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Enhanced surface patterning of chalcogenide glass via imprinting process using a buffer layer

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ABSTRACT

In an effort to enhance transcriptability of quasi-three-dimensional patterns present in silicon stamp onto the surface of 'bulk' chalcogenide glass, a buffer layer was introduced during the replication process via imprinting. Dissimilar patterns with diverse depths along the surface normal direction were imprinted with or without the buffer layer, and the resulting patterns on the glass surface were compared with regard to the transcription quality in both the lateral and vertical directions. After assessing the processing conditions appropriate for imprinting bulk As_2S_3 glass especially in terms of temperature and duration, candidate materials suitable for the buffer layer were screened: Commercially available polydimethylsiloxane was then chosen, and impact of this buffer layer was elucidated. The imprinted patterns turned out to become more uniform over large surface areas when the buffer layer was inserted. This finding confirmed that the use of buffer layer conspicuously enhanced the transcriptability of imprinting process for bulk chalcogenide glass.

1. Introduction

Imprinting technique is considered as a viable option that is capable of facilely fabricating two-dimensional structures (or patterns) on the surface of chalcogenide glass in the form of either film or bulk. To be practically applied to photonic devices, the surface patterns typically need to be defined in a length scale of a few micrometers in the direction vertical to the surface in order to secure optical confinements. It was experimentally demonstrated already that an imprinting process performed to the surface of chalcogenide glass film was able to introduce a rib-type waveguide structure satisfying such a vertical length scale [1–3]. It is worth mentioning that the planar waveguides demonstrated in these previous works were aimed to exploit optical contrast between the chalcogenide film and the substrate, so that the film-type geometry was preferred. On the other hand, it is easy to envisage that for some specific applications a quasi-three-dimensional pattern needs to be introduced on the surface of chalcogendie glass, which would require transcription of the stamp pattern down to a deeper distance from the top surface of glass, and a more abundant mass flow of chalcogenide glass under imprinting needs to be accompanied. Bulk chalcogenide glass taking shape of, e.g., rectangular plate or circular disk is likely to be more adequate for this purpose. Advantages associated with the imprinting process of bulk chalcogenide glass were well addressed already in Ref. [4]. However, in this case, both the top and bottom surfaces of the bulk chalcogenide glass should be prepared to be parallel with each other. If the top surface is not parallel to the bottom one, then lateral resolution of the imprinted pattern would be deteriorated. This deterioration would become more severe when the size of the imprinted pattern is large and/or when the imprinter is poorly aligned. In this regard, in this study, we mainly aimed to mitigate this problem caused by the both surfaces not in parallel. Specifically, transcriptability was experimentally verified to be significantly improved when another material with a proper viscosity in the temperature range for imprinting bulk chalcogenide glass, named buffer layer in this study, was placed below chalcogenide glass disk to be imprinted. Silicon stamps with various patterns consisting of different shapes and depths were imprinted onto the surface of chalcogenide glass with or without the buffer layer, and the resulting surface patterns were analyzed in terms of transcription quality.

2. Experimental

Several Se-based or S-based chalcogenide glasses have been tested in this study for their applicability as to imprinting pro-

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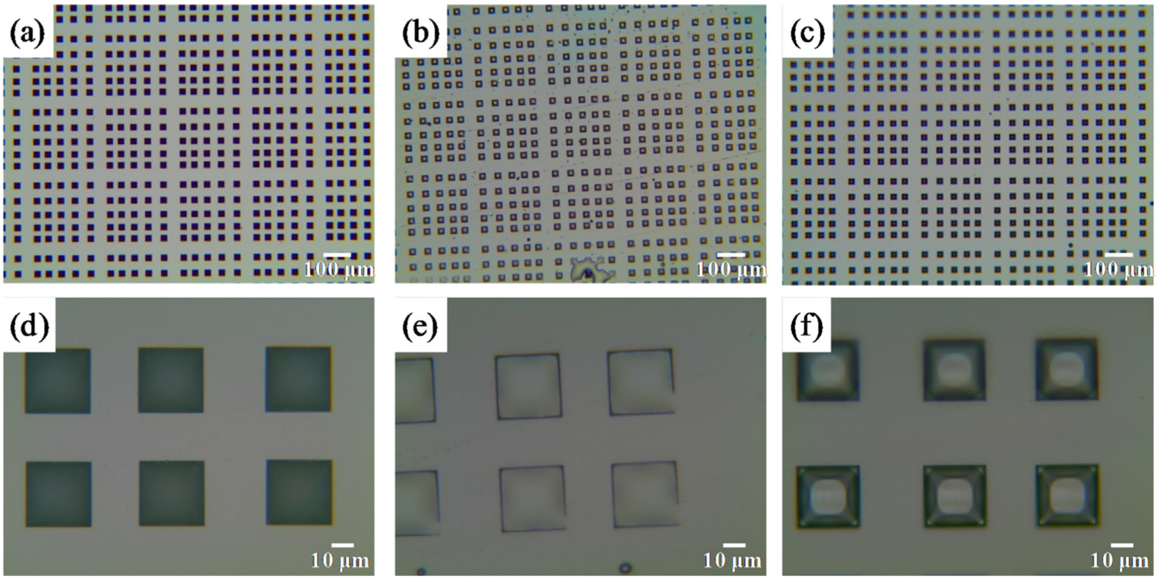

