Induction heating ferromagnetic particles embedded PDMS mold for microstructure embossing

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Abstract
Polydimethylsiloxane (PDMS) is an excellent soft mold material with the advantages of precise replication, easy demolding, and low production cost. However, the strength and hardness of PDMS are relatively low, and PDMS cannot be directly inductively heated. In this study, PDMS is embedded with ferromagnetic powders to increase its hardness and make it heatable. Direct induction heating of the PDMS mold can raise its inherent temperature, increase the heating efficiency by 100% compared with pure PDMS, and improve the shortcomings of uneven surface temperature distribution from high thermal resistance. Furthermore, adding the ferromagnetic metal powder to PDMS can improve its conductivity and make the mold a high-low surface temperature gap as low as 1.6 °C. Adding nickel powder to the PDMS mold makes the hardness 2.29 times higher than that of pure PDMS and can withstand a pressure of 7 kg cm⁻², which is very conducive to hot embossing. This study used a self-designed five-sided cladding iron block base and a PDMS mold with ferromagnetic metal powder for hot embossing. This heating apparatus can quickly raise the PDMS surface temperature and emboss deep V-groove microstructures on the polycarbonate (PC) film; the replication performance can reach more than 97%.

1. Introduction
The microstructures on the polymer surface are very attractive for commercial applications ranging from biomedical field, optical components, and brightness enhancement modules. Micro-hot embossing is the most commonly used method to fabricate microstructures on the polymer surface due to its low cost and easy operation. Generally, metal molds are widely used for micro-hot embossing due to their high strength and induction heating capability. But the production process of metal mold is cumbersome, resulting in increased cost and time-consuming. PDMS is currently the most commonly used silicone elastomer for soft molds. PDMS has good replication capability and can directly replicate the feature sizes of the microstructures. The cost-effective production of PDMS mold is relatively simple, accurate, and time-saving. However, the strength and hardness of PDMS are relatively low. Using an external heat source to treat the PDMS for hot embossing, indirect heating is generally adopted. The indirect heating methods of PDMS mold for hot embossing include vacuum oven heating [1], electric hot plate heating [2, 3], infrared lamp heating [4], graphene composite heater [5–7], and mobile induction heating stainless steel metal interlayer, etc [8]. The heating conduction is indirect, and the efficiency is poor. In addition, PDMS itself is an insulator, non-ferromagnetic, and cannot be directly inductively heated.

As for the hot embossing of PDMS, several teams have developed some hot embossing techniques for soft PDMS molds. For example, Narasimhan et al [2] used a general plate-to-plate (P2P) commercial embossing machine to complete the hot embossing PMMA of the PDMS mold for the microfluidic devices. Goral et al [9] used two 3/4-inch-wide binder clips, glass slides, and a standard oven heating to imprint a 2 mm thick PDMS mold. Gao et al [10, 11] proposed an air-cushioned imprinting method. Weng et al [12–15] developed an electromagnetically controlled magnetic soft mold imprinting technique. Although the research can use PDMS...
In this study, the thermomechanical properties and hardness of PDMS mold play a vital role in the hot embossing process because the harder the mold and the more uniform temperature distribution, the higher the quality of the imprinted microstructure. Kim et al. [17] changed the mixing ratio of AB agents from 10:1 to 5:1 with 85 °C hardening bake for three days; PDMS hardness can be improved by 32%. The increase of curing agent makes the PDMS molds still have thermal stability at a temperature up to 300 °C, allowing hot embossing under higher temperatures and pressures, which is more favorable to improving the replication rate.

