



3D-interconnect: Visualization of extrusion and voids induced in copper-filled through-silicon vias (TSVs) at various temperatures using X-ray microscopy

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ABSTRACT

Visualization of voids and copper extrusion in copper-filled through-silicon vias (TSVs) under different annealing conditions is greatly enhanced using X-ray microscopy. In addition, the dimensions of the TSVs after Cu deposition can also be measured by 3D reconstruction. Surface inspection found mushroom defects on the top of TSVs after deposition of the etch stop layer, forming a small bump on top of the TSV. The ~50 nm etch stop layer is part of the dielectric stack used before metal 1 is deposited on top of the TSVs. The TSVs are annealed, then undergo chemical mechanical polishing (CMP) before the etch stop layer is deposited at 350 °C. It is assumed that after CMP the etch stop layer on top of the TSVs is planar. However, cross-sectional scanning electron microscopy (XSEM) shows mushroom defects but no particles at the interface of the TSV and the etch stop layer.

This study investigates the changes in copper extrusion and induced voiding that result from different annealing temperatures. Round TSVs ~25 μm deep and 4–5 μm in diameter were filled with copper by electrochemical deposition (ECD), followed by chemical mechanical polishing and pre-annealing to 150 °C for one hour. Based upon the material properties of TSVs, the high density copper absorbs photon energy more strongly than silicon or air. This results in good contrast within the copper-filled vias when X-ray micrographs are taken and allows voids within the material to be seen. A lab-based X-ray microscope with 8 Kev X-ray energy allows a penetration of ~50 μm into the silicon. Consequently, sample preparation requires wafers to be backside polished from an initial thickness of 775 μm to <50 μm. When scanned with a lab-based X-ray microscope, a set of TSVs is imaged and the 3D X-ray tomography shows voids or seamlines at the bottom of the TSVs. This sample was annealed to 225 °C and 300 °C, respectively. Upon scanning the same TSVs, the X-ray micrograph shows the seamline changes shape, voids are induced at the center of TSVs, and copper extrudes on top of the TSVs. This suggests the copper extrusion causes the etch stop layer to form a defect at the top of the TSVs. An X-ray microscope offers the potential to inspect voids in multiple vias at the same time without a physical cross section of the TSVs. This technique can be used to advance the study of different stress-induced voids used for 3D interconnects.

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1. Introduction

Through silicon vias (TSVs) are important in creating 3D integrated circuits and 3D packages in which they provide an electrical connection for two or more die and wafers. The structure of the TSV is deeper and wider than a damascene via. These TSVs are filled with copper (Cu), which has a lower resistivity than aluminum. Recent scanning electron microscopy (SEM) surface inspection

has revealed a mushroom defect (small bump) when the etch stop layer is deposited on top of the TSVs at 350 °C. This defect deplanarizes the dielectric layer, which can affect the uniform etching of the dielectric layer for metal 1 and impede its Ohmic contact with the TSV. Fig. 1 illustrates the defect-free etch stop layer (a) and mushroom defect (b). The cross-sectional SEM suggests the mushroom defect is caused by copper extruded from the TSV. This study applied thermal stress by annealing the TSVs to various temperatures to observe whether copper extrusion is thermally induced on the same TSVs.

A high resolution imaging technique is needed to visualize the changes by thermal effects on copper-filled TSVs. The source of the technique must be able to non-destructively penetrate through

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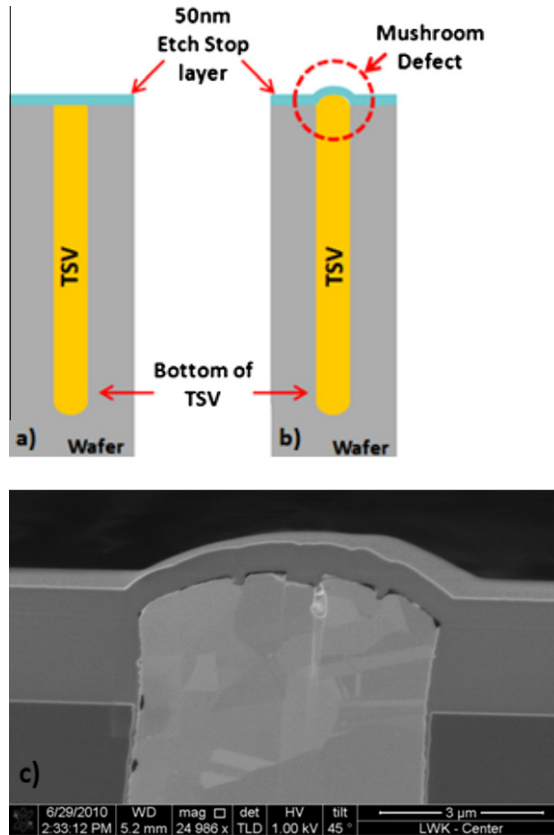


