Conformal photo-resist coating technique in the through-silicon via of the semiconductor devices with the rotary atomising aerosol spray

Yoshiyuki Seike*

Technology Coordination Department,
Asahi Sunac Corporation,
5050, Asahimae-cho, Owariasahi,
Aichi pref., 488-8688, Japan
E-mail: seike@sunac.co.jp
*Corresponding author

Masanori Ohtsubo

Art Science and Technology Center for Cooperative Research,
Collaborative Research Division,
Kyushu University,
6-1, Kasuga Park, Kasuga,
Fukuoka pref., 816-8580, Japan
E-mail: ohtsubo@astec.kyushu-u.ac.jp

Futoshi Shimai and Kenji Maruyama

Equipment Marketing Section,
Marketing Division,
Tokyo Ouka Kogyo,
150, Nakamaruko, Nakahara-ku, Kawasaki,
Kanagawa pref., 211-0012, Japan
E-mail: f-shimai@tok.co.jp
E-mail: k-maruyama@tok.co.jp

Hiroyuki Akenaga and Yoshinori Kobayashi

NC Technical Center,
New Component Div.,
Asahi Sunac Corporation,
5050, Asahimae-cho, Owariasahi,
Aichi pref., 488-8688, Japan
E-mail: akenaga@sunac.co.jp
E-mail: kobayashi@sunac.co.jp

Keiji Miyachi

New Component Div.,
Asahi Sunac Corporation,
5050, Asahimae-cho, Owariasahi,
Aichi pref., 488-8688, Japan
E-mail: miyachi@sunac.co.jp

Masahiko Amari

Asahi Sunac Corporation,
5050, Asahimae-cho, Owariasahi,
Aichi pref., 488-8688, Japan
E-mail: Amari@sunac.co.jp

Toshiro Doi

Art Science and Technology Center for Cooperative Research,
Collaborative Research Division,
Kyushu University,
6-1, Kasuga Park, Kasuga,
Fukuoka pref., 816-8580, Japan
E-mail: doi@astec.kyushu-u.ac.jp

Syuhei Kurokawa

Department of Mechanical Engineering,
Faculty of Engineering,
Kyushu University,
744 Motooka, Nichi-ku, Fukuoka,
Fukuoka pref., 819-0395, Japan
E-mail: kurobe-@mech.kyushu-u.ac.jp

Abstract: Semiconductor devices are increasingly sophisticated in their application of three-dimensional laminated chips inside through-silicon via (TSV). TSV is a technology that connects the stacked chips using through electrodes instead of higher integrated circuits densities. In this report, we invented a new photo-resist coating method inside the TSV using the rotary atomising aerosol spray. We measured the characteristics of the flying droplets from the atomising aerosol spray nozzle by a shadow Doppler particle analyser (SDPA) to indicate that the new method is capable of coating the photo-resist inside the TSV. Furthermore, we tried to make the prototype coating system with the rotary atomising aerosol spray nozzle and run the experiments in to coat the photo-resist inside the TSV test element group (TEG) wafer. Results indicate that the photo-resist is coated on the TSV TEG wafer having opening diameter of 50 μm to 200 μm, depth of 50 μm and square pyramid via holes with tapered angle of 54.7 degrees by using the prototype equipment. It is confirmed that coating along the inner walls of via holes is feasible.

Keywords: through-silicon via; TSV; 3D stack semiconductor device; rotary atomising aerosol spray; photoresist coating; flying droplets; shadow Doppler particle analyser; SDPA; resist thickness; film thickness; photo-resist; conformal resist coating.

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Biographical notes: Yoshiyuki Seike is a Manager of Technology Coordination Department and Quality Assurance Department at Asahi Sunac Corporation. He engages in the research and development of the semiconductor production machine. He earned his PhD in Mathematics Electronics and Informatics from Saitama University, Japan and is focused on the surface conditioning technology in the electrical devices.

Masanori Ohtsubo is a Technical Staff in the Art Science and Technology Center for Cooperative Research Division (KASTEC); realm of optoelectronics functional materials, at Kyushu University. His current research is focused on the high efficiency polishing process for the wide-gap semiconductor substrates such as SiC, etc. He was educated at Virginia Polytechnic Institute and State University, USA.

Futoshi Shimai is a Manager of Equipment Section in the Marketing Division at Tokyo Ohka Kogyo Co., LTD. He engages in the development of the semiconductor production machine and the research of the process semiconductor devices.

Kenji Maruyama is a Manager of Equipment Section in the Marketing Division at Tokyo Ohka Kogyo Co., LTD. He engages in the development of the semiconductor production machine and the research of the process semiconductor devices.

Hiroyuki Akenaga is an Engineer of New Components Technology Center in the New Component Division at Asahi Sunac Corporation. He engages in the research of the semiconductor production process. He was educated in the Materials Science at Kitami Institute of Technology, Japan.

Yoshinori Kobayashi is a Manager of New Components Technology Center in the New Component Division at Asahi Sunac Corporation. He engages in the research of the semiconductor production process. He was educated in the Department of Mechanical Engineering at Nagoya Institute of Technology, Japan.

Keiji Miyachi is the Division Director in the New Component Division at Asahi Sunac Corporation. He engages in the management of the study in the research of the semiconductor production process. He earned his PhD from the Department of Mechanical Engineering at Kyushu University, Japan.