In addition to the indirect heating of the PDMS through an external heat source, another heat source is generated inside the PDMS through internal high-frequency induction heating. At present, PDMS embedded magnetic particle induction heaters are mainly used in the research of micro-heating chips in the biomedical field [18-20]. The purpose of this research is to use ferromagnetic powder such as iron, nickel, etc, to mix with PDMS A and B agents evenly, and then through the appropriate baking time to increase the PDMS hardness for preventing soft mold deformation during the hot embossing process. In this study, the different wt% of Fe or Ni powder relative to PDMS is mixed with 5:1 PDMS, embedded into a PDMS mold, and then with the proper baking time, the final hardness can be increased by 129%. In addition, this research also designed a five-sided cladding induction heating apparatus. The heating method is different from the general traditional single-sided contact heating method. The iron block is used as the heating base surrounding the PDMS mold in five contact sides to make the heating more efficient, the surface temperature distribution more uniform. The hot embossing system attached a high-density silicone sheet to the PDMS, Fe-PDMS, or Ni-PDMS surface with a mold size of 30 mm × 30 mm × 2.5 mm (thickness); a pneumatic cylinder is tuned to emboss the V-groove microstructure onto a PC substrate. Under the same system and heating conditions, the heating efficiency of 70 wt% Ni-PDMS composite can be improved 100% higher than that of pure PDMS. The maximum temperature of Ni-PDMS composite is also 25.6 °C higher than that of pure PDMS. Next, an infrared thermal imager camera was used to analyze the temperature difference in surface uniformity distribution between pure PDMS and Ni-PDMS composite. The experiment found that the surface temperature difference of Ni-PDMS composite is between 1.6 °C ∼ 3.2 °C, which is better than 9.8 °C ∼ 30 °C of pure PDMS. The replication rate confirms that Ni-PDMS composite with Shore A hardness 87 HA can bear the embossing pressure over 6.8 atm during plate-to-plate (P2P) embossing without affecting the quality of the imprinted product. The success of Ni-PDMS composite embossing will create opportunities for future roller embossing and new applications.

2. Experimental methods

2.1. Hardness experiment

2.1.1. PDMS hardness experiment

PDMS Sylgard 184 Silicone elastomer kit, containing silicone elastomer base A and curing agent B, was purchased from Dow Corning Corp. USA. The manufacturing steps of PDMS mold are shown in figure 1: mixing, stirring, degassing, casting, curing, demolding, and then baking. In this experiment, we test the different mixing AB ratios of agents from 3:1 to 20:1. After curing for one hour at 85 °C, an additional 24 to 72 h of hardening baking experiment was performed at 85 °C once again to improve the hardness of PDMS, thereby enhancing the thermomechanical properties of PDMS and making it more suitable for hot embossing.

2.1.2. PDMS embedded with iron particles

In this study, the 2–4 μm ultrafine iron powder, purity 99%, was added to PDMS to enhance its hardness further. In addition, ferromagnetic material iron and the Fe-PDMS composite mold can be inductively heated. Fe weight percentages (30%, 50%, 66%, 80%) wt% are mixed with PDMS AB agent of 5:1 ratio to obtain the better hardness than PDMS as the same procedure shown in figure 1.
2.1.3. PDMS embedded with nickel particles
Since Vale T123 nickel powder has a smaller skin depth and higher bulk density than Fe powder, high-purity ultrafine nickel powder, 99.8%, is also tested for the hardness experiment. The spherical particle size of 3.5–4 μm, Ni (30%, 44%, 50%, 70%) wt% repeats the same experiment as the previous mixing procedures. Figure 2 shows the cross-sectional SEM image of nickel powder uniformly mixing with PDMS to form a Ni-PDMS composite mold.

2.2. Induction heating system
Figure 3 shows the design of the induction heating system with a five-sided induction heating iron block. The main mechanisms of the machine are a pneumatic cylinder, aluminum block, high-density silicon sheet, five-sided cladding iron block base, and spiral induction coils. After the iron block is heated by induction heating, the heat is transferred to the PDMS embedded in the iron block. The PDMS inside the iron block is a five-sided cladding heat conduction to the PDMS, and its coverage area to the PDMS is up to 57%. The heat transfer
efficiency is better than that of the single-sided contact heating surface. This design can reduce the heat dissipation and heat loss of the PDMS surface, lower the temperature difference on the surface, and maintain the imprinting temperature of the PDMS mold. The hot embossing system uses a low-cost and straightforward P2P embossing with the pneumatic cylinder, aluminum block, and high-density silicone sheet. With this setting, we carry out a series of induction heating, heating efficiency, temperature uniformity, pressure control, and hot embossing experiments between Ni-PDMS composite and pure PDMS.

2.3. Heating experiment
PDMS itself is a non-conductor, and the general heating method is indirect heating by an external heat source. In this experiment, a five-sided cladding iron block is used to increase the temperature by induction heating, and then the heat is transferred to the embedded PDMS mold. Induction heating the iron block with a frequency of 80 kHz and a power of 7.5 kW for 30 s, the temperature of the iron block can quickly rise to about 400 °C and conduct heat to the PDMS mold. The thermal conductivity of PDMS is 0.15 W/m.K, with high thermal resistance and difficulty in heat-conducting. Therefore, if we want to heat the PDMS mold to the PC glass transition temperature ($T_g$) 150 °C, or even higher replication rate temperature 175 °C, heating with pure PDMS is still too slow, as shown in figure 4. Finally, the heating efficiency can be improved by incorporating ferromagnetic particles into the PDMS to solve the heating problem. Therefore, we conducted a series of heating efficiency experiments to compare 70 wt% Ni-PDMS composite with pure PDMS.

