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Rapid replication of polymeric and metallic high aspect ratio microstructures using PDMS and LIGA technology

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Abstract This paper present a method of rapid replication of polymeric high aspect ratio microstructures (HARMs) and a method of rapid reproduction of metallic micromold inserts for HARMs using polydimethylsiloxane (PDMS) casting and standard LIGA processes. A high aspect ratio (HAR) metallic micromold insert, featuring a variety of test microstructures made of electroplated nickel with 15:1 height-to-width ratio for 300 μm microstructures, was fabricated by the standard LIGA process using deep X-ray lithography (DXRL). A 10:1 mixture of pre-polymer PDMS and a curing agent were cast onto the HAR metallic micromold insert, cured and peeled off to create reverse images of the HAR metallic micromold insert in PDMS. In addition to the replication of polymeric HARMs, replicated PDMS HARMS were coated with a metallic sacrificial layer and electroplated in nickel to reproduce another metallic micromold insert. This method can be used to rapidly and massively reproduce HAR metallic micromold inserts in low cost mass production manner without further using DXRL.

1
Introduction

The demand for high aspect microstructures (HARMs) for sensor and actuator applications has been increasing in the MEMS community. HARMs offer a number of advantages to general MEMS applications including increased structural rigidity, low driving voltage, higher actuation force, and larger displacement in actuator systems, higher sensitivity in sensor applications by virtue of large mass, and large magnetic forces for magnetic MEMS due to large volume. Common techniques for fabricating HARMS include LIGA (a German acronym for lithography, electroplating, and molding) (Cheng et al. 1999), LIGA-like (Lorenz et al. 1998), and deep reactive ion etching (DRIE) (Chung et al. 2000) processes. The LIGA process is a well-known technique to create HARMs with aspect rations of up to 100:1 with a few millimeters in height. A major drawback of this technology is the need of synchrotron radiation source. For low-cost HARMs fabrication, a LIGA-like process is available with the epoxy-based resist SU-8 (Lorenz et al. 1998) with the compensation of lower resolution and lower aspect ratio. Another alternative to realize HARMs is to make deep silicon trenches using DRIE based on inductively coupled plasma (ICP) (Chung et al. 2000).

Recently there have been many investigations in the MEMS community to realize plastic high aspect ratio MEMS devices, especially for the emerging application areas such as lab-on-a-chip and biomedical MEMS. Common methods to realize polymeric HARMs include injection molding and hot embossing. Typical polymer replication materials include polycarbonate (PC) (Becker et al. 1999), polyvinyl chloride (PVC) (Lin et al. 1998), polymethyl methacrylate (PMMA) (Lin et al. 1998), and polyethylene (PE) (Despa et al. 1999). Replication using most thermoplastic materials (PC, PVC, PE, and PMMA) requires either a modified or conventional injection molding machine and/or hot embossing machine. Polydimethylsiloxane (PDMS), however, can be replicated

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by direct casting without using injection molding/hot embossing machines for microstructures replication. PDMS replication has been utilized mostly in microfluidics with the use of epoxy-based photoresist SU-8 as mold inserts. A fluidic switch and a side channel flow controller have been fabricated in PDMS against a SU-8 master for micro total analysis systems (μTAS) (Duffy et al. 1999). Three-dimensional micro-channels in PDMS with a sandwich-molding configuration have been developed for complex 3-D channel paths (Jo et al. 2000).

In this work, PDMS was used for a massive replication of polymeric HARMs against a high aspect ratio metallic micromold insert fabricated by the standard deep X-ray lithography (DXRL) process in a low cost production manner. Such plastic HARMs can be used in a variety of MEMS applications, especially in μTAS applications. A novel method to massively reproduce multiple metallic micromold inserts with quality nearly equivalent to the original metallic micromold inserts prepared by the standard LIGA process without further using the DXRL process was also investigated.