Masahiko Amari is the President at Asahi Sunac Corporation. He has managed Asahi Sunac Corporation, which has developed the coating equipment, the forging machinery and manufacturing machine of electrical devices. He obtained his PhD from the Department of Mechanical Engineering at Waseda University, Japan.

Toshiro Doi is a Professor in the Art, Science and Technology Center (KASTEC) at Kyushu University, Japan. He earned his PhD in Polishing Technology from the University of Tokyo, Japan. He is currently Professor Emeritus of Kyushu University as well as of Saitama University. His research covers precision processing including chemical mechanical polishing (CMP) technology. He is a Fellow, distinguished Chairman of the Planarization CMP Committee of the Japan Society for Precision Engineering (JSPE), Chairman of the 136 Committee on Future-Oriented Machining of the Japan Society for the Promotion of Science (JSPS), Electrochemical Society, and a member of some other national and international associations.

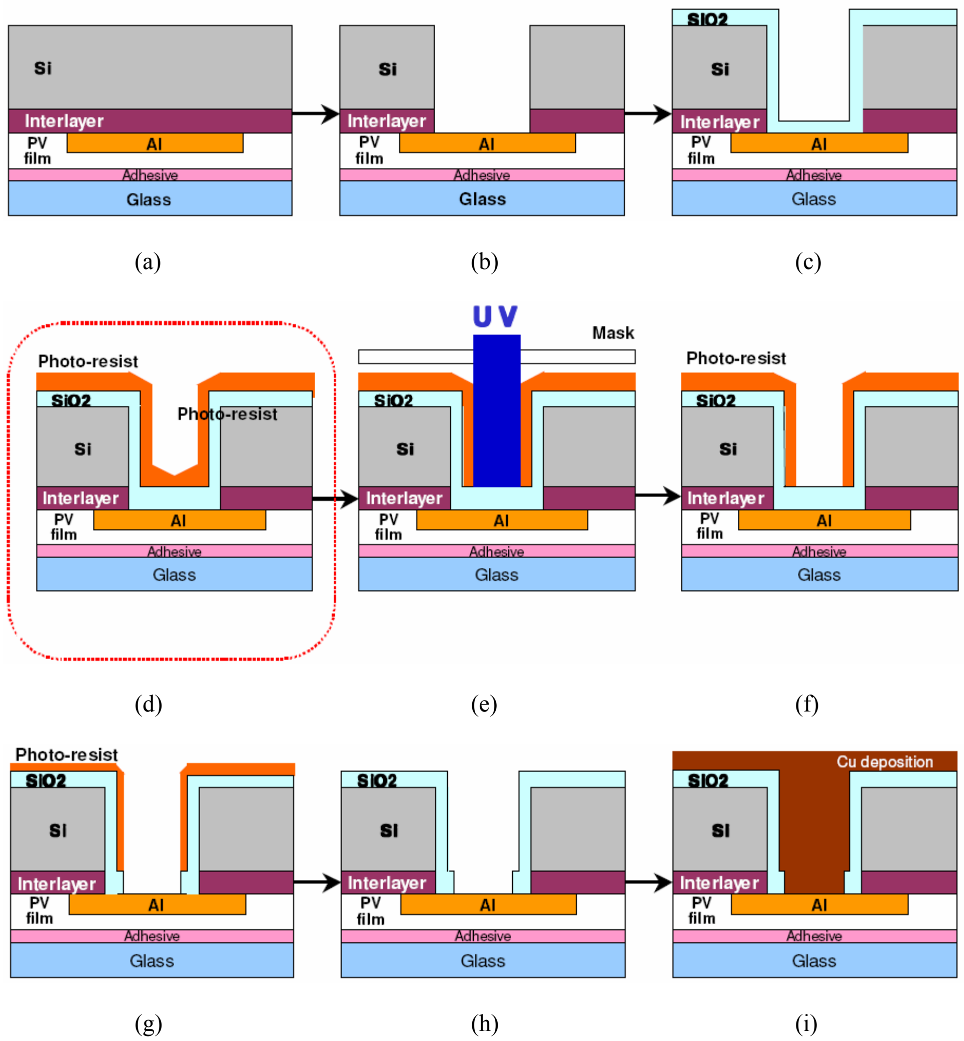
Syuhei Kurokawa is currently a Professor in the Department of Mechanical Engineering at Kyushu University, Japan. He earned his PhD in Production Engineering from Kyushu University. He acted as a Visiting Professor of Laboratory for Machine Tools and Production Engineering (WZL) of the RWTH Aachen in Germany in 1998. His research fields include measurement and evaluation of gear accuracy, characterisation of surface topography, nanomachining of micro machine elements and measuring devices, and planarisation CMP technology of device wafers. He is a member of the Japan Society of Mechanical Engineering (JSME), the Japan Society for Precision Engineering (JSPE), the Japan Society for the Promotion of Science (JSPS) and the Japan Society for Abrasive Technology (JSAT).

1 Introduction

In recent years, studies of three-dimensional stack packaging have been making progress for miniaturisation, further enhancement in performance and reducing costs of semiconductor devices (Denda, 2011; Tominaga et al., 2010). Semiconductor devices having three-dimensional stack structure achieve high density by utilising through-silicon via (TSV) as an interlayer electrode on multiple layers of devices stacked vertically in the unit of silicon wafers or chips.

