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Spray coating of photoresist for pattern transfer on high topography surfaces

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Abstract

In this paper, a new method of photoresist coating, direct spray coating, is studied. This method is especially suited to coat high topography surfaces for some special applications in microelectromechanical systems, radio frequency components and packaging. The most suitable photoresist type and coating process are found. The influence of several coating parameters on the thickness and uniformity of the photoresist layer is investigated. A model describing the dependence of the thickness on the major parameters is presented. Very promising results are obtained using spray coating for the fabrication of several three-dimensional structures.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Photoresist coating is one of the most frequently used process steps in microelectronic processing. Spin coating is the most common method of coating planar surfaces. However, for the fabrication of microelectromechanical system (MEMS) devices quite often uniform coating of photoresist on nonplanar surfaces is required. In this case, special coating methods will be needed.

To date, several photoresist coating techniques have been introduced for this purpose. Spin coating, the most conventional coating method applied to standard flat wafers, can also be used for high topography surfaces if certain modifications are made such as adapting the chuck for coating of nonplanar wafers [1] or changing the spin speed to allow sufficient time for the solution to flow and spread into the deep features [2]. However, the uniformity of the coated layer is not as good as for planar wafers and not always sufficient for the required resolution. Techniques for coating a flat panel display substrate such as the meniscus and extrusion coating have been recently developed [3]. In these techniques, solution flows either through a slot over a moving substrate or through a rolling applicator tube, leaving behind a thin film of deposited layer. Although these techniques consume less coating material, a lot of parameters influence the uniformity of the coated layer. Furthermore, their applicability to IC technology is limited due to the handling complexity and low throughput. Another new method is plasma deposition of photoresist. This method involves a monomeric coating

material, which may be evaporated at ambient temperatures and gets its polymeric form after deposition on the substrate [4]. But only coating of parylene and its derivatives is available at the moment. Alternative methods that are potentially suitable for coating nonplanar surfaces are electrodeposition and direct spray coating of photoresist. Electrodeposition of photoresist has been reported as an attractive method for 3D stacks of chips [5, 6]. This technique is based on the electrodeposition of emulsified organic material onto a conductive substrate. A dedicated plating system with an electrochemical cell is used. As the wafer is used as an electrode, it requires a conductive layer on the surface to be coated. Direct spray coating of photoresist¹ appears to be a promising technique for coating irregular surfaces. It does not require a conductive layer and thus can be used at all stages of the process.

In this paper, we present a direct photoresist coating method using the EVG101 spray coater (see footnote 1). A model has been proposed to define the thickness of a coated photoresist layer on a flat wafer. The model has been generated using important parameters of the spray process. Further, spray coating for MEMS applications has been investigated through the coating of high topography surfaces, i.e. on wafers with etched cavities up to 400 μ m deep.

The thickness and uniformity achieved are sufficient to pattern contact windows in very deep cavities and fine lines across shallower cavities. Examples of patterned structures

¹ EV Group, E. Thallner GmbH, DI Erich Thallner Strasse 1, A-4780 Schaerding-Austria (http://www.EVGroup.com).

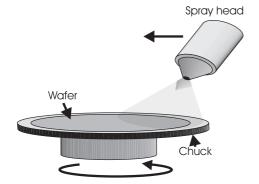


Figure 1. Schematic drawing of the spray system.

using spray coating are presented to illustrate the advantages offered by this method in MEMS applications.

2. Spray system

The Electronic Visions direct spray coating system EVG 101 has an ultrasonic spray nozzle with a patented droplet filter and innovative dispensing method. Figure 1 depicts a schematic drawing of the spray unit. During the spray coating process, the wafer is rotated at low angular velocity (30-60 rpm), while the swivel arm of the spray coating unit is moved across the wafer. The spray head is kept at about 45° angle to the wafer. Using low rotation speed can minimize the centrifugal forces on the outer substrate area. The EVG system uses a syringe pump that provides similar precision and repeatability to a commercial resist pumping system that automatically dispenses directly from a photoresist bottle but offers the flexibility to use very small quantities of photoresist. The syringe pump is therefore appropriate for experimental purposes. The EVG coating system is operated through Windows-based software that allows editing of the coating recipes.

