Applying X-ray microscopy and finite element modeling (FEM) to identify the mechanism of stress-assisted void growth in through silicon via (TSV)

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APPLYING X-RAY MICROSCOPY AND FINITE ELEMENT MODELING (FEM) TO IDENTIFY THE MECHANISM OF STRESS-ASSISTED VOID GROWTH IN THROUGH SILICON VIA (TSV)

by

Lay Wai Kong

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APPLYING X-RAY MICROSCOPY AND FINITE ELEMENT MODELING (FEM) TO IDENTIFY THE MECHANISM OF STRESS-ASSISTED VOID GROWTH IN THROUGH SILICON VIA (TSV)

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This project is dedicated to my Parents who have never given up on me or failed to give my moral support under any condition.
ABSTRACT

Fabricating through-silicon vias (TSVs) is challenging, especially for conformally filled TSVs, often hampered by the seam line and void inside the TSVs. Stress-assisted void growth in TSVs has been studied by finite element stress modeling and X-ray computed tomography (XCT). Because X-ray imaging does not require TSVs to be physically cross-sectioned, the same TSV can be imaged before and after annealing. Using 8 keV laboratory-based XCT, voids formed during copper electroplating are observed in as-deposited samples and void growth is observed at the void location after annealing. We hypothesize that the mechanism generating voids is hydrostatic stress-assisted void growth. Stresses in a copper-filled TSV with a pre-existing void were simulated by finite element methods. The peaks of the hydrostatic stress and its gradient are shown to be around the edge of the void. Comparing simulated results and experimental data shows that void growth in TSVs is stress-assisted: vacancies diffuse and coalesce at the void as a result of the hydrostatic stress gradient.
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CHAPTER 1
Introduction

1.1 3D-Interconnect: The Through Silicon Via Approach

In 1965, Gordon Moore first predicted that transistor density on integrated circuits (IC) would double every two years. Since then, the semiconductor industry has pushed the limits of 2D scaling in an effort to maintain Moore’s law. In response to the challenges facing the scaling of complementary metal-oxide-semiconductor (CMOS) technology, the International Technology Roadmap for Semiconductors (ITRS) now includes roadmaps for extending CMOS and for technologies beyond CMOS. A key part of maintaining the trend of increasing CMOS capability is the introduction of improved on-chip and chip to chip interconnection. Thus, the ITRS has traditionally included both Interconnect and Packaging Roadmaps. The demand from consumers for electronic devices with better performance and portability has forced a new paradigm known as “More than Moore,” which includes the use of three-dimensional integrated circuits (3DICs) approaches. 3DICs are a relatively new packaging technology consisting of stacking different integrated circuits vertically to achieve higher speed, better form factor, increased functionality, and heterogeneous functions in one package. However, the realization of this increased functionality is difficult with conventional approaches for IC fabrication and packaging methods. One of the main limiting factors in package size comes from a chip’s interconnects. Figure 1 is a SEM image of a 3DICs, which uses wires as IC-IC connection [1]. Technology that heavily uses wire bonds may save precious space on a PCB, but wastes much more 3D space. This is because nothing can come in contact with IC-IC, thus forcing them to be spatially separated from each other preventing a shorting of the device. Additionally,
more material is used to keep electrical isolation between ICs.

![Figure 1.1: Wire bonding technology for 3DICs.](image)

The leading alternative to wire bonds is the utilization of Through-Silicon-Vias (TSVs). Typically, TSV’s contains a high-aspect-ratio metal via embedded in silicon and electrically isolated from the silicon by a layer of dielectric liner hence forming a metal-oxide-semiconductor structure. It functions as 3D interconnect that passes through a wafer or chip allowing an electrical connection through the substrate. This technology is beginning to be commercially utilized in many different devices and shows a promising future in many prospective applications including digital and video cameras [2, 3], as well as DRAM [4].
A simple TSV process flow is shown in Figure 1.2. First, dielectric layer and photoresist are deposited on a piece of wafer (a). Under a mask, the resist is exposed to a radiation source of a specific wavelength. This allows the exposed resist to be removed when placed in a developer solution and transferring the pattern on the mask to the photoresist (b). Then, the patterned resist is placed in a developer solution to etch away exposed material besides dielectric layer (c). With reactive ion etching method (RIE), the exposed wafer area (TSV arrays pattern) is etched to the targeted TSV dimension (d). The TSV array trenches are deposited with liner, barrier, and Cu seed layer (e). The TSV trench is then deposited with copper (f). Finally, chemical mechanical polishing (CMP) is used to polish away the overburden of copper (g). These TSV structures are deeper and wider than a damascene via, which is in the sub-micron range. Therefore, filling the TSV can take more than 10 times the damascene via filling time [5]. Two methods have been actively studied by researchers and engineers to fill the TSV’s: 1) Conformal deposition and 2) Bottom
up or (aka superconformal deposition).

With the demand for TSVs growing, making reliable TSV for 3DICs is important. The purpose of this research is to analyze the effect of thermal stresses on through silicon vias using novel inspection techniques to observe voids without cross sectioning the sample and utilize finite element modeling to study these stresses throughout the vias and around voids.
1.2 Hypothesis

Figure 1.3: Voids close to the bottom of the TSV after pre-annealing at 150°C (a). SEM image suggests voids are generated at the center of TSV after annealing at 300°C (b).

Figure 1.3 shows a cross section scanning electron microscope (SEM) image of a 5µm diameter TSV after deposition at room temperature (a) using a conformal plating method. Voids are apparent at the bottom of the TSV. The SEM image of another TSV from the same piece of wafer annealed to 300°C shows bigger voids at the bottom of the TSV and smaller voids along the center (b). A comparison of the pre- and post annealed TSV cross-sectional images suggests that voids grow in the TSV after annealing. The growth of such a void could cause device degradation and eventually reliability issues. This void growth in conformally filled TSV is hypothesized as diffusion and coalescence of vacancies to the pre-existing void area under
the driving force of hydrostatic stress gradients. To identify the mechanism of stress-assisted void growth in the TSV, the following objectives have been investigated.
1.3 Objectives

There are many challenges faced by new technologies during their initial stages. This research is motivated by the need of new inspection technique and the choice of TSV filling method as part of the effort to optimize the reliability of TSV. At this point, thermal testing of TSV’s filled using different methods is necessary. This allows for a better understanding of impact of thermal stress on the TSV (e.g. void growth as shown in Figure 1.3). Without this analysis, 3DICs with TSV would not be rendered fully reliable. This also causes immature design parameters and operational conditions, which could lead to the failure of 3DICs at the early life stage.

To accomplish this study and investigate the hypothesis, it is necessary to visualize and measure the changes induced by thermal stresses in TSV’s without physically cross sectioning the TSV. This allows the same TSV before and after annealing to be compared, thus providing a more accurate analysis of the changes induced by annealing. Silicon wafers are opaque to visible light; therefore, sub-surface imaging techniques are needed to replace visible light microscopy as an inspection technique. To accomplish the objective, different sub-surface imaging techniques are studied. A finite element model that mimics the thermal test condition and geometry of TSV is analyzed. Since finite element analysis only provides an estimate of the thermal stress distribution, the simulation results are compared with the experiment measurements.

1.3.1 Sub-surface Imaging Techniques For TSV

The first objective is to determine the best void inspection method to inspect and make an accurate measurement on the thermally induced changes in TSV. The inspection technique has to be a sub-surface imaging technique with the ability to provide 3D structural information. Furthermore, this technique should not require physically cross sectioning the TSV thus allowing the same TSVs to be compared
before and after thermal treatment. This is obviously not capable by any visible light microscope, or high resolution SEM or transmission electron microscopy (TEM).

Figure 1.4 illustrates the penetration capability of different wavelengths in bonded wafers. Infrared (IR), acoustic, and x-ray microscopes are studied and evaluated. IR and acoustic microscopes are not only used by researchers for material science, but also as in-line metrology tools for defect and overlay inspection at the interface of bonded wafers. X-ray microscope have been widely use in medical studies and failure analysis. Evaluation of these techniques is based on their capabilities which include resolution, penetration depth, material interaction, requirement of sample preparation, and destructiveness.

The second objective is to obtain a thorough set of characterization data for
comparison with the predictions from FEM simulations. Conformal and bottom up filled TSV arrays are inspected using sub-surface microscopy techniques before and after thermal treatment by annealing to different temperatures. Inspection is repeated on the same TSV arrays before and after annealing, so comparisons can be made at the identical TSV arrays. Comparison in this manner is considered more accurate since a direct visualization and understanding of thermal stress effect on TSV can be observed.

1.3.2 Finite Element Analysis

To further the hypothesis investigation, a finite element modeling of hydrostatic stress and accompanying gradient in the TSV is analyzed as the third objective. This stress analysis is based on the large difference in thermal expansion coefficients between metals and dielectrics or semiconductors. Experiment evidence from literature reveals that thermal stress induced void formation can affect interconnects reliability, and accelerates electromigration from an earlier void nucleation time. The complications of early onset void formation could seriously degrade with electromigration, causing larger voids and open circuits [6]. Some studies found that Cu-based films shrunk relative to the Si substrate, resulting in an alteration of the film footprint on the substrate after going through thermal treatment [7]. This suggests the thermal expansion coefficients could alter the copper-filled TSV as well as the pre-existing defects in TSV. Void growth as seen in Figure 1.3 could causes degrade with electromigration. Therefore, an understanding of the void growth phenomenon in TSV is necessary to assess which filling method is optimal for producing reliable 3DICs.

A finite element analysis on vacancies diffusivity under the presence of hydrostatic stress and hydrostatic stress gradient is needed. Therefore, the simulated hydrostatic stress and hydrostatic stress gradient in TSV is used as the driving force in diffusion models to analyze high vacancy concentration location in TSV. To complete the investigation, experiment measurements and finite element analyses are compared.
1.4 Outline of Subsequent Chapters

In this chapter, an introduction of the TSV approach to 3D Interconnect is presented. As the cross sectional SEM images of TSVs illustrate, the conformal filling process used to fabricate the TSVs often results in voids and seems at the center of the TSV. One objective is to investigate the hypothesized mechanism for void growth. If the proposed mechanism is correct, this information can contribute to the selection of improved fabrication processes. X-ray microscopy is the best sub-surface imaging technique for inspecting the voids in TSVs. Before further evaluation of this technique, the principles of x-ray microscopy are explained in chapter 2. Understanding the principles of this technique is important because it provides the necessary background for sample preparation and data analysis. Afterwards, an evaluation of the strengths of x-ray microscopy for TSV characterization can be made. In chapter 3, the x-ray microscopy resolution, penetration depth, material interaction, requirements for sample preparation, and destructiveness are evaluated using TSV samples. Internal and external inspection of TSV arrays is also presented. In addition, the capability of x-ray microscopy for resolving the liner is shown. In chapter 4, measurements from experimental and finite element analysis results are discussed. After that, both experimental and finite element analysis results are compared. Other sub-surface imaging techniques, IR and acoustic microscopies of TSV are studied in chapter 5. The principles of how these sub-surface imaging techniques are explained in detail. Attempts to detect TSVs and defects are made and presented in this chapter. Finally, a summary of each objective and conclusions of this research are drawn. Finally, suggestions for future work are offered.
CHAPTER 2
X-ray Microscopy

2.1 X-ray

X-rays (or X-radiation) are electromagnetic radiation that appears between Ultraviolet and Gamma rays as shown in the electromagnetic spectrum above (Fig 2.1). X-rays have a wavelength in the range of $10^{-2}$nm to 1nm. The X-ray was first systematically studied by a German physicist, W. C. Rontgen. While working on a cathode ray tube, Rontgen shielded the tube with heavy black paper and passed a high electric voltage through the tube, resulting in a green colored light displayed on a screen sitting a few feet away from the tube. He then found that the new ray would pass through some solid objects and named this new ray an X-ray where “X” signifies an unknown type of radiation. Figure 2.2 was one of his first experiments in 1895 where his wife’s hand and her ring was captured [8].
The X-ray is categorized to two categories: soft and hard x-ray’s due to their penetrating abilities. Soft x-ray’s have a range from about 0.12 to 12keV (10 to 0.10nm wavelength), while “hard” X-ray’s range from 12 to 120keV (0.10 to 0.01nm wavelength) [9]. The idea of x-ray computed tomography (CT) was patented in 1972 by Godfrey Hounsfield [10]. That same year, the first X-ray computed tomography scanner for medical use was debuted [11]. Ultimately, for their contributions, Hounsfield and Cormack shared the Nobel Prize in Medicine in 1979. Since then, much interest and effort from researchers have made a significant improvement to CT scanners. Devices now allow spatial resolution down to hundreds of micrometers, which is appropriate for medical purposes only. For materials science applications, there are many commercially available X-ray CT microscopes that have been manufactured. The resolution of these microscopes has increased to \(~1\mu\)m or less, which is greater than that of standard medical CT machines. Shown in Table 2.1 is a comparison of commercially available x-ray CT microscopes by different manufacturers [12–17]. Due to competitive advantage in the market place, certain details about the tool are non-disclosable.
Table 2.1: Commercially available XCT systems from different manufacturers.