Fig. 1. Defect-free etch stop layer (a), mushroom defect (b), SEM of mushroom defect (c).

the high density copper-filled TSVs to compare the same TSV before and after annealing. A conventional optical microscope that uses visible or infrared light as a light source cannot do this because metal is opaque under the light source. An acoustic microscope, a good sub-surface imaging technique, applies ultrasound waves that can penetrate any elastic material but its resolution is limited by its wavelength to $\sim 60 \mu\text{m}$ with 110 MHz frequency [1]. High resolution transmission electron microscopy (TEM) is able to transmit copper, but the sample must be polished to at least 100 nm for penetration. X-ray energy at around 8 keV has better transmission capability than TEM and uses a Fresnel zone plate applied with the X-ray microscopy optical system, allowing for a res-

olution of $\sim 50 \text{ nm}$ [2,3]. Therefore, a lab-based X-ray microscope that generates 8 keV X-ray energy was used to image the thermally induced changes in the TSVs without physical cross-sectioning into the TSV. A finite element model (FEM) was made to compare the simulated extrusion with the X-ray microscope measurements.

2. Experiment procedures

2.1. Sample

TSVs arrays 4 and 5 μm in diameter are etched to $\sim 26 \mu\text{m}$ deep with reactive ion etching (RIE) on a 300 mm silicon wafer. With plasma-enhanced chemical vapor deposition (PECVD), a layer of 200 nm liner (TEOS) is deposited on the TSV wells to prevent the Cu from interacting (i.e., leakage) with the Si substrate. Then 10 nm of a copper diffusion barrier layer (Ta/TaN) is deposited on the TEOS-SiO₂ by physical vapor deposition (PVD). Using the same method, a 500 nm copper seed layer is deposited after the barrier layer. Finally the rest of the TSV well is plated by electrochemical deposition (ECD) of copper at 40 °C. After ECD, the whole surface of the 300 mm wafer is left with a layer of copper overburden. Chemical mechanical planarization (CMP) removes this overburden layer and keeps the surface smooth to prepare the wafer for dielectric layer deposition. Finally, the wafer undergoes a 150 °C anneal for one hour without depositing the dielectric layer used before metal 1 deposition.

2.2. Sample preparation setup for imaging

Different X-ray energies have different transmission capabilities in materials. At $\sim 8 \text{ keV}$, the transmission capability is $\sim 50 \mu\text{m}$ for silicon wafers with copper-filler TSVs. This can be calculated by Beer Lambert's law,

$$I = I_0 e^{-\alpha x} \quad (1)$$

where the transmitted X-ray energy, I , through a material with thickness, x , is related to the incident X-ray energy, I_0 , and the material attenuation coefficient, α . The TSV arrays on the wafer are cleaved into $5 \text{ mm} \times 5 \text{ mm}$, which are polished from $775 \mu\text{m}$ to $\sim 50 \mu\text{m}$ from the back with sand paper. The TSV arrays that remain in the $5 \text{ mm} \times 5 \text{ mm} \times 50 \mu\text{m}$ wafer are then scanned by Xradia's lab-based X-ray microscope, nano X-ray computed tomography (NanoXCT). Its $\sim 8 \text{ keV}$ X-ray energy is generated from a copper anode. A condenser condenses and focuses the emitted X-ray energy to the sample, which is attached to a rotating sample stage. The transmitted X-ray energy is then refocused into the detector by a