An example of process to form image sensor by using TSV is shown in Figure 1. Via hole patterns are formed in the silicon substrate by photolithography-etching method in advance [Figure 1(a) and 1(b)]. After forming a silicon dioxide film [Figure 1(c)], photo-resist layer is formed at the inner face of via holes [Figure 1(d)]. Through holes are formed again by photolithography-wet etching method [Figures 1(e) to 1(h)]. Inside the holes are copper plated for electric connection. Shape of TSV via hole varies depending on the kind of device; they may be pillar-shaped, cone-shaped, square pyramid or others. Nagarajan (2006) and Wimplinger (2008) reported that the opening diameter of via hole is 1 μm to 150 μm, and the depth is 10 μm to 400 μm. Gap filling method, a combination of spin coat method and vacuum technology, is widely used in the conventional photo-resist film formation process at the inner surface of via holes [shown in Figure 1(d)]. This method fills photo-resist in the via holes by dropping photo-resist in the centre of a wafer, followed by high-speed spinning of the wafer to spread photo-resist with centrifugal force, and then exposing the wafer in vacuum environment. Since this method uses deforming action by vacuuming inside the via holes after spin coating photo-resist, conformal film can not be formed along the inner walls of via holes tens of μm in diameter. In order to form conformal film for practical use, processing time of photolithography becomes longer, requiring special photoresist material, thus there is a cost problem.

Figure 1 A flow diagram of the TSV manufacturing process, (a) a wafer with wiring (b) photo-lithograph and etching (c) chemical vapour deposition of SiO_2 (d) coating of the photo-resist (e) UV exposure (f) alkali development (g) etching (h) ashing (i) copper deposition (see online version for colours)



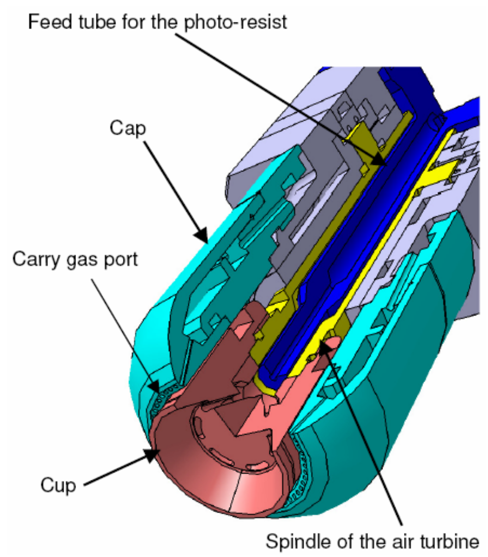
As a solution to the problems described above, we have originated a new photo-resist film forming method that uses a new rotary atomising aerosol spray designed for TSV wafers for image sensors with square pyramid via holes having the opening diameter of 50 µm to 200 µm, depth of 50 µm and tapered angle of 54.7 degrees. By using this method, smaller droplets can be generated compared to the conventional aerosol spray method, allowing conformal film to be formed along the inner walls of via holes because smaller droplets will be sprayed into the via holes. Based on this idea, a prototype of rotary atomising aerosol spray nozzle was made, and the characteristics of the generated droplets were measured by using a shadow Doppler type particle analyser (SDPA). It is confirmed that least square mean of 16 µm diameter droplets are generated. Since this average diameter is about 1/3 to 1/2 of the diameter of via holes, it is anticipated that the conformal film will be able to be formed inside the via holes. After that, a prototype of coating equipment with newly developed rotary atomising aerosol spray nozzle, which founded by Seike et al. (2010), was made; it is confirmed that conformal film of photo-resist on the inner walls of via holes can be formed as a result of coating experiment on TSV test element group (TEG) wafers.

2 Proposal of new TSV coating method

2.1 Proposal of coating method using rotary atomising aerosol spray

Conventional spin coating method drops photo-resist on a wafer and spread it with centrifugal force, allowing the film to be formed on the wafer surface. Yet, conformal film cannot be formed inside via holes having opening diameter of 50 µm to 200 µm because some holes are filled and some holes are void. Therefore, we thought spraying photo-resist directly onto the TSV wafer might generate conformal film on the inner walls of via holes. However, Lefebvre (1989) described the conventional two-fluid nozzle generates larger droplets, which cannot go into via holes having 50 µm to 200 µm in diameter, so we need to generate smaller diameter droplets. If droplets smaller than 50 µm is generated and the diameter of droplets can be controlled, we thought it is easy to determine the film forming conditions. Also, in order for the droplets of photo-resist to reach the bottom face of via holes and attach to it, we thought it is necessary to control flying speed of droplets.

Figure 2 A cross-section image of an inventable rotary atomising aerzol nozzle (see online version for colours)



We proposed an idea of a coating method using rotary atomising aerosol spray that rapidly rotates the cup with photo-resist to atomise it with centrifugal force, and spray atomised photo-resist to silicon wafer by carrier gas. A rotary atomising aerosol spray nozzle shown in Figure 2 is designed based on this idea. This nozzle is capable of generating droplets 15 μm in diameter with centrifugal force by rotating a cup at maximum speed of 70,000 min^{-1} using air turbine. We thought that the droplets could reach inside of TSV wafer via holes by letting them blown by carrier gas. Further, if the rotating speed of cup and flow rate of carrier gas can be controlled individually, diameter and speed of flying droplets can be controlled independently, enabling to control the film thickness of photo-resist on the walls of via holes. If there is pulsation at feeding photo-resist to the cup even at the flow rate of 1 cc/min, inconsistency of film thickness becomes significant. Therefore, it is required to minimise pulsation at pressure feeding. In order to prevent metal contamination of the photo-resist, the pressure feeding part should be made with resin.