(1) Maximum quoted resolution, may not be available for maximum sample size

(2) ABS: absorption contrast, PC: phase contrast

(3) Field of view depends on resolution mode

(4) Energy selectable, photon energies not provided

(5) SEM add-on, parameters depend on SEM used

—- indicates no information located

In this study, three X-ray microscopes that operate at different x-ray energy are evaluated (Table 2.2). These x-ray microscopes are: micro x-ray computed tomography (MicroXCT), nano x-ray computed tomography (NanoXCT), and synchrotron transmission x-ray microscope (synchrotron TXM). In this chapter, these x-ray microscopes principle which includes the x-ray attenuation, resolution, tomography technique, x-ray source and x-ray beamline is discussed.
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<th>X-ray Microscope</th>
<th>Resolution Based On</th>
<th>Fresnel Zone Plate</th>
<th>X-ray Source</th>
<th>X-ray Energy</th>
<th>Tomography</th>
</tr>
</thead>
<tbody>
<tr>
<td>MicroXCT</td>
<td>Projection Imaging</td>
<td>No</td>
<td>Lab-based</td>
<td>150keV</td>
<td>Yes</td>
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<tr>
<td>NanoXCT</td>
<td>X-ray Optics</td>
<td>Yes</td>
<td>Lab-based</td>
<td>8keV</td>
<td>Yes</td>
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<tr>
<td>Synchrotron TXM</td>
<td>X-ray Optics</td>
<td>Yes</td>
<td>Synchrotron</td>
<td>400eV-1.8keV</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Table 2.2: Different X-ray microscopes.
2.2 Attenuation Coefficient

The X-ray imaging microscope works by detecting the transmitted x-rays after passing through a material. X-ray transmission is determined by how far into a material the X-rays will penetrate (penetration depth), and also what materials can be distinguished from each other. Generally the higher the X-ray source energy, the greater the potential for X-rays to transmit through denser elements (heavy metal). The ability for X-rays to transmit through a material is characterized by the attenuation coefficient. When an incident x-ray photon, $I_o$, passes through a material with thickness, $x$, part of the x-ray is transmitted and part of it is attenuated in the material by scattering. The scattering of X-ray’s can happened in three different ways: 1) Photoelectron generation (Fig 2.3a), where the X-ray photon is completely absorbed by the system, and an electron with the same energy is ejected (such that momentum is conserved), 2) Elastic scattering (Fig 2.3b), also known as Rayleigh or Thomson scattering, is where the x-ray photon is deflected, but the energy is unaffected, and 3) Inelastic scattering (Fig 2.3c), also called Compton scattering, results when some of the X-ray energy is partially absorbed by the system, ejecting a photoelectron and deflecting the incident X-ray photon [18].
Figure 2.3: Photoelectron generation (a), Elastically scattering (b), and Inelastic scattering (c).
Similar to optical microscopy, the image contrast in x-ray microscopy comes from variations in absorption of X-ray’s. In absorption contrast mode, the contrast comes from the variation in optical density, $D$ [19]. Assuming x-rays are traveling straight through the material along the $z$-axis, the optical density can be expressed by:

$$D(x, y) = -\ln(T(x, y))$$
$$= - \int \mu(x, y, z) \, dz$$  \hspace{1cm} (2.1)

Where:
- $T(x, y) = \text{Transmission}$
- $\mu(x, y, z) = \text{Absorption coefficient}$

From the atomic point of view, the absorption coefficient $\mu(x, y, z)$ is related to atomic density $N_k(x, y, z)$ and absorption cross-section, $\mu_k^a$.

$$\mu(x, y, z) = \sum_k N_k(x, y, z) \mu_k^a dz$$  \hspace{1cm} (2.2)

Absorption cross-section is determined by the classical electron radius ($r_o$), x-ray wavelength ($\lambda$), and the imaginary part of the atomic scattering factor ($f = f_1 + if_2$) where $f$ measures scattering amplitude of a wave by an isolated atom.

$$\mu_k^a = 2r_o \lambda f_2$$  \hspace{1cm} (2.3)

However, in practice, each of the parameters in equation 2.2 will vary with position within the sample, and the attenuation may not be the product of only the
atomic density and cross section. Once the beam exits a sample, it has been attenuated by some amount, and the exiting beam follows the generalized Beer-Lambert Law, given by equation 2.4:

\[ I = I_o e^{-\mu x} \]  \hspace{1cm} (2.4)

where the initial x-ray intensity, \( I_o \), passes through a material with thickness, \( x \), with an attenuation coefficient, \( \mu \) [18, 20]. The attenuation coefficient is dependent upon the mass density and the chemical composition of a material. Materials with a small attenuation coefficient mean the material is relatively transparent to the x-ray, while large attenuation coefficients mean the x-ray will attenuate fast in the material.
2.3 Tomography

Computed Tomography (CT) is the reconstruction of a series of projections on the object being studied. The tomogram provides 3D structural information which enables further analysis such as virtual cross sectioning, pore and phase analysis, and 3D visualizations. During imaging, the object is rotated by 180° or 360° in certain angle steps. The collected projections are then reconstructed by tomography methods. One of the well-known algorithms, Filtered Back Projection (FBP), is a widely used technique for converting a series of attenuation contrast data into a projection. By a series of transformations on the measured data, a function that represents the scanned slice \( f(x, y) \) or the FBP reconstruction is [18]:

\[
f(x, y) = B[F^{-1}(\text{abs}) * R(f)]
\]

where \( \ast \) is the convolution operator and \( R[g(x, y)] \) is the Radon transform which gives a projection of \( g(x, y) \) taken from an angle \( \theta \) (Fig. 2.4) [18]. It is defined by equation (2.6) where \( B[g(x, y)] \) is the back projection transform, given by equation (2.7):

\[
R[g(x, y)] = \int_{-\infty}^{\infty} f(x, y) \, du
\]

\[
B[g(x, y)] = \int_{0}^{\pi} g(x \cos \theta + y \sin \theta, \theta) \, d\theta
\]
Figure 2.4: \(f(x, y)\) and its \(\theta\) projection.

\[ F^{-1}(\text{abs}) \] represents a real-valued function (of a real variable) whose Fourier transform is the absolute value function. Since there is no such function, this is in practice approximated either numerically or with a function whose behavior is similar enough. In order for this algorithm to be computer-friendly, all functions have to be converted into discrete forms, i.e. changing all integrals to sums over a finite number of projections. Besides FBP, there are algebraic reconstruction methods which start off by treating the slice’s function as a digital image, broken up into a finite number of (square) pixels. Each pixel represents a basis function whose value is ‘1’ inside the pixel and ‘0’ in every other pixel in the image. Algorithms using this reconstruction technique determine which of those pixels is active in order to display the image data. One of the appealing aspects of this method is that most of the steps perform calculations on sparse matrices, which can result in very fast reconstructions on modern computing systems.
2.4 Resolution

Most of the commercial available lab-based x-ray computed tomography microscopies are based on the principle of point projection where the incident x-ray source is transmitted through the sample and onto a detector, while some of the microscopies are based on x-ray optical system. The resolution of these x-ray microscopies is mainly depend on three approaches to increasing imaging resolution are (1) reduction in the x-ray spot size, (2) use of higher resolution detectors or (3) employment of x-ray optical elements. Projection imaging without the use of lenses has the advantage of simplicity (e.g. MicroXCT), while x-ray microscopy using optical system (e.g. NanoXCT and Synchrotron TXM) increases limitation of sample size. The resolution of x-ray microscopy based on projection imaging and x-ray optical system is discussed in this section.

2.4.1 Micro-scale Resolution Based on Projection Imaging

Illustrated in Figure 2.5 [21] is the geometric configuration of the projection type x-ray microscope [12]. The achievable resolution is driven by a function of both x-ray source spot size and detector resolution. As the distance \(a\) between the source
(S) and object gets smaller, the magnification of the image increases, but the blur in the detector plane, \( r_s \), increases as well. This blurring, also known as geometric un-sharpness, can be explained in equation 2.8:

\[
  r_s = \frac{b}{a} \quad (2.8)
\]

Where \( b \) is the distance between object and detector. The geometric magnification, \( M \) is given by:

\[
  M = \frac{a + b}{a} \quad (2.9)
\]

With equation (2.8), (2.9), and the detector resolution, we can then get an approximated achieved resolution, \( r_{\text{total}} \), as shown in equation (2.10).

\[
  r_{\text{total}} = \frac{\sqrt{r_{D}^2 + r_{S}^2}}{M} \quad (2.10)
\]

Where : \( r_D = \) Detector resolution

The \( r_D \) is typically about 120\( \mu \)m for most conventional x-ray projection-based systems. Based on the equations, it is clear that increasing the magnification onto the detector by reducing the distance, \( a \) will increase the blurring at the same time. To reduce the blurring, the spot size of the x-ray source has to be very small. However, small spot size sources severely limit the sample size that can be imaged for tomography. To maximize the resolution without increasing the blurring, a high res-
olution detector is applied. Now the system resolution, \( r_{\text{total}} \), in equation (2.10) is no more limited by the source spot size [22]. X-radia MicroXCT system applies a high resolution detector (2048 x 2048 pixel CCD detector) which possesses 0.65 \( \mu \text{m} \) resolution [23]. With this detector, the system able to maintain a larger spot size source so extended size of samples can be imaged. This also allows 100-150mm working distance at \( a \) and \( b \). The sub-micron resolution detector combined with a micro-focus x-ray source allows the MicroXCT system to achieve 1 \( \mu \text{m} \) resolution. Graph shown in Figure 2.6 is the effect of the combination of detector and x-ray spot size on system resolution [22].

![Graph showing system resolution as a function of geometric magnification with different source spot size and detector resolution.](image)

Figure 2.6: System resolution as a function of geometric magnification with different source spot size and detector resolution.
2.4.2 Nano-scale Resolution Based on X-ray Optics

The ability to achieve nanoscale resolution with transmission x-rays laboratory source and synchrotron source through conventional x-ray point projection is not very practical. X-ray’s with a refractive index that are very close to one make them weakly refracted by optical lenses, and thus a different technique is required to focus and achieve a high resolution image. To accomplish this task, an improved X-ray optical system is used. The geometric configuration of x-ray microscopy using optical system is shown in Figure 2.7 [21]. A zone plate (or Fresnel zone plate) is implemented which uses diffraction instead of refraction to focus the x-rays. Thus, the propagated rays not altered at the boundary between media of different densities, but altered by ”bend around corners” of the zone ring. By far the most common imaging lenses in industrial XCT systems and synchrotron TXM are micro-Fresnel zone plates (μFZP), so named because their overall dimensions typically do not exceed 1mm (millimeter) [24]. The μFZP used in NanoXCT and Synchrotron TXM is shown in Figure 2.8 [22].
Figure 2.8: Schematic of a zone plate showing the focal distance, \( f \) and the radially decreasing spacing, \( dR_n \).

The zone plate consists a set of symmetric circular rings which alternate between opaque and transparent. The rings in the zone plate decrease in width as they increase in radius toward the outermost zone ring which has the smallest width. The radii of the zone plate edges \( (r_n) \) are given by:

\[
  f^2 + r_n^2 = \left( f + \frac{n \lambda}{2} \right)^2
\]  

(2.11)

Where:

- \( n \) = Zone number
- \( f \) = First order focal length
- \( \lambda \) = Wavelength
After expansion, the relationship can be expressed by:

\[ r_n^2 = n\lambda f + \left(\frac{n^2\lambda^2}{4}\right) \tag{2.12} \]

When using a monochromic light source, the Fresnel lens follows the thin lens equation:

\[ \frac{1}{p} + \frac{1}{q} = \frac{1}{f} \tag{2.13} \]

Where: 
- \( p \) = Object distance
- \( q \) = Image distance

The magnification, \( M \) is:

\[ M = \frac{p}{q} \tag{2.14} \]

Figure 2.9 is a Fresnel zone plate lens with plane wave illumination showing first order diffraction. Sequential zones of radius are specified so that the incremental path to the focal point is \( n\lambda/2 \). Lens characteristics such as focal length \( f \), diameter \( D \), and numerical aperture \( NA \) are described in terms of \( \lambda \), \( N \) (number of zones), and \( \Delta r \), the outer zone width [25]. Light passing through will bend around (diffract) the opaque ring and the diffracted light constructively interferes at a desired focus point. Generally, zone plate optics are usable up to energies of about 10keV, but can be extended to \( \sim50 \text{keV} \) by stacking several zone plates on top of one another [26]. This helps improve resolution in the hard X-ray ranges which require thicker plates, where the aspect ratios of the outer radii get very large, very quickly [27, 28]. The resolution of the image is approximately determined by the outermost zone width.
and is expressed in equation 2.15:

\[
\sigma = 1.22R_n
\]  

(2.15)

Where:  
\( \sigma \) = Resolution  
\( R_n \) = outermost zone width
The resolution of the image is no more wavelength dependent, but is limited by how small the zone widths can be made [12, 29, 30]. For a zone plate with outermost zone width of 35nm, the theoretical Rayleigh resolution obtained is $\sim 42.7\text{nm}$. However, the resolution is practically found to be $\sim 50\text{nm}$ [31, 32].

Like most of the synchrotron-based x-ray microscopes, the source of synchrotron TXM is below 2keV [33]. This low x-ray energy limits the penetration and depth of focus (DOF). The NanoXCT uses x-ray energies at 8keV to provide greater penetration and DOF than Synchrotron TXM. The DOF is a function of x-ray wavelength and the numerical aperture ($NA$) of focusing optics, which is described in equation (2.16).

$$DOF = \pm \frac{\lambda}{2(NA)^2} = \frac{2\delta r^2}{\lambda}$$

The determining factor of the x-ray microscope resolution comes from the fabrication capability of the zone plates. Currently the outer zone width of 15nm has been achieved for soft x-ray zone plates [34]. For hard x-rays, the zone plate lenses are more difficult to fabricate because of the required high aspect ratio (>10) zone thickness necessary to attenuate high energy x-rays. Typically, the higher the aspect ratio, the higher the diffraction efficiency. The NanoXCT system has stacked zone plates, with each outermost zone having a width of 30nm and zone height of 450nm. Figure 2.10 shows SEM images of an Au (gold) Fresnel zone plate used in hard X-ray microscopy. The outermost zone is about 50nm wide and the zone height is about 450nm. Two zone plates can be bonded together to increase efficiency [31].
Figure 2.10: An Au Fresnel zone plate used in hard x-ray microscopy.
2.5 X-ray generation and Beamline

2.5.1 Laboratory X-ray Microscope: NanoXCT and MicroXCT

Laboratory X-ray sources are generated in an x-ray tube as shown in figure 2.11. High velocity free electrons are scattered by a heated cathode (filament), which is heated with a very high voltage. A strong electrical field formed between cathode and anode causing ionization of gas molecules. Due to the bombardment of positive ions, the cathode emits “cathode rays” (electrons) which are accelerated toward the anode (metal target). When electrons hit the heavy metal, some of them will approach the nucleus of the metal atoms. The interaction between these electrons and the nucleus cause electrons to lose energy by radiating X-rays with well defined energy [23].

Figure 2.12 is the beamline of the Xradia MicroXCT which uses a microfocusing x-ray source in the hard x-ray range up to 150keV. This x-ray source penetrates through the sample mounted on a high-resolution stage which allows translation in x–y–z directions. High-resolution imaging is achieved using a 2048 × 2048 pixel CCD detector paired with lenses of varying magnification coupled to scintillating crystals, producing an effective detector pixel size ¡1µm [23]. Scintillators are materials that emit low-energy (usually in the visible range) photons when struck by a high-energy charged particle. The incident x-ray is absorbed, creating a hole, and then a multi-stage recombination process results in a scintillation photon being emitted. This process is used to convert the transmitted x-ray to a visible light image before being captured by the CCD detector [35].
Figure 2.11: X-ray Tube diagram of MicroXCT and NanoXCT.

Figure 2.12: MicroXCT beamline.
The beamline of NanoXCT system is illustrated in Figure 2.13. Incident x-rays at 8keV are initially focused by a reflective capillary optic or condenser zone plate, designed to condense a selection of the rays to a small spot [36] onto the sample. The lens is often made with a blocking ring in the center [24] which illuminates the sample with a partially coherent hollow cone beam. After transmitted through the sample, the rays begin to diverge. A diffractive focusing optic (i.e. Fresnel zone plate) is located about 20mm past the sample position capturing the diverging rays and focusing the transmitted image of the sample several hundred millimeters downstream [37]. The x-rays are then converted to a visible light image by a scintillating crystal, and finally captured by a high-resolution CCD detector [23].
2.5.2 Synchrotron X-ray Microscope: BESSY II Synchrotron Transmission X-ray Microscope (TXM)

Figure 2.14: Diagram of Synchrotron circular track.
Shown in Figure 2.14 is the simplified diagram of circular track and beamline branch of BESSY II Synchrotron TXM (Transmission X-ray Microscope) at Berlin, Germany. A synchrotron is a type of cyclic particle accelerator. In the circular track, electric field is applied to accelerate the particles (close to speed of light) and magnetic field (bent-magnet radiation) is used to turn these accelerated particles so they circulate around the circular track. These particles then emit a very brilliant and highly focused light. X-rays a continuous spectrum of energies allowing researchers to choose the desired wavelength used to study their samples. The BESSY II synchrotron uses undular radiation in their synchrotron storage ring. The Undulator consists of a periodic structure of dipole magnets. Electrons that traverse the periodic magnets are forced to oscillate resulting in radiated energy. This radiation is highly intense and concentrated in narrow energy bands [38].

