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EFFECT OF TEMPERATURE AND TIME OF BAKING PHASE ON SU-8 PR FILM USED AS A HARD MASK FOR A DEEP LITHOGRAPHY

ẢNH HƯỞNG CỦA NHIỆT ĐỘ VÀ THỜI GIAN SẤY ĐẾN LỚP VẬT LIỆU CẢN QUANG SU-8 SỬ DỤNG LÀM MẶT NẠ CỨNG TRONG KỸ THUẬT QUANG KHẮC SÂU

Bui Tuan Anh

ABSTRACT

The surface profile of Fresnel lens during the fabrication process depends on many factors including the thickness, flatness, and pattern clarity of a photoresist (PR) film used as a hard mask for the Si lithography. SU-8 PR with a high viscosity, which is capable of creating large thickness films, can be used as a hard mask in the Fresnel lens fabrication processes. However, it is not easy to achieve a film thickness of about 10 µm with the requirement of a flat surface, free of air bubbles after the baking phases. This paper presents the effect of temperature and baking time on the quality of SU-8 PR film. Accordingly, to achieve the required quality of PR film, a two-step baking phase was employed. The time and temperature for a soft-baking corresponds to 60 minutes and 60°C, respectively, while a post exposure baking (PEB) phase must be kept at 90°C for about one hour. The experimental results show that the defects including PR cracking, bubble problem on the PR layer are eliminated. Thus, these baking parameters are feasible for the application used a thick PR film as a hard mask in a deep lithography process.

Keywords: *SU-8 PR, film thickness, baking temperature, baking time.*

TÓM TẮT

Biên dạng bề mặt của thiết bị hội tụ Fresnel trong quá trình chế tạo phụ thuộc vào nhiều yếu tố bao gồm độ dày, độ phẳng và độ rõ nét hoa văn của lớp cản quang (PR) được sử dụng làm mặt nạ cứng trong kỹ thuật quang khắc Si. Vật liệu cản quang SU-8 với độ nhớt cao, có khả năng tạo ra các lớp cản quang có độ dày lớn, có thể được sử dụng làm mặt nạ cứng trong quy trình chế tạo thiết bị hội tụ Fresnel. Tuy nhiên, để đạt được độ dày màng khoảng 10µm với yêu cầu bề mặt phẳng và không có bọt khí sau các giai đoạn sấy là rất khó khăn. Bài báo này trình bày ảnh hưởng của nhiệt độ và thời gian sấy lên chất lượng của màng vật liệu cản quang SU-8. Theo đó, để đạt được chất lượng cần thiết của lớp cản quang, giai đoạn sấy gồm hai bước đã được sử dụng. Thời gian và nhiệt độ cho sấy mềm tương ứng là 60 phút và 60°C, trong khi giai đoạn sấy sau phơi sáng phải được giữ ở 90°C trong khoảng một giờ. Kết quả thử nghiệm cho thấy các khuyết tật trên lớp vật liệu cản quang bao như các vết nứt, bọt khí đã được loại bỏ. Do đó, các thông số sấy này là phù hợp cho ứng dụng đã sử dụng màng cản quang dày làm mặt nạ cứng trong quy trình quang khắc sâu.

Từ khóa*: Vật liệu cản quang SU-8, chiều dầy màng, nhiệt độ sấy, thời gian sấy.*

School of Mechanical Engineering, Hanoi University of Science and Technology
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1. INTRODUCTION