Table 1. Composition of the nickel electroplating solution

Component	Grams or liter per 1 liter of DI water
Ni $(SO_3NH_2)_2$ (nickel sulfamate)	450 ml
H_3BO_3 (boric acid)	37.5 g
$C_{12}H_{25}NaO_4S$ (Sodium dodecyl sulfate or lauryl sulfate)	3 g

2
Experiment

2.1
PDMS HARMs replication

The standard LIGA process (Marques et al. 1997) using synchrotron radiation source at the Center for Advanced Microstructures and Devices (CAMD) at the Louisiana State University was used to prepare a metallic micromold insert that features a variety of test patterns. A detailed process was described elsewhere (Kim et al. 2000). In particular, nickel was over-electroplated to create metallic micromold inserts for PDMS replication. Table 1 shows the composition of the nickel electroplating solution, a nickel sulfamate electrolytic solution buffered with boric acids. Typically, the electroplating bath was heated to 55 °C and a current density of 10 mA/cm^2 was used.

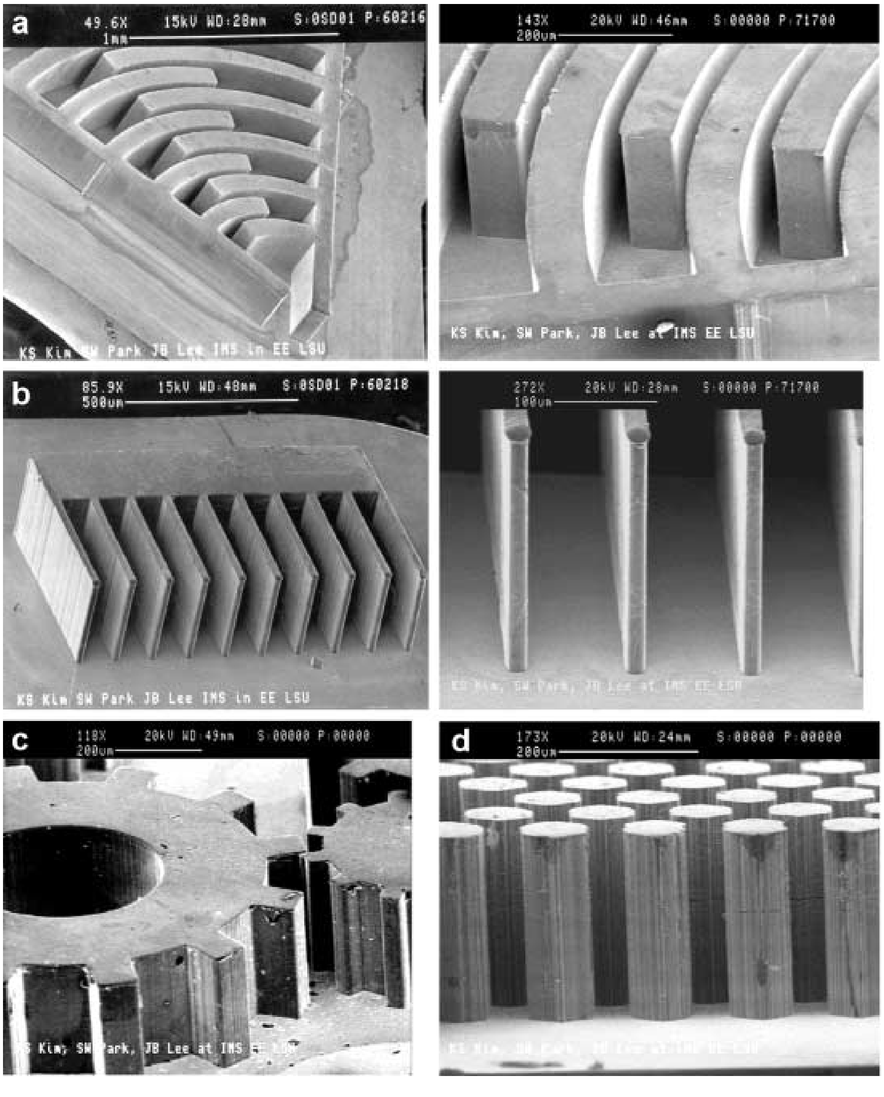

Fig. 1a–d. SEM photomicrographs of test patterns in an electroplated nickel micromold insert with a height of 300 μm and maximum aspect ratio of 15:1. **a** Angular comb fingers; **b** comb fingers; **c** micro gears; **d** honeycombs