Figure 2.15: Synchrotron TXM beamline.

On the right of Figure 2.15 is the BESSY II Synchrotron TXM beamline. The transmission x-ray microscope (TXM) that is able to produce broadband x-rays up to \(\sim 1.8\text{keV}\) as seen in one of the branches or segments shown in Figure 2.14. The M1 as a plane mirror in the first switching mirror unit is used to direct the beam into the X-ray microscope beamlines. In the second switching mirror unit there are two plane mirrors: M2.1 and M2.3 which direct the beam into the transmission X-ray microscope. The condenser-monochromator of the TXM consists of an off-axis transmission zone plate (OTZ) and three plane mirrors. The beam is then focused on the sample for inspection [33].
Visible light optical microscopy and electron microscopy are most commonly used for direct imaging of structures. However to non-destructively image optically opaque samples or those that are too thick for electrons to penetrate, these two techniques are not applicable. X-ray microscopy complements the two techniques in that it has better resolution compared to visible light due to its short wavelength, and better penetrating power than electrons since they do not interact with matter as strongly as a charged particle [31]. Unlike electron microscopy, metal coating and vacuum compatibility are not required. This is especially advantageous for soft or biological samples because they can be imaged at their natural state. X-ray microscopy is also ideal for defect analysis on irreplaceable samples which cannot be modified [39].

A comparison of methods that can be used to inspect the voids and liner in copper filled TSVs is made and listed in table 3.1. It describes the capabilities of x-ray microscopy and its potential as a void metrology technique in copper filled TSVs. All the methods shown in the Table 3.1, x-ray microscopy, SEM and TEM have the capability to inspect voids in the copper filled TSV. High energy and short wavelength range of x-ray’s in the electromagnetic spectrum make it ideal for penetrating all materials easily, though different x-ray energies have different penetration depths on various materials.
The laboratory based NanoXCT and synchrotron TXM has a high resolution at \( \sim40 - 50\text{nm} \) and greater probing depth than SEM and TEM. Because the x-ray has high penetration capabilities, no physical cross sectioning into the TSVs is needed, but minimum sample preparation is required as explained in a later section. Although SEM and TEM have better resolution than x-ray microscopy, the probing depth is very low, thus physical cross sectioning into the TSV is required. For TEM, the sample preparation is extensive and requires the sample to be thinned to at least \( \sim100\text{nm} \) for the possible transmission. X-ray microscopes are not only able to take two dimensional (2D) images, but also able to reconstruct three dimensional (3D) tomography’s made from a series of images taken from different angles of the sample. This allows 3D structural information of the sample to be obtained. SEM alone doesn’t have a 3D tomography capability, and requires a FIB to keep ionizing and imaging the TSV layer by layer as the 3D tomography progresses. This process is not only time consuming but also damages the TSVs. TEM has 3D tomography capabilities, but the volume from the tomography is so small that only \( \sim0.02\% \) of TSV 3D structural information can be produced from a tomography. In addition to soft x-ray microscopy, a laboratory based hard x-ray MicroXCT is capable of detecting voids in

<table>
<thead>
<tr>
<th>Techniques</th>
<th>Spatial Resolution</th>
<th>Probing Depth</th>
<th>Sample Preparation</th>
<th>3D Imaging</th>
</tr>
</thead>
<tbody>
<tr>
<td>NanoXCT</td>
<td>( \sim50\text{nm} )</td>
<td>( \sim60\mu\text{m} )</td>
<td>Minimal (thickness of sample ( \sim60\mu\text{m} ))</td>
<td>Yes (60\mu\text{m})(^3)</td>
</tr>
<tr>
<td>MicroXCT</td>
<td>( \sim1\mu\text{m} )</td>
<td>( &gt;775\mu\text{m} )</td>
<td>No</td>
<td>Yes (( &gt;775\mu\text{m} ))(^3)</td>
</tr>
<tr>
<td>Synchrotron TXM</td>
<td>( \sim40\text{nm} )</td>
<td>( &lt;3\mu\text{m} )</td>
<td>Massive (lamella preparation by FIB)</td>
<td>Yes (( &lt;3\mu\text{m} ))(^3)</td>
</tr>
<tr>
<td>SEM</td>
<td>( \sim1-10\text{nm} )</td>
<td>( &lt;10\text{nm} )</td>
<td>Medium (cross sectioning)</td>
<td>No, needs FIB</td>
</tr>
<tr>
<td>TEM</td>
<td>( \sim0.1\text{nm} )</td>
<td>( &lt;100\text{nm} )</td>
<td>Extensive (thickness of sample ( &lt;100\text{nm} ))</td>
<td>Yes, but very small (( \text{nm}^3 ))</td>
</tr>
</tbody>
</table>

Table 3.1: Methods comparison.
TSVs without any requirement for sample preparation, but the resolution is limited at $\sim 1\mu m$. The capability of x-ray microscopy techniques to inspect voids in the TSV without physically cross sectioning the TSV is used to inspect the changes of voids in TSVs under different annealing condition.

X-rays can pass through virtually anything, but the transmission of materials is energy dependent. The x-ray energy used in NanoXCT is monochromatic at 8keV, while Synchrotron TXM uses broadband energy from $\sim 400eV - 1.8keV$. These x-ray energies transmit differently in materials. Shown in Figure 3.1 is the energy dependent transmission graph of materials used to fabricate TSVs. This transmission graphs show the potential for NanoXCT and synchrotron TXM to measure x-ray transmission or absorption on TSV. The thickness of the materials used to plot the graph is $\sim 3\mu m$. Based on the transmission, TSV image contrast generated by NanoXCT and synchrotron TXM can be estimated. The barrier layer ($TaN/Ta$) with atomic density, 16.7$g/cm^3$ has the highest absorption using 8keV x-ray energy in NanoXCT. This is followed by the second highest absorption material, copper ($Cu$), with atomic density at 8.96$g/cm^3$. Silicon ($Si$) with density 2.33$g/cm^3$ reaches 90% of transmission, while Tetraethyl Orthosilicate ($TEOS$) has the highest transmission with 0.94$g/cm^3$ atomic density. At 400eV - 1.8keV x-ray energy, $TEOS$ has the highest transmission follow by $Si$, but both barrier and $Cu$ absorb all x-ray energies. When x-ray micrograph contrast is generated based on absorption, the highest transmission material will be shown with brightest contrast and vice versa.
Figure 3.2 is the hard x-ray range energy dependent transmission graph of TSV materials. When the x-ray energy operated at hard x-ray range, low atomic density material appears to have \( \sim 100\% \) transmission. As shown in graph, Si and TEOS with low atomic density appear to have the same transmission after \( \sim 16\text{keV} \). This implies that x-ray microscopy using x-ray energy more than 16keV is unable to distinguish the absorption of these two materials. At 30keV, Cu and TaN/Ta absorb energy differently, but these absorptions are expected to be similar as the energy increase to 150keV. This indicates the Si and TEOS occur in same contrast in MicroXCT, as well as the contrast of Cu and TaN/Ta.
Figure 3.2: Energy dependent transmission from 10keV-30keV.
3.1 Sample Description

To evaluate the capabilities of the x-ray microscopies on void inspection, TSV arrays with known defect as shown in Figure 3.3 is used.

![Figure 3.3: 4\(\mu\)m (a-b) and 5\(\mu\)m (c) TSV arrays with known defects.](image)

TSVs shown in Figure 3.3 are FIB cross sections of \(\sim 4 \times 26\mu\)m (a-b) and \(5 \times 27\mu\)m (c) TSV arrays with known defects. The dimension of the TSV arrays is etched by reactive ion etching (RIE). After that, liner and barrier are deposited with plasma-enhanced chemical vapor deposition (PECVD) and physical vapor deposition (PVD) respectively. Finally, the remaining TSV volume is electrochemically deposited with copper. The 4\(\mu\)m TSVs is electroplated in a way that incomplete filling is created at the bottom. These TSVs are filled from top to bottom at a depth of 17\(\mu\)m leaving a
5x9\(\mu\text{m}\) void unfilled (Fig 3.3a-b), while the 5\(\mu\text{m}\) TSV is plated with 1x9\(\mu\text{m}\) void at the center and close to the bottom of TSV.
3.2 Sample Preparation

Before sample preparation, a calculation of the materials transmission should be made using Beer’s law equation. This calculation is important to understand how the image contrast quality changes with the thickness of sample. To do this, the attenuation coefficient of materials that interact with x-ray’s at 8KeV is needed. Copper-filled TSVs are denser and have a higher attenuation than the surrounding silicon wafer. Table 3.2 indicates the 8KeV x-ray transmission of silicon and copper at different thicknesses.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm³)</th>
<th>Absorption Length (µm)</th>
<th>Transmission(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>50µm</td>
</tr>
<tr>
<td>Silicon</td>
<td>2.33</td>
<td>70.82</td>
<td>49.4</td>
</tr>
<tr>
<td>Copper</td>
<td>8.96</td>
<td>22.89</td>
<td>10.6</td>
</tr>
</tbody>
</table>

Table 3.2: X-ray transmission of different thickness of materials.

The thickness is estimated based on the tomography rotating angle setup. During the tomography scan, the sample is rotated in a range of angles while images are continuously taken. The thickness of the sample that the x-rays must transmit through is constantly changing due to the different angles of rotation. At 50µm, the transmission of Si is ~49% and ~10% for Cu. This shows that the absorption contrast between the two is substantial. Therefore the silicon wafer is polished to a thickness of ~50µm, so the whole TSV array remains in a single piece of wafer. Figure 3.4a-b illustrates the thickness of materials that x-rays need to transmit through at different angles.

In Figure 3.4a-b is a TSV 5µm in diameter and 30µm deep embedded in a piece of 50µm thick wafer. If the tomography is set to take images from 0° to 180°, the 0° angle (a) will be the minimum path length (5µm) for x-rays to pass through in...
copper. At $90^\circ$ (b), the path length for x-rays to transmit in copper is at a maximum of 30$\mu$m.

![Figure 3.4: Minimum (a) and maximum (b) of x-ray path in copper.](image)

### 3.2.1 NanoXCT

As discussed previously, the intensity of transmitted x-ray is material dependent; the denser a material, the less penetration. Based on transmission calculations of silicon and copper at various thicknesses, TSVs in a full thickness wafer are required to be thinned from 775$\mu$m to about 50$\mu$m by polishing. The flow of sample preparation is shown in Figure 3.5a-d.

First, a TSV array from a wafer is cleaved to 5 x 5mm (a). The face of this piece of wafer is attached to a polishing holder (b). Using a die level polishing method, the back of the wafer is polished manually on diamond sand paper from 775$\mu$m to $\sim$100$\mu$m thick (c). Finally, polishing of the wafer continues to $\sim$50$\mu$m thickness...
with a dimple grinder using SiC diamond paste (particle size: 1µm) applied to the dimpling wheel.

Figure 3.5: NanoXCT sample preparation requirement.

Shown in Figure 3.6 is the 3D (a) and side view (b) of the sample after sample preparation. The entire TSV array remains in the ~50µm thick wafer after non-destructively preparing each array.

Figure 3.6: TSV arrays remain in the ~50µm thick wafer after sample preparation.
3.2.2 Synchrotron TXM

Similar to the NanoXCT, calculations of transmission using the synchrotron TXM are necessary in order to know the thickness at which x-ray can transmit through a material before being attenuated. The broadband X-rays from a Synchrotron TXM range from 400eV-1.8keV. The attenuation length of copper and silicon is very small in this range of x-ray energies where the ability for transmission is limited to \(<3\mu m\) for copper. Therefore only TSVs below \(3\mu m\) in diameter can be scanned by the TXM which operates at energies between 400eV and 1.8keV. Therefore, the sample has to be prepared in lamella form using a focused ion beam (FIB). Figure 3.7 shows the sample dimensions after preparation by FIB cut. The dimension of the lamella is \(~27x3x36\mu m\) and contain two TSVs (\(~2\mu m\) diameter) embedded in the piece of lamella.

![Figure 3.7](image)

**Figure 3.7:** Dimension of lamella after sample preparation.
3.3 Void Inspection by X-ray Microscopy

![Figure 3.8: Incomplete filling at the bottom of 4μm TSV.](image)

Shown in Figure 3.8 and Figure 3.9 are the x-ray micrographs generated by NanoXCT from two TSV arrays that are embedded in a piece of ~50μm wafer. These TSV arrays with dimension ~4x26μm and ~5x27μm respectively are fabricated with known defects as described in previous section. This piece of wafer was attached to a rotating sample stage. The 8keV x-ray was condensed by the condenser before being focused on the sample. The x-ray was continually emitted at the sample for 20 hours. During the 20 hours, the sample was rotated from -70° to +70° with 0.44° angle steps. A total of 321 images were collected at an average ~3.7 minutes exposure time of each projection. After reconstruction of the series of images, 3D structural information is generated. This allows multi-directional (XY, YZ and XZ planes) visualization of TSVs. Figure 3.8 and Figure 3.9 are the XY plane at the center of TSV arrays. The defects observed in these TSVs were incompletely filled at the bottom of the 4μm TSV array (Fig 3.8) and large voids at the bottom of 5μm TSV array (Fig 3.9) which is agree with the FIB images shown in Figure 3.3a-c.
Since imaging the TSV with NanoXCT does not require cross sectioning on TSV, imaging back the same TSV arrays after annealing is possible. This is essential to understand the effect of thermal stress on the copper-filled TSV. The TSV arrays shown in Figure 3.8 and Figure 3.9 were annealed to 300°C for 24 hours under pure nitrogen environment. After 24 hours, the sample was relaxed to room temperature. The same imaging setup was repeated on the same TSV arrays after annealing, so a new series of 321 images were collected and reconstructed. Comparing the x-ray images of identical TSV arrays at before and after annealing, thermal stress has significant impact on the pre-existing defects. Demonstrated in Figure 3.10a-b are pair of TSVs from the 4μm and 5μm TSV arrays respectively. The shape of the pre-existing void in 5μm TSV is changed and a small extrusion from the top is indicated by the reference line (a), while larger extrusion is observed on the 4μm TSV (b). Delamination is observed at the side wall around both TSVs when inspecting the internal structure of the TSVs. The external view of the TSV arrays can be observed by the 3D image shown in Figure 3.11.
Figure 3.10: Comparison of same $4\mu\text{m}$ (b) and $5\mu\text{m}$ (a) TSVs at before and after annealing.

