Resolving fabrication issues in photolithography and thin film deposition

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ABSTRACT

This study discusses the issues in two fabrication processes, viz. photolithography and thin film deposition, and their solutions. In the photolithography process using photoresists AZ-10XT and AZ-4562, the issue of bubble formation is observed after UV exposure. This issue was resolved by carefully adjusting the soft-bake parameters to ensure the solvent was completely removed without damaging the photoresist. Similarly, with the thin film deposition of Ti/Au using the electron beam evaporation technique, the issue of poor adhesion of the films was observed in the tape-peel test. It is inferred that the presence of moisture on the substrate is a reason for the poor adhesion of the deposited films. Hence, in-situ pre-deposition heating of the substrate is carried out inside the EBES to remove the moisture adsorbed on the surface of the substrate. The tape-peel test clearly shows that the adhesion quality of deposited films improved after the pre-deposition heating.

KEYWORDS

Photoresist,
Thin Film Deposition,
Electron Beam Evaporation.

1. Introduction

The fabrication processes are crucial for the semiconductor and microelectromechanical systems (MEMS) industries to create complex devices (Goodge, 1983; Hajare et al., 2022; Judy, 2001). These processes directly influence the performance, reliability, and cost-effectiveness of the device. As the device sizes are becoming smaller and smaller, precision and control of fabrication processes are essential to create intricate patterns and structures. Any issues in these processes, such as the ones we are addressing in this study, can significantly impact the product, making the need for effective solutions crucial. This study aims to contribute to this understanding by investigating and resolving specific issues encountered in two fundamental fabrication processes: photolithography and thin film deposition. Specifically, we investigate issues related to bubble formation in photolithography and poor adhesion in thin film deposition.

Similarly, the issue of bubble formation is discussed in section 2. In the photolithography process with AZ-10XT and AZ-4562 positive photoresists (PR), bubbles were formed in PR after UV exposure. The causes of the observed bubble formation and how to avoid this issue are discussed. It was shown that modifying the soft-bake process to ensure complete solvent removal from the photoresist before exposure solves this issue. Similarly, we discuss the issue of poor adhesion in the thin film deposition of Ti/Au using electron beam evaporation (EBES) is discussed in section 3. The poor adhesion was attributed to the presence of moisture on the substrate surface. To improve the adhesion, an in-situ pre-deposition heating step within EBES was introduced to eliminate adsorbed moisture. The results demonstrate a significant improvement in film adhesion after implementing this pre-heating technique.

It is to be noted that the experiments related to photolithography and thin film deposition were carried out in class 100 and class 1000 clean rooms, respectively, and all the clean room protocols (Whyte, 1999) were followed while performing the experiments.

2. Bubble Formation in AZ-10XT and AZ-4562 Positive Thick Photoresists During Photo-Lithography Process

Photolithography is one of the critical steps of semiconductor and MEMS fabrication. It involves

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Table 1
Photolithography process steps for AZ-10XT and AZ-4562 (ref. MicroChemicals).

AZ-10XT	AZ-4562
➢ Spin coating ○ Step 1: 500 RPM / 7 sec ○ Step 2: 2300 RPM / 45 sec ○ Step 3: 500 RPM / 7sec ➢ Soft bake at 110°C for 2min 45sec ➢ Re-hydration time: 10 min ➢ Exposure ○ Intensity: 18.6 mJ/cm^2 ○ Total exposure dose:651 mJ/cm^2 ○ Exposure time: 7 sec ○ Cycles: 5 ➢ Development with AZ726MIF developer ➢ Development time: 6min 45sec ➢ Final bake at 90°C for 5 min	➢ Spin coating ○ Step 1: 500 RPM / 5 sec ○ Step 2: 4000 RPM / 40 sec ○ Step 3: 500 RPM / 5 sec ➢ Soft bake at 110°C for 1min ➢ Re-hydration time: 10 min ➢ Exposure ○ Intensity: 18.6 mJ/cm^2 ○ Total exposure dose: 513 mJ/cm^2 ○ Exposure time: 6.9 sec ○ Cycles: 4 ➢ Development with AZ726MIF developer ➢ Development time: 3 min ➢ Final bake at 110°C for 2 min

