces of samples showed good appearance with no defect such as the reverse taper of microstructure. The etched depth measured using interferometric surface profiler was approximately 10 μm.

For a given mold, complete filling conditions were investigated with different force at the same temperature of 275 °C ($T_g$ of polyimide) on the basis of the DMTA test results. Good replication could be achieved at temperature about 275 °C with the press pressure at 5 MPa and the embossing time of 300 s, as shown in Fig. 3. The reusability of the molds strongly depended on its surface quality as well as aspect ratio and cleanness. Regarding the anti adhesion treatment of mold surfaces, there is need for the further study because most mold release agents such as self-assembled monolayers (SAMs) did not provide sufficient anti-adhesion effect under such high temperature condition

Table 1
ICP etching conditions for samples

| Source power (W) | Bias power (W) | $SF_6$ | | $C_4F_8$ | | $O_2$ | | Valve pos (%) | Pressure (Pa) | He Pressure (mBar) | Temp (°C) | Process time (min) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Flow (sccm) | Time (s) | Flow (sccm) | Time (s) | Flow (sccm) | Time (s) | | | | | |
| 800 | 37 | 250 | 2 | 200 | 1.3 | 12.5 | 0.5 | 60 | 2.5 | 10 | 10 | 7.5 |