Figure 3.11: 3D image shows the external view of TSV arrays.
As explained previously, synchrotron TXM has limited transmission capability due to an operating x-ray energy <2keV. Therefore, FIB is needed to prepare the sample into a lamella for synchrotron TXM imaging as described in Figure 3.7. First, 1keV x-ray energy is focused at the lamella. Two TSVs with different filling profile can be observed in Figure 3.12b. The contrast image is measured based on the transmission of material. Materials with large atomic density will be shown in darker contrast. The TSV on the left of Figure 3.12b was filled to the center and unfilled the rest of the volume. The TSV at the right side sealed up at the top and the barrier/Cu seed layer can be clearly observed. Higher x-ray energy, 1.8keV was focus at the filled area of the left TSV. More transmission reveals that a void is embedded in copper as shown in Figure 3.12a. Both the Synchrotron TXM and NanoXCT have nano-scale resolution based on x-ray optics. Comparing the results measured from 400eV-1.8keV and 8keV, Synchrotron TXM resolves the detail in TSVs better than the NanoXCT, but its transmission is limited at below 3µm thick lamella. NanoXCT
with larger transmission results in a larger field of view at nano-scale resolution. This makes NanoXCT a powerful x-ray microscope able to inspect the thermally induced changes (e.g. void growth, extrusion, and delamination) of the same TSV before and after annealing without physical cross section on TSV.
The micro-scale resolution of MicroXCT is based on projection imaging without x-ray optics. With operating x-ray energies up to 150keV, and projection imaging methods, the x-ray microscope allows for larger working distance and greater transmission capability. Larger pieces of wafer can be handled by the working distance and no polishing is needed. A piece of wafer with 5\( \mu \)m TSV arrays underwent \( \sim \)10 hours of tomography by MicroXCT. The virtual cross section of the TSV arrays shown in Figure 3.14a-b are corresponding to the vertical red line and horizontal green line respectively in Figure 3.13. Incomplete filling at the bottom of the TSV arrays is observed in a more detailed view shown in Figure 3.15 and Figure 3.16. The advantage of using MicroXCT for inspecting TSV arrays without thinning the wafer is great, but at a resolution of \( \sim \)1\( \mu \)m it is only possible to detect the incompletely filled areas, and doesn’t have the ability to resolve it. Therefore, NanoXCT becomes the best technique to study void growth mechanisms in TSVs.

Figure 3.13: Full thickness wafer is inspected by MicroXCT.
Figure 3.14: The virtual cross section of the red line (a) and green line (b) of Fig 3.13.

Figure 3.15: Detail view of yellow box in Fig 3.14a.
Figure 3.16: Detail view of red box in Fig 3.14a.
3.4 Liner Inspection by X-ray Microscopy

![Graph showing energy dependent transmission of TEOS, Si, and Cu.](image)

Figure 3.17: Energy dependent transmission of TEOS, Si and Cu.

The energy dependent transmission of TSV materials shown in the graph of Figure 3.2 indicates TEOS has the greatest transmission and should be able to be measured with x-ray energies ranging from 400eV-1.8keV and 8keV. Therefore, the attempt of measuring the liner by synchrotron TXM and NanoXCT was performed. From 400eV to ∼510eV x-ray energy shown in the graph of Figure 3.17, transmission in Si and Cu is ∼0%, while TEOS transmission increases before reaching its absorption edge at ∼540eV. This absorption edge occurs because TEOS is a chemical compound with the formula Si(O(CH₃)₂)₄ containing oxygen (O₂). The absorption edge of oxygen K shell is at 536eV [40], where a large absorption will happen, causing very low transmission. Thus the area of interest is focused below 550eV x-ray energy.
The condition of 0% transmission of $Si$ and $Cu$ makes $TEOS$ contrast distinguishable from other materials. Imaging the liner at these x-ray energies is then based on the ability of the x-ray optics. Non-filled 1, 3, and 5$\mu$m in diameter TSV arrays with $TEOS$ liner, $TaN/Ta$ barrier, and $Cu$ seed layer are deposited on a piece of wafer. Before the wafer is milled by FIB, epoxy is injected into the non filled TSV arrays. The purpose of filling the TSVs with epoxies is to avoid particles dropping into the TSV during milling. Figure 3.18 illustrates the sample preparation for synchrotron imaging. As explained previously, the penetration depth of Synchrotron TXM is limited below $\sim3\mu$m. The area of interest has to be milled into a lamella dimension. Shown in the red box (a) of Figure 3.18 are the TSVs selected to mill by FIB. After sample preparation, side and top view of the lamella are shown in Figure 3.18b-c.
Figure 3.19: Images corresponding to 452eV (a), 510eV (b and d) and 555eV (c).

Three energies; 452eV, 510eV and 555eV are chosen to image the 1μm TSV liner in the lamella. The images corresponding to the chosen energies are shown in Figure 3.19. At 452eV (a), transmission of x-ray on Si is 9.58x10^{-3}% or ~0% and TEOS is 4.82%. This weak signal is able to be detected by the TXM, but not able to be resolved.
When x-ray energy is increased to 510eV (b), the Si remains \( \sim 0\% \) transmission but TEOS increases to \( \sim 11\% \), which is significant high enough to be resolved by TXM, and view the detail as shown in Figure 3.19 (d). As the energy continues to increase to 555eV, the transmission on Si starts to increase to 0.4\%, while transmission on TEOS decreases to 3.18\%. The decreasing absorption of X-rays on TEOS is the consequence of absorption edge at \( \sim 540\)eV. The suddenly high absorption of X-ray’s by oxygen causes a reduction of x-ray transmission through the TEOS, preventing detection using the TXM.
As x-ray energy continues to increase, more transmission through the same materials occur. At 8keV x-ray energy, the transmission of Si is 96% and TEOS is 99.6% or ∼100%. This transmission signal should be substantial enough to be detected by the high sensitivity contrast detection system of NanoXCT. TSV arrays from the same piece of wafer are cleaved into 5x5mm and polished from the back to ∼60µm. The piece of wafer is tilted at a small angle of <30°. Without tomography, a 2D transmission image shown in Figure 3.20 is generated with exposure time <10s. A tomography based on absorption projections from the same TSVs was made with total time ∼2 hours. Figure 3.21 is the cross sectional view of the center of a TSV. Because the projection is measured based on x-ray absorption, the contrast of the materials is inversed from the projection shown in Figure 3.20.

Figure 3.21: Cross section view at the center of TSV.
CHAPTER 4
Void Growth In TSVs

In this chapter, experimental and finite element analyses are carried out to establish the validity of the hypothesis. Experimental data and finite element analysis results are compared and discussed. Then, a set of conformally filled and bottom-up filled TSV are annealed to different temperature from 200°C to 300°C. These TSV arrays are measured by NanoXCT and compared.

4.1 Background of Void Growth in Conformally Filled TSV

There are three conditions needed to cause void growth phenomena in copper filled TSV: 1) Pre-existing void or void nucleation, 2) Vacancy source, and 3) Driving forces. Void nucleation is vacancy motion leading to stress induced voiding, which is controlled by the diffusional mechanisms active within a given material. Pre-existing voids can easily appear as filling method defects (e.g. conformally filled TSV) and filling parameters. The vacancy source can easily come from the copper grain boundaries in TSV, where small voids are located on the grain boundaries. These vacancies are assumed in local equilibrium along the TSV. When driving forces are introduced to the system, it produces a gradient in the vacancy chemical potential causing the vacancies to diffuse toward the void.
Figure 4.1: Void growth mechanism.

Figure 4.1 illustrates the mechanism of void growth in TSV with pre-existing voids or seam lines. After the TSV is copper-plated at room temperature, the grain size and orientation are random (Fig 4.1a). When annealed at a higher temperature, these grains grow larger (Fig 4.1b). Grain growth results in the reduction of the number of grain boundaries which are of lower density than the surrounding material. The excess grain boundary volume is transformed into a tensile stress with an attendant higher vacancy concentration. These excess vacancies easily diffuse under the influence of a driving force. Consider the case in which a pre-existing void and seam line appear in the TSV as a result of conformal plating. As this TSV is annealed to a higher temperature, stresses are induced in the TSV due to the difference in the thermal expansion coefficients of the materials. To relieve the stress, the seam line and pre-existing voids are modeled as a stress sink to relieve the stresses where they vanish at the void surface, thereby creating a stress gradient (Fig 4.1c). This stress gradient is the driving force that causes vacancies to diffuse into the pre-existing void [41]. The stress relaxation over time through the diffusion of vacancies leads to
the growth of pre-existing voids (Fig 4.1d), which is expected to abate once the stress gradient is relieved.

Figure 4.2: Active diffusion volume on TSV.
From a mass transport perspective, three major volumetric scales can be used to define the diffusion problem: 1) Interconnect Volume, 2) Diffusion volume, and 3) stress gradient regions. The first is the interconnect volume given by the product of the length, height, and width of interconnect at the damage area. Secondly, diffusion volume depends on the active diffusion mechanism present, e.g., grain boundary (GB) diffusion, interface diffusion, and atomic migration. This diffusion pathway occurs under a sufficient driving force. Lastly, the stress gradient region depends on the geometry of interconnect, the materials properties, and the local stress levels developed at the annealing temperature. Only vacancies within the active diffusion volume are able to participate in the void growth process under a driving force. Since interconnects are not present, only diffusion volume and stress gradients are considered.

Figure 4.2 shows the TSV after being annealed to 300°C. The white dash circle line indicates the approximate active diffusion volume, a sphere with the diameter, $2X_D$. However, the copper volume within the sphere is the volume of active diffusion volume. The diffusion length proportional to the square root of diffusivity, and annealing time. This diffusion length is expressed in equation 4.1.

$$X_D = \sqrt{Dt} \quad \text{(4.1)}$$

Where: $X_D = \text{Diffusion length}$  
$D = \text{Diffusivity}$  
$t = \text{Annealing time}$
The diffusivity \((D)\) shown in equation 4.2 can be calculated with the diffusion coefficient \((D_o)\), diffusion activation energy \((E_A)\), and the annealing temperature \((T)\). Note that the diffusion activation energy is diffusion mechanism dependent. Typically, the \(E_A\) for grain boundary diffusion is \(\sim 0.9\text{eV}-1.1\text{eV}\). The \(E_A\) for interface diffusion and atomic migration is \(\sim 0.74\text{eV}\) and \(\sim 2.07\text{eV}\) respectively [42].

\[
D = D_o e^{-\left(\frac{E_A}{RT}\right)}
\]  

(4.2)

The estimated active diffusion volume, \(2X_D\), on the TSV in Figure 4.2 is \(\sim 16\mu m\), where the major grain growth area is observed. With \(X_D \sim 8\mu m\), \(t=24\text{ hours}\), the diffusivity is found to be \(\sim 7.4\times10^{-10}\text{cm}^2/\text{s}\). The diffusion activation energy is found to be \(\sim 0.98\text{eV}\), which is in the range of grain boundary diffusion activation energy. The grain boundaries can be clearly seen around the void (Fig 4.2, left), which indicate that excess vacancies can easily diffuse toward the pre-existing voids. Therefore, the primary active mechanism for vacancy diffusion in TSVs with pre-existing voids should be grain boundary diffusion under the driving force of hydrostatic stress gradients.

To visualize and identify a void growth mechanism, a technique with high resolution and the ability to non-destructively penetrate through the high density copper-filled TSVs is needed [43]. In this study, we used a high transmission 8keV x-ray imaging technique to inspect TSV changes caused by thermal stresses. Three dimensional finite element modeling are used to simulate the hydrostatic stress, and the corresponding gradients based on the coefficient of thermal expansion mismatch between the silicon wafer and copper-filled TSV under different annealing temperatures. Two geometries, an ideal TSV and a TSV with pre-existing voids, were studied. The experimental data from x-ray microscopy and numerical results were analyzed and compared.
4.2 Experiment Details

![Diagram showing TSV trench with liner, barrier, and seed layer before and after CMP.](image)

Figure 4.3: TSV trench deposited with liner, barrier and seed layer (a) as seen before CMP (b) and after CMP (c).

TSV arrays 5µm in diameter and 26µm deep were etched onto 300mm silicon wafers. These TSVs underwent a reactive ion etch (RIE) to the desired dimension. A layer of ∼200nm tetraethyl orthosilicate (TEOS) was deposited as a liner on the TSV well with plasma-enhanced chemical vapor deposition (PECVD). Before the TSVs were copper-filled by electrochemical deposition (ECD) at 40°C, a 10nm copper diffusion barrier layer of Ta/TaN was deposited on the TEOS by physical vapor deposition (PVD) and a 500nm copper seed layer was deposited after the barrier layer. Afterwards, the TSVs were copper-filled with conformal method, causing the entire surface of the wafer to be left with a layer of copper overburden, which was removed by chemical mechanical planarization (CMP) after pre-annealing at 150°C for one hour [44]. A 5mm x 5mm piece of wafer with TSV arrays was cleaved from the same 300mm wafer. This piece was then polished from the backside to ∼50µm so that only the 26µm deep TSVs remained. Finally, the TSV array was inspected using an 8keV lab-based X-ray computed tomography (NanoXCT) from Xradia. Transmission calculations and experimentation indicated the high resolution of the 8keV X-ray microscope has the capability to transmit through the TSV arrays without the requirement of physical cross sectioning of the TSVs as discussed in previous studies [43, 44]. Furthermore, the NanoXCT uses a Fresnel zone plate that functions as a lens to refocus the transmitted X-rays from the sample to the detector, allowing
a 50nm resolution to be achieved [31, 32, 45]. The X-ray imaging setup for TSV arrays is discussed in chapter 3. Two different TSV array samples were scanned by NanoXCT before and after post-annealing at 225°C and 300°C, respectively, to compare the changes caused by different annealing temperatures on the same TSV array to be captured and compared.

Figure 4.4: X-ray micrograph of TSV arrays at before and after annealing.
Because X-ray tomography allows observation in all directions, 2D and 3D structural information can be analyzed in detail. The TSV image contrast generated by the 8keV X-ray microscope is brighter for copper and darker for silicon than air or voids. Shown in Figure 4.4 is the TSV arrays at before (Fig 4.4a-b) and after (Fig 4.4c-d) annealing to 225°C and 300°C respectively. By comparing the TSV after 225°C (Fig 4.4c) and 300°C (Fig 4.4d) annealing, the observations suggest the total volume of void growth in both TSV arrays is similar. However, higher annealing temperatures will induce greater driving forces and thus the total volume of void growth should increase with annealing temperature. Therefore, the ability for x-ray microscopy to generate non-destructive tomography is needed in this study. This allows TSV analysis at any plane and cross section. A more detail analysis of these TSVs is shown in Figure 4.5 and Figure 4.6 [46].

Two X-ray images from different planes of the same TSV were used to study void growth (see Fig. 4.5 and Fig 4.6). The TSV in Figure 4.5 was annealed at 225°C, whereas the TSV in Figure 4.6 was annealed at 300°C. Before annealing at a higher temperature, the pre-existing void or seam line (Fig 4.5a and Fig 4.5b) is caused by conformal plating as clearly seen at the bottom of the TSV (Fig 4.5). After annealing at 225°C for 24 hours, growth of the voids is seen in the XY (Fig 4.5c) and YZ (Fig 4.5d) planes of the same TSV. Similar results are observed in the TSV annealed at 300°C.