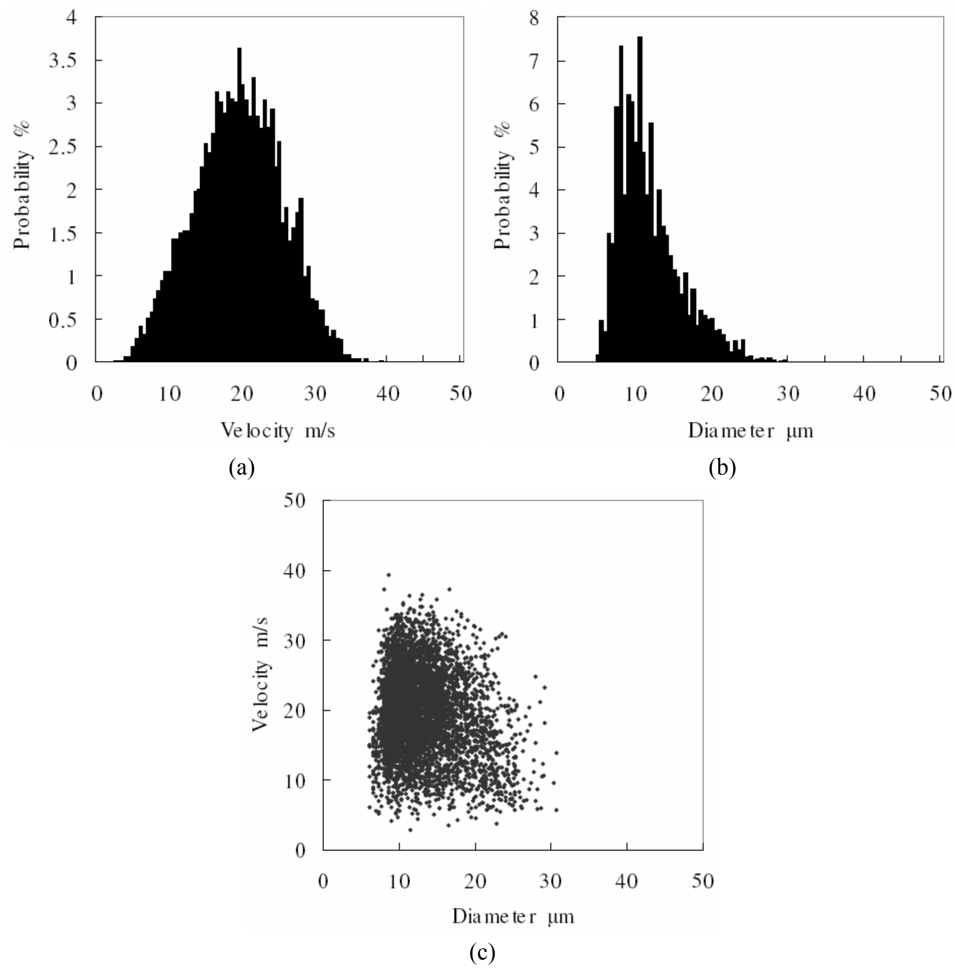
2.2 Verification of flying droplets' control factor

Based on the concept of Figure 2, a prototype of rotary atomising aerosol spray nozzle was made, and the condition of flying droplets sprayed from the nozzle was recorded by using a high-speed video camera (IDT, model Motion X). The picture example is shown in Figure 3. The spraying conditions: Rotating speed of cup is 30,000 min^{-1}, compressed air of 0.2 MPa is used as carrier gas, and flow rate of DI water is 30 ml/min.

Figure 3 An image of the flying droplets captured by a high speed camera

You can see the droplets atomised by the centrifugal force of the cup, which is pushed out downward by carrier gas. Thus, we made repetitive study on the shape of nozzle for liquid to be discharged from the cup as well as the position and direction of carrier gas to be discharged by using the high-speed video camera. We found the discharge hole should be as small as possible and there should be many holes, liquid discharge should be consistent, and gas current plate should be provided at the cup to discharge uniform carrier gas. Secondly, we measured the characteristics of droplets by using the SDPA, which was reported by Ueyama et al. (2003). This particle analyser is capable of simultaneously analysing size and speed of flying droplets by transmitting laser beam to the sprayed flying droplets and detecting their shadow by using optical detector.

Figure 4 Characteristics of the flying droplets from developed the rotational atomising aerozol nozzle corresponding to cup rotational speed is 70,000 min^{-1}, carry gas pressure is 0.12 MPa, flow rate of photo-resist is 5 g/min and stand off distance is 30 mm, (a) velocity distribution (b) diameter distribution (c) scatter diagram relationship between the diameter and the velocity



