# Feasibility Study: Inkjet Filling of Through Silicon Vias (TSV)

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# Abstract

Three-dimensional stacking and integration (incl. Through Silicon Vias - TSV) of microelectronic devices are modern techniques with great potential in industrial application, but face severe cost-disadvantages. In this paper, a new filling technique for TSV based on inkjet printing with a high cost-saving potential is proposed. We give a brief explanation of this new technique and present first results of processed samples.

Our TSV processing technique can be summarized in three major steps. In step one, multiple structures are pre-etched in standard silicon wafers. These structures require an electrical isolation from the surrounding substrate. In step two, a standard piezoelectric inkjet nozzle is used to print our self-developed silver ink into these structures, forming the vias. After the ink is dried, in the third and final step, the samples are sintered. Finally the TSV samples are analyzed by cross sectional polishing and a scanning electron microscope.

This paper proves the feasibility of inkjet printing of TSVs by homogeneous covering of via side walls with silver particles.

#### Introduction

The shrinkage of semiconductor devices' footprint and package size has been the major cost driver in semiconductor industries over the last decades [1]. Through Silicon Vias (TSV) permit the vertical interconnection of 3D-stacked semiconductor devices by directing the electrical signal from the front side through the substrate to the backside of the device [2]. Hence, TSV enable more chip packaging concepts than common 2Dintegration techniques and offer promising solutions to multi chip packaging challenges. Micro-electromechanical systems (MEMS), e.g. acceleration sensors, require interconnections to their signalprocessing circuits. Further on, logic and memory devices are combined by several approaches requiring interconnections. Wire bonding is a common technique for semiconductor interconnections but faces difficulties with the increasing demand of a higher signal speed, less power consumption and lower heat dissipation. Besides, bond pads require a major part of a chip's footprint. Bond wires can also act as inductors and may, thus, cause electromagnetic compatibility issues.

In recent years, inkjet printing of particle inks has drawn a lot of attention due to its applicability in various fields. For printing, particles with a size ranging from a few microns to only some nanometers [3] are dissolved in suitable solvents. Concerning thick film microelectronic components, conductors, resistors, capacitors and inductivities [4] have been realized. Used substrates vary from ceramics to a variety of flexible substrates [5].

We present a novel TSV metallization technique using inkjet printing, which shows a high cost-saving potential. In addition to its cost-efficiency, inkjet printing offers many advantages compared to complex thin-film processes for TSV metallization. Being a non-contact and additive process, inkjet printing has a great flexibility in terms of substrate characteristics like topography and wafer bow. Also, horizontal wire connections may be printed in the same process step as the TSV metallization, saving time and lithography layers.

# Materials & methods

## Wafer preparation

Our TSV metallization technique addresses a via-last process, i.e. fabricating the vias after CMOS and MEMS processing. Hence, the TSV process has a temperature budget of 400 °C and must be non-destructive to the MEMS structures. The silicon wafers are fabricated by standard MEMS processes. Anisotropic deep reactive ion etching (DRIE) allows for a high aspect ratio at TSV formation. In order to achieve large via depths, etching selectivity is enhanced by depositing a tetraethylorthosilicat (TEOS) hardmask prior to the DRIE. The electrical sidewall isolation separating the silver ink from bulk silicon is deposited from TEOS sub-atmospheric chemical vapor deposition. Before filling the vias with silver ink, the TEOS passivation layer is opened at the via bottom by back-etching to enable an electrical contact.

# Ink preparation & properties

Three main aspects have to be considered when designing a particle loaded inkjet ink: wetting behavior of the substrate, requirements of the specific printing system and sintering behavior of the particles.

When printing TSVs an ink featuring a high wettability, i.e. a low contact angle on the substrate is desirable as this will allow for ink spreading over the whole via column. In particular, ink flow into the pre-etched vias will be supported, thus allowing the particles to enter the via. Wettability strongly depends on the polar and dispersive surface tension of the substrate and the ink. The particles do not affect the inks surface tension [6]. Hence, measurements are conducted without particle loading. Figure 1 shows the side view of a contact angle measurement of the used solvent, diethylene glycol monobutyl ether (BC), on a silicon wafer using a KRÜSS DSA 100 drop shape analysis system. The spreading on the substrate indicates the desired wettability for printing TSVs.

Printability is a boundary condition placed by the printhead. For the presented study the single nozzle piezo actuated printhead MD-K-140 (microdrop Technologies GmbH, Germany) is used. Printability is strongly influenced by the ink viscosity and the dispersed particles (size and solid content). The requested viscosity range of 0.4 to 100 mPas is given by the printhead manual. A higher solid content leads to a higher viscosity. Generally viscosity can be adjusted by heating the ink. However, heating is limited due to increased solvent evaporation at the nozzle, which might lead to clogging.