A comparison of the XY planes (Fig 4.5c and Fig 4.6c) of the two TSVs that annealed to 225°C and 300°C respectively indicates that the total volumes of void growth are similar. However, the YZ plane of both TSVs (Fig 4.5d and Fig 4.6d) clearly shows the volume of void growth for the 300°C annealed TSV is greater than that of the 225°C annealed TSV. This points to a need for an inspection method that can produce volumetric data to compare the volume of the voids with void growth. ImageJ with the TXM Reader plug-in was used to calculate the volume of void growth from the 3D reconstructed file, based on measuring the pixels of the dark area in the TSV. Calculations show that before post-annealing, the TSV in Figure
4.5 has a volume of $\sim 0.11 \mu m^3$ compared to $\sim 0.41 \mu m^3$ after 225°C annealing. The TSV in Figure 4.6 had a volume of $\sim 0.13 \mu m^3$ before being annealed to 300°C. After annealing, the volume in the same TSV was found to be $\sim 1.63 \mu m^3$. The total volume of void growth was found to be $\sim 0.3 \mu m^3$ and $\sim 1.5 \mu m^3$ for the TSVs annealed at 225°C and 300°C, respectively. These results suggest that the volume of void growth with a stress-free temperature at 150°C increases as the annealing temperature rises. The hydrostatic stress gradient as driving force is thus assumed to be a greater driving force at higher annealing temperatures.

Figure 4.5: 2D X-ray image at XY (a) and YZ (b) plane of TSV before annealing. X-ray image at XY (c) and YZ (d) of the same TSV after being annealed to 225°C.
Figure 4.6: 2D X-ray image at XY (a) and YZ (b) plane of TSV before annealing. X-ray image at XY (c) and YZ (d) of the same TSV after being annealed to 300°C.
4.3 Finite Element Analysis

4.3.1 Driving Force of Hydrostatic Stress and Hydrostatic Stress Gradient

The net vacancy flux along the copper-filled TSV depends on four driving forces: 1) atom concentration gradients, 2) electrical current (electromigration), 3) temperature gradients, and 4) mechanical stress gradients [47]. A few assumptions were made to simplify the finite element modeling (FEM) analysis in this study. Since no electrical current is applied to the TSV, the driving force from the electrical current can be eliminated. Furthermore, the sample is loaded into the furnace after the desired temperature is reached and is kept in the furnace at a constant anneal temperature for 24 hours. Therefore, the sample is considered as having no heat transfer during annealing so that the impact from the temperature gradient can be eliminated as well. Only the mechanical stress and concentration gradients were taken into consideration. The total atomic flux, $J_{\text{tot}}$, under these two driving forces is shown in equation (4.3) [47, 48],

$$J_{\text{tot}} = -D\nabla C + \frac{DC\Omega}{KT}\nabla\sigma_{Hs}$$  \hspace{1cm} (4.3)

Where:
- $D$ = Atom diffusivity
- $C$ = Atom concentration
- $\Omega$ = Activation volume
- $T$ = Absolute temperature
- $K$ = Boltzmann’s constant
- $\sigma_{Hs}$ = Hydrostatic stress
The highest tensile hydrostatic stress region is known to be more favorable for void nucleation, while the hydrostatic stress gradient is known to be the driving force for void growth. The site where the hydrostatic stress gradients are the most concentrated is thought to be the most probable for void growth [49–51].

Because the TSVs were conformally plated, copper is grown layer by layer from the sidewall of the TSV toward the center. In this process, a seam line or void can form in the center of the TSVs. These seam lines and voids are seen in conformal-filled TSVs and exist before any annealing or further processing is done. We therefore concentrated our study on the impact of hydrostatic stress gradients on the void growth of conformal-filled TSVs with pre-existing voids. The FEM analysis used a TSV structure with a pre-existing void to analyze the role of the hydrostatic stress gradient distribution around the void in driving the vacancies to diffuse toward the void. The hydrostatic stress is described as the average of three normal stresses. The stress and its gradient are illustrated in equations (4.4) and (4.5), respectively [52, 53].

\[
\sigma_{Hs} = \frac{\sigma_{xx} + \sigma_{yy} + \sigma_{zz}}{3} \quad (4.4)
\]

\[
\nabla \sigma_{Hs} = \left( \frac{\delta \sigma_{Hs}}{\delta x}, \frac{\delta \sigma_{Hs}}{\delta y}, \frac{\delta \sigma_{Hs}}{\delta z} \right) \quad (4.5)
\]

The 3D finite element analysis in this study was carried out using the COMSOL software package to simulate the hydrostatic stress and gradient distribution in a 5\(\mu\)m TSV structure. Since the test structure is simply an array made by repeating TSVs at every 5\(\mu\)m of silicon, a simplified model with only a single TSV in a 15x15x35\(\mu\)m volume of silicon was used as one of the unit segments. The dimensions of the TSV are 5\(\mu\)m in diameter and 25\(\mu\)m deep. Two TSV geometries were used in the FEM analysis. The first is an ideal TSV geometry with \(~100\)nm liner at the interface between the Si and Cu. In the second, an ellipse (1\(\mu\)m x 4\(\mu\)m) is added at the bottom.
of the TSV to represent a pre-existing void. The bottom of the TSV was rounded to mimic the geometry of the TSV observed in the X-ray micrograph. The geometry described above is illustrated in Figure 4.7a-c.

Figure 4.7: TSV 3D model used in the finite element analysis.
Figure 4.8: The boundary condition: Symmetry (a), prescribed displacement (b), and free surface (c).

Figure 4.8 describes the boundary condition of the TSV model used in FEM. To consider the TSVs are arrayed periodically, mirror symmetry was used as a boundary condition and applied to the four outer most surfaces of the model (Fig 4.8a). The bottom surface of the model was described as prescribed displacement ($Z=0$) condition to prevent rigid-body movements, but not affect the stress distribution (Fig 4.8b). On the other hand, the top surface of the model was free to move during deformation (Fig 4.8c). All the interfaces were assumed to have perfect adhesion.
Each domain shown in Figure 4.9 is represented by different materials. These materials are the materials used to deposit the TSVs. The properties of these materials used in the calculation are summarized in Table 4.1 [54–57]. Additionally, elastic-plastic behavior was applied to the copper with a linear isotropy strain-hardening, $h$, and temperature-dependent yield strength, $\sigma_y$, which is expressed in equations 4.6 and 4.7, respectively [58].
\begin{table}
\centering
\begin{tabular}{|l|c|c|c|}
\hline
Properties & Silicon (Si) & Copper (Cu) & TEOS \\
\hline
Young Modulus(GPa) & 131 & 115 & 71.7 \\
\hline
Poisson’s Ratio & 0.27 & 0.35 & 0.16 \\
\hline
CTE (ppm/°C) & 2.61 & 17 & 0.51 \\
\hline
\end{tabular}
\caption{Thermo-mechanical properties of the materials used in the finite element calculations.}
\end{table}

\begin{equation}
Y(\varepsilon^{-p}) = Y_o + h\varepsilon^{-p}
\end{equation}

\begin{equation}
\sigma_y = 222.4 - 0.35T
\end{equation}

Where:

\begin{itemize}
\item $Y$ = yield stress (MPa)
\item $\varepsilon^{-p}$ = plastic strain
\item $h$ = tangential modulus (MPa)
\end{itemize}

It is suggested by the Micro-Raman spectroscopy studies of TSVs that the initial yield stress is 172.3MPa and the tangential modulus is 517MPa [58–61]. X-ray micrographs were taken after the TSVs were pre-annealed at 150°C, and after they were post-annealed at 225°C and 300°C, respectively. The simulations likewise used the referenced temperature (stress-free temperature) of 150°C for the whole domain. The domain was then annealed up to 225°C and 300°C respectively, and cooled down to room temperature ($T_{room}$). The FEM analysis was simulated with the COMSOL thermal structural interaction module to analyze stresses caused by thermal expansion.
when samples are annealed to higher temperatures. With this FEM, the area where most concentrated hydrostatic stresses exist (compressive or tensile) can be predicted during the annealing or cooling process. However, the simulated numerical value will differ from the experimental stress measurement, due to the assumptions used to simplify the FEM analysis.

Figure 4.10: Hydrostatic stresses plotted along the horizontal of pre-existing void (The path is illustrated by black dashed line in the right figure).
Figure 4.11: Hydrostatic stresses plotted along the vertical of pre-existing void (The path is illustrated by black dashed line in the right figure).

For the numerical analysis of TSV with pre-existing voids, the hydrostatic stresses are plotted horizontal and vertical to the center of the void as illustrated in Figure 4.10 and Figure 4.11 [46]. The domain of the void used in the model is air, which is assumed to be a stress-free body. This expectation is seen in the finite element results wherein zero hydrostatic stresses are found in the pre-existing void. The numerical results in Figure 4.10 and Figure 4.11 show the copper TSV domain is under compressive hydrostatic stresses when the temperature rises as in the case of A1 (150°C to 225°C) and B1 (150°C to 300°C). The compressive stress in B1 is higher than in A1 in both the horizontal (maximum 327MPa) and vertical (maximum 393MPa) plots from each figure. This domain is under tensile hydrostatic stress when cooling down, as shown for A2 (225°C to $T_{room}$) and B2 (300°C to $T_{room}$) in both
the horizontal and vertical directions. B2 experiences higher tensile stress than A2 in both the horizontal and vertical plots with peaks at \( \sim 1800 \text{MPa} \) and \( \sim 1300 \text{MPa} \), respectively for B2 and A2.

![Figure 4.12: Hydrostatic stresses gradients contour plot of an ideal TSV structure (a) with 100nm liner, and TSV structure with pre-existing void (b) cooled from 300°C to room temperature.](image)

Compressive hydrostatic stress is known to suppress or delay the creation of voids, while tensile hydrostatic stress accelerates their formation. This gradient is the primary factor in void growth [62]. This implies that voids may form as the annealing temperature is cooled to \( T_{room} \) in the highest tensile hydrostatic stress region. Since the TSV has a pre-existing fill-induced void (Fig. 4.5a-b and Fig 4.6a-b), we therefore concentrate on the role the gradient of tensile hydrostatic stress has in void growth. The hydrostatic stress gradients after cooling of an ideal TSV and TSV with a pre-existing void were compared. The hydrostatic stress gradient contour plots of an ideal TSV with \( \sim 100 \text{nm} \) of liner and a TSV with a pre-existing void are shown in Figure 4.12a-b [46] respectively. The hydrostatic stress gradient around the
void of the TSV with a pre-existing void is found to be greater than in the ideal TSV (Fig 4.13).

Figure 4.13: Hydrostatic stresses gradients plotted along the horizontal of TSV with pre-existing void and ideal TSV (The path is illustrated by black dashed line in the right figure).
Figure 4.13 [46] is the hydrostatic stress gradient plotted along horizontal (black dashed line) planes of the ideal TSV and TSV with pre-existing void from numerical studies. The stress gradients for the ideal TSVs cooled from 225°C and 300°C to $T_{room}$ reach their peak stress at $\sim 70\text{MPa}/\mu\text{m}$ and $\sim 140\text{MPa}/\mu\text{m}$, respectively. The FEM analysis results illustrate that higher hydrostatic stress gradients are found around the interface of the $\text{Si} - \text{SiO}_2 - \text{Cu}$ around the TSV and gradually vanish into the wafer. The gradient in the TSV region is $\sim 0\text{MPa}/\mu\text{m}$ for ideal TSVs. At the interfaces, the coefficients of thermal expansion (CTE) of the materials are different (Table 4.1). When annealed to higher temperatures, thermal stress makes the materials expand differently, and consequently, stresses concentrate at the interface, which could easily impact stress-induced void creation (SIV) and void growth [63].

For the TSV with a pre-existing void, the stress gradients around the void reach their maximum stress at $\sim 1300\text{MPa}/\mu\text{m}$ and $\sim 4000\text{MPa}/\mu\text{m}$ for TSVs cooled from 225°C and 300°C to $T_{room}$, respectively. When the copper-filled TSV is annealed at a higher temperature than its stress-free temperature, then cooled to room temperature, the copper undergoes high tensile stress [63–65]. The pre-existing void serves as a stress sink, thereby relieving the stress and generating the hydrostatic stress gradients between the void and other points in the TSV. These hydrostatic stress gradients are shown in Figure 4.12b. The vacancies are assumed to be in local equilibrium, so that the stress gradients induce a gradient in the vacancy concentration or chemical potential [43]. Consequently, the high stress gradients around the void relax over time as the vacancies diffuse, causing the pre-existing void to grow. This void growth after annealing a TSV with pre-existing voids is seen in the experimental data from reconstructed X-ray micrographs. Thus, void growth occurs at the pre-existing void because the hydrostatic stress gradient is highest near the void.
Figure 4.14: Hydrostatic stresses gradients plotted along the vertical of TSV with pre-existing void (The path is illustrated by black dashed line in the top figure).

Not only do we see that the hydrostatic stresses and gradients around the pre-existing void are higher than at the interfaces around the TSV (Fig 4.10), but when TSVs with a 150°C stress-free temperature are annealed at a higher temperature, higher stresses and gradients occur during cooling. Both the horizontal plot (Fig 4.13) and vertical plot (Fig 4.14) [46] clearly illustrate this. In the horizontal plot, the peak of the tensile hydrostatic stresses (Fig 4.10) and gradients when cooling from 300°C to $T_{room}$ is found to be higher than when cooling from 225°C, with a $\sim$460MPa and $\sim$2800MPa/µm difference, respectively. For the vertical plot, stresses (Fig 4.11) and gradients when cooling from 300°C are greater than when cooling from 225°C, with a $\sim$400MPa and $\sim$6x10^4MPa/µm difference, respectively. Hence, cooling from a higher annealing temperature to $T_{room}$ could increase the stress gradients or the
driving force. Therefore, the X-ray micrographs and calculations show that the total volume of void growth at 300°C is greater than the volume at 225°C (Fig 4.5 and Fig 4.6).

Furthermore, higher stresses and gradients are shown to occur more frequently at the vertical edges (Fig 4.14) than the horizontal edges (Fig 4.13) of the elliptical void, as was seen in the FEM analysis of a TSV with pre-existing void. As shown in Figure 4.13 and Figure 4.14, the gradients in the horizontal plot are in the range of $10^3$MPa/µm, while in the vertical plot, they are in the range of $10^4$MPa/µm. In the elliptical void, the end in the vertical direction is sharper than in the horizontal direction implying a greater concentration of stresses and gradients appearing in sharper corners, leading to greater driving forces.

### 4.3.2 Diffusion FEM

#### 4.3.2.1 Background [66]

Under a constant driving force, chemical potential, concentration gradient and flux, atomic diffusion phenomena can be described by the Fick’s first law. The Fick’s first Law states that the flux, $J$, of a component of concentration, $C$, across a given plane is proportional to the concentration differential across that plane, and is expressed by equation 4.8:

$$J = -D \Delta C \quad (4.8)$$

It fits to explain the flux goes to zero as the inhomogeneous specimen becomes homogeneous under driving force. However, in most of the diffusion problem, the atomic diffusion flux changes with position and time. The Fick’s first law cannot be
used to describe diffusion where flux varies with position and time, or where the flux has a changing driving force. To handle such a non-steady-state problem, a continuity equation (Gauss theorem assumes that the derivatives are continuous functions) is derived from the principle of conservation of mass. To do this the Gauss theorem or divergence theorem is used. The divergence theorem states that the sum of all sources minus the sum of all sinks gives the net flow out of a region.