There are several adjustable parameters involved in spray coating that influence the thickness, uniformity and quality of the photoresist layer. Among them, the most important parameters are

- (1) Photoresist solution composition, i.e. the solids content of the spray solution and the solvent. The solids content represents the densities of raw materials used in the photoresist solution. The solids content of the solution can be reduced by adding solvent to the solution, thus reducing the viscosity. The EVG 101 can spray solutions with viscosity lower than 20 cSt.
- (2) The volume of dispensed photoresist, which is controlled by the dosage pump.
- (3) The scanning speed of the atomizer: lower speed produces thicker layers. The scanning speed varies as the spray head sweeps over the wafer. This is due to the fact that the area requiring coverage reduces when the spray head is near the center of the wafer. A constant speed is not appropriate in this case. The scanning is divided into multiple segments or indices, across the wafer, each with its own speed. On this machine, the scanning of the atomizer is divided into 15 segments across the wafer. An example of the scanning profile in a test recipe is shown in figure 2.

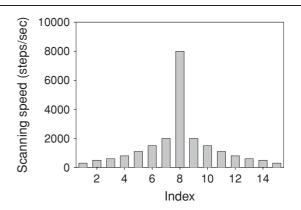


Figure 2. Scanning speed profile across the wafer.

(4) The transfer efficiency (TE) of the machine. TE is a measure of the percentage of the actual coating material that is deposited on the substrate with respect to the total volume sprayed, i.e.,

$$TE = \left(\frac{\text{volume of solids deposited on wafer}}{\text{volume of solids sprayed}}\right) \times 100\%$$
(2.1)

This value indicates the coating efficiency of the machine because during spraying, only part of the photoresist is deposited on the wafer, the rest is wasted material. TE is one of the most important parameters in coating as a small improvement in efficiency will yield a significant reduction in waste disposal (solvent evaporation) and benefit costs.

- (5) Substrate size and geometry. For example, coating small substrates reduces the TE. Coating a flat substrate results in thicker layer than for a substrate with high topography because a flat substrate has less surface area.
- (6) Distance from the spray nozzle to the substrate.
- (7) Spinning velocity of the wafer, which mainly has an effect on the layer uniformity.
- (8) The spray pressure, that will have an effect on the droplet size of the photoresist.

Understanding the parameters that influence the spray process is essential to attain desired and controllable results.

3. Photoresist type

In principle, the spray coating system can be operated using solutions with viscosity lower than 20 cSt in order to get the proper droplet size distribution. To date, no commercial photoresist specific for this system is available. A commercial photoresist solution used for electrostatic spray coating, AZ4823, can be used for spray coating on flat wafers. For special applications such as coating nonplanar surfaces, a specific photoresist solution is required. Several photoresist solutions have been investigated in order to find the most suitable one for the purpose of coating high topography surfaces. Most available photoresists have a viscosity higher than 20 cSt but they can be diluted with photoresist compatible solvents if the following issues are taken into consideration:

- same or similar chemistry as main photoresist solvent
- no chemical reaction with the photoresist or during exposure should take place

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Table 1. Photoresist types and their characteristics.				
Photoresist	Viscosity (cSt at 25 °C)	Solids content (%)	Main solvent	Added solvent
AZ4823 AZ4562 AZ4562-PGMEA AZ4562-MEK	5 440 <20 <20	15 39.5 5–17 5–15	PGMEA PGMEA	PGMEA MEK

high evaporation rate

• low toxicity and compatibility to IC technology.

Three types of photoresist solutions have been selected and studied, namely: (1) photoresist AZ4823 (commercial photoresist from Clarian Corp)², and various mixtures of AZ4562 (Clarian Corp) photoresist (see footnote 2) diluted in (2) PGMEA (methoxy-propyl acetate) solvent or (3) MEK (methyl-ethyl ketone) solvent. The main characteristics of these solutions are summarized in table 1.