2.4. Thermal analysis
IR thermal imaging camera uses photoelectric technology to detect the radiant heat energy of an object and then converts the heat energy into an infrared band signal, converts this signal into a recognizable image pattern, and can further calculate the temperature value. This infrared detection technology of radiant heat emitted by the object allows the user to observe the temperature distribution and changes on the object surface only through the screen display. The range of different temperature scales can be divided into various colors to distinguish on...
display. This experiment uses the F20 infrared remote temperature thermal imaging analyzer produced by NEC to analyze the heating status and temperature uniformity. The system with IRM F30 software can analyze and capture IR images of surface temperature during the induction heating of PDMS and Ni-PDMS composite mold. We record the temperature change at different periods and then get the temperature uniformity distribution data of the mold surface.

2.5. Hot embossing
In this experiment, a Ni-PDMS composite V-groove replica mold with 5:1 PDMS and 70 wt% 3.5 μm nickel powder was replicated from a metal mold. Then the embedded five-sided cladding system in figure 3 is used for induction heating experiments. The iron block is heated to 400 °C for 40 s through the induction coil, and then the temperature is transferred to the Ni-PDMS composite by the five-sided contact surfaces of the iron block. Adjust the setting embossing temperatures and pressures as shown in table 1. The temperature must be higher than the glass transition temperature \((T_g)\) of the PC film. Then, use the pneumatic cylinder directly to apply pressure to the microstructure of Ni-PDMS composite mold and PC film with 4 ~ 7 kgf cm\(^{-2}\) for 10 min. And under the action of the constant pressure, the PC material is deformed to flow and fill the microstructure. After the filling is completed, the system is cooled below the glass transition temperature of the PC under holding pressure and finally cooled down to room temperature to release the pressure and demold.

3. Results and discussion

3.1. PDMS mold hardness
Generally, we use the mixing ratio of Dow Corning Sylgard 184 AB agent 10:1. However, because PDMS is a soft mold, the Shore A hardness is only 38 HA. To increase the hardness of PDMS to 60 HA, we can change the AB agent’s mixing ratio from 10:1 to 5:1 and at least 24 h baking for hardening after curing. Moreover, we can further improve PDMS’s hardness and thermal conductivity by adding ferromagnetic metal particles such as iron, nickel to increase the hardness to 87 HA.

3.1.1. The hardness of pure PDMS
The method of PDMS curing is generally to mix AB agents at a ratio of 10:1 uniformly. After baking for 1 h or standing for 24 h, they will be cured and formed. Its Shore A hardness is about 38 HA, equivalent to an eraser’s hardness.
hardness. It is the commonly used soft mold PDMS hardness if the imprinting pressure is too large, and the mold can easily deform. Table 2, the PDMS hardness can be improved by changing the mixing ratio of the AB agent from 10:1 to 5:1. The mixing ratio of 5:1 can obtain the best hardness no matter after curing or hardening baking. The hardening experiment reached the highest hardness point of 60 HA after baking for 48 h, an increase of 58% compared with the 10:1 after curing.

3.1.2. The hardness of Fe-PDMS composite
Table 3, 80 wt% of Fe and PDMS AB agent ratio 5:1 are mixed and then curing at 85 °C for 1 h. After 72 h of baking, the PDMS hardness reaches the highest point, 86 HA, equivalent to the heels’ hardness of shoes, 2.26 times the 10:1 pure PDMS in table 1. After baking 72 h, the hardness of Fe-PDMS composite increased by 126%, compared with 10:1 pure PDMS, 38 HA.

3.1.3. The hardness of Ni-PDMS composite
The weight percentage of Ni, wt% (30%, 44%, 50%, 70%) mixed with PDMS AB agent 5:1, and the results are shown in table 4. The hardness value of 70 wt% of Ni-PDMS composite can reach 87 HA after baking for 72 h, and the Shore A hardness value is near the value of Fe-PDMS composite 80 wt%. The reason is the bulk density of nickel powder is 1.9 ~ 2.3 g cm⁻³, which is 53%–90% larger than the bulk density of iron powder, 1 ~ 1.5 g cm⁻³.