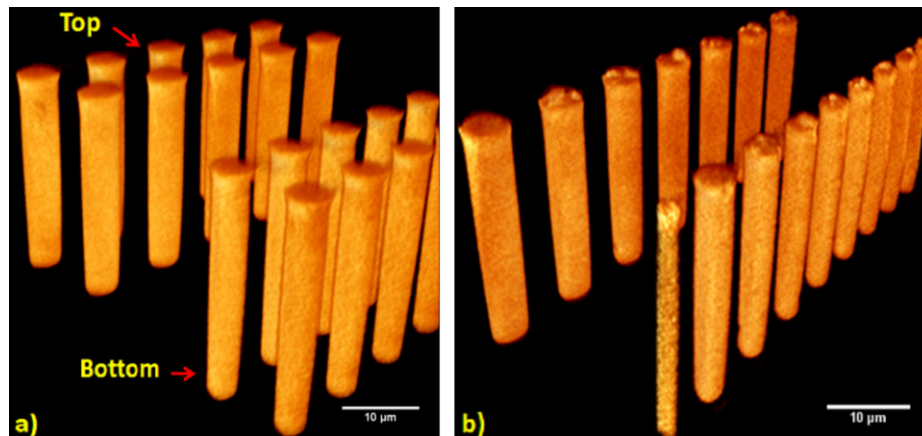


Fig. 2. External view of TSVs before annealing (a) after 300 °C annealing. Extrusion effect at the top of TSVs (b) after 300 °C annealing.

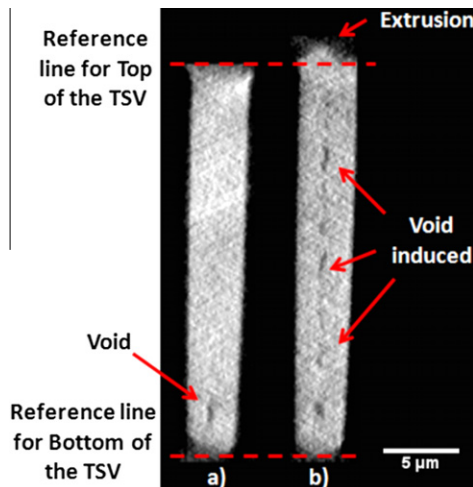


Fig. 3. XY plane at the center of 4 μm TSV before (a) and after 225 °C annealing (b).

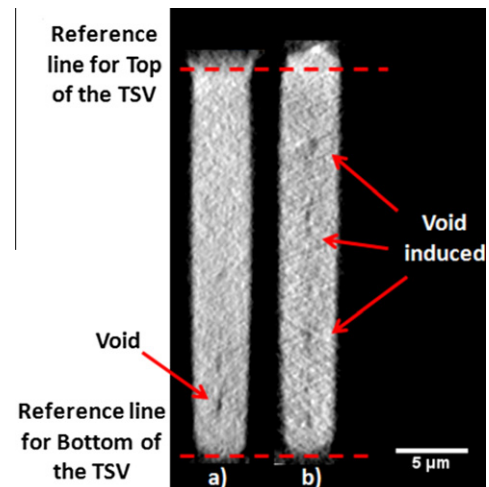


Fig. 5. XY plane at the center of 4 μm TSV before (a) and after 300 °C annealing (b).

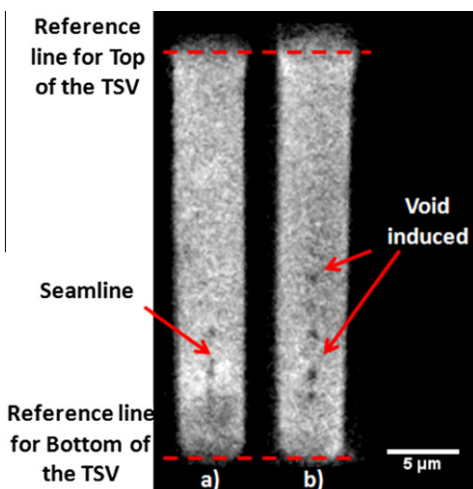


Fig. 4. XY plane at the center of 5 μm TSV before (a) and after 225 °C annealing (b).

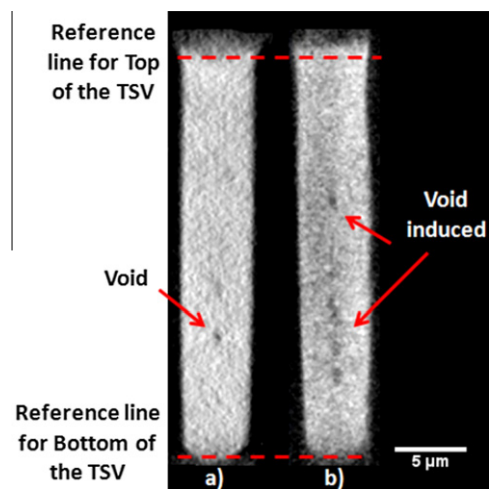


Fig. 6. XY plane at the center of 5 μm TSV before (a) and after 300 °C annealing (b).