The first one, AZ4823, is a commercial photoresist for electrostatic spray coating with a low viscosity of 5 cSt and a solids content of 15%. The other photoresist, AZ 4562, is a high viscosity (440 cSt) photoresist normally used to form a thick layer by spin coating. In order to use it in the spray system we have to dilute the AZ4562 photoresist to lower the viscosity. At first, we used PGMEA as solvent. The AZ4823 and the AZ4562 diluted in PGMEA have been successfully used for spray coating on flat wafers and on wafer with narrow etched cavities [7, 8]. However, for the spraying on high topography wafers, they present some limitations due to the flow of the photoresist. In fact, after the photoresist is spray coated on the wafer, it tends to flow locally, resulting in accumulation of photoresist at the bottom corner and reduction at the top corner of cavities. To minimize this flowing effect, the drying of the sprayed layer should be accelerated. There are two ways to achieve this. One is to use a spraying solution containing a solvent with high evaporation rate and the other is to use a heated chuck while spraying. The main solvent contained in AZ4823 and AZ4562-PGMEA solutions is PGMEA. This solvent does not evaporate sufficiently fast to avoid or limit the flowing effect. For this photoresist solution, the heated chuck (60-80 °C) must be employed to assist the drying of sprayed layer. However, when using a heated chuck, the surface of the coated layer becomes rougher [7, 8].

For wafers with deep cavities, even better results can be obtained with photoresist solutions containing a fast evaporating solvent. A third type of photoresist solution was prepared using AZ4562 photoresist diluted in MEK solvent. MEK evaporates faster than PGMEA (the boiling point of MEK is 80 °C, whereas the boiling point of PGMEA is 145 °C). Several solutions of AZ4562-MEK with different solids content have been tested. The best results are achieved with a AZ4562-MEK solution with a solids content of 10%.

4. Model for film thickness

Based on the parameters mentioned in section 2, we propose a model to determine the thickness of a photoresist layer formed

by spray coating on a flat wafer. The model takes into account most of the observed coating variables. It can be used to predict, understand and control the coating process.

4.1. Model description

The proposed model is derived for coating a flat wafer substrate.

The photoresist film thickness formed after spray coating can be calculated as

$$d = \frac{V}{S} \tag{4.1}$$

where d is the film thickness, V is the volume of photoresist on the wafer and S is the area of the wafer.

The final photoresist volume on the wafer is only a part of the total volume that is consumed by the spray coater. The total real consumed volume is denoted as V_{tt} . The photoresist solution contains two main parts: solids and the solvent. Only the solids contribute to the film formation. The solids content of the solution is reflected by c.

As mentioned in the previous section, during the spray coating process, a part of photoresist is lost and the actual photoresist on the wafer is measured by the TE. The TE of the normal spray technique is in the range of 15–40% and varies with the method of coating and the coating tool [9]. There are several factors that affect the TE. The main factors impacting TE are size and geometry of substrate, equipment maintenance, method of spray and machine configuration such as atomizing air pressure, spray head, etc. Thus, TE = f (solution, substrate, equipment).

In general, the TE of specific coating equipment can be empirically determined or provided by the equipment supplier. The TE of the EVG101 is empirically determined and is about 20%. Correct operation of the spray system is also one of the key parameters that can improve the TE. Thus, the volume of photoresist on the wafer is

$$V = V_{\rm tt} c {\rm TE} \tag{4.2}$$

The total consumed volume $V_{\rm tt}$ can be expressed as follows:

$$V_{\rm tt} = V_{\rm dis} t_{\rm sc} \tag{4.3}$$

where V_{dis} is the dispensed volume (μ l s⁻¹) of the photoresist, which can be controlled by the photoresist pump and be set through software and t_{sc} is the total scanning time of the spray head, which sweeps from one side to the other side of the wafer. The scanning movement of the EVG 101 spray head is divided into 15 segments as explained earlier and controlled by the step motor. Therefore, the scanning time can be calculated as

$$t_{\rm sc} = \sum_{n=1}^{15} t_n = \sum_{n=1}^{15} \frac{k}{v_n}$$
(4.4)