From the above experiments, after adding the ferromagnetic powder to PDMS 5:1 to form Ni-PDMS composite 70 wt%, the hardness reaches the highest value 87 HA after baking 72 h, 129% higher than PDMS 10:1 curing one hour. The hardness is equivalent to the eraser’s hardness rising to a harder one than the sole’s heel, as shown in figure 5. Therefore, adding the ferromagnetic powder to improve PDMS hardness is beneficial to subsequent induction heating and high fidelity replication rate. Ferromagnetic particles embedded in PDMS can also bear the embossing pressure of 6 ~ 7 kgf cm⁻², the same applied pressure to a rigid metal mold for the hot embossing process.

3.2. Temperature uniformity
In the same induction heating experiment, Ni-PDMS 70 wt%, compared with pure PDMS by an infrared camera for surface temperature uniformity analysis. As the surface temperatures were heating to near 175 °C, the IR camera shows that the high-low temperature difference on the surface of pure PDMS is less than 12.4 °C. In contrast, the high-low temperature difference of Ni-PDMS composite with 70 wt% nickel powder is only

| Table 2. PDMS shore A hardness scale at different mixing ratios and baking time. |
|-----------------|-----|-----|-----|-----|-----|-----|-----|
| PDMS A/B Ratio  | 3:1 | 4:1 | 5:1 | 6:1 | 10:1| 15:1| 20:1|
| Curing 85 °C 1 h (HA) | 40 | 48 | 52 | 48 | 38 | 21 | 13 |
| Baking 85 °C 24 h (HA) | 47 | 54 | 57 | 56 | 49 | 33 | 23 |
| Baking 85 °C 48 h (HA) | 60 | 60 | 60 | 60 | 60 | 60 | 60 |
| Baking 85 °C 72 h (HA) | 60 | 60 | 60 | 60 | 60 | 60 | 60 |

| Table 3. Fe-PDMS composite Shore A hardness scale with different Fe wt% and baking time. |
|-----------------|-----|-----|-----|-----|
| Fe ratio (wt%)  | 30% | 50% | 66% | 80% |
| Curing 85 °C 1 h (HA) | 47 | 53 | 66 | 75 |
| Baking 85 °C 24 h (HA) | 60 | 60 | 73 | 83 |
| Baking 85 °C 48 h (HA) | 85 | 85 | 85 | 85 |
| Baking 85 °C 72 h (HA) | 86 | 86 | 86 | 86 |

| Table 4. Ni-PDMS composite Shore A hardness scale with different Ni wt% and baking time. |
|-----------------|-----|-----|-----|-----|
| Ni ratio (wt%)  | 30% | 44% | 50% | 70% |
| Curing 85 °C 1 h (HA) | 53 | 67 | 70 | 75 |
| Baking 85 °C 24 h (HA) | 62 | 74 | 77 | 83 |
| Baking 85 °C 48 h (HA) | 86 | 86 | 86 | 86 |
| Baking 85 °C 72 h (HA) | 87 | 87 | 87 | 87 |
1.6 °C (±0.8 °C), as shown in figure 6. In this experiment, with the same induction heating condition, 7.2 kW, the PDMS and Ni-PDMS composite were comparative analysis, respectively, and use the thermal infrared camera to inspect 155 °C, 165 °C, 175 °C, 185 °C surface temperature distribution. The surface temperature uniformity analysis shows that the distribution range of temperature difference on the surface of pure PDMS is between 9.8 °C and 30 °C, and it increases with the increase of surface temperature. The temperature difference between the surface of the Ni-PDMS composite is only 1.6 °C ~ 3.2 °C, in figure 7. The temperature distribution on the Ni-PDMS composite surface is highly uniform.

Induction heating can quickly heat the Ni particles embedded in the PDMS mold, thereby significantly improving PDMS’s shortcomings, such as high thermal resistance and low thermal conductivity, and making the surface temperature distribution of PDMS more uniform. Improving the PDMS surface temperature uniformity is also very beneficial to hot embossing temperature control and replication rate.

### 3.3. Heating rate

In the heating efficiency experiment, PDMS takes 110 s to reach 160 °C. Ni-PDMS composite only takes 55 s to attain 160 °C under the same 7.2 kW, 40 s heating conditions. Figure 8 shows that under the same heating conditions, the heating efficiency of Ni-PDMS composite is increased by 100% (2.0 times) than that of PDMS. According to figure 8, comparing PDMS and Ni-PDMS composite heated to their highest temperature under the same heating conditions, Ni-PDMS composite can be 25.8 °C higher than PDMS temperature, which is 16% higher. According to the experimental results, we select Ni-PDMS composite 70 wt% as the best choice for our hot embossing mold because Ni-PDMS composite is far superior to pure PDMS in heating efficiency and maximum temperature.