Fresnel zone plate that determines the resolution of the image. Samples attached at the sample stage rotate from -70° to $+70^\circ$, for a total of 141° during the tomography scan. Three-hundred twenty-one X-ray images are taken during the 141° rotation with an average ~ 3 s exposure per image. The images are reconstructed using Xradia's 3D reconstruction software. The same sample is annealed to 225 °C, and the tomography scans are repeated. The same procedure is repeated on a different piece of wafer with an annealing temperature of 300 °C. 3D tomography of the same TSV arrays before and after annealing capture changes in the same copper-filled TSVs; the reconstructions are available in 2D and 3D.

3. Results and discussion

3.1. X-ray external inspection of embedded TSV arrays

Fig. 2a is the 3D reconstruction of the TSV array before annealing. The large field of view ($60\ \mu\text{m} \times 60\ \mu\text{m}$) of the X-ray microscope allows for more than 20 TSVs to be captured in a single tomography scan. The external view of the TSV array shows planarization on top of the TSVs. After the 150 °C annealing, no deformation is observed from the top of the 4 and 5 μm TSVs. After

225 °C and 300 °C (b) annealing, copper extrusion is apparent at the top of the TSVs. This thermally induced extrusion is suggested as the root cause of the mushroom effect that appears after the 50 nm etch stop layer is deposited at 350 °C.

3.2. X-ray internal inspection of the embedded TSV array

From the same tomography scan, a series of 2D images at different XY, YZ and XZ planes can be obtained to analyze the internal of TSVs. Because the X-ray microscope measures at absorption mode, higher density material will have a brighter contrast. In this case, copper will have brighter contrast than the silicon wafer and voids in the TSV. The 2D images shown in Figs. 3 and 4 are internal views of 4 and 5 μm diameter TSVs before and after 225 °C annealing. The figures show the XY plane at the center of the TSVs. In Fig. 3, a void is observed at the bottom of the 4 μm TSV (a); the depth before annealing to 225 °C is measured at 25.7 μm. After 225 °C annealing (b), the XY plane at the center of the same TSV clearly exhibits an extrusion effect at the top of TSV (see the top reference line). Induced voids are found along the center of the TSV as seen in Fig. 4a, which shows a seamline at the bottom of the 5 μm TSV. This 26.71 μm deep TSV was then annealed to 225 °C. The shape