![Figure 4.15: The flux through a cube in Cartesian coordinate system.](image)

Now, consider a flux, $J_1$ going in and out of a cube in a period $\Delta t$, the number of atoms entering or leaving, $\Delta N_1$ a surface, $A_1$ as shown in Figure 4.15 will be

$$\Delta N_1 = J_1 A_1 \Delta t$$  \hspace{1cm} (4.9)
By summing all the fluxes, $J_i$ passing through each surface of area or all the atoms in and out of the cube volume, the net change of total atoms inside the cube is shown in equation 4.10.

\[
\sum_{i=1}^{6} J_i A_i \Delta t = \sum_{i=1}^{6} \Delta N_i = \Delta N \tag{4.10}
\]

If the volume of the cube is $V$ and $C$ is the concentration in the cube, the changes of concentration, $\Delta C$ is described in equation 4.11.

\[
\Delta N = V \Delta C \tag{4.11}
\]

With equation 4.10 and 4.11, equation 4.12 is obtained.

\[
\sum_{i=1}^{6} J_i A_i = \frac{dN}{dt} = V \frac{dC}{dt} \tag{4.12}
\]
According to the Gauss theorem, the left term is equal to the divergence of flux inside the cube volume, thus we get equation 4.13.

\[ \sum_{i=1}^{6} J_i A_i = (\nabla J)V \quad (4.13) \]

In general, the Gauss theorem applies to an arbitrary shape of volume enclose by an area. The summation shown in the left term in equation 4.13 can be expressed as integration over a close surface, which is shown in equation 4.14.

\[ \oint_A (J \cdot n) dA = (\nabla J)V \quad (4.14) \]

With the combination of equation 4.12-4.14, the Fick’s second law predicts how diffusion causes the concentration to change with time is derived and shown in equation 4.15-4.16.

\[ V \frac{dC}{dt} = \oint_A (J \cdot n) dA = (\nabla \cdot J)V \quad (4.15) \]

\[ V \frac{dC(x, t)}{dt} = -(\nabla \cdot J) \quad (4.16) \]

In this case, the influx is assumed less than the outflux, so the concentration
inside the cube is decreases with time. Therefore, a negative sign is on the flux divergence term.

### 4.3.2.2 Vacancy Concentration Under Hydrostatic Stress Gradients

The purpose of this section is to analyze the changes of vacancy concentration with position under the driving forces of concentration and stress gradients, using the diffusion module in COMSOL. Other driving forces (e.g. temperature gradient and electromigration) are negligible. Equation 4.16 (Fick’s second law) is the only equation provided in the diffusion module, which is used to explain the non-steady state situation. Upon substitution of the total diffusion or atomic flux, $J_{tot}$ by the driving forces of concentration and stress gradients (Equation 4.3), into equation 4.16, equation 4.17 is obtained.

\[
\frac{\delta C(x,t)}{\delta t} = \nabla (D \nabla C) - \nabla \left[ \frac{DC}{kT} (\Omega \nabla \sigma_{Hs}) \right] \quad (4.17)
\]

Since the interest is to analyze the vacancy concentration changes with position under the concentration and stress gradients, the FEM can be simplified to a steady state system without time dependence as shown in equation 4.18-4.19.

\[
\frac{\delta C(x,t)}{\delta t} = - (\nabla \cdot J) = 0 \quad (4.18)
\]

\[
0 = \nabla (D \nabla C) - \nabla \left[ \frac{DC}{kT} (\Omega \nabla \sigma_{Hs}) \right] \quad (4.19)
\]
In the diffusion module, the simulated hydrostatic stress gradient in the previous section is used in the second term of equation 4.19. An initial $1 \text{mol/m}^3$ of vacancies is assumed to be distributed throughout the TSV (Fig 4.16a). A constant $D (7.4 \times 10^{-10} \text{cm}^2/\text{s})$ is used as the diffusivity of vacancies in the TSV. This value $D$ is optional based on equation 4.19. The boundary conditions around the TSV and void is set as no flux and the vacancies are assumed to be in thermal equilibrium along the TSV with the hydrostatic stress gradient. Therefore, the hydrostatic stress gradient produces a gradient in vacancy concentration without relaxation, which is shown in Figure 4.16b. The highest vacancy concentration is occurring at the location where the highest hydrostatic stress gradient is concentrated.

However, in reality, this situation would not last since the void surface is a perfect sink/source for vacancies so the vacancies would annihilate at the surface rapidly. This plot (Fig 4.16b) is a good approximation of the chemical potential for vacancies in the presence of the void and the applied stress. This indicates that we would expect to see preferential vacancy flow to the areas of stress concentrations, thus enlarging the void.
Figure 4.16: FEM color contour plot of vacancy concentration.
4.4 Bottom-up vs Conformally Filled TSV

The experimental measurements and finite element analysis results have good agreement showing that pre-existing voids or seamlines present in conformally filled TSVs are prone to void growth due to the filling method. To investigate the affects of the bottom-up filing method as a function of void growth compared to the conformal filling, experiments are repeated on bottom-up filled TSV. The TSV arrays are 5x50\mu m. Copper is filled from the bottom of the TSV trench toward the top of TSV in this bottom up procedure, preventing the formation of a pre-existing void or seamline occurring at the center of the TSV. These bottom-up filled TSV arrays are inspected with NanoXCT before and after 200°C, 225°C, 250°C, and 300°C annealing respectively. These TSV arrays underwent 24 hours annealing in pure nitrogen environment for each of the annealing temperatures. An x-ray tomography of each TSV array is generated and compared as shown in Figure 4.16. Before the TSVs were processed for any thermal treatment, no pre-existing void was observed in the TSV as shown in Figure 4.16 a, c, e, and g. After these same TSVs were annealed to different temperatures respectively, x-ray tomography was generated for each of these TSVs resulting in no voids or void growth being observed (Figure 4.16 b, d, f, and h).

Another two conformally filled TSV arrays were annealed to 200°C, and 250°C respectively to complete the same four different annealing temperatures of TSV arrays as bottom up-filled TSV. These conformally filled TSVs were inspected with NanoXCT and is shown in Figure 4.17, which includes before (Fig a, c, e, and g) and after annealing (Fig 4.17b, d, f, h). The images clearly show the pre-existing voids present in conformally filled TSVs before annealing except TSV in Figure 4.17e. After annealed to 200°C, 225°C, 250°C, and 300°C respectively, void growth is observed and total volume of void growth increases with annealing temperature. The comparison between bottom up-filled TSVs (Fig 4.16) and conformally filled TSV (Fig 4.17) is solid evidence that conformally filled TSV are prone to void growth which is of great concern for device degradation and failure at early life stages. Therefore,
bottom up-filled TSV is preferable to avoid any void growth issues associated with conformally filled TSVs.

Figure 4.17: Bottom up-filled TSV at before (a, c, e, and g) and after (b, d, f, and h) annealed to different temperatures respectively.
Figure 4.18: Conformally filled TSV at before (a, c, e, and g) and after (b, d, f, and h) annealed to different temperatures respectively.
CHAPTER 5
Other Sub-surface Microscopy
Techniques of TSVs

Besides x-ray microscopy, there are other potential sub-surface imaging techniques to image the TSV non-destructively. These sub-surface techniques are (1) acoustic and (2) infrared microscopy. These techniques are used as in-line metrology to inspect the interface of bonded wafers for defects and overlay measurement. Acoustic and infrared might not have similar resolution as x-ray microscopy, but they are able to detect some TSV defects at micro-scale resolution. In this chapter, the principle and TSV imaging of these microscopies will be discussed.

5.1 Principles of Scanning Acoustic Microscope
(Sonix)

5.1.1 Ultrasound

Sound waves are disturbances which travel through a medium. As the wave travels, the medium will experience local oscillations, but the particles in the medium do not travel with the wave. The disturbance may take any of a number of shapes, from a finite width pulse, to an infinitely long sine wave. Sound waves are mechanical vibrations that are able to travel through all forms of elastic medium: gases, air, liquids, solids and plasma, but cannot travel in vacuum medium. They travel through materials under the influence of sound pressure. Because molecules or atoms of an elastic material are bound elastically to one another, the excess pressure results in
a wave propagating through the material. Sound waves are travelling waves which oscillate and are composed of frequencies that transmit through the medium [67]. The sound range diagram in Figure 5.1 shows the different type of sound waves.

![Figure 5.1: Sound range diagram.](image)

The figure above shows that audible sounds detected by humans occur between infrasound and ultrasound. These audible sounds are in the range of 12Hz-20KHz. Ultrasound occurs at higher frequency than audible sounds, and will be the focus of this study. Like other sound, ultrasound transmits through gases, liquids and plasma as longitudinal waves, also called compression waves. In solids, ultrasound can propagate in four principle modes based on the way the particles oscillate: (1) longitudinal wave, (2) transverse wave (or shear waves), (3) surface wave, and in thin materials as (4) plate waves. However, longitudinal and transverse waves are the two modes of propagation most widely used in ultrasonic testing. Various types of waves that vibrate elliptically could also be occurring at the surfaces and interfaces [67].

### 5.1.2 Longitudinal Wave

In longitudinal waves, the oscillations of particles occur in the longitudinal direction or parallel to the direction of wave propagation. The particles do not move along with the wave; they simply oscillate back and forth about their individual equilibrium positions as the wave passes. Since compression and dilation forces are
active in these waves, they are also called pressure or compression waves. They are also sometimes called density waves because their particle density fluctuates as they move. Compression waves can be generated in liquids as well as solids, because the energy travels through the atomic structure by a series of comparison and expansion (rarefaction) movements [67].

5.1.3 Transverse Wave

In the transverse or shear wave, the particles oscillate perpendicularly to wave direction or transverse to the direction of propagation. Same as longitudinal waves, the particles in transverse waves do not move along with the wave, they simply oscillate up and down about their individual equilibrium. Shear waves require an acoustically solid material for effective propagation; therefore, these waves do not effectively propagate in materials such as liquids or gasses. Shear waves are relatively weak when compared to longitudinal waves, and are usually generated in materials using some of the energy from longitudinal waves.

5.1.4 Acoustic Impedance

A scanning acoustic microscope works on the principle of propagation and reflection of ultrasonic waves at interfaces where a change of acoustic impedance, $Z$, occurs [68, 69]. This acoustic impedance is expressed as the product of the density and the speed of sound in the medium, as shown in equation (5.1) [68].

$$Z = \rho V$$  
(5.1)
Where: 

\[ Z = \text{Acoustic impedance of a material} \]
\[ \rho = \text{Density of the material} \]
\[ V = \text{Acoustic wave velocity in the material} \]

We can see from the above equation, the impedance is governed by the acoustic wave velocity and density of the material. The speed of sound is unique in various materials due to the differences of mass of the atomic particles and the spring constants. The mass of the particles is related to the density, and the spring constant is related to the elastic constants of a material. The velocity can be represented as a function of the materials density and elastic constants as shown in equation (5.2) [68].

\[ V = \left(\frac{C}{\rho}\right)^{\frac{1}{2}} \quad (5.2) \]

Where:

\[ V = \text{Speed of sound} \]
\[ C = \text{Elastic constant} \]
\[ \rho = \text{Material density} \]

Table 5.1 [67, 70] shows the density, velocity, and acoustic impedance for different materials. It can be used to explain why sound waves cannot travel through vacuum, and a medium is needed to transmit the sound wave. When there is a mismatch of impedance at the boundary between two materials, part of the sound wave will reflect. The intensity of the reflected wave increases as the mismatch in acoustic impedance increases. This can be explained from the equation (5.3) and (5.4). From Table 5.1, the mismatch of the acoustic impedance between air and silicon is large causing nearly all of the wave energy generated from the transducer to be reflected, while very little of wave is transmitted into the specimen. A couplant or coupling medium is needed to facilitate the transmission of ultrasonic energy from the trans-
ducer into the specimen. Compared to air, a couplant like water allows more sound energy into the specimen so a usable ultrasonic signal can be obtained.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm³)</th>
<th>Longitudinal wave velocity (m/s)</th>
<th>Acoustic Impedance (kg/m²s)(10⁶)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>1.00</td>
<td>1483</td>
<td>1.48</td>
</tr>
<tr>
<td>Alcohol</td>
<td>0.79</td>
<td>1168</td>
<td>0.92</td>
</tr>
<tr>
<td>Air</td>
<td>0.0013</td>
<td>344</td>
<td>0.4472</td>
</tr>
<tr>
<td>Silicon</td>
<td>2.33</td>
<td>8600</td>
<td>20.04</td>
</tr>
<tr>
<td>Gold</td>
<td>19.3</td>
<td>3240</td>
<td>62.53</td>
</tr>
<tr>
<td>Copper</td>
<td>8.90</td>
<td>4700</td>
<td>41.83</td>
</tr>
<tr>
<td>Aluminum</td>
<td>2.70</td>
<td>6260</td>
<td>16.90</td>
</tr>
<tr>
<td>Epoxy Resin</td>
<td>1.20</td>
<td>2600</td>
<td>3.12</td>
</tr>
<tr>
<td>Resin (IC Pkg)</td>
<td>1.72</td>
<td>3930</td>
<td>6.76</td>
</tr>
<tr>
<td>Glass (Quarzt)</td>
<td>2.70</td>
<td>5570</td>
<td>15.04</td>
</tr>
<tr>
<td>Alumina</td>
<td>3.80</td>
<td>10410</td>
<td>39.56</td>
</tr>
</tbody>
</table>

Table 5.1: The acoustic impedance of different materials.
5.1.5 Transmission and Reflection

The piezoelectric transducers of the in-house scanning acoustic microscope from Sonix are typically made to transmit and receive longitudinal waves. To generate or transmit the ultrasound waves, a high voltage pulse from the transmitter is given to the transducer. The transducer vibrates and generates a plane wave of a unit amplitude. This plane wave is refracted by a lens and travels as a longitudinal wave in the coupling medium. When it reaches the surface of any solid specimen, part of the wave remains as a longitudinal wave, and part of the wave travels as a transverse wave at a typical angle to propagate through the specimen.

Figure 5.2: Transmission and reflection wave from transducer.
Figure 5.2 shows an elastic material (specimen) that is immersed in a coupling medium (water), and a transducer generating an ultrasound toward the specimen, also known as the initial ultrasound wave. The ultrasound will travel in the water as a longitudinal wave before refracted by the lens. This initial ultrasound wave is carrying the maximum wave energy generated by the transducer and will keep travelling until it reaches the boundary in between the water and specimen. At this boundary, the acoustic impedance of water, $Z_1$, is different from the acoustic impedance of specimen, $Z_2$, due to the change of materials density and the velocity of sound in different materials. Part of this initial wave is reflected from the boundary and received by the transducer, while part of it continues to propagate through the specimen reaching another boundary where a change in acoustic impedance occurs again.