**Figure 1**. Ejecting BC onto a silicon wafer (time elapsing from top to bottom) showing the desired low contact angle.

Realization of TSVs requires electrical conduction from the top to the bottom of the via. Thus, sintering of the particles has to result in a suitable conductivity. Applied temperatures are limited because of temperature sensitive components placed on the wafer in earlier stages of the wafer production. Conveniently, sintering of nanoparticles requires much lower temperatures than sintering of macroscopic particles. Sintering at temperatures of between 200 and 300 °C for 30 minutes suffice for the connection of the chosen silver particles (Silver Powder #11000-10, Ferro GmbH, Germany) with a mean diameter of 300 nm (Figure 2).

These three aspects are taken into account while designing the ink. The result is an ink based on diethylene glycol monobutyl ether with a solid substance content of silver particles of 30 % by weight and a viscosity of 8.2 mPas (at a temperature of 35 °C and a shear rate of 10 000 s<sup>-1</sup>). Additionally, the ink comprises a small amount of ethyl cellulose for ink stabilization. The particles have, as mentioned above, a mean diameter of 300 nm. Optical measurements as well as weight measurements are conducted concerning drop volume. We measured drop volumes of  $178 \pm 2 \,\mu\text{m}^3$  and  $162 \pm 10 \,\mu\text{m}^3$  respectively. Optical measurements are advantageous in time needed for measuring and reliability. Thus, they are to be preferred for future experiments and calculations.



Figure 2. SEM cross sectional image of sintered (300 °C for 30 min) silver nanoparticles on a silicon wafer.

#### Printing process & sintering

A self-developed printing station is used in this study. It consists of the single nozzle printhead incl. its ink reservoir, an

underpressure/electrical control system and a heatable substrate table. A camera is coaxially aligned with the nozzle for substrate observation and positioning.

The printhead control system is used to determine and maintain optimum printing parameters (piezo voltage, pulse length and nozzle temperature). For this purpose, the printhead is moved to an observation station. A second camera system allows for the analysis of emitted drops by means of a stroboscope diode, which is triggered time-delayed by the piezo voltage. This camera is used to determine drop volume as well. Appropriate printing parameters strongly depend on the ink used (i.e. its viscosity, dispersed particles, surface tension). Once parameters providing steady, reproducible drops are found, printing can start.

Substrate table movement and triggering of the piezo voltage are controlled by a LabView program designed specifically for our printing station. Layouts with freely selectable resolutions ( $\mu$ m per pixel) can be printed by loading a monochrome bitmap. In order to print, the substrate table is moved along under the nozzle line by line. Each pixel value specifies whether the piezo is triggered at a specific location.

For filling of the TSVs it is necessary to print a large number of drops. Subsequent drops have to be placed with a time delay allowing the solvent to evaporate. Because of the low solid content concerning volume (silver particles of 30 % by weight in BC come up to only 3.7 % by volume), omitting the time delay would result in overflooding of the via, thus contaminating the area around it.

Two ways of printing multiple drops at one location are realized. First, the whole layout is printed multiple times. In between successive layers, the substrate table is positioned so that the substrate camera can be used to observe the printing progress of a specific area after each layer. Before the substrate table is moved to print the next layer, an image of the printing progress is saved and the nozzle is cleaned. Cleaning is carried out by triggering the piezo multiple times (e.g. 1000 times with a frequency of 1 kHz). This prevents the nozzle from clogging because of solvent evaporation which is mainly caused by the substrate tables heating. Thus, printing with any waiting time and any number of layers can be carried out without the necessity of supervising the process while still allowing to document each layers progress. The second way of adjusting the ink amount is printing multiple drops at one location before moving on to the next. Both processes can be combined as well. The effect of different combinations is discussed

As mentioned above, sintering is used to create electrically conducting connections between the printed particles (Figure 2). For sintering, the temperature is raised with 10 K/min to at least 300 °C. After keeping peak temperature for 30 minutes, cooling-down to room temperature is realized with 10 K/min again.

## Characterization methods

Printed and sintered wafers are analyzed in top and cross sectional views. Top view images are obtained using an optical microscope. Cross sectional images of grinded vias are obtained using a scanning electron microscope (SEM).