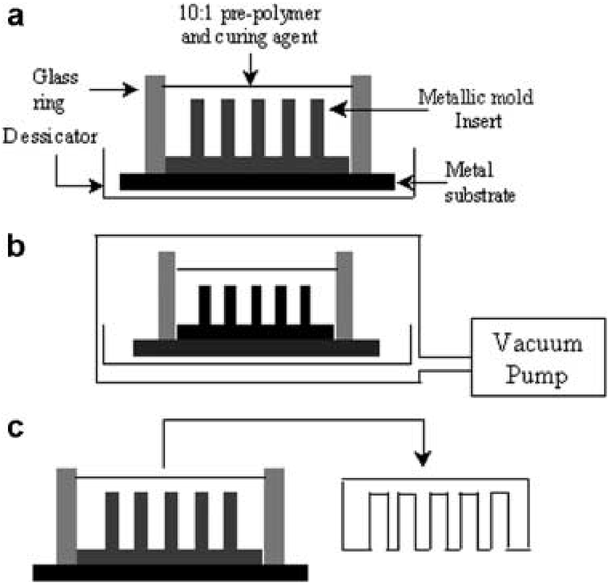

Fig. 2a–c. The process sequence for PDMS HARMs replication. **a** Casting of 10:1 mixture of pre-polymer and curing agent; **b** removal of trapped air bubbles; **c** curing and peeling off replicated PDMS HARMs

Figure 1 shows scanning electron microscope (SEM) photomicrographs for selected test patterns. All of the electroplated nickel HARMs shown are 300 μm in height and the aspect ratio for angular comb fingers, gears, honey combs and straight comb fingers shown in the Fig. 1 is 15:1 The gap between angular comb structures in (a) is 20 μm and the width of comb fingers in (b) is 20 μm.

The PDMS used in this experiment is a commercially available silicone rubber compound from General Electric (Waterford, New York, USA). It is microelectronics compatible (neutral cure), durable, optically transparent, and usable over a wide range of temperatures from −100 to 100 °C (Lotters et al. 1997). It has unique chemical and physical properties including a low glass transition temperature ($T_g \approx -125$ °C), a high flexibility (the shear modulu is between 100 kPa and 3 MPa), a very low loss tangent ($\tan \delta \ll 0.001$), and high dielectric strength ($\sim$14 V/μm) (Lotters et al. 1997).

A set of RTV 615A (pre-polymer) and RTV 615B (a curing agent) were mixed with a 10:1 weight ratio and degassed using a vacuum pump to remove air bubbles entrapped during mixing. It was directly cast onto the electroplated nickel micromold insert which was mounted on a metallic substrate with a glass ring on it. The glass