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² Technical information for AZ products, Clarian Corp. Clariant Corporation (USA), Business Unit Electronic Materials, 70 Meister Avenue, Somerville, NJ 08876, USA.

where t_n is the scanning time of segment n(s), v_n is the scanning speed of spray head in segment n (steps s⁻¹) and k is the length of one scan segment (steps).

Combining equations (4.2), (4.3), (4.4) and (4.1), we finally obtain the photoresist film thickness:

$$d = \frac{kc\text{TE}}{S} \times 10^{-3} \sum_{n=1}^{15} \frac{V_{\text{dis}}}{v_n}$$
(4.5)

where *d* is the photoresist thickness (μ m), *c* is the solids content (%), *k* is the distance of one scan segment (steps), TE is the transfer efficiency (%), *S* is the wafer area (cm²), *V*_{dis} is the volume dispensed (μ l s⁻¹) and *v_n* is the scanning speed of the spray head in segment *n* (steps s⁻¹) (*n* = 1, 2, ..., 15).

The value 10^{-3} comes from the unit converter.

Equation (4.5) clearly reflects the dependence of the film thickness on process parameters. The first five parameters mentioned in section 2 are included in the equation. The last three parameters, i.e. the distance from the spray nozzle to the substrate, the spinning velocity of the wafer and the spray pressure are either fixed by the equipment manufacturer or if they can be varied will only have an effect on the film uniformity and not on the thickness.

From equation (4.5), S and k are constants for a certain wafer diameter. Thus, for 4 inch wafers

$$S = \pi r^2 = 78.5 \,(\text{cm}^2)$$
 and $k = 4 \times 10^4 / 15$.

Considering that TE for the EVG101 is about 20%, the expression for film thickness can be simplified as

$$d = 0.679c \sum_{n=1}^{15} \frac{V_{\text{dis}}}{v_n}.$$
 (4.6)

Although this is an approximation of the actual spray process, it does indicate that the solids content (c), the dispensed volume (V_{dis}) and the scanning speed (v_n) are the major variables to be considered, making the process more elucidative.

4.2. Model verification and discussion

Experiments to spray coat planar wafers have been carried out by using AZ4823 and AZ4562 diluted photoresist with different solids contents. Silicon substrates, 4 inch $\langle 100 \rangle$ orientation, were used for the experiments. After spray coating, the wafers were baked on a hot plate at 95 °C for 3 min. The photoresist thickness was then measured by using the reflectance spectrometry technique. The photoresist thickness was averaged over a 10-point measurement. The experimental data were then compared to the values calculated by equation (4.6) to verify the model.

4.2.1. Photoresist thickness versus dispensed volume. The first experiment used AZ4823 photoresist. The solids content of this solution is 15%. The scanning speed v_n is programmed as illustrated in figure 2. Only the dispensed volume is a variable. Figure 3 shows the experimental data and the predicted values of photoresist thickness. The dispensed volume used ranged from 10 to 100 μ l s⁻¹. Two sets of experiments were performed. Excellent agreement between the model and the corresponding experimental data is obtained as demonstrated in figure 3.

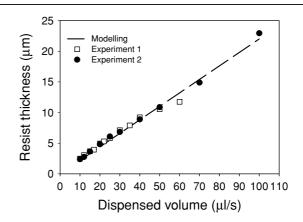


Figure 3. Photoresist thickness versus dispensed volume (calculated versus experimental data) using photoresist AZ4823 (solids content 15%).

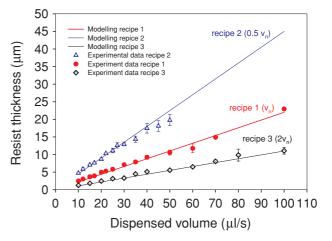


Figure 4. Photoresist (AZ4823) thickness versus dispensed volume at different scanning rates (calculated versus experimental data).