Explore why the heating efficiency and maximum temperature of Ni-PDMS composite are far higher than that of PDMS. The main reason is that in addition to the external heat source from the heat transfer of the iron block, the Ni-PDMS composite has an internal intrinsic heat source under induction heating due to the addition of nickel powder. After Ni-PDMS composite undergoes the same induction heating conditions, the maximum temperature can rise to 45 °C after 7.2 kW and 40 s heating. To confirm this hypothesis, we replaced the five-sided cladding iron block with a Teflon carrier, performed the same heating experiment on the Ni-PDMS composite, and observed the temperature rising trend of its intrinsic heat source, as shown in figure 9. It explains why Ni-PDMS composite is better than PDMS regardless of heating efficiency and maximum temperature under the same heating condition.

### 3.4. Replication of V-grooves on polymeric substrates

We divided the PC film test piece into two upper and lower areas for the data measurement. In the discussion of the effect of temperature and pressure on the replication rate, we first fix the pressure parameter to 5 kgf cm⁻² and change the imprinting temperature range to 155 °C, 165 °C, 175 °C, 185 °C; the average replication rate and
Figure 6. At the surface temperature near 175 °C, the IR camera shows (a) the difference between the high-low temperature variation on pure PDMS surface is less than 12.4 °C. (b) The temperature difference of Ni-PDMS composite 70 wt% is only 1.6 °C (±0.8 °C).

Figure 7. The surface temperature difference distribution of pure PDMS ranges from 9.8 °C to 30 °C, increasing with the surface temperature rising, while Ni-PDMS composite surface temperature difference is relatively uniform between 1.6 °C and 3.2 °C.
trend with the temperature obtained in figure 10. When the embossing temperature is increased from 155 °C to 185 °C, the average replication rate has an increasing from 5.9% to more than 98%.

In discussing the replication rate at a fixed temperature, we set the temperature at 175 °C and changed the imprint pressure to the range of 4 ~ 7 kgf cm$^{-2}$ for experiments. Figure 11 shows the replication rate trend with

Figure 8. The comparison of the heating efficiency between PDMS and Ni-PDMS composite through induction heating the five-sided cladding iron block for 40 s.

Figure 9. (a) A Teflon base replaced the five-sided cladding iron block stage using the same heating condition. (b) The temperature rising was through the internal induction heating source of Ni-PDMS composite.

Figure 10. Temperature versus replication rate variation trend at a static pressure of 5 kgf cm$^{-2}$. 
embossing pressure at a fixed temperature 175 °C. At 4 kgf cm$^{-2}$ embossing pressure, the replication rate can reach 97%. The higher the embossing pressure, the higher the replication rate obtained. Compared with the influence of temperature on the replication rate, the embossing pressure has a less significant impact on the replication rate. And because the hardness of Ni-PDMS composite is higher than that of pure PDMS, even if the pressure is increased to 7 kgf cm$^{-2}$, there will be no deformation of the soft mold due to excessive embossing pressure, which will affect the replication rate. The 3D profile of the imprinted PC film surface is measured by the Laser Scanning Confocal Microscope (LSCM), shown in figure 12.

4. Conclusion

We conducted a comprehensive study of the pure PDMS and Ni-PDMS composite materials, including hardness comparison, temperature uniformity, heating rate, maximum temperature, and replication rate. Ni 70 wt% was mixed with PDMS AB agent 5:1, cured and baked at 85°C for 72 h. The hardness is up to 87 HA, which is 2.29 times that of pure PDMS. Regarding heating efficiency, Ni-PDMS composite material is 100% higher than pure PDMS, and the maximum temperature is also 25.8 °C higher than pure PDMS. Ni-PDMS composite surface temperature distribution is more uniform, and the surface temperature difference can go...
down to 1.6 °C. Hot embossing of Ni-PDMS composite mold, as the temperature rises from 155 °C to 185 °C, the replication rate also increased from 5.9% to 98.6%. The experimental data also pointed out that the pressure has a minor impact on the replication rate than temperature. However, embossing pressure still affects the degree of the fidelity of the embossed structures to the original mold. The verification result confirms that the Ni-PDMS composite can withstand the pressure of 6 ~ 7 kgf cm⁻² (5.8 ~ 6.8 atm) during hot embossing without mold distortion.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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