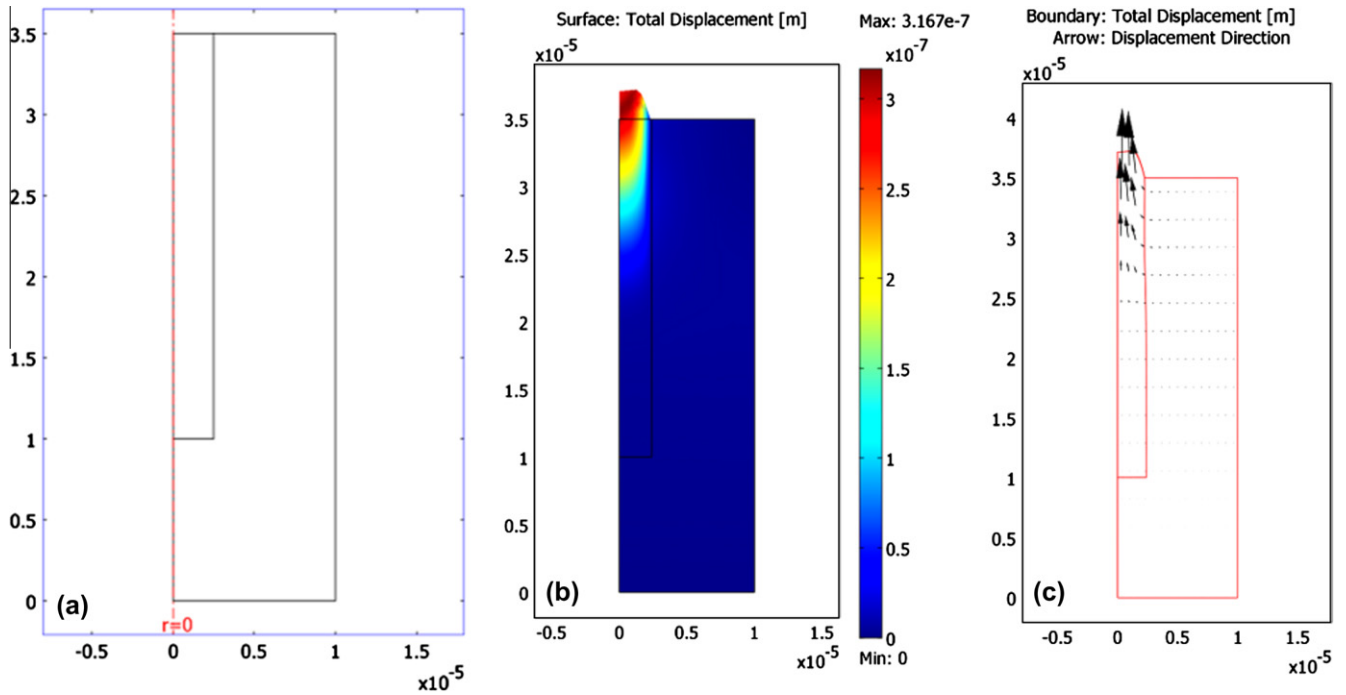


Fig. 7. The TSV geometry using 2D axial symmetry coordination (a), the displacement contour (b), the direction of extrusion of copper (c).

of the seamline changes and voids are observed at the center of the TSV, but fewer than in the 4 μm TSV. The same thermal stress effects are observed at 300 $^{\circ}\text{C}$, which are shown in Figs. 5 and 6.

3.3. Modeling

A 2D axial symmetry FEM was modeled based on the same dimensions and loading conditions as the measured sample. Its geometry is shown in Fig. 7a. Elastic–plastic copper was used as the TSV and silicon as the wafer. The linear isotropic strain-hardening and temperature-dependent yield strength (σ_y) of the elastic–plastic copper are expressed in Eqs. (2) and (3), respectively [4]:

$$Y(\varepsilon^{-p}) = Y_0 + h\varepsilon^{-p} \quad (2)$$

$$\sigma_y = 222.4 - 0.35T \quad (3)$$

From Eq. (2), Y is the yield stress (MPa) as a function of plastic strain (ε^{-p}) and h is the tangential modulus (MPa). Some studies show that the yield stress of elastic–plastic copper ranges from 80 MPa to 300 MPa [5,6]. Others have reported the initial yield stress and tangential modulus at 172.3 MPa and 517 MPa, respectively, as measured by μ -Raman Spectroscopy, which are corroborated by thermo-mechanical studies [7–9]. The strain-hardening and temperature-dependent yield strength is then applied to the elastic–plastic copper in simulation. The Comsol thermal structural interaction module is used in the FEM to compare the deformation as shown in the FEM result and X-ray microscope data. Fig. 7a, the FEM result, which shows copper extruding from the top of the TSV after a 300 $^{\circ}\text{C}$ anneal. These results are based on the difference in the coefficients of thermal expansion of copper and silicon and the plastic deformation of copper TSV. In the FEM result, copper TSV extrudes from the top of the TSV at a maximum of $\sim 0.32 \mu\text{m}$, which is close to the X-ray measurement, where indicating the depth after annealing becomes $\sim 0.4 \mu\text{m}$. This $\sim 0.4 \mu\text{m}$ extrusion is great enough to cause a mushroom defect at the 50 nm etch stop layer. In Fig. 7c the (red) boundary line shows

the extrusion, while the arrows (black) indicate the displacement or extrusion direction. The size of the arrow is proportional to the amount of displacement. FEM results show greatest displacement occurs at the top and toward the center of the TSV.

4. Conclusion

We have demonstrated the visualization of extrusion and voids in copper-filled TSVs at various temperatures using an 8 keV lab-based X-ray microscope. The 3D structural information generated by the X-ray microscope clearly shows extrusion from the top of the TSV and voids at the center after samples were annealed to 225 $^{\circ}\text{C}$ and 300 $^{\circ}\text{C}$. This makes the X-ray microscope a powerful tool to inspect the thermally induced changes in the same TSVs without having to physically cross-section them. Furthermore, results from a 2D FEM based on thermal structural interactions of materials were found to fit the X-ray measured profiles. From these matching results, it is suggested that the deformation or extrusion is predominantly strain-induced and depends on the elastic–plastic behavior of the copper. The effect of voids and other defects in the TSVs on the extrusion phenomenon should be made a topic of further study.

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