4.2.2. Photoresist thickness versus scanning speed. For this experiment, the photoresist AZ4823 was used and three different scanning speed profiles were employed. The scanning speed can be different at each segment. In order to obtain a uniform layer over the wafer, a profile like the one shown in figure 2 is needed. Three recipes with different scanning speeds were prepared. Recipe 1 employs the same profile as in figure 2 with the scanning speed (v_n). Wafers were coated with different dispensed volumes from 10 to 100 μ l s⁻¹.

The other two recipes also have the same scanning profile, but the scanning speed v_n is different. For recipe 2 v_n is only half $(0.5 v_n)$ and for recipe 3 is double $(2 v_n)$.

In figure 4, the experimental and calculated data of the photoresist thickness versus dispensed volumes are plotted for all three recipes. For the same value of dispensed volume, the photoresist thickness increases with decreasing scanning speed, i.e. lower scanning speed produces thicker photoresist layers. For recipes 1 and 3, the experimental data fit well with the calculated thickness. On the other hand, when using recipe 2 the measured values deviate somewhat from the calculated ones for a dispensed volume higher than 20 μ l s⁻¹. This suggests that a lower scanning speed might allow thicker photoresist layers, but when approaching a critical value, other

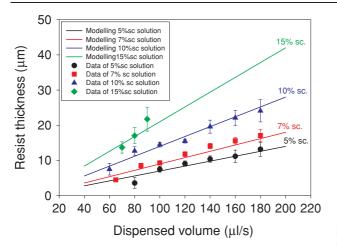


Figure 5. Photoresist (AZ4562 with MEK solvent) thickness versus dispensed volume at solutions with different solids content (calculated versus experimental data).

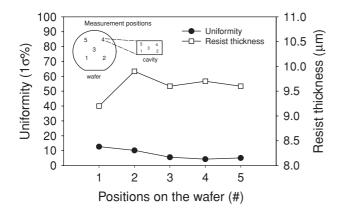


Figure 6. Photoresist (AZ4562-MEK solids content 10%) thickness and uniformity measured at the bottom of 375 μ m deep cavities at different positions on the wafer.

effects, such as the exhaust system, might have a bigger effect on the final thickness. A high scanning speed when using low dispensed volumes should also be avoided as this might result in a pinhole-rich layer.

4.2.3. Photoresist thickness versus solids content. То investigate the dependence of photoresist thickness on the solids content of the solution, a number of photoresist solutions with different solids contents were prepared. For this experiment, we used the thick photoresist AZ4562 (39% solids content) diluted in MEK solvent to make several photoresist solutions with the solids contents of 5%, 7%, 10% and 15%. For each solution, a set of wafers were spray coated with different dispensed volumes ranging from 60 to 180 μ l s⁻¹. Lower or higher volumes were not used as it would result in either large density of pinholes or too thick a layer. The scanning speed profile was kept constant as indicated in figure 2. The experimental data on thickness versus dispensed volume of different solutions are shown in figure 5. The thickness increases with the dispensed volume and solids content of the solution.

Depending on the envisioned application, one can choose a solution with the most suitable composition. For example, a

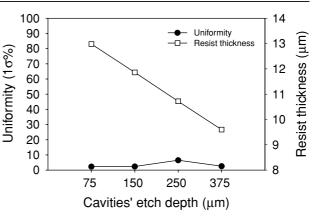


Figure 7. Photoresist (AZ4562-MEK solids content 10%) thickness and uniformity on wafer with cavities of different etch depths.