An example of measurement result is shown in Figure 4. Measurement conditions: Cup speed is 70,000 min^{-1}, compressed air with pressure of 0.12 MPa is used as carrier gas, and its flow rate is 128 L/min (ntp). Real photo-resist (PMER P-LA900 manufactured by Tokyo Ohka Kogyo) was used as spray liquid. The photo-resist is a mixture of novolac resin, propylene glycol mono methyl ether acetate (PGMEA) and sensitive material. Its viscosity was 6.0 mPa · S measured at an ambient temperature of 20°C. When spraying environment changes, viscosity of the photo-resist changes, resulting in the possible change in diameter of droplets. Thus, it was measured at an ambient temperature of 20°C. Spray rate was 5 g/min and distance between the tip of nozzle to the measuring position (stand off distance) was 30 mm. Distribution of flying droplets is shown in Figure 4(a). According to the figure, average speed is 19.66 m/s and standard deviation is 5.98 m/s. Distribution of droplets' diameter is shown in Figure 4(b). According to the figure, average diameter is 15.82 µm and standard deviation is 4.13 µm. Figure 4(c) shows the correlation between the droplets' diameter and their flying speed. You can see that large number of 10 µm diameter droplets exists in 20 m/s speed. The average droplets' diameter discharged from conventional and widely-used external mixing type two-fluid spray nozzle is 50 µm or larger, which is highly inconsistent, although it depends on the spraying conditions. Compare to the conventional nozzle, the flying speed of droplets sprayed from the newly developed nozzle is 20 m/s, and the standard deviation is 4.13 µm with the average diameter of 15 µm, so the inconsistency is small.

Secondly, we measured the speed and size of the flying droplets by SDPA by varying carrier gas pressure from 0.12 MPa to 0.21 MPa [flow rate from 128 L/min (ntp) to 200 L/min (ntp)]. The measurement result is shown in Table 1. Upper line shows the average and lower line shows the standard deviation.

Figure 5 shows the relation between carrier gas pressure and the droplets' average flying speed. When the pressure is increased from 0.12 MPa to 0.21 MPa at each cup's rotating speed, the droplets average flying speed increased from 20 m/s to 25 m/s. According to Table 1, as carrier gas pressure is increased at each rotating speed, standard deviation increases and inconsistency of speed also increases. The results indicate that the speed of flying droplets can be arbitrarily controlled to find out the optimum coating condition of TSV wafer by adjusting carrier gas pressure.

Table 1 Results of the measuring droplets by SDPA

Gas pressure (Gas quantity)	*Velocity of the droplets m/s*			*Diameter of the droplets µm*		
	Rotary speed min^{-1}			*Rotary speed min^{-1}*		
	30,000	*50,000*	*70,000*	*30,000*	*50,000*	*70,000*
0.12 MPa	19.09	19.68	19.66	28.51	15.84	15.82
128 L/min (ntp)	5.76	5.60	5.98	9.82	4.38	4.13
0.16 MPa	19.25	21.08	23.14	19.46	18.50	16.49
165 L/min (ntp)	6.74	7.14	7.08	5.67	5.49	4.48
0.21 MPa	21.99	21.40	25.86	17.07	17.12	16.69
200 L/min (ntp)	8.02	8.05	8.54	4.79	4.99	4.74

Notes: Upper: mean value; lower: standard deviation

Table 2 Thickness of the coated photo-resist in the TSV via holes

	Thickness of the photo-resist µm						
Measuring point	*a*	*b*	*c*	*d*	*e*	*f*	*g*
Width: 50 µm	16.73	2.83	3.35	1.03	0.77	7.09	7.46
Width: 100 µm	3.14	1.92	1.67	1.83	1.67	7.71	7.43
Width: 150 µm	3.11	3.47	3.47	0.63	0.99	6.67	6.22
Width: 200 µm	1.62	3.90	3.9	1.21	1.21	7.03	6.49

Figure 5 Relationship between carry gas pressure and mean velocity of the flying droplets corresponding to each rotational speed of the air turbine, which were summarised from the results of measurement using the shadow Doppler particle analyser (see online version for colours)

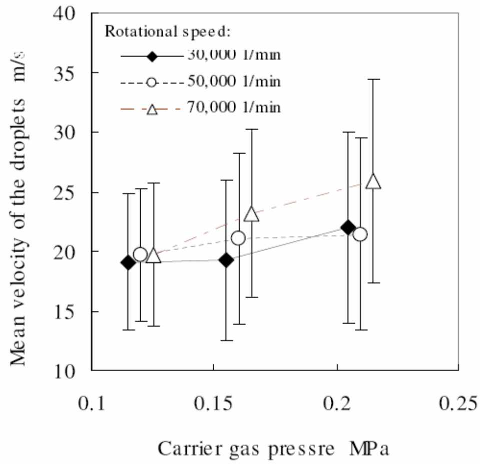


Figure 6 Relationship between rotational speed of the air turbine and mean diameter of the flying droplets corresponding to each carry gas pressure, summarised from the results of measurement using the shadow Doppler particle analyser

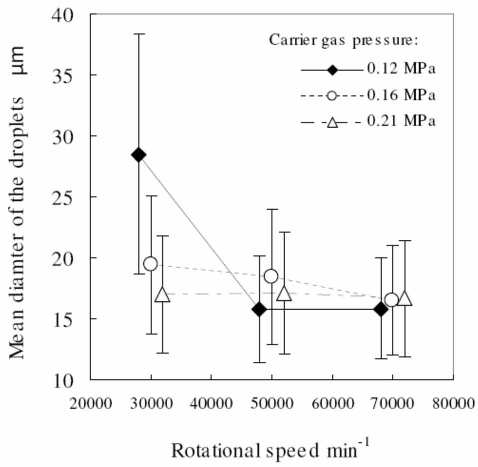


Figure 6 shows relation between cup's rotating speed and average diameter of flying droplets. As the cup's rotating speed is increased at each carrier gas pressure, average diameter of droplets tends to become smaller. The average diameter is 16 μm when the cup's rotating speed is 70,000 min^{-1}. According to Table 1, as the rotating speed of cup increases at the standard deviation of flying droplets diameter, number of larger diameter droplets tends to decrease and inconsistency of droplet size tends to become smaller. Also, as carrier gas pressure increases, average droplets diameter becomes smaller even in the cup's low rotating speed zone. It can be explained as the phenomenon resulting from reatomisation of droplets by carrier gas that takes place concurrently with rotation of cup whose speed controls the droplet size. These results show the cup's rotating speed should be set as fast as possible to make small flying droplets in order for the droplets to directly go into via holes with the opening diameter of around 20 μm to form coating film inside the via holes. Further, it is important to control flying droplets speed properly