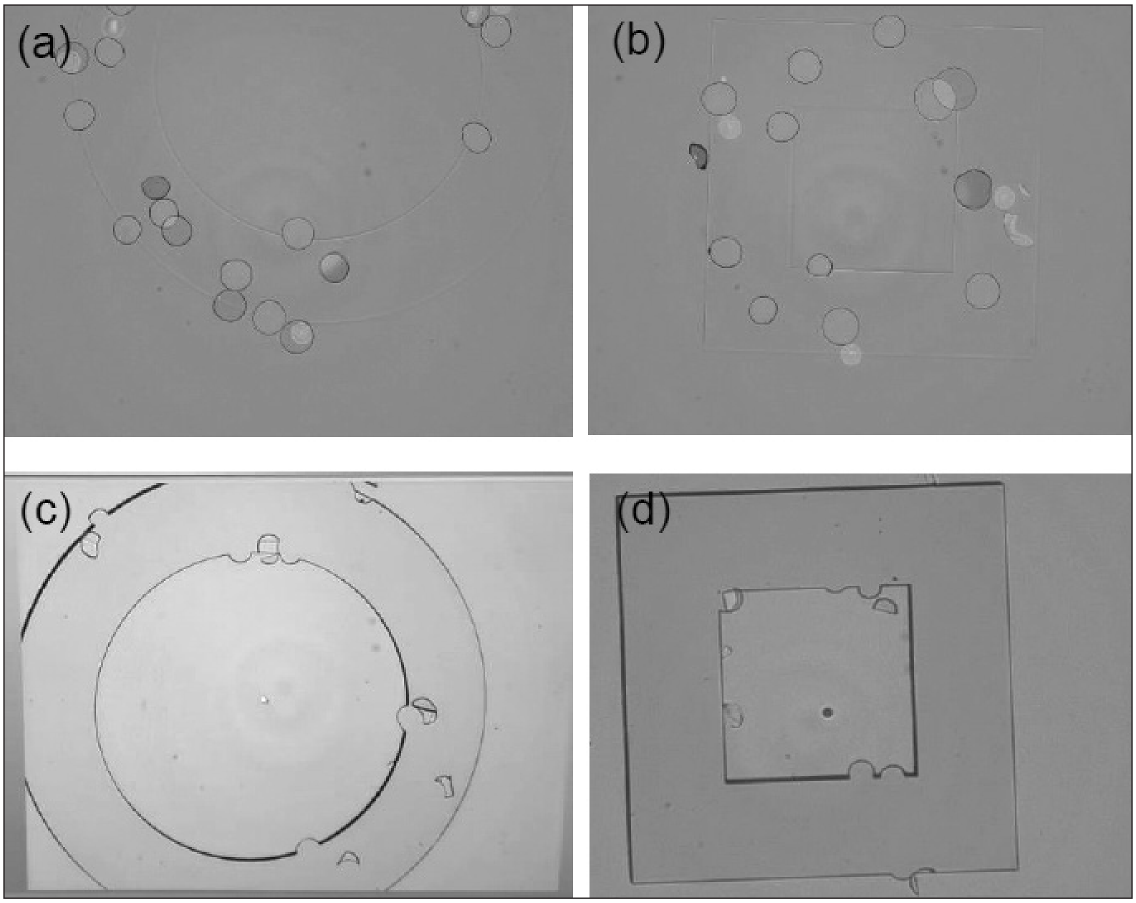

Fig. 1. The formation of bubbles in photoresist.
(a) and (b): after UV exposure, (c) and (d) : after the pattern development.

generating a pattern of photoresist (PR) on the substrate by exposing it to the light of a well-defined wavelength (typically UV). Usually PR contains three main ingredients: (a) resin, which acts as a binder for adhesion, chemical resistance, etc.; (b) sensitizer, which has photo-sensitive properties; and (c) solvent, which keeps the resist in the liquid form. PR have two types based on how they change after exposure to light. In positive PR, the light weakens the cross-linkages and makes the initially insoluble PR soluble in the developer solution. Meanwhile, in the case of a negative PR, the initially soluble PR becomes insoluble in the developer solution after exposure to light. Here, the issue of bubble formation in PR after UV exposure was discussed, along with the ways to avoid it.

Here, we present the issue of bubble formation in PR, which was observed after exposure to UV, discuss its reasons, and propose a solution to this issue.

The photolithography process starts with coating the PR on a substrate, such as a silicon wafer, using the spin coater. After coating, it is necessary to remove the excess solvent from the coated PR. This is especially important for thicker PR to ensure a well-defined photoresist pattern with higher resolution. Therefore, the sample is soft-baked on the hot plate at temperatures high enough to evaporate the solvent from PR. After soft-bake, the samples are selectively exposed to UV light by using a mask or UV-based maskless writer. It is observed that the optimal amount of water molecules in the PR helps achieve fast development rates and high contrast. Therefore, it is advised to wait for a few minutes after soft-bake to allow the rehydration of PR before exposing it to UV light. After the exposure, the sample is dipped in the developer solution, which selectively removes the unwanted PR and makes the desired PR pattern.