The fraction of this initial wave intensity that is reflected from surface of specimen can be calculated with equation (5.3) [68] if the acoustic impedances of the materials on both sides of the boundary are known.

$$R = \frac{(Z_2 - Z_1)}{(Z_2 + Z_1)}$$

(5.3)

This value that is produced called the reflection coefficient, $R$. If this value is subtracted from 1, the remaining value will be equal to the transmission coefficient which can be calculated from equation (5.4) as below [5].

$$T = \frac{2(Z_1)}{(Z_2 + Z_1)}$$

(5.4)
5.1.6 Resolution

The spatial resolution [68, 71], $W$ of the acoustic microscope is based on Rayleigh criterion and is given as:

$$W = \frac{0.51\lambda_o}{NA} \tag{5.5}$$

Where:

$NA$ = numerical aperture

$\lambda_o$ = Wavelength, given by $V_o/f$ ($V_o$ is the velocity of sound)

The resolution is proportional to the wavelength of the ultrasound, which is dependent upon frequency and the velocity of sound in the material. This gives one the idea that unlimited resolution can be obtained if the frequency is increased infinitely. However, the ultrasonic attenuation, which is proportional to the square of the frequency, doesn’t allow this happen. The attenuation per distance traveled is shown in equation (5.6) [68].

$$A = \alpha_o f^2 \tag{5.6}$$

Where:

$A$ = Attenuation

$\alpha_o$ = Attenuation coefficient of the material

$f$ = Frequency of sound in the material
When ultrasound travels through a medium, its intensity reduces with distance. This reduction is caused by a combination of scattering and absorption, also known as attenuation. Scattering is the reflection of the sound in the direction other than the original propagation direction, and absorption is the conversion of the sound energy to other forms of energy. Absorption is also known as the decay rate of the wave as it propagates through material. We know that higher frequencies and shorter wavelengths can give better resolution, but what is the highest resolution that can be obtained? It is very important to understand how attenuation allows one to determine the value of the highest frequency and shortest wavelength.

When the acoustic wave is generated from the vibration of the transducer, it will propagate through the lens. Single crystal sapphire is a type of material that acoustic waves can propagate through with relatively small loss of energy or small attenuation. As we have discussed earlier, the acoustic wave needs a medium to support its propagation. It is important to choose a right material and coupling medium for the wave to propagate between the lens and the specimen. If the coupling material possesses high attenuation, the leftover acoustic wave has an insufficient amount of energy and causes poor resolution. Now, let us see how the attenuation constrains the highest frequency and shortest wavelength to obtain the maximum resolution. If a pulse length is given, the minimum focal length is given as below:

\[ FL = \frac{V_o t_o}{2} \]  

Where: 
\( FL \) = Focal length  
\( V_o \) = Velocity  
\( t_o \) = Time interval between echoes
From equation (5.7) [68], we know that the attenuation is proportional to the square of the frequency. An acceptable attenuation, $\alpha_{acc}$, is needed to determine the maximum frequency that can be used. The constrained frequency by this $\alpha_{acc}$ is shown in equation (5.8) [68].

\[
F \leq \sqrt{\frac{\alpha_{acc}}{2\alpha_o FL}} \quad (5.8)
\]

Where:
- $f$ = Frequency
- $\alpha_{acc}$ = Acceptance attenuation
- $\alpha_o$ = Attenuation coefficient

This maximum frequency is then defined by the shortest wavelength, $\lambda_{min}$, that can be used as shown in equation (5.9) [68]

\[
\lambda_{min} = \frac{V_o}{f}
\]
\[
= \sqrt{\frac{V_o^3 \alpha_o t_o}{\alpha_{acc}}} \quad (5.9)
\]

\[
R_c = |\sqrt{V_o^3 \alpha_o}|
\]
\[
= \lambda_{min} \sqrt{\frac{\alpha_{acc}}{t_o}} \quad (5.10)
\]
As seen in equation (5.10) [68], the resolution coefficient is proportional to the shortest wavelength that can be used with the given $\alpha_{acc}$ and $t_o$. Also, from equations (5.7-5.10) we can make a short conclusion that higher frequencies will have less penetration and lead to shorter focal lengths, while allowing an improvement in resolution. Figure 5.3 below gives a short comparison between low and high frequency which is important when dealing with samples of different thicknesses. It gives a picture of how to choose the right transducer for our sample.

Figure 5.3: Comparison between high and low frequency transducers.
<table>
<thead>
<tr>
<th>Fluid</th>
<th>Temperature T(K)</th>
<th>Velocity $V_s$(μm/ns)</th>
<th>Impedance $Z$ (Mrayl)</th>
<th>Attenuation $\alpha_0$ (dB/μm GHz$^2$)</th>
<th>Resolution coefficient $R_0$(μm dB$^{1/2}$/ns$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>298</td>
<td>1.495</td>
<td>1.49</td>
<td>0.191</td>
<td>0.799</td>
</tr>
<tr>
<td>Water</td>
<td>333</td>
<td>1.551</td>
<td>1.525</td>
<td>0.086</td>
<td>0.566</td>
</tr>
<tr>
<td>Methanol</td>
<td>303</td>
<td>1.088</td>
<td>0.866</td>
<td>0.262</td>
<td>0.581</td>
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<tr>
<td>Ethanol</td>
<td>303</td>
<td>1.127</td>
<td>0.89</td>
<td>0.421</td>
<td>0.776</td>
</tr>
<tr>
<td>Acetone</td>
<td>303</td>
<td>1.158</td>
<td>0.916</td>
<td>0.469</td>
<td>0.853</td>
</tr>
<tr>
<td>Carbon Tetrachloride</td>
<td>298</td>
<td>0.93</td>
<td>1.482</td>
<td>4.67</td>
<td>1.94</td>
</tr>
<tr>
<td>Hydrogen peroxide</td>
<td>298</td>
<td>1.545</td>
<td>2.26</td>
<td>0.087</td>
<td>0.566</td>
</tr>
<tr>
<td>Hydrogen</td>
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<td>1.19</td>
<td>0.08</td>
<td>0.049</td>
<td>0.287</td>
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<td>Carbon disulphide</td>
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<td>0.087</td>
<td>0.442</td>
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<td>Mercury</td>
<td>297</td>
<td>1.449</td>
<td>19.7</td>
<td>0.050</td>
<td>0.391</td>
</tr>
<tr>
<td>Gallium</td>
<td>303</td>
<td>2.87</td>
<td>17.5</td>
<td>0.0137</td>
<td>0.57</td>
</tr>
<tr>
<td>Air (dry)</td>
<td>273</td>
<td>0.33145</td>
<td>0.43x10$^{-4}$</td>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td>Air (dry)</td>
<td>293</td>
<td>0.34337</td>
<td>0.41x10$^{-3}$</td>
<td>1.6x10$^5$</td>
<td>80</td>
</tr>
<tr>
<td>Air (dry)</td>
<td>373</td>
<td>0.386</td>
<td>-----------</td>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td>Argon (4MPa)</td>
<td>293</td>
<td>0.323</td>
<td>0.023</td>
<td>3.58</td>
<td>0.347</td>
</tr>
<tr>
<td>Argon (25MPa)</td>
<td>293</td>
<td>0.323</td>
<td>0.145</td>
<td>0.721</td>
<td>0.156</td>
</tr>
<tr>
<td>Xenon (4MPa)</td>
<td>293</td>
<td>0.178</td>
<td>0.042</td>
<td>8.28</td>
<td>0.216</td>
</tr>
<tr>
<td>Oxygen</td>
<td>90</td>
<td>0.9</td>
<td>1.0</td>
<td>0.086</td>
<td>0.25</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>77</td>
<td>0.85</td>
<td>0.68</td>
<td>0.12</td>
<td>0.271</td>
</tr>
<tr>
<td>Neon</td>
<td>27</td>
<td>0.60</td>
<td>0.72</td>
<td>0.201</td>
<td>0.208</td>
</tr>
<tr>
<td>Helium</td>
<td>4.2</td>
<td>0.183</td>
<td>0.023</td>
<td>1.966</td>
<td>0.110</td>
</tr>
<tr>
<td>Helium</td>
<td>1.95</td>
<td>0.227</td>
<td>0.033</td>
<td>0.610</td>
<td>0.084</td>
</tr>
<tr>
<td>Helium</td>
<td>0.4</td>
<td>0.238</td>
<td>0.035</td>
<td>0.015</td>
<td>0.014</td>
</tr>
<tr>
<td>Helium</td>
<td>0.1</td>
<td>0.238</td>
<td>0.0345</td>
<td>4x10$^{-5}$</td>
<td>7x10$^{-4}$</td>
</tr>
</tbody>
</table>

Table 5.2: Acoustic parameters of various fluids.
Table 5.2 [68] has listed the resolution coefficient for various fluids as medium. Cryogenic liquids offer a better resolution than water, but in most applications water is chosen as the coupling medium for acoustic microscopy. Since the acoustic properties of the water change at different temperatures, the resolution increases proportionally to the temperature as seen in Table 5.3. From this table, the velocity of the waves increases with temperature from 0°C to 70°C, while the attenuation decreases with temperature. However, the rate of attenuation is reducing at higher temperature.

<table>
<thead>
<tr>
<th>Temperature T(K)</th>
<th>Velocity $V_o$ (µm/ns)</th>
<th>Impedance $Z$ (Mrayl)</th>
<th>Attenuation $\alpha_o$ (dB/µm GHz$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.40239</td>
<td>1.402</td>
<td>0.469</td>
</tr>
<tr>
<td>10</td>
<td>1.44727</td>
<td>1.447</td>
<td>0.321</td>
</tr>
<tr>
<td>20</td>
<td>1.48234</td>
<td>1.48</td>
<td>0.226</td>
</tr>
<tr>
<td>30</td>
<td>1.50913</td>
<td>1.503</td>
<td>0.165</td>
</tr>
<tr>
<td>40</td>
<td>1.52886</td>
<td>1.517</td>
<td>0.13</td>
</tr>
<tr>
<td>50</td>
<td>1.54255</td>
<td>1.524</td>
<td>0.104</td>
</tr>
<tr>
<td>60</td>
<td>1.55099</td>
<td>1.525</td>
<td>0.086</td>
</tr>
<tr>
<td>70</td>
<td>1.5548</td>
<td>1.52</td>
<td>0.075</td>
</tr>
<tr>
<td>80</td>
<td>1.55449</td>
<td>1.511</td>
<td>0.076</td>
</tr>
<tr>
<td>90</td>
<td>1.55048</td>
<td>1.497</td>
<td>0.063</td>
</tr>
<tr>
<td>100</td>
<td>1.54311</td>
<td>1.479</td>
<td>0.062</td>
</tr>
</tbody>
</table>

Table 5.3: Change of acoustic properties of water with temperature.
The velocities shown in Table 5.3 can be derived by polynomial expansion in terms of temperature, $T$, and polynomial coefficients, $K$, as shown in equation (5.11) [68].

$$v_o = \sum_{i=0}^{N} K_i T^i$$

(5.11)

Where:
- $K$ = polynomial coefficients
- $T$ = Temperature
5.2 Principles of IR Microscope

5.2.1 Infrared

Like visible light, infrared light has a range of wavelengths. The range of infrared lies between the visible and microwave range in the electromagnetic spectrum. Infrared light that is closer to visible wavelength is called Near Infrared which has shorter wavelength and the longer wavelengths, Far Infrared, are closer to microwave wavelength. The infrared range starts from near infrared (∼700nm, ∼10^{14}Hz) to far infrared (1mm, ∼10^{12}Hz) [72, 73]. The range of infrared light in comparison to the full electromagnetic spectrum is shown in Figure 5.4 below.

![Electromagnetic spectrum](image)

Figure 5.4: Electromagnetic spectrum.

The wavelength increases from gamma radiation to radio. The frequency of the radiations is inversely proportional to the related wavelength as shown in equation (5.12). This implies that the radiation energy decreases when moving from gamma radiation to radio radiation as seen from equation (5.13).

\[ f \propto \frac{1}{\lambda} \] (5.12)
Infrared radiation is emitted by any object that radiates heat. Every day, we experience far infrared radiation in the form of heat from the sun, radiators, fires and etc. It is very useful in oxygenation, promotes the rebuilding of injured tissue, relieves nervous tension etc. On the other hand, the near infrared wavelengths are not easily felt by the temperature-sensitive nerves in our skin. One of the common applications of near infrared waves is the remote control of TV’s. Nowadays, infrared is used in the Semiconductor industry as a non-destructive test (NDT) method. In this study, we will concentrate on the application of infrared microscopy on bonded wafer.

5.2.2 LASER (Light Amplification by Stimulated Emission of Radiation)

Infrared radiation is located at the long wavelength range in the electromagnetic spectrum. From the previous section, we understand that longer wavelength light possesses much lower energy than visible light. This ordinary infrared light is too weak to be used in microscopy applications. Without any amplification, the infrared applications are limited due to low penetration depth and low resolution. The in-house Olympus Confocal IR Laser Scanning Microscope employs high intensity monochromatic light sources. It amplifies the light signal by stimulated emission; a natural effect that was deduced by considerations relating to thermodynamic equilibrium. The result of this amplified light is a high intensity beam and directional with a very pure wavelength or frequency. An understanding of fundamental laser principles is very important for explaining the operation of any laser.

Let us start from the quantization of energy in an atom. This quantization results from the electrons only associated with discrete energy levels. This is very
different from the classical physics which assumes that the energy of an electron can vary continuously, and can have any amount of energy. If an electron lies at an energy level that is higher than ground state then it will spontaneously drop to the lower level. When this happens, energy in the form of a photon will be released. This energy can be calculated by taking the energy difference between the two energy levels [74]. With this in mind, we can relate it to stimulated emission shown in Figure 5.5. Stimulated emission is a process when an excited electron is perturbed by an incoming photon having the same amount of energy, causing the excited electron to return to the lower state and emit another photon. This second photon is created with the same frequency, phase, direction and polarization. Now we see how a single photon perturbed by an excited electron results in two photons being emitted. This amplifies the intensity of the original light [74].

![Figure 5.5: Stimulated emission process.](image)

5.2.3 Confocal

In the basic optical microscope, the condenser lens collects light from the light source and directs it to a small area of an interested specimen. The emitted light
from the area of interest is then received by the detector. The detector receives both in-focal point and out-of-focal point light of an objective lens. The images created by a conventional microscope are rather blurred. This is overcome by a confocal system where the confocal system excludes most of the light from the specimen that is out of the focal plane, resulting in a sharp image where better contrast images are achieved [75, 76].

Figure 5.6: Basic microscope receives various emitted light.
Deviating from a conventional microscope, the confocal microscope uses the idea of point-by-point specimen illumination. This is done by placing a pinhole aperture behind the objective lens allowing only the in-focus emitted light from the specimen to pass through the pinhole and collected by the detector. By moving the specimen or adjusting the optical system, the area of interest is scanned and assembled in whole. Figure 5.6 and Figure 5.7 illustrate how the photodetector receives light from various points from the specimen, and how the pinhole aperture rejects out-of-focus light that is emitted from the specimen.

Figure 5.7: Out-of-focus light is rejected by a pinhole aperture in confocal system.