3 Experimental

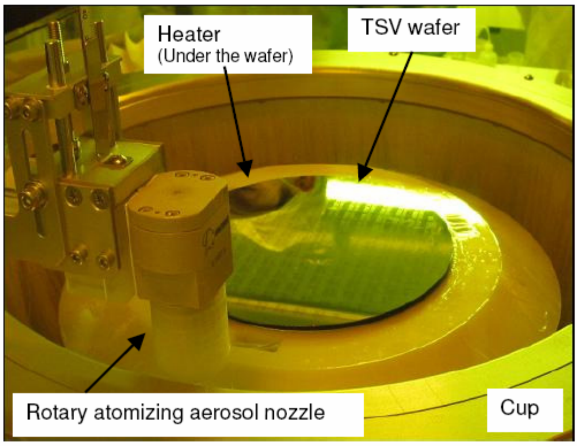
We made a prototype coating equipment incorporated with a newly developed rotary atomising aerosol spray nozzle mentioned in the previous section, and conducted a TSV wafer coating test. The prototype coating equipment is shown in Figure 7. Figure 7(a) shows the equipment overview. The equipment is divided into the following parts, namely, coating chamber (left top), actuator chamber (right top), machine control section (bottom) and photo-resist feeder. Exterior is made of stainless steel. Figure 7(b) shows the coating chamber. TSV wafer is set on a hot plate with a vacuum chuck inside the spray cup bath so that the wafer temperature can be changed arbitrarily. The rotary atomising aerosol spray nozzle installed on the X-Y actuator moves above the TSV wafer in a zigzag pattern to coat entire surface of the wafer. The bottom face of spray cup bath's interior is laden with solvent to collect the mist-form photo-resist that remained without becoming a coating film. The configuration of this system is designed to enable recycling of solvent containing dissolved waste photo-resist, which is collected and separated by the circulating system, and then only the solvent is fed back to the side panel of the spray cup bath. HEPA filter is provided on the top of the coating chamber to keep inside the chamber in Class 1000 cleanliness. As photo-resist is sensitive to ultraviolet ray, LED light to block ultraviolet ray is mounted.

To protect the rotary atomising aerosol spray nozzle, the most essential part of this equipment, from metal contamination, the section wetted by photo-resist is made of fluorine resin, and the air turbine is made of poly ether ether ketone (PEEK) resin. In addition, a double-barrelled plunger pump with 180 degrees phase-shift is adopted to cancel pulsation while feeding photo-resist. Inner diameter of the feed tube in the nozzle is 0.5 mm to increase fluid resistance in the liquid passage and reduce pulsation.

Photo-resist coating test on the TSV TEG wafer was conducted by using a newly developed prototype photo-resist coating equipment. Scanning electron microscope (SEM) image of TSV TEG wafer used in this test is shown in Figure 8. The TEG wafer is 200 mm in diameter, having groups of via holes with four different opening sizes, namely, 50 × 50 μm, 100 × 100 μm, 150 × 150 μm and 200 × 200 μm formed on its surface. Figure 9 shows the cross sectional view of the via holes. The via holes are formed by wet etching with tapered angle of 54.7 degrees after photolithography, which have inverted square pyramid cupped shape. Depth of via holes is 50 μm regardless of the opening size. Since the wafer remains unprocessed after wet etching, natural oxide films are stacked on its surface. After the coating film is formed, film thickness is measured at the points from 'a' to 'g' by using SEM.

Figure 7 The prototype coating system, (a) an appearance of TSV coating equipment (b) inside the coating room (see online version for colours)

(a)



(b)

Coating conditions: Cup's rotating speed of rotary atomising aerosol spray nozzle is 70,000 min^{-1}, and compressed air of 0.19 MPa is used as carrier gas. The stand off distance is 30 mm, nozzle's moving speed is 10 mm/s and overspray pitch is 5 mm. The stage temperature is kept constant to 40 °C. Photo-resist (PMER P-LA900 manufactured by Tokyo Ohka Kogyo) having ratio by weight of 10% was used as spray liquid. Discharge rate of the photo-resist was computed from solid content of the photo-resist and the moving speed of the nozzle to form 7 μm thickness coating on the wafer surface. After that, it was verified at the preliminary test and finally decided to be 6 g/min.