The photolithography was carried out on the thermally oxidized Silicon samples using AZ-10XT and AZ-4562 positive thick PR. Table 1 gives the process steps for these PRs.

However, bubble formation on the samples was observed after the exposure step. Figure 1 shows the samples seen through the optical microscope after the exposure and development stages. Although the bubble formation can be from the possible contaminants on the surface of the substrate, these substrates were cleaned with acetone, isopropyl alcohol and deionized water inside the class 100 clean room environment before starting the photolithography process. In addition, it is important to note that the bubble formation happens during the exposure step and not while doing the soft-bake. Such type of bubble formation during the exposure of positive PR can also be formed due to nitrogen (N_2) generation in the PR when exposed to UV. During the process of UV exposure, N_2 gas gets released (Adam & Hashim, 2012) from the naphthoquinone diazide molecule while it get converted into an indene carboxylic acid. This may happen if the exposure dose is very high or the soft bake is insufficient to remove the solvent from the PR.

Therefore, a process optimization is carried out to solve the issue of bubble formation. It is observed that the increase in the soft bake time and temperature indeed reduces the problem of bubble formation. This is because the initial soft bake time and the temperature were probably insufficient to completely remove the solvent from PR, despite the process being as per the required conditions (ref. Micro Chemicals - APPLICATION NOTES). This can happen if the substrate heating time is not considered. Similarly, for PRs with higher thicknesses (such as AZ-10XT and AZ-4562 of this study), the optimum soft bake parameters may deviate from the technical specifications due to the dependency on the baking setup as well as the environmental conditions such as gas pressure, moisture etc. (ref. STRANGE APPEARANCE OF THE PHOTO RESIST). In such cases, it is necessary to optimize the parameters in lab conditions to ensure the removal of PR solvent before UV exposure and avoid bubble formation. Finally, the process for both PRs was optimized to get a clear pattern without bubbles. Table 2 provides the optimized process parameters, while the other parameters are the same as Table 1. Figure 2 shows the photoresist patterns with the optimized parameters.

3. Adhesion of E-beam Evaporated Thin Films by In-Situ Pre-Deposition Substrate Heating

An Electron Beam Evaporation System (EBES) is a widely used technique for thin film deposition, especially during fabrication in various domains such as MEMS, semiconductors, optics, etc(van Dorp & Hagen, 2008). It uses an electron beam

Table 2
Optimized soft bake parameters.

AZ-10XT		AZ-4562	
before	**after**	**before**	**after**
Soft bake at 110°C for 2min 45sec	Soft bake at 120°C for 3min	Soft bake at 110°C for 1min	Soft bake at 140°C for 2min

Table 3
The deposition parameters.

Material	Thickness (nm)	Melting point	Input Current (mA)	Deposition Rate (Å/s)	Source to substrate distance (mm)	Substrate RPM
Ti	10	1660 °C	300	1.5	700	22.7
Au	50	1064 °C	110	0.2	600	23.2

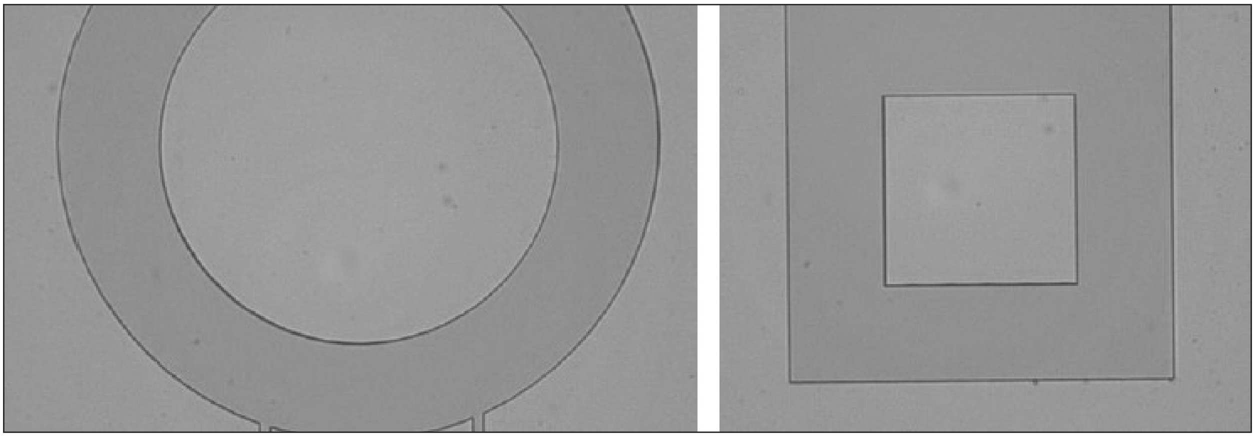

Fig. 2. Clearly defined PR patterns with no bubble formation after the increased soft-baking time and temperatures to the values mentioned in Table 2.

to heat a target material until it evaporates, and the vaporized atoms are then deposited onto substrates (viz., Silicon, glass, alumina, ceramics, sapphire, etc.). EBES typically can deposit thin films from sub-nanometres to a few hundred nanometres of thickness owing to their growth rates in Å/sec.