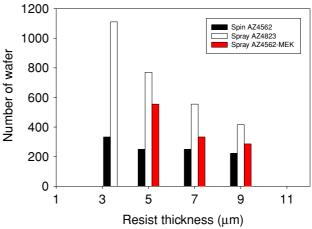


Figure 8. Amount of coated wafers per 1 liter of photoresist.

low solids content (5% or 7%) cannot form a very thick layer but it gives a better uniformity layer as can be seen by the small thickness deviation observed. This is a preferable condition to coat flat wafer. A higher solids content solution can create a thicker layer but the photoresist uniformity over the wafer is not as good as low solids content solutions. A solution of 10% solids content has been used and proved to be quite suitable to coat high topography wafers. With a dispensed volume of less than 120 μ l s⁻¹, the spray coating can form a uniform layer up to 15 μ m (small deviation is observed). This solids content value is sufficient to reduce the flowing effect, thus providing a good coverage over high topography surfaces. Higher values such as 15% are not always desirable because the photoresist thickness variation is larger in this case. In fact, a high solids content photoresist contains a low quantity of solvent and thus the smoothening of the layer is reduced, making the surface rougher.

5. Spray coating for MEMS applications

5.1. Photoresist thickness and uniformity

For this experiment, the photoresist AZ4562-MEK solution (solids content 10%) was used with the scanning speed (v_n) programmed as in figure 2. This recipe was used as the starting

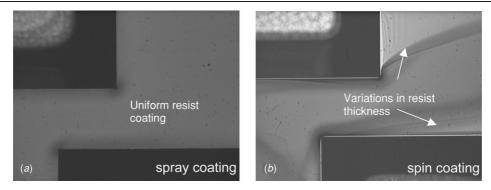


Figure 9. Images of the wafer surface between two cavities: (*a*) using spray coating (photoresist AZ4562-MEK) and (*b*) using spin coating (photoresist AZ4562).

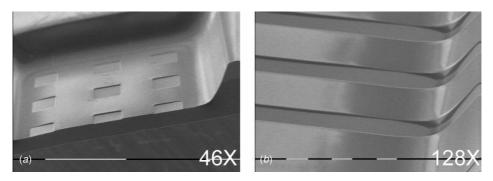


Figure 10. Patterning of spray coated photoresist (AZ4562-MEK solids content 10%) in and across a 375 μ m deep cavity: (a) contact windows at the bottom of the cavity and (b) lines running over the cavity.

point for the wafers with cavities. It uses a dispensed volume of photoresist of 80 μ l s⁻¹ (this resulted in a ~10 μ m thick layer on the flat wafer). Silicon wafers with deep-etched cavities ranging from 75 to 375 μ m were tested. After spray coating, the wafers were placed on a hot plate at 90 °C for about 3 min. A lithography step was applied to pattern structures in and across the cavities. Exposure was carried out in the EV-420 contact aligner for 25 s (UV source, intensity 625 mJ cm⁻²). In order to open the structure at the bottom of cavities, high exposure energy is needed. Immersion development was performed using a potassium-based alkaline developer (AZ400K) diluted 1:4 in deionized (DI) water. The photoresist thickness for these wafers is the thickness at the bottom of the cavities. At each cavity, the thickness was measured at five points and averaged. The photoresist coating layer is evaluated through the uniformity $(1\sigma\%)$:

$$1\sigma\% = \frac{\sigma}{\text{average}} \times 100$$

The photoresist thickness and uniformity for a 375 μ m deep cavity are shown in figure 6. The photoresist thickness is around 9 μ m and the uniformity within a cavity (1 σ %) is 10%. That is, higher than on the flat wafer but acceptable when the dimensions of patterned structures are in the range of tens to hundreds of microns. The photoresist thickness and its uniformity over the wafer were also measured. Figure 7 illustrates the average thickness and uniformity for 75, 150, 250 and 375 μ m deep cavities. The photoresist thickness decreases with an increase in the etch depth of the cavities. In most cases, the uniformity 1 σ % is around 10%.

The thinner photoresist layer in a deeper cavity can be explained by the fact that the coating area in a deeper cavity is larger than in a shallow cavity.

The uniformity of the photoresist layer obtained by this spray coating method is better than that obtained with the spin coating method. Moreover, the reproducibility from wafer to wafer is suitable for batch production.