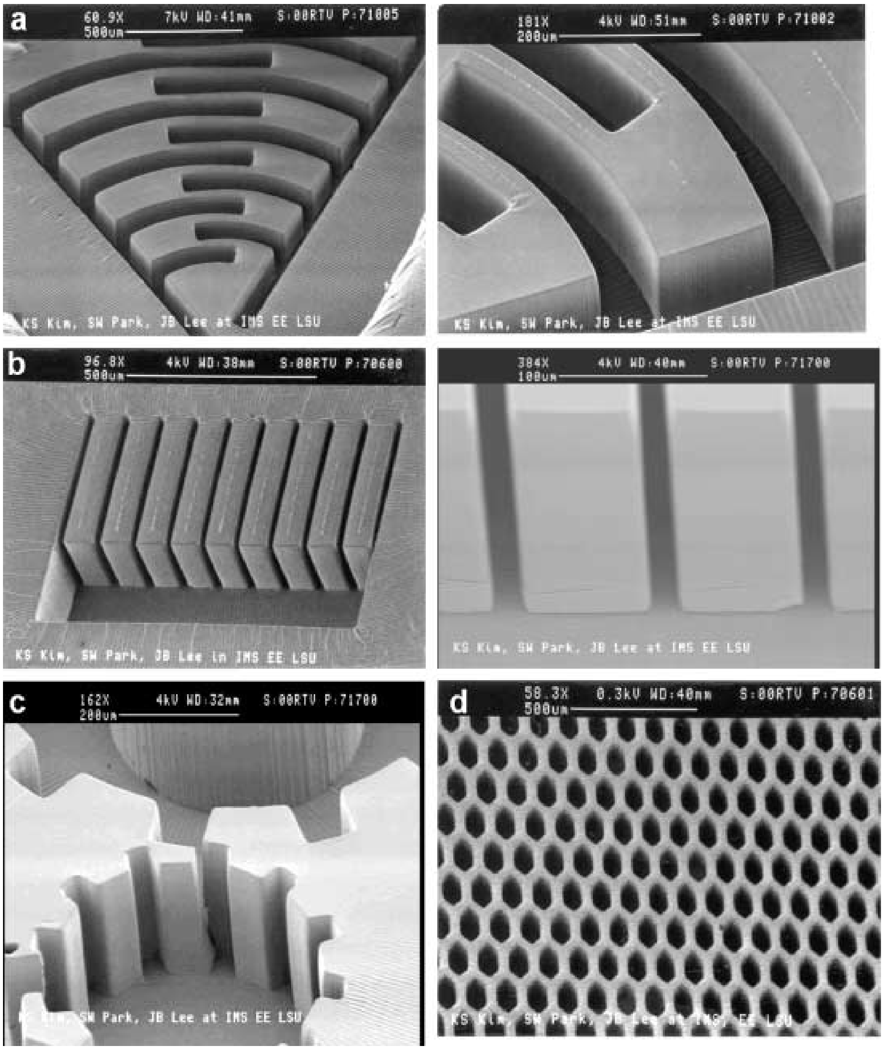

Fig. 3a–d. SEM photomicrographs of test patterns in a replicated PDMS (reverse image of nickel micromold insert in Fig. 1) with a height of 300 μm and maximum aspect ratio of 15:1. **a** Angular comb fingers; **b** straight comb fingers; **c** micro gears; **d** honeycombs

ring was sealed to the substrate with AZP 4620 photoresist (Clariant Corp.) and baked in a convection over (Fig. 2). Sample were placed in a plastic desiccator and degassed again, using a vacuum pump to remove trapped air bubbles generated during casting inside the deep trenches. Then it was cured at 65 °C for 4 h to create replicated PDMS HARMs. The curing time can be reduced to 1 h if the temperature is increased to 100 °C. Replicated PDMS HARMs could be simply peeled off from the metallic micromold insert.

Since the volume shrinkage of the PDMS after the curing is only 0.2% (GE Silicones), there were no observable dimensional changes in replicated PDMS HARMs compared to dimensions in the original metallic micromold insert. Figure 3 shows SEM photomicrographs of replicated PDMS HARMs which correspond to metallic micromold insert test patterns in Fig. 1. Most of the processing steps were carried out in non-cleanroom environment.

2.2
Metallic micromold insert reproduction using replicated PDMS

Although the LIGA technique was successfully used to deliver high quality microstructures for more than two decades, there has been a sustained debate if this technique is suitable for mass production. Some critics argued the fact that access to synchrotron radiation source is limited and processing cost is relatively higher than other micromachining techniques. However, when the LIGA technique is utilized to provide high quality molds and manufacturing of final microstructures is carried out by hot embossing and/or injection molding, the LIGA technique can become one of suitable low cost MEMS manufacturing techniques. Even in the case of hot embossing and/or injection molding plastic microstructures manufacturing, metallic micromold inserts fabricated by LIGA must be continually provided for mass production due to mechanical wear of micromold insert.

As a part of this research, we investigated a method of inexpensive, rapid, massive reproduction of multiple numbers of metallic micromold inserts from one LIGA processed metallic micromold insert without further use of a synchrotron radiation source. The fabrication sequence for the metallic micromold insert reproduction is shown in Fig. 4 and the process is explained as follows:

(1) A metallic micromold insert is fabricated by over-electroplating using standard LIGA process;
(2) Multiple numbers of PDMS HARMs are replicated using process sequences described in Sect. 2.1 (Fig. 2);
(3) Sacrificial layers (2 μm thick copper or titanium/gold) are deposited on the surface of the replicated PDMS using sputtering or evaporation (Fig. 4-c);
(4) Nickel was electrodeposited on the copper-coated replicated PDMS using the bath composition described in the Table 1 with a current density of 10 mA/cm^2. The electroplated nickel microstructure was a replica metallic micromold insert (Fig. 4-d);
(5) After the electroplating, the PDMS mold is peeled off to release the replica metallic micromold insert (Fig. 4-e);