This study discusses the adhesion quality of Ti/Au thin films deposited using the EBES. Initially, we observed poor adhesion of Ti/Au films deposited on sapphire substrate. The adhesion quality was improved successfully by pre-deposition in-situ heating of the sapphire substrate.

The thin films of Ti (10 nm) / Au (50 nm) were deposited on the 2-inch diameter Sapphire substrate using the EBES (KENOSISTEK, Italy). The substrates were cleaned using Acetone, Isopropyl alcohol, and deionized water before loading into the load-lock chamber of EBES. The load-lock chamber helps transfer the samples inside the deposition chamber of EBES without breaking the vacuum. This allows the deposition of Ti and Au films at a high vacuum (base pressure < 10^{-6} Torr). The deposition parameters of Ti and Au materials are given in Table 1. The substrate temperature during deposition is typically < 45 °C.

We have done the tape-peeling test using the pressure-sensitive adhesive tape to check the adhesion quality of deposited films (Bartlett et al., 2023). In this, we have fixed the deposited samples onto the transparent adhesive tape made of polyethylene terephthalate (PET) material (Adwill D-series tapes), as shown in Fig. 3 (a). After this, we removed the tape to see the peeling of the deposited material. It was observed that the materials adhered to the tape and got peeled along with the tape. The image of the tape after peeling from the sample is shown in Fig. 3 (b), which shows the peeled materials onto the tape. Figure 3 (c) shows the remaining Ti/Au film on the sapphire test after peeling the tape. Therefore, the tape-peeling test indicates that the quality of adhesion of Ti/Au on the sapphire substrate