In an ultrasonic device, the Fresnel focusing lens, which can be used to focus ultrasound energy, has been investigated and fabricated for an application of acoustic ink printing [1-4]. The Fresnel lenses can offer a planar geometry and relative ease of fabrication in comparison to other forms of focusing lens. However, the geometry is critical for efficient focusing, and thus tight thickness control of the lens elements is usually needed. A design of Fresnel lenses which use multiple-phase levels to approximate the curvature of spherically focusing field offer high efficiencies was investigated by B. Hadimioglu et al. in 1993 [5]. In 2011, M-C Pan et al. presented a design and fabrication of four-level Fresnel lenses with operating frequency of 100MHz and 200MHz through micro-electro-mechanical systems (MEMS) [6]. The authors showed a three-mask fabrication process using silicon dioxide (SiO_2) as a hard mask in a deep etching process. The 100MHz multi-level Fresnel lens was designed with a step height and maximal radial distance of the lens are h = 4.55 and r_{max} = 244µm, respectively. Besides, a two-mask fabrication process employing SU-8 photoresist (PR) in the lithography was used to fabricate the 100 MHz Fresnel lenses with purpose of addressing the difficulty of non-uniform photoresist coverage because of the high aspect ratio of the lens [7]. With high viscosity and good photosensitivity, SU-8 negative PR is widely used in the lithographic applications employing a thick film and high aspect ratio of a micro-structure. Tens and even hundreds of micrometer thickness of the photoresist layer, which can be obtained in a single coating process, is one interest in our application. Besides, other advantages of SU-8 in micro-electro-mechanical systems such as lowcost, low optical absorption in the near-UV range, stability of thermal and chemical, and good resolution with vertical sidewall profiles are also considered [8]. In this fabrication, SU-8 2010 PR was used due to its good properties and suitable for deep reactive-ion etching (DRIE). Besides, SU-8 is a negative PR used in this experiment so that the PR regions, which will be remained or striped after taking lithography, must be noticed when designing the mask. Hence, the photoresist region exposed will be kept after development process. A patterning process with negative and positive photoresists can be illustrated as Figure 1.

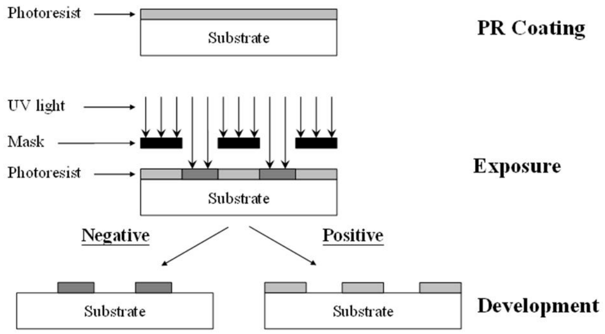


Figure 1. Patterning process with negative and positive photoresists (adapted from [9])

The precise profile of Fresnel lens depends on the uniform thickness of PR layer in the lithography. The PR thickness is determined by spin speed and PR viscosity. Hence, a slow speed or PR with high viscosity is necessary to obtain a thick PR layer. A more detailed investigation of the influence of PR (eg., viscosity, coating speed, etc.) on the profile of the focusing lens are ongoing. Despite the high viscosity of SU-8 PR, it is not easy to create a thick PR layer over 10µm with a flat surface. Especially on the small area as in the process of making Fresnel lens operating with frequency of 100MHz. An attention should be paid that the influence of temperature and time factors during the baking period must be considered. This article presents the effect of baking temperature and baking time on the quality of SU-8 PR film, which is used as a hard mask for the fabrication of 100MH Fresnel lens, to find the most feasible condition for making such a good PR film.

2. EXPERIMENT PROCEDURE

The Si wafers with diameter of 4-inch was used to prepare the substrate for fabricating the Fresnel lenses. The specifications of 4-inch Si wafers are shown in Table 1. The wafer was firstly cut into some square samples with a dimension of 40mm × 40mm before being rinsed in acetone, IPA and DI water to remove any particles or chemical substances on their surfaces. Subsequently, nitrogen gas was used to remove most of DI water on the surface of silicon substrates before taking a prebaking at a temperature of 120°C for 15 minutes on a hotplate with the purpose of full dehydration. The samples were kept on the hotplate to gradually cool down to room temperature. The sequent steps of the fabrication may be illustrated in Figure 2.