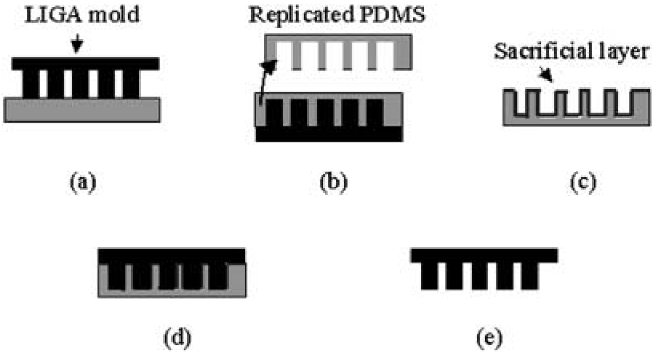

Fig. 4a–e. Fabrication sequence of replica metallic mold inserts using replicated PDMS molds

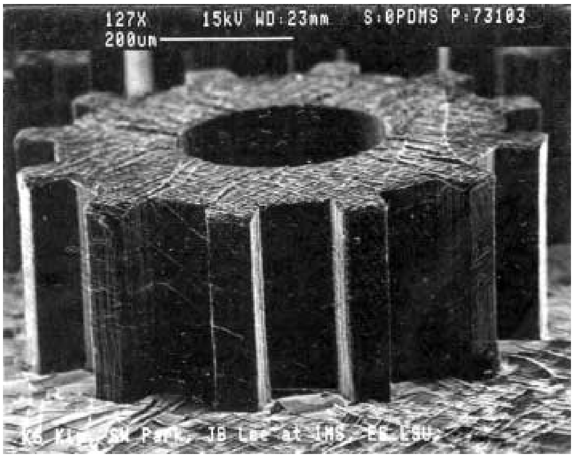

Fig. 5. An SEM photomicrograph of the reproduced metallic micromold insert

(6) The metallic seed layer for the electroplating was selectively etched away after the separation. In the case of a copper sacrificial layer, an etchant composed of cupric sulfate ($CuSO_4 \cdot 5H_2O$) saturated with ammonium hydroxide (NH_4OH) was used. In the case of titanium/gold sacrificial layers, diluted hydrofluoric acid (HF) and potassium iodide (KI) based etchants were used;
(7) The backside of the over-electroplated reproduced micromold insert was planarized as needed.

Figure 5 shows SEM photomicrographs of a micro gear on the reproduced high aspect ratio metallic micromold insert made using a sputtered copper sacrificial layer. The copper sacrificial layer deposition (step 3) on the PDMS molds could be carried out either by sputtering or evaporation. When sputtering was used, the surface of the replicated PDMS could be attacked by the high power plasma and created micro-cracks. The surface of the micro gear in the Fig. 5 shows such micro cracks.

In order to avoid micro crack formation, electron beam evaporation of copper was used. Since the thin film deposition profile using evaporation is directional (anisotropic, not conformal), the replicated PDMS sample needed to be tilted and constantly rotated during the evaporation to conformally deposit the seed layer on the surface of mold. Figure 6 shows SEM photomicrographs of