For grinding, the silicon wafer is cleaved into several wafer pieces. The samples are placed into a sample holder and embedded using an epoxy resin as molding material, which is liquid at room temperature. After baking out the epoxy resin a cross section polish is prepared. First, grinding is carried out with a coarsegrained grinding wheel to subtract material. Such abrasive materials have grain sizes of between 15  $\mu$ m and 30  $\mu$ m. Next, the sample is cleared of deformations and damages by mechanical polishing to approach the TSV to be analyzed. For this step, diamond polishing is a suitable method, using diamond particles with a grain size of 1  $\mu$ m to 9  $\mu$ m. The cross section is finalized by chemical mechanical polishing. Oxide particles like silicon dioxide or aluminum oxide with grain sizes of about 0.05  $\mu$ m dispersed in alkaline solution permit fine and precise mechanical polishing of the last few microns before reaching the target cross section. Moreover, the chemical mechanical polishing removes smallest defects or polishing artifacts like scratches. In addition, applying the alkaline solution leads to a better material contrast for SEM studies and further analysis.

The acquired images are used for evaluation of different approaches to inkjet printing of TSVs.

# **Results & discussion**

The aim of electrically conducting silver filled TSVs can be achieved by filling the via completely or by coating its walls. Feasibility of both methods is discussed by interpreting the results of three printing variants.

## Variant I

In variant one ink is ejected drop by drop into the vias (depth  $300 \,\mu\text{m}$ , diameter  $75 \,\mu\text{m}$ ) of a wafer, which is heated to a temperature of 80 °C. Waiting time between the drops is set to 30 s. About 780 drops are needed to fill the via with silver (considering silver bulk density). However, after the deposition of 120 drops an silver cap forms on top of the via.

Investigation on cap formation is carried out by variation of substrate temperature and waiting time. It shows, that the formation of caps depends strongly on these parameters (Table 1). Lower substrate temperatures as well as shorter waiting times reduce the occurrence of caps, i.e. the more solvent is present while printing, the less caps are formed. It has to be taken into account that the sample size of 9 to 17 for each pair of parameters is not sufficient for reliable statistical conclusions. Sample size explains the lower than expected percentage of capped vias at operating point 70  $^{\circ}$ C/ 60 s.

Cross sectional SEM images (Figures 3 and 4) of the sintered vias confirm, that the silver particles are not carried down to the via bottom, thus, forming the caps. As mentioned before, vias can be realized by complete filling or by wall coating. The SEM images show large cracks in the solid silver caps. These have to be avoided because cavities decrease an electrical components lifetime. The occurrence of cracks is ascribed to sinter shrinkage.

Printing series of single drops with the given parameters discloses two important aspects of printing TSVs. First, fast evaporation at the vias top requires larger amounts of solvent than a single drop can provide. Second, complete filling with particles comprises the disadvantage of cracks in the via, thus, wall coating is the preferred method.

Table 1. Variation of substrate temperature and waiting time strongly influence the formation of via caps. For each pair of parameters 9 to 17 vias are analyzed.

Temperature in °C	Waiting time in s	Capped vias in %
	15	11
60	30	55
	60	100
70	15	78
	30	89
	60	(67)
80	15	100
	30	100
	60	100
00	30	100
90	60	100



**Figure 3**. SEM cross sectional image of a via after sintering at 300  $^{\circ}$ C for 30 min. 120 drops deposited with a waiting time of 15 s and a substrate temperature of 70  $^{\circ}$ C.



**Figure 4**. SEM cross sectional image of a via after sintering at 300 °C for 30 min. 120 drops deposited with a waiting time of 60 s and a substrate temperature of 90 °C.

#### Variant II

To overcome the difficulties of printing variant one, i.e. cap forming, the printing process is adjusted to achieve wall coating in the vias. Additionally, shallower vias are filled (depth  $100 \,\mu$ m, diameter 75  $\mu$ m). The number of drops needed for an overall silver coating of 4  $\mu$ m thickness is calculated. It amounts 16 drops for a via with a depth of 100  $\mu$ m and a diameter of 75  $\mu$ m. This number is fractioned in multiple drop series. The result of (a) printing all 16 drops at once (frequency of 1 kHz) is compared to the result of (b) printing 8 fractions of 2 drops with a waiting time in between fractions of 60 s. The analyzed samples are sintered at 400 °C.

The wafers surface is contaminated with silver particles in both cases. Using process (a) leads to a much larger contaminated area. This can obviously be explained by the larger amount of solvent present after drop deposition. As printed, sixteen drops have a volume of about 30 vias. Thus, overflooding of a large area around the via is inevitable.