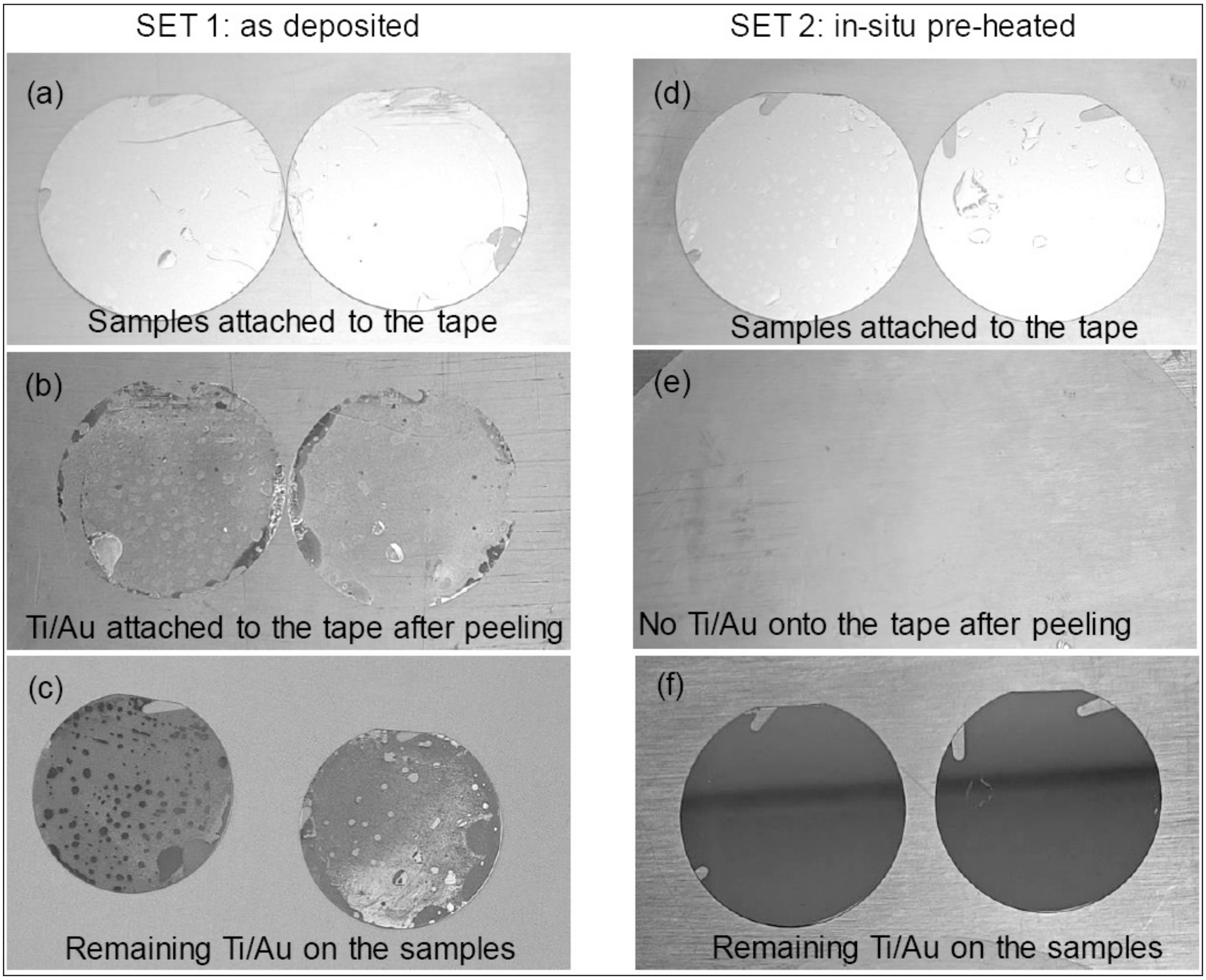

Fig. 3. (a)-(c) correspond to the samples without in-situ substrate heating, and (d)-(f) correspond to the samples with in-situ pre-deposition annealing at 100C/30 minutes before deposition of Ti/Au thin films on sapphire substrates. Figures (e) and (f) clearly show an improved adhesion quality of Ti/Au thin films with pre-deposition substrate heating.

is poor. It is possible to get poor adhesion in the presence of contaminations, such as oils, particles, etc., on the surface. However, as mentioned before, the sapphire substrates were thoroughly cleaned before loading into the EBES, and these cleaning procedures are known to remove such contaminants.

It has been reported that moisture inside the vacuum chamber affects the deposition quality (Möller et al., 2002; Serpini et al., 2017; Tsuchiya et al., 2000). As mentioned earlier, the depositions were carried out in a high vacuum with base pressure < 10^{-6} Torr, meaning the moisture content inside the deposition chamber is negligible. However, suppose the moisture in the atmosphere is adsorbed on the surface of the substrate before loading it in the EBES. In that case, the OH- group present on the surface of a substrate can affect the quality of deposition. In addition, a previous study reported that the quality of Au deposition on the Ti surface is not good if the Ti surface gets oxidized (Todeschini et al., 2017). Therefore, we believe that the reason for the poor adhesion of Ti/Au films is the moisture adsorbed on the substrate.

To remove the moisture content from the surface of the substrate, we performed in-situ preheating of the samples inside the EBES deposition chamber. After loading the sapphire wafers inside the deposition chamber of EBES, we heated them at 100 °C for 30 minutes and let them cool naturally to room temperature. The deposition procedure was started only after the temperature of the sapphire wafers reached below 35°C. The tape-peeling test was once again performed on these in-situ pre-heated samples to check the adhesion quality of Ti/Au films. Figures 3 (d), (e) and (f) show the samples attached to the tape, tape and samples after the peeling test, respectively. No trace of any Ti/Au film is found on the tape peeled off from the samples, as indicated in Fig. 1(d), and the deposited Ti/Au film remains intact on the sapphire substrate. Therefore, we conclude that the adhesion quality of Ti/Au films deposited on the sapphire substrates improved after in-situ pre-heating of bare sapphire substrates before the film deposition.

4. Conclusion

Here, we present the issues observed during the fabrication processes of photolithography and thin film deposition and the solutions to those problems. We observed bubbles forming in the photoresists after exposure to UV light. It is found that such bubble formation occurs if the solvent does not evaporate properly. Therefore, the time and temperature of the soft-bake step of the photolithography process were increased to ensure the proper evaporation of the solvent, which removed the formation of the bubbles.

Similarly, we have reported an issue of poor adhesion of Ti (10 nm) /Au (50 nm) films deposited in an electron beam evaporation system. It is inferred that the poor adhesion in the films is due to the presence of moisture on the surface of a substrate. Therefore, the substrates were pre-heated to 100°C inside the EBES system at a base pressure of < 10^{-6} Torr to ensure moisture removal from the surface. The tape-peeling test showed that the adhesion quality improved after the pre-deposition substrate heating.

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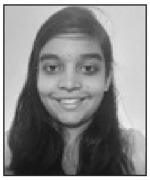

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