Figure 8 The surface of a TSV TEG wafer used in the experiments (see online version for colours)

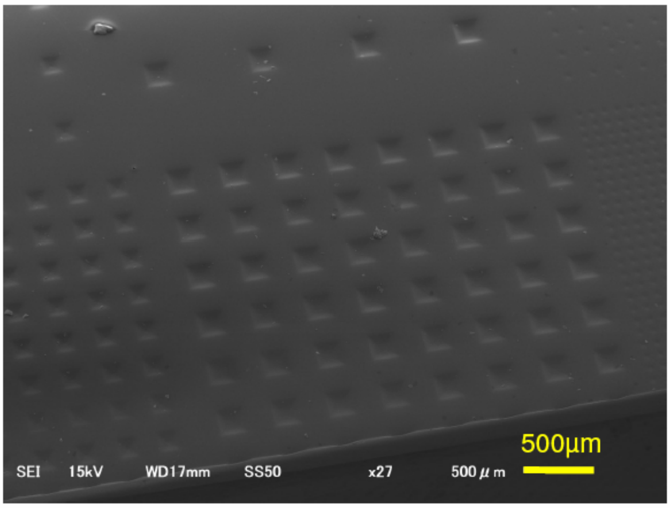
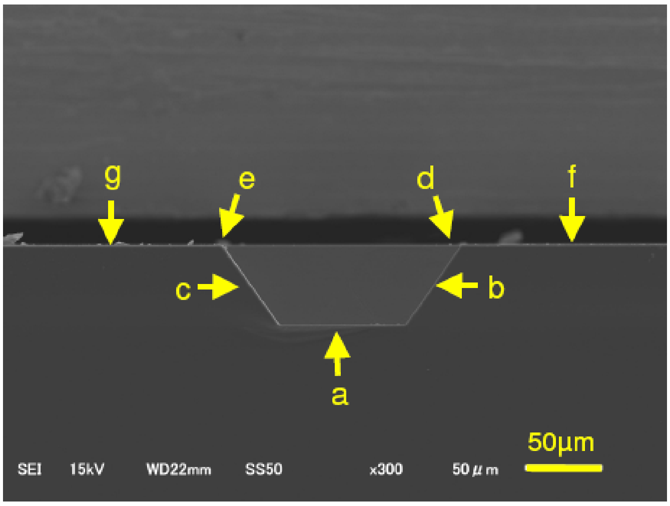


Figure 9 A cross-section of the via of the TSV wafer and measuring point of the films thickness (see online version for colours)

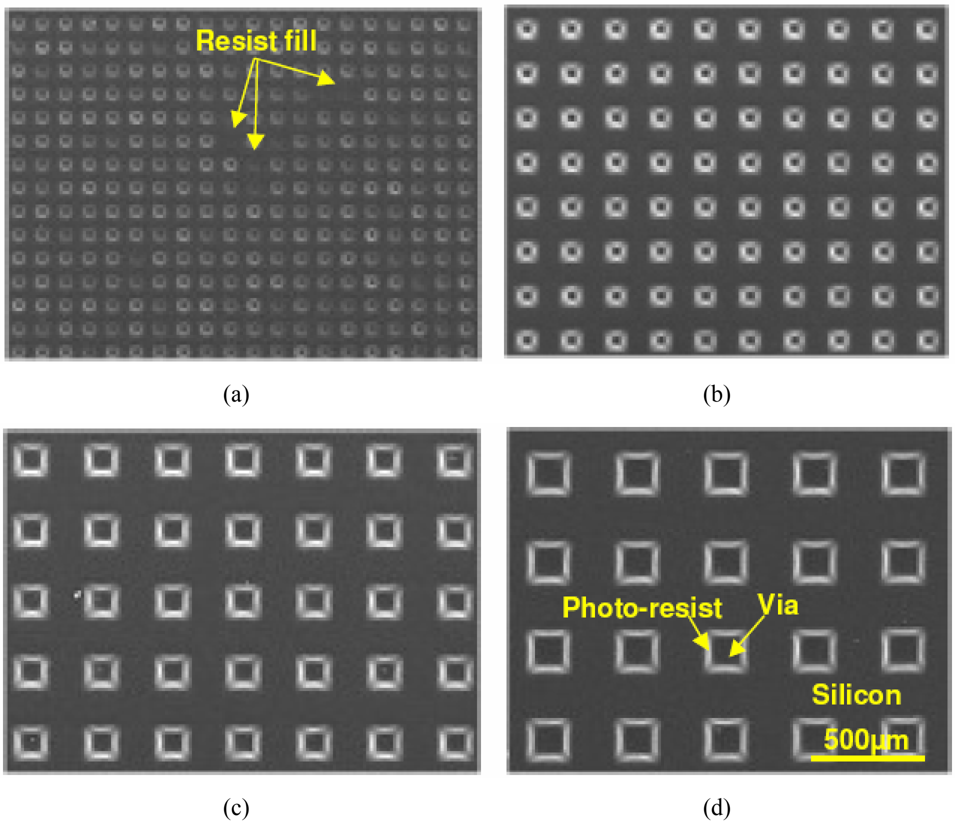


4 Results and discussions

SEM image of TSV wafer after coating photo-resist is shown in Figure 10. The image shows that every opening is coated. However, from the standpoint of yield at real production line, coating should be evenly formed inside via holes on all wafers. To ensure that, we counted the number of uncoated via holes and filled via holes (clogged via) as shown in Figure 10(a), and number of coated via holes by using SEM to compute coating rate (coated via holes/total via holes). As the result, the coating rate was 92.5%

for 50 µm opening, and 100% for 100 µm, 150 µm and, 200 µm openings. Since this method only checks the wafer from its surface, detailed coating state such as coating distribution on the TSV via holes' inner surface is not verified. We need to develop a non-contact measurement method in future.