Table 1. Specifications of 4-inch silicon wafers

Specification	Value
Diameter	100 ± 0.2mm (6")
Thickness	525 ± 25µm
Method	CZ
Type	N
Dopant	Phosphorus
Orientation	(100)
Resistivity	1 - 10 (Ω.cm)

As shown in Figure 2, the fabrication of Fresnel lens using two masks associated with two etching steps is indicated. Our previous experiments showed that two- and three-mask processes employing a SiO_2 layer as a hard mask in DRIE have been used in the fabrication [6]. However, the disadvantage of non-uniform photoresist coverage because of the high aspect ratio (ratio of the feature height to its width) of the lens was faced. Hence, one or two outer ring group of the fabricated Fresnel lenses was not taken shaped as designed. Therefore, a two-mask process employing SU-8 in the lithography was applied with the purpose of addressing those difficulties.

Several sets of cleaned substrates were separated to examine the relationship between spin coating speed and thickness of the PR layer. Each sample was coated at a low speed, which was about 1000rpm, for 30 seconds and a higher speed for 60 seconds in order to obtain a particular thickness of PR layer following by a soft-baking. With a high viscosity PR, a thick PR film has been created that is expected to have a good protection of the unetched region of silicon surface in the deep etching.

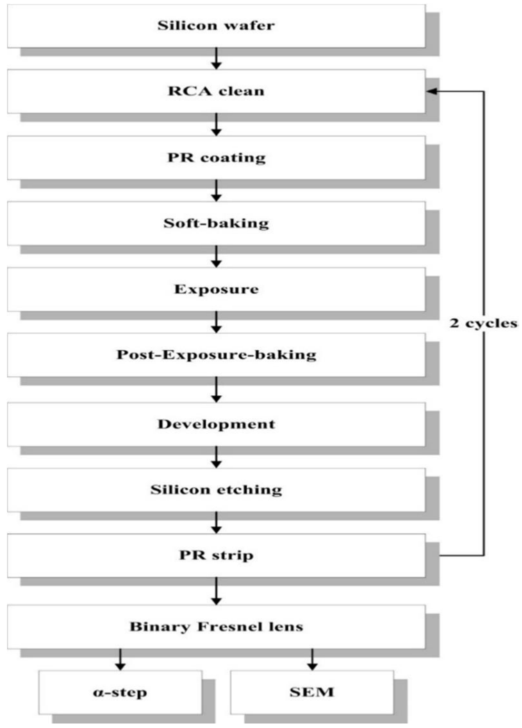


Figure 2. Sequent steps of fabrication of Fresnel lens employing SU-8 PR

As known, every parameter of the lithography also affects the quality of the photoresist film; hence, the samples were baked at various manners to investigate their influences on the PR films. Those are at 60°C to 90°C with different baking times. Especially, the rest time lasting days before soft-baking was really needed to improve the uniformity of the PR layer. Similarly, exposure dose is also one of most important factors that determine the resolution of exposed patterns, and therefore, various exposure doses were applied to see their influence. Post exposure baking (PEB) and development time were also investigated with the same purpose. In this experiment, a mask aligner machine (Machine type: Quintel Aligner Q4000) is employed for exposure. The I-line with its wavelength 365nm is used and the light intensity is set at 11mW/cm^2. Hence, the exposure time would be varied to obtain an exposure dosage required. To consider the influence of temperature and time of baking stage on the PR film, the development time and exposure dosage need to choose in advance. In our previous experiments, the feasible exposure dosage, which is about 220mJ/cm^2 was suggested for the exposure with PR film thickness about 10µm. the samples were immersed in a beaker containing developer solution for 4 minutes in an ultrasonic cleaner. Hence, the baking time and temperature in first experiment was set from 1 to 2 minutes at 65°C for the pre-soft bake and from 2 to 3 minutes at 95°C for the soft bake as the suggestion of the PR manufacturer; and then cooling down to room temperature. In addition, a two-stage of baking was also considered for other experiments, which include 60°C in 60 minutes and 90°C for another 60 minutes and gradually cool down to room temperature.