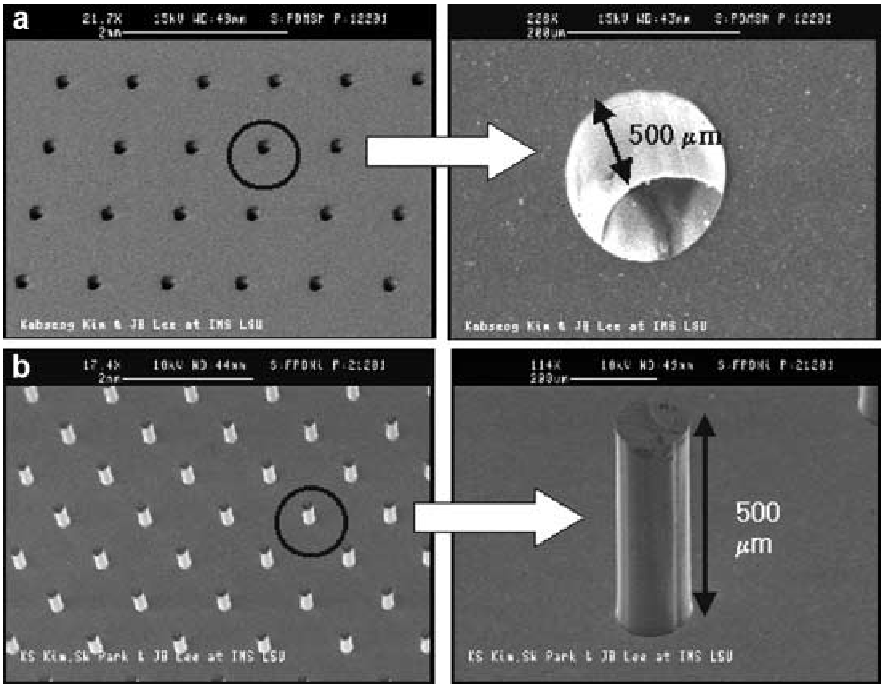

Fig. 6a,b. SEM photomicrographs of the replica metallic micromold insert made using electron beam evaporation of the copper sacrificial layer. **a** After the copper sacrificial layer deposition on the surface of the PDMS mold; **b** after the etching of the sacrificial layer showing the surface of the replica metallic micromold insert

copper deposited PDMS (Fig. 6-a) and the resulting replica micromold insert (Fig. 6-b) by electron beam evaporation of a sacrificial layer. There was no micro-crack problem in this method.

3 Characterization

In order to characterize the surface quality of the PDMS mold, a Wyko interferometric surface profiler was used to measure the surface roughness of the original nickel micromold insert prepared by the standard LIGA process and of the replicated PDMS HARMs. Multiple measurements were made for the surface areas of the nickel micromold insert and the corresponding surface areas of the replicated PDMS HARMs. The average surface roughness on the nickel micromold insert and the replicated PDMS HARMs were found to be 204 and 215 nm, respectively (Fig. 7). It is concluded that high precision HARMs pattern transfer has been obtained in a reliable manner. Key geometrical dimensions were measured throughout the process, during both PDMS replication and the metallic micromold replication, but there were no observable dimensional changes.

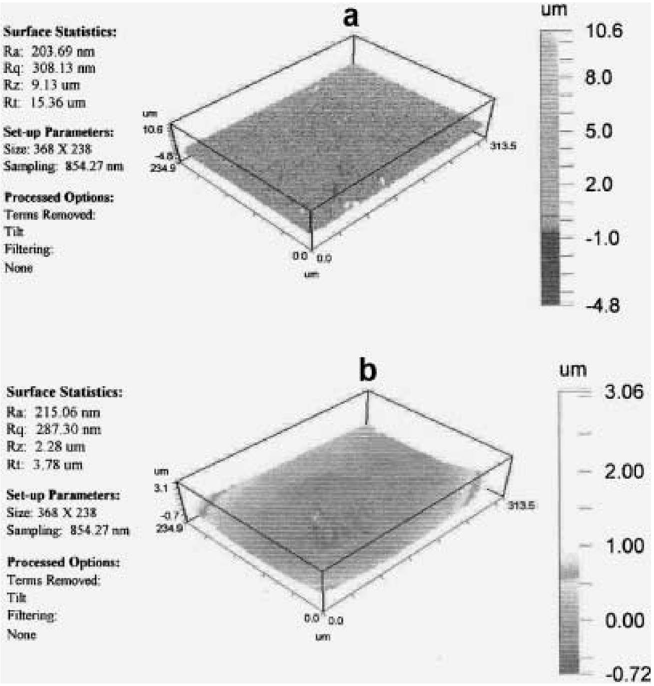

Fig. 7. Typical surface roughness of **a** the metallic micromold insert and **b** the replicated PDMS mold

4 Conclusions

Massive replication of polymeric HARMs using PDMS casting in a non-cleanroom environment was demonstrated. Surface roughness of both the metallic micromold insert and the replicated PDMS molds were measured using an interferometric surface profiler. No major surface wear or roughness was observed on either the metallic micromold insert or the PDMS HARMs. Such PDMS-based polymeric HARMs can be used for a variety of μTAS applications due to their excellent hydrophobic characteristics. In addition, a novel mold insert replication technique using molded PDMS to massively reproduce high quality, high precision metallic micromold inserts in a cost production manner was demonstrated.

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