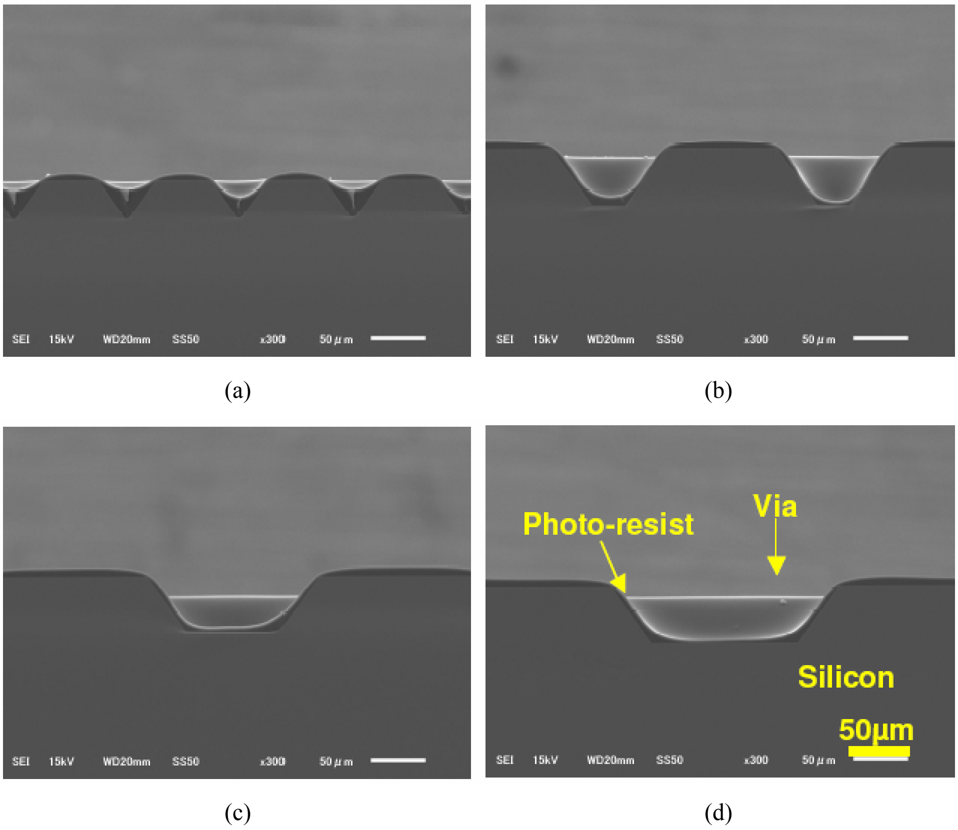
Figure 10 The surface of the TSV TEG wafer coated with the photo-resist, (a) width: 50 µm (b) width: 100 µm (c) width: 150 µm (d) width: 200 µm (see online version for colours)



We broke up the arbitrary spot of the via holes of each opening size on TSV TEG wafer. The cross sectional image of these via holes taken by SEM is shown in Figure 11. Conformal coating of thickness 1.2 µm to 3.2 µm formed along the shape of via hole can be observed on each via hole's cross section image. However, film thickness at bottom section of 50 µm via hole is relatively thick due to its narrow area. Also, coating thickness on the side walls 'b and c' of the via hole and opening parts 'd and e' are thinner than bottom face 'a' and top faces 'f and g' of TSV wafer. This phenomenon is assumed that photo-resist attached to the side walls and corner part of via holes has flown into the bottom part.

The test conducted with fixed coating condition is reported here. Since the optimum condition was found by repeating multiple experiments, systematic optimisation has not been carried out. Systematic analysis of relation between the coating condition and coating state should be done in future. And we will try to coat the photo resist films in smaller and the higher aspect TSV holes in the next step.

Figure 11 The cross section of the TSV TEG wafer coated with the photo-resist, (a) width: 50 µm (b) width: 100 µm (c) width: 150 µm (d) width: 200 µm (see online version for colours)



5 Conclusions

In this study, a new coating technique on semiconductor devices having three-dimensional stack structure was suggested and its prototype equipment was developed.

The suggested coating technique is a rotary atomising aerosol spray method which rotates the cup at the speed of up to 70,000 min^{-1}, atomises the photo-resist with centrifugal force of the cup, sends and sprays the atomised droplets with carrier gas. In this report, photo-resist coating on inner walls of the TSV on semiconductor devices having three-dimensional structure was studied.

1 Distribution of diameter and flying speed of photo-resist droplets sprayed by rotary atomising aerosol spray are measured by using a SDPA. According to the result, droplets diameter was 15.82 µm and flying speed was 19.66 m/s when the cup's

rotating speed is 70,000 min^{-1} and carrier gas pressure is 0.12 MPa. We have made a hypothesis that since the size of flying droplets is about 1/3 to 1/12 of TSV opening size (50 μm to 200 μm), it is small enough for the droplets to reach inside the via hole and form a coating film inside the hole.

2 Characteristics of droplets were measured by varying cup' rotating speed and carrier gas pressure of the rotary atomising aerosol spray nozzle. According to the result, as the cup speed increases, the average diameter of flying droplets decreases and inconsistency of droplet diameter becomes smaller. It is confirmed that when the carrier gas pressure is increased, average speed also increases.

3 Based on the measured droplets' characteristics, prototype coating equipment was made. This prototype equipment is capable of coating entire surface of a wafer while holding and heating a 200 mm wafer on the stage and letting the nozzle move above the wafer in a zigzag pattern.

4 Photo-resist is coated on the TSV TEG wafer having opening diameter of 50 μm to 200 μm, depth of 50 μm and square pyramid via holes with tapered angle of 54.7 degrees by using the prototype equipment. It is confirmed that coating along the inner walls of via holes is feasible.

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