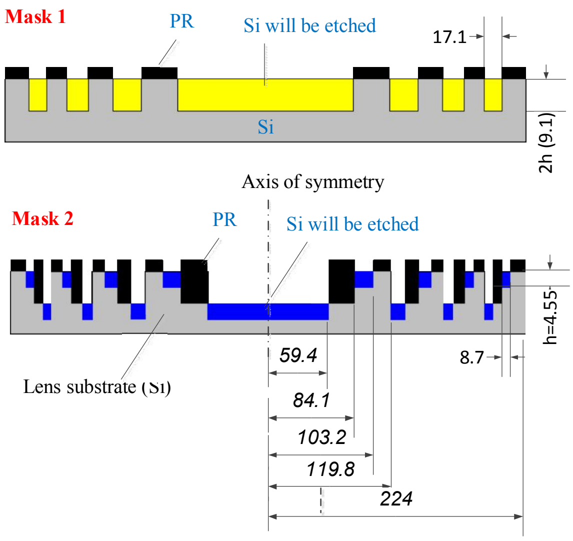


Figure 3. Two-mask fabrication process of Fresnel lens employing SU-8 PR as a hard mask

The fabrication processes of acoustic focusing lens are carried out by two cycles corresponding to two different masks. In the first cycle fabrication processes, the Si substrate was etched with the depth of 2h, where h = 4.55µm is the step height of Fresnel lens. And then, the wafer was aligned and exposed with 2nd mask and repeated the same processes with the depth of Si etching h in the second cycle. The fabrication process of Fresnel lens employing SU-8 PR film as a hard mask is briefly described in Figure 3 (unit in µm).

After each silicon etching, the PR layer is striped and cleaned for the subsequent measurements. Hence, the samples are placed in a dissolved solution tank in 20 min, which includes sulfuric acid and hydrogen peroxide with a volume ratio H_2SO_4:H_2O_2 = 3:1. The solution is heated until 150°C and kept for 20 min to ensure all PR stripped. The subsequent necessary measurement is to make sure whether the fabrication results meet the requirement or not to continue taking the next steps of the fabrication.

3. RESULTS AND DISCUSSIONS

The manner has revealed some advantages in the fabrication such as a thick PR film was obtained in a single spin coating; vertical sidewall of a high aspect ratio feature was also confirmed after exposure and development. However, some difficulties such as bubble problem on PR layer after soft-baking, non-uniform film after spin coating, diffraction, and partial cross-link of PR when exposure, overdevelopment, etc were faced. To confirm the relation between film thickness and spin-coating speed, some experimental trials were performed. The spin-coating at 1000rpm for 30 seconds and at a higher speed for 60 seconds were employed, the film thickness is shown in Table 2. Hence, a desired thickness can be determined. Through the trials, it showed that the PR film spin-coated at over 6000rpm that satisfied the expected thickness, which is about 10µm, with smooth surface, and then it was used in all experiments. As known, it is difficult to achieve a very flat surface with a high viscosity PR, especially, on a square sample. Taking spin-coating at such high speed and longtime could make the PR layer more uniform and smoother.

Table 2. SU-8 PR film thickness vs spin-coating speed

Spin-coating speed	Film thickness (µm)					
(rpm)	1	2	3	4	5	Mean
3000	14.10	13.86	13.85	13.98	13.95	13.95
4500	10.70	10.35	10.62	10.46	10.50	10.53
6000	8.93	9.20	9.31	9.05	9.10	9.12
7500	8.64	8.76	8.89	8.90	8.80	8.80

In the first experiment, the baking time and temperature was set as: 1 - 2 minutes at 65°C for the pre-soft bake; 2 - 3 minutes at 95°C for the soft bake as the suggestion from the data sheet of PR manufacturer; and then cooling down to room temperature. However, a phenomenon of bubble formation on the PR film surface, which appeared during soft baking, was a serious problem. It directly affects the fabrication result. Hence, the coated samples were transfer to a hotplate to bake at a temperate of 95^{o}C for several minutes due to its thickness [10]. Bubbles suddenly appeared after first several seconds of baking. That is probably the solvent evaporation was not completed because the solvent in the top layer of PR was first evaporated, this condensed the top layer and kept the solvent inside the deeper planes within PR film. In addition, a short baking time also made the PR films contain high solvent content that will generate high film stress during post-exposure baking (PEB) [11]. Therefore, a longer time and lower temperature of the baking were considered to let the solvent gradually evaporate to avoid being kept inside the PR film. Thus, a PEB with temperature of 95^{0}C for one hour was used, the result showed that a cracking problem appeared on the PR surface (as shown in Figure 4) . This may cause by the internal stress within PR film due to short PEB time. Thus, a longer PEB time was applied for those samples, which was one hour at 60^{0}C and increased to 90^{0}C for several last minutes to improve cross-linking in the exposed areas. Hence, the problem of film cracking was completely solved.