SEM images show the great advantage of variant two printing processes compared to variant one (Figures 5 and 6). A large amount of particles is deposited on the side walls of the via as well as on the bottom forming incomplete, probably non-conducting, but homogeneous silver layers. As the same number of drops are printed in both variant two processes, more silver particles are carried into the via using process (b) due to reduced overflooding.

Some overflooding is necessary in order to allow for an electrical connection from the via to the wafer surface. However, the occurrence of excessive overflooding is disadvantageous in two aspects. First, calculating the number of drops necessary to inject a



**Figure 5**. SEM cross sectional image of a via printed with variant II process (a) after sintering at 400 °C for 30 min.16 drops deposited without a waiting time. Substrate temperature 80 °C.



**Figure 6**. SEM cross sectional image of a via printed with variant II process (b) after sintering at 400 °C for 30 min. 16 drops deposited in 8 fractions of 2 drops with a waiting time of 60 s. Substrate temperature 80 °C.

specific amount of silver into a via is hindered, if not impossible. Second, wide spread coverage of the wafer surface enlarges space consumption of a single TSV and can interfere with other components on the wafer.

Printing variant two, though providing considerable improvement compared to variant one, has to be adjusted to form closed layers.

#### Variant III

In order to investigate the reason for incompleteness of the silver layers, variant three introduced some slight modifications. Printing process (b) is used again with severely reduced sintering temperature (200 °C) for one sample and the number of drop series is doubled for another, i.e. 16 fractions of 2 drops. A via printed with the last-mentioned parameters can be seen in top view in Figure 8.



**Figure 8**. Via in top view after the deposition of 32 drop in 16 fractions of 2 drops with a waiting time of 60 s. Substrate temperature 80 °C. Depth of field from wafer surface to via bottom.

Doubling the amount of deposited ink results in better coverage inside the via (Figure 9). Total layer thickness ranges from 2.9 to  $15 \,\mu$ m. Small cavities between chunks of silver still exist and a connection from inside the via to the wafer surface is weak. In addition, bulges are formed half way down the via. Possibly, this is a result of the solvent evaporating mainly in this part of the via.

Reducing the sintering temperature results in a more homogenous coverage than sintering at 400 °C (Figure 10). No large cavities can be found. Bulges are present in this sample as well. Layer thickness ranges from 1 to  $8.6 \,\mu\text{m}$ . However, after sintering at 200 °C the nanoparticles appear to be welded but not coalesced. The effect of sintering temperature on conductivity can only be investigated by electrical measurement. Therefore, wafers with pre-processed daisy-chains (i.e. conductors connecting the bottoms of two vias inside the wafer) will be used for subsequent printing trials.

Two conclusions can be drawn from these two variations. First, sintering temperature has to be chosen below 400 °C to avoid small cavities. Second, the amount of solvent present at a time, i.e. the number of drops deposited at once, can be used to control where most of the particles will accumulate inside the via. Changing the number of drops deposited at once while printing might, thus, result in homogeneous layers concerning thickness while avoiding any cavities by selecting an appropriate sintering temperature.



**Figure 9**. Variant III: SEM cross sectional image of a via after sintering at 400 °C for 30 min.32 drops deposited in 16 fractions of 2 drops with a waiting time of 60 s. Substrate temperature 80 °C.



**Figure 10**. Variant III: SEM cross sectional image of a via after sintering at 200 °C for 30 min.16 drops deposited in 8 fractions of 2 drops with a waiting time of 60 s. Substrate temperature 80 °C.

# **Conclusions & Outlook**

This paper proves the feasibility of inkjet printing of through silicon vias. While complete filling of the vias is undesirable due to cracks appearing during sintering, side wall coating of the vias is a promising approach. Descriptions on how to achieve homogeneous coverage with silver particles in the via are given.

Further experiments have to investigate the influence of the amount of ink printed per time and of the sintering temperature on the conductance of the vias. Shifting the drop rate might greatly increase homogeneity and quality of the conductive coating. Pre-processed daisy-chains will allow for electrical characterization of future samples. Corresponding electrical measurements require contact areas on the wafer surface. Coverage of the via edge has to be realized, e.g. by printing a small number of drops slightly shifted to the via on purpose.

The evaluation of faster, non-destructive characterization techniques concerning silver distribution in the via, e.g. using x-rays, is in operation. Non-destructive characterization will be a useful tool when investigating the vias stability in the field, e.g. its ability to withstand temperature cycles.

Additionally, future investigations will take different particles into account, e.g. adding smaller nanoparticles into our ink might improve binding of the silver when sintering at low temperatures like 200 °C.

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