**Proceedings of the** 

# **Advanced Architectures in Photonics**

September 21–24, 2014 Prague, Czech Republic

Volume 1

Editors

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Involved Ltd. Siroka 1 537 01 Chrudim Czech Republic

Proceeding of the Advanced Architectures in Photonics http://aap-conference.com/aap-proceedings

ISSN: 2336-6036 September 2014

Published by **Involved Ltd.** Address: Siroka 1, 53701, Chrudim, Czech Republic Email: <u>info@involved.cz</u>, Tel. +420 732 974 096



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## Electric nanoimprint to oxide glass containing alkali metal ions

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Fine patterns were electrically imprinted onto an alkali ion containing glass using a two-dimensional mold with 700 nm period by applying a DC voltage of 120 V for 60 s at 100 °C below the glass transition temperature ( $T_g$  = 550 °C) under the pressure of 0.02 or 3 MPa.

Electrical nanoimprint is known as the low temperature process for the formation of fine structures on alkali ion containing glasses below their glass transition temperatures [1–3]. Recently, we reported the alkali ion diffusion from anode side to cathode side in a soda-lime glass only in the contacted area of the mold fine structure [3]. This paper reveals the correlation between the alkali diffusion and the fine structure formation.

Fig. 1 shows the imprint time dependences of current and the variations of glass surfaces after the electrical nanoimprint under the pressures of 3 MPa and 0.02 MPa. Each photograph was obtained by interrupting the applied voltage at the predetermined point. As recognized from the diffraction image of illuminated visible light, the patterned area expanded gradually, and the uniform transfer of fine pattern was achieved when the total charge exceeded 20 mC. Therefore, we can conclude that the imprint pressure is not so important for the electrical nanoimprint.

Fig. 2 denotes the model to explain the fine pattern transfer by the electrical nanoimprint. The right hand in the figure exemplifies the flatness of mold and glass plate. The warpage of the latter, which is 150 nm in vertical interval, is much larger than the hole depth formed by the imprint. The imprint pressure of 0.02 MPa should be insufficient



**Figure 1.** Time dependences of applied voltage and current (upper), and electrically imprinted glass surfaces (lower) at 450°C under the pressure of (a) 3 MPa and (b) 0.02 MPa. The specimen size is 10×10×1 mm.



**Figure 2.** (a) Model of fine pattern formation by electrical nanoimprint. (b) and (c) are surface curvatures of mold substrate and soda-lime glass plate used for the imprint, respectively.

to achieve the perfect contact between mold and glass plate even if at 450 °C. We considered that the mold should contact to the glass plate partially at the initial stage of DC voltage application. The sodium deficient layer with negative charge is formed below the mold contacted area during the voltage application.



**Figure 3.** AFM views of glass plates imprinted under (a) 3 MPa and (b) 0.02 MPa, before and after KOH etching. Total charge estimated after electric imprint is 20.8 mC for (a) and 22.8 mC for (b).



Figure 4. Experimental setup of electrical nanoimprint.

Simultaneously, an electrostatic attractive force should be generated between the deficient layer and the mold. Such phenomena have been reported in the studies on anodic bonding used for the fabrication of MEMS [4]. The electrostatic attractive force induces the penetration of mold pillar to the sodium deficient area, and the next neighbor pillars contact to the glass surface, forming another sodium deficient area. After the repetition of these steps, the fine pattern transfer proceeds in the entire area of the plate under low imprint pressure.

The alkali deficient layer, which was estimated to be 400 nm in depth using a ToF-SIMS, can be selectively etched in a KOH solution. Fig. 3 shows the AFM views before and after the KOH etching of the electrically imprinted glass plates. No pressure dependence was recognized in the surface morphologies before and after the etching. Therefore, the electrical nanoimprint is advantageous process for the fine pattern transfer onto alkali ion containing glasses below  $T_{q}$ .

Fig. 4 exemplifies the experimental setup of electrical nanoimprint. A carbon coated SiO<sub>2</sub> mold with two-dimensional pillar with 700 nm period was contacted to a soda-lime glass ( $T_g = 550$  °C) on the cathode stage at 450 °C in N<sub>2</sub>, and then a positive DC voltage of 120 V was applied to the mold for 60 s.

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