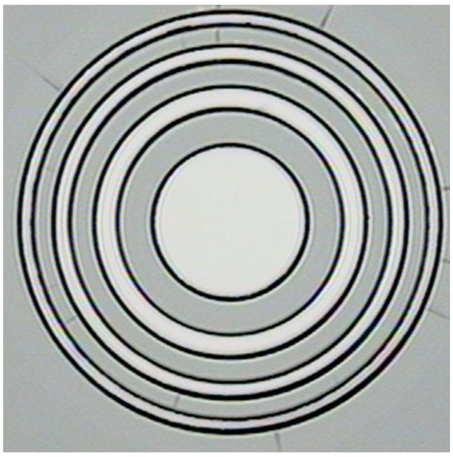
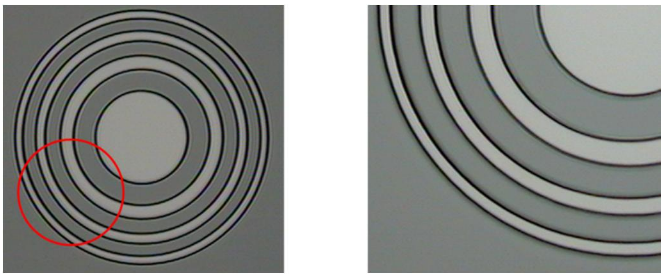


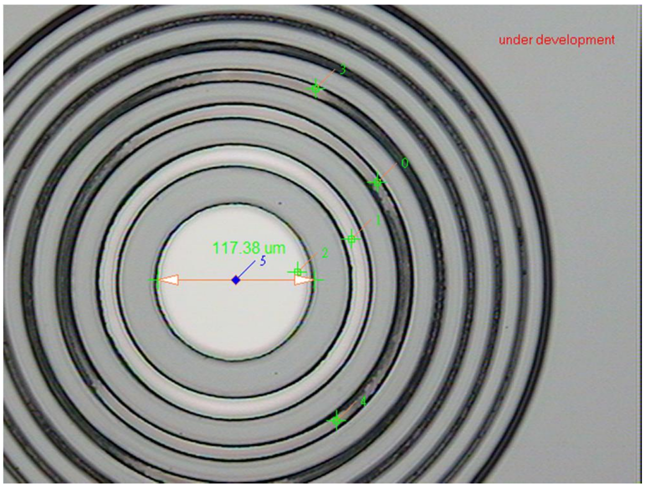
Figure 4. PR cracking in post exposure bake at 95^{0}C for 1 hour

In addition, a two-stage of baking was considered for other experiments, which include 60^{o}C in 60 minutes and 90^{o}C for another 60 minutes and then turn the hotplate off to gradually cool down to room temperature without taking the samples out of the hotplate. However, the bubbles still appeared in the first minutes but at a lower density in comparison with the previous manner. Especially, the size of bubbles progressively decreased while baking time was increased. Moreover, this caused the sample surface much roughness and affected the resolution of following exposure. Thus, a consideration of reduction of evaporation difference between surface and deeper regions of the PR film was performed. That is, leaving the samples in a very plat-leveled position for a day before having a two-step soft-baking process. High temperature and long PEB time may partially reflow the PR films; this may make the resist films more uniform. The problem of bubbles on the film surfaces was completely solved.