5.2. Applications

Spray coating can be used to coat both planar and nonplanar surfaces and it presents some advantages over the spin coating method. First, this technique uses much less photoresist than spin coating. In fact, for spin coating, the wafer is flooded with photoresist but due to the high rotation speed, only a small amount of photoresist remains on the wafer. For spray coating, very fine droplets of photoresist are deposited directly on the wafer to form the layer. The photoresist loss in the system is the small part that sprays out into the air or to the exhaust unit. This provides cost savings in the amount of photoresist used, together with reduced waste disposal costs. The amount of wafers coated per liter of spray coating technique is higher than with spin coating. Figure 8 shows a comparison between spin coating of AZ4562, spray coating of AZ4823 and spray coating of AZ4562 diluted in MEK (10% solids content). Depending on the target thickness of the photoresist layer, the amount of wafers spray coated with AZ4823 is two to three times larger than that of spin coated AZ4562.

Secondly, the reproducibility of spray coating is much better than spin coating. The photoresist thickness is repeatable over all cavities of the same size, regardless of

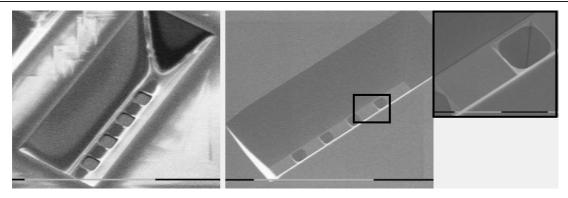


Figure 11. SEM photographs of: (a) photoresist pattern of contact windows at the bottom of a 375 μ m deep cavity (magnification ×66) and (b) through-wafer holes are formed by silicon etching in KOH.

the position of cavities on the wafer. Due to an even distribution of photoresist over the wafer while spraying, the shape of the cavity has a negligible influence on the photoresist uniformity. Spray coating has no thickness variation caused by a directional effect of spinning, thus no striation and no damaged structures are observed. Figure 9 shows two images of a photoresist layer on the surface between two cavities: (a) using spray coating and (b) using spin coating. The spray coated photoresist layer is clearly uniform, while the spin coated surface shows some striation caused by photoresist thickness variation. Thirdly, direct spray coating does not require a special underlayer and can be applied on both insulating and conductive layers. Thus, spray coating can be used at all stages of the process and gives rather encouraging results, especially for patterning structures at the bottom of deep cavities.

To illustrate the potential of this coating technique, contact openings at the bottom of cavities as deep as 400 μ m and photoresist lines in and across such cavities have been realized. Figures 10(*a*) and (*b*) show the patterning of spray coated photoresist for both contact windows and lines in and across a 375 μ m deep cavity.

Another example of photoresist patterns at the bottom of a narrow cavity is illustrated in figure 11. Although this cavity is narrow at the bottom, an acceptable resolution is still achieved. The photoresist patterning has been applied in a post-process bulk micromachining module to form through-wafer holes for contacting backside to frontside of the wafer [10]. The patterns are well defined, thus allowing a second silicon wet etch step to form through-wafer holes as illustrated in figure 11(b).

6. Conclusions

Direct spray coating is a promising technique to form a conformal photoresist layer on irregular surfaces or to obtain very thick ($\sim 25 \ \mu$ m) photoresist layers on flat wafers. A model has been proposed to determine the thickness of the sprayed layer. This model is also a means to predict, understand and control the coating process and estimate the consumable cost of spray material. The model can be applied not only to photoresist but also to other coating

solutions. One of the main benefits of the spray coating method is the possibility of forming a layer with good coverage over very high topography surfaces. The photoresist solutions as well as parameters of the spray system have been optimized for photoresist patterning on 3D structures. The thickness and uniformity of the spray coated photoresist layer are suitable for several MEMS applications as illustrated by the fabrication of various patterns in and across deep cavities. The results achieved so far clearly show the characteristics and potential of this technique.

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