Fig. 1. Optical microscopic images for the pattern consisting of the inverted square pyramids: (a), (d) silicon stamp; (b), (e) imprinted glass surface processed without the buffer layer; (e), (f) imprinted glass surface processed together with the buffer layer.

cess [5,6]. However, experiments concerning the effects of buffer layer were mainly performed with As_2S_3 glass, since this glass is one of the prototypical chalcogenide glasses [7]. As starting materials, arsenic (As) and sulfur (S) with purities of 99.999% were used in their elemental form to fabricate 'bulk' glass via the conventional melt-quenching route [8–10]. After being carefully weighed, each batch was vacuum-sealed inside a silica ampoule and then melted at 1000 °C for 24 h using a rocking furnace. Quenching was conducted through immersing the ampoule into ice water, and annealing was subsequently undertaken at 150 °C for 2 h. Glass transition temperature of our As_2S_3 glass was measured to be 170 °C, similar to those previously reported [11]. Glass rods thus prepared were cut and polished to make disk specimens with diameter of 12 mm and thickness of ~2 mm. It is noteworthy that the cutting and polishing was not the best that we could possibly achieve, and thereby both faces of the resulting disks were not parallel with each other. In many cases, the disks became tapered so that the corresponding difference in the thickness amounted to approximately 10 μm for 2-mm-thick disks.

Various patterns were devised and engraved onto *p*-type Si wafer. The size of a stamp was typically of 10 × 10 mm^2. It needs to be mentioned that the depth of entrenched regions for some patterns was not constant but set to vary in order to make quasi-three-dimensional patterns. After imprinting, the height of prominent regions in the surface morphology of the glass was measured and compared with the corresponding depth of depressed regions in the Si stamp. Conventional dry- or wet-etching process was employed to realize such patterns onto the Si stamps [12]. In particular, a wet-etching technique was applied to define the smoothly varying depth in the pattern consisting of inverted pyramids shown in Figs. 1 and 2.

Imprinting was carried out with a homemade hot press in which a chalcogenide glass disk was placed between two stainless plates. Temperature of the two plates was controlled within uncertainty of ±1 °C, and imprinting was achieved by letting the upper plate move downward to a desired displacement. Based on our preliminary experiments performed to set suitable imprinting conditions, i.e., temperature, duration and displacement, imprinting at 255 °C resulted in a best performance. This temperature corresponded to a viscosity of $\sim 10^8$ Pa s for As_2S_3 glass [13], which belonged to the typical viscosity range previously suggested for imprinting this group of chalcogenide glass [1,3,14]. For all of the imprinting experiments in this study, temperature was thus fixed at 255 °C, but duration was allowed to vary typically from 600 s to 1800s according to geometry of the patterns.

Table 1
Surface roughness of the four-story pagoda pattern in the Si stamp and the imprinted glass.

Region	RMS (nm)	
	Si stamp	As_2S_3 glass
A	16	23
B	27	38
C	25	67
D	60	170

The surface morphology of the stamps and the imprinted glasses were monitored using optical microscope (BX51M, OLYMPUS) and/or scanning electron microscope (JSM-6700F, JEOL). In addition, to get more precise information as to transcriptability, atomic force microscope (XE-100, PARK SYSTEMS) was utilized for a specific pattern which is detailed below.

3. Results and discussion

The positive impact of the buffer layer is visually demonstrated in Fig. 1. In this instance, an array of inverted square pyramids introduced to the Si stamp (Fig. 1(a)) exemplifies such an enhancement in the surface morphology after imprinting with the buffer layer. An inverted square pyramid in the Si stamp was of 20 × 20 μm^2 in its base area and the length from the base plane to the apex was 30 μm, as shown in Fig. 2 (a) and (b). Imprinting this stamp without the buffer layer (Fig. 1(b)) resulted in a relatively poor transcriptbility compared to the case with the buffer layer (Fig. 1(c)). Specifically, the imprinted pattern became not only more uniform along the lateral direction but also more defined along the vertical direction after the introduction of buffer layer, as can be noticed in Fig. 1(e) and (f). The relatively poor transcription mainly caused by the unparallel faces of glass disk was mitigated to a great extent by using the buffer layer. In other words, difficulty in precise alignment due to the uneven chalcogenide glass resulted in deterioration of lateral and/or vertical resolutions in the imprinted patterns. How-

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