Besides, exposure dosage and development time are also important parameters that need to be considered. Low exposure dosages tend to make the feature become slopped with the top wider than the bottom because the exposure dose decreases as the transferred depth was increased. That means the exposure does not create enough acid to enable sufficient cross-linking during post exposure baking. Shown in Figure 5 (a), the PR in outer trenches was not tripped during the development process after a higher dosage was used in exposure. This probably caused by over-exposure, that means the dosage with a strong energy has made the top layer of the exposed area become cross-linked and changed its refraction index. The UV source transferred through that layer will be refracted to consolidate the bottom part of the lateral area of such small trenches. Figure 5 (b) shows an under-development phenomenon in the second cycle of Fresnel lens fabrication process. In which, the development time was not enough to remove the exposed PR pattern out the surface, especially, in small and deep trenches of the PR patterns. This problem was solved as the development time was increased.



(a)



(b)

Figure 5. Phenomenon of under development

In addition, a two-step of soft-baking shows its advantages in ensuring a uniform PR layer and eliminates bubbles on the surface. However, the quality of the different patterns in one test are quite different. Therefore, the temperature in different regions on the hot plate is measured, the results show that, when the temperature needed is about 60 or 90°C (shown in Figure 6), we need to adjust the hot plate temperature at 65 and 95°C, this is also a limitation of heating equipment during the baking phase.

Therefore, samples located at temperatures approximately 60°C in the first baking stage and 90°C in the second baking period were tested to compare with samples in other locations. In fact, the samples at the required temperature locations were tested, the results showed that the patterns of the PR were clear and satisfied the quality requirements for the fabrication process of Fresnel lens (shown in Figure 7).

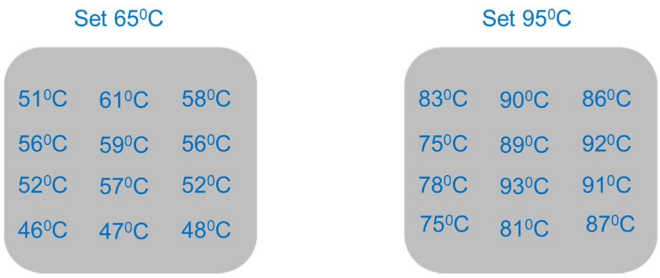


Figure 6. Temperature distribution on the hot plate

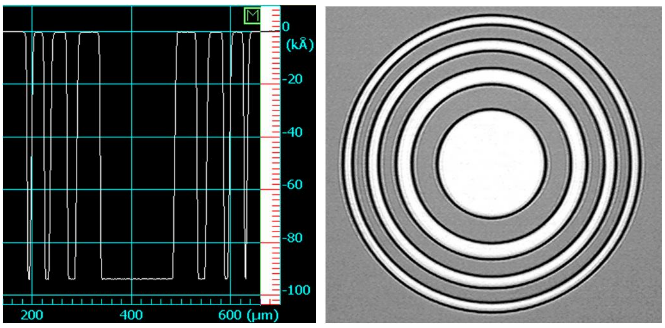


Figure 7. SU8-PR film after using a two-step of soft-baking

This shows that the selected baking temperature and time are large enough for the PR layer to stabilize, especially the gradual heating allow the solvent in the PR layer to have enough time to evaporate and slowly escape the surface, thereby eliminating the bubbles on the surface of the film.

4. CONCLUSION

In the fabrication process of Fresnel lens, a SU-8 PR film, which was used as a hard mask with a thickness of approximately 10µm, was employed. A RP film, which was coated on a Si substrate and ensured the quality requirements after the exposure and development phases, has been successfully manufactured. Beside the pre-selected fabrication parameters, the effect of temperature and time during baking on film quality has been shown. Accordingly, to ensure the best quality under the laboratory conditions, it is necessary to use a two-step baking phase, with the time and temperature for soft-baking corresponding to 1 hour and 60°C, respectively. At the PEB stage, the required temperature is about 90°C and maintained for about 60 minutes to eliminate PR cracking problems as well as bubbles on the film surface. Thus, this baking condition is most feasible with purpose of getting a smooth, thick, and clear pattern of PR film after exposure and development that can be used for the deep Si lithography. This can be also used for other applications that need a thick PR film with those requirements.

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THÔNG TIN TÁC GIẢ

Bùi Tuấn Anh

Viện Cơ khí, Trường Đại học Bách khoa Hà Nội