Figure 5.8 shows a simplified optical system of the confocal infrared (IR) laser scanning microscope. The laser of the system provides a very intense excitation light as seen by the green color in the figure. It passes through the pinhole in front of the laser and reaches the dichromatic mirror. The dichromatic mirror functions as a
beamsplitter which lets the excited light get reflected toward the specimen, and allow the emitted light from specimen to pass through it before reaching the detector. After the excited light is reflected by the dichromatic mirror, it becomes the incident light source focused on the specimen by the objective lens.

Figure 5.8: A simplified system of the Olympus Confocal IR Laser Scanning Microscope.
All the emitted light from the specimen shown in red is then reflected, and travels in the opposite direction of the incident light toward the photomultiplier detector. In a confocal microscope, not all the emitted light from the specimen is collected by the detector. There is another pinhole placed in front of the detector. This pinhole is conjugate to the focal point of the lens. It blocks most of the out-of-focus light allowing the in-focus emitted light to pass through and be collected by the detector. This resulting image formed by the out-of-focus light is significantly attenuated compared to the image of the in-focus light [76].

As mention previously, there is never a complete image of the specimen in confocal system because at any instant only one point of the specimen is observed. To collect the full image, motors are needed to drive the mirrors and scan the laser across the area of interest on the specimen. To visualize a full image, a computer is used to build the images collected by the detector.

### 5.2.4 Resolution

In optical physics, resolution is defined as the ability to distinguish the separation between two closest points. Usually all optical microscopes have their limitation in resolution. Same as an ordinary microscope, the resolution of the confocal microscope is limited by the aperture of the lens, diffraction, and the intensity of incident light. However, there is one more component that affects the resolution in confocal microscopy, pinhole size, which is not utilized in an ordinary microscope [75, 76].

Figure 5.9a and 5.9b illustrate the diffraction patterns that form by a point source with a confocal system and conventional microscope respectively. The center of the diffraction pattern has the highest intensity and is called the Airy disk, shown on the left of figures. On the right shows a simple 2D intensity point spreading function where the intensity of light is a function of radius of an Airy disk. The intensity of light of confocal microscope is higher when compared to a conventional
microscope. This means the energy at the central part of the Airy disk for a confocal system is higher than that of a conventional system.

Figure 5.9: Diffraction pattern intensity profiles of a confocal (a) and conventional (b) microscope.
According to the Rayleigh criterion, optical resolution is defined as the minimum separation between two Airy disks (Fig 5.10) that can be distinguished. The radius or resolution, $R$ of a conventional optical microscope is given in equation (5.14) [77, 78].

$$R = \frac{1.22\lambda f}{nD}$$

(5.14)

Where $\lambda$ is the wavelength of the incident light, $n$ is the refractive index of the medium where the light travels through, $f$ is the focal length of the lens, and $D$ is the lens’s diameter. This equation can be rewritten to substitute the numerical aperture, $NA$ of the lens. The numerical aperture is simply a function of focal length ($f$) and diameter ($D$) as shown in equation (5.15) [77]. The rewritten equation is then shown in equation (5.16) [77].

$$NA = \frac{2f}{D}$$

(5.15)
The resolution for a confocal system is slightly increased which is defined in equation (17) [77, 78].

\[ R = \frac{0.61\lambda}{nNA} \]  \hspace{1cm} (5.16)

\[ R = \frac{0.44\lambda}{nNA} \]  \hspace{1cm} (5.17)

In Figure 5.10, the Airy disk of two point-like objects overlap with each other. Since the Airy disk is the brightest spot in the intensity point spreading function, it cannot be resolved by the optical system. The image from these two point-like objects is then represented by the total intensity of both objects shown in red. Contrast can be used to distinguish the separation between them if these two point-like objects are separated with sufficient distance, causing minimum intensity to occur in the space between each object. This implies that sufficient separation will allow the first zero or lesser intensity to occur in their combined intensity distribution.
5.3 Acoustic Microscope of TSV: Wide Angle Transducer (PVA TePla – SAM TECH)

Although acoustic microscopy waves can propagate through any elastically material, a standard transducer cannot resolve the TSV arrays. For example, resolution of a standard transducer using 110MHz frequency is \(\sim 60\mu m\), which couldn’t resolve the TSV arrays. As explained previously, the XY resolution coefficient is proportional to the shortest wavelength (higher frequency) that can be used with the given acceptable attenuation and time interval between echoes. Thus, the attenuation should be reduced in order to increase the resolution. Therefore a wide angle acoustic transducer operated at 110MHz was used to detect the defects on TSV.

Figure 5.11: Wide angle at 60°.

Figure 5.11 shows the wide angle of 60° at the transducer. This transducer is made with 2.5mm focal length with 60° of the cone angle. Advantages of using this wider angle are seen by increasing the Z spacing resolution, in addition to an increase in the XY resolution with shorter focal lengths by reducing the attenuation.
Rewriting equation (5.8) we then get the linear proportion between acceptable attenuation and focal length as below:

\[ \alpha_{acc} = 2FLf^2\alpha_o \] (5.18)

Where:
- \( f \) = Frequency
- \( \alpha_{acc} \) = Acceptance attenuation
- \( \alpha_o \) = Attenuation coefficient
- \( FL \) = Focallength

This explains why reducing the focal length will reduce the acceptable attenuation which increases the intensity of ultrasound resulting in better resolution.

![Figure 5.12: Target to be inspected: SEMBar, AG and AE reticle.](image)

Shown in Figure 5.12 is the TSV at SEMBar and via chain from AE and AG reticle (Fig 5.12), which will be inspected by scanning acoustic microscope with a
wide angle transducer. The via chains from AE and AG reticle are connected by Metal 1 and Metal 2 layers at the top and the bottom of the 5μm TSV respectively. The line width of Metal 1 and 2 of AE’s via chain is 30μm, and the metal line width for AG’s via chain is 10μm.

The TSV is first electroplated on a single wafer (bottom wafer) and followed by Metal 1 deposition on top. After this, a carrier wafer is attached, and the bonded wafer stack is then polish from the back from an initial thickness of ∼775μm to ∼25μm where the bottom of the TSV is exposed. Metal 2 layer is then deposited on the exposed TSV. The cross section of these via chains is described in Figure 5.13 below, where Metal 1 and Metal 2 are connected with TSV’s.

![Figure 5.13: The cross section of via chain.](image-url)
5.3.1 Result of Wide Angle Transducer With SAM Tech

Figure 5.14: SEMBar SAM image. Red arrows show the detectable defects at TSV.

Figure 5.14 above shows the SAM image focused at the sub-surface of the TSVs at the SEMBar. The SEMBar consist of 1, 3, 5, 10, and 15\textmu m diameter TSVs. This SEMBar area is cleaved off from a single patterned wafer and further polished from the back to \(\sim\)50-100\textmu m. The transducer is then scanned from the back through the 50-100\textmu m silicon piece. In this image, the 10 and 15\textmu m TSVs (red arrows) are detected, but not the 1, 3, and 5\textmu m TSVs. Some defects are detected by the SAM which are shown by red arrows.

A large field of view image is taken on part of the die and shown in Figure 5.15. The pattern of the die is clearly seen with the Metal 2 layer wafer on the top and the Metal 1 layer is at the bottom. Circled in the red box is the area of interest, recticle AE and recticle AG. The scan is continued by focusing between the TSV from the top of Metal 2. Figure 5.16 below shows the 5\textmu m round TSVs from the via chain without Metal 1 and Metal 2.
Figure 5.15: Large field of view of via chains at reticle AE and AG (red box).

Figure 5.16: 5µm round TSVs from via chain of AE and AG recticle.
The close up view in Figure 5.17 shows the 5µm TSVs in AE are detected and resolved better than the TSVs in AG. A possible explanation of this result is the distance between two TSVs in AE is larger than the AG. The resolution is defined as the ability to distinguish the separation between two closest points. Obviously the wide angle and shorter focal length transducer gives a better resolution, but based on the result, the resolution is estimated to be between 10-20µm. Therefore it cannot fully resolve the 5µm TSV, but is able to detect it. The separation between two TSVs in AG is much smaller than the AE, resulting in a more blurry image of AG TSVs.
5.4 IR Microscope of TSV

The discussion in principle of IR explains the limitation of conventional optical microscopy and how to improve the resolution with the combination of confocal and laser techniques. Olympus confocal IR laser scanning microscope (FR3220-IR) improves the XY resolution to sub-micron. It has been shown that the IR microscope can resolve 0.5\(\mu\)m spacing between two metal lines [Appendix]. This resolution has made the IR microscope a very good wafer level overlay measurement techniques. Because silicon is not opaque to IR light, defect inspection and defect review at the bonded interface is capable with the IR microscope, but it cannot be used to inspect the internal structure of TSVs as shown previously using an x-ray microscope. However, it can be used to detect non-filled and incomplete filled TSV as shown in Figure 5.18 and Figure 5.19 respectively.

![Figure 5.18: Low magnification IR imaging reveals non-filled TSVs.](image)

Figure 5.18 is an IR micrograph taken at the bonded interface through bonded wafers. The IR light propagates through the top wafer before reaches the surface of the bottom wafer. When IR reaches copper, most of the light will reflect back to detector while some of the light is continue penetrate through in silicon wafer.
Therefore, brighter contrast is seen on metal structures and darker contrast on non-metal materials. In Figure 5.18, the IR light is focused at the top surface of bottom wafer (focal plane), where the SEMBar is deposited. Clearly, some non-filled 1µm and 3µm TSVs in the copper-filled TSV arrays are seen, where no reflected light is detected by detector.

![Image of IR micrographs showing incomplete TSVs](image)

**Figure 5.19:** Low magnification IR image reveals incomplete TSVs.

Incomplete-filled TSV are observed in 10µm and 15µm TSV arrays as shown in Figure 5.19. The IR micrograph indicates incomplete filling at the center of the TSVs. An x-ray tomography is generated on these TSV arrays non-destructively. Incomplete filling is found at the center of the TSV arrays, which is shown in the TSV cross section in Figure 5.20. Because the TSVs are filled with conformal method, copper is deposited from the side walls of TSV trench toward the center. Therefore, incomplete filling is most probably occurring at the center of the TSV. However, none of these defects are observed on 5µm TSV array. This could be explained by the filling parameter, which is designed to fill the 5µm TSV arrays. Thus, filling defects are found in other TSV arrays.
Figure 5.20: X-ray micrograph shows incomplete filled TSV.
CHAPTER 6
The Final Summation

6.1 Summary

The hypothesis of stress-assisted void growth in copper-filled TSV was investigated. In the first part of this study, the capabilities of x-ray microscopies at different ranges of x-ray energies were evaluated. The 450eV-1.8keV synchrotron TXM has the best resolution at \(~40\text{nm}\), can resolve and inspect voids in copper, barrier, liner and filling defects, but its probing depth or transmission is limited at below \(~3\mu\text{m}\). This limitation restricts TSV with diameters \(>3\mu\text{m}\) from being inspected. The MicroXCT operated at \(~50\text{keV}\) has very good probing depths, which requires no sample preparation. However, its resolution is limited to \(~1\mu\text{m}\). Voids below \(1\mu\text{m}\) cannot be resolved by MicroXCT. Therefore, the 8keV laboratory based x-ray microscope (NanoXCT) is chosen to visualize the voids growth in copper-filled TSV’s in this study. The NanoXCT capabilities for TSV’s are describe as below:

- No physical cross sectioning of TSV
- 2D and 3D structural information from computed tomography image.
- Internal and external inspection on TSV arrays
- Good contrast for \(Si\), \(Cu\), and voids
- Minimal sample preparation
- Large X-ray penetration depth
- High resolution at \(~50\text{nm}\)
- Able to measure the volume of material
- Able to resolve TEOS Liner

With these capabilities, the same conformally filled TSV arrays were compared before and after being annealed to 200°C, 225°C, 250°C and 300°C. The experimental results show that total volume of void growth of TSV with stress free temperature at 150°C increases with annealing temperature.

The hydrostatic stress and its gradient FEM analysis shows the experimental data derived from X-ray tomography and computation are in good agreement. Three conditions were shown to determine where hydrostatic stresses and gradients are likely to concentrate. First, the numerical results show that an ideal TSV has its maximum stress gradients around interfaces, but they are not as high as the stress gradients at the edge of pre-existing voids in the modeled TSV. This suggests that the void growth phenomenon most probably occurs at pre-existing voids at the center of the conformal-filled TSV as shown in the X-ray micrograph. Second, the finite element analysis shows that the tensile hydrostatic stresses and gradients increase as the annealing temperature cools. This indicates that the total volume of void growth increases as the annealing temperature rises, as evidenced in the X-ray micrographs. Finally, higher hydrostatic stresses and gradients are observed at the vertical edge than at the horizontal edge of elliptical pre-existing voids. This suggests that the site at which stresses concentrate is geometry dependent.

Using the simulated hydrostatic stress gradients in diffusion FEM, vacancy concentration changes with position under the driving force was analyzed. Highest concentration of vacancies is found around the pre-existing void area, which again agrees with the experimental results.

All these FEM and experimental results a great agreement to the hypothesis, where void growth in conformally filled TSV is hypothesized as diffusion and coalescence of vacancies to the pre-existing void area under the driving force of hydrostatic stress gradients.
6.2 Conclusion

To inspect the changes in copper-filled TSV by thermal stress, the 8keV laboratory based x-ray microscope is currently most useful after comparing with other x-ray, acoustic and infrared microscopes. It has been shown that the occurrence of void growth phenomenon in conformally filled TSVs (pre-annealed to 150°C) after annealed to higher temperatures occurs. However, this technique is limited to small sample sizes where the wafer has to be cleaved, and is currently not available as an in-line metrology.

![Figure 6.1: No pre-existing void or seamline is observed (a). Void growth is observed at the same TSV after annealing (b).](image)

With the great agreement of FEM analysis and experimental results to the hypothesis, it is concluded that the conformal filling method is prone to void growth. The conformal filling method is high risk of void, seamline or weak attached surface at the center of the TSV. This causes the TSV prone to void growth at the center after annealing. Shown in Figure 6.1a is a TSV pre-annealed at 150°C, no pre-existing void or seamline is observed. However, after annealed to 255°C, void growth is occurred at
the center of the TSV (Fig 6.1b). The measured total volume of void growth might not cause immediate failure to TSVs 5µm in diameter (Fig 6.2a), but could easily cause failure to TSVs below 3µm in diameter (Fig 6.2b). In contrast, bottom up filling methods are preferable. Evidence of these experimental results have shown where a series of bottom-up filled TSV arrays were inspected before and after annealing to 200°C, 225°C, 250°C and 300°C respectively.

Figure 6.2: 2µm TSV (red dash line) could fail by the total volume of void growth.
6.3 Future Work

6.3.1 Bottom-up Filled TSV

Conformal deposition methods (Fig 6.3a) fill copper from the sidewall toward the center of the TSV trench layer by layer. It is a fast filling method, but high risk seamlines and voids at the center. Furthermore, a thick overburden layer of copper is deposited on the top of the wafer. This overburden can be polished away by CMP, but will affect the CMP cost. On the other hand, bottom up deposition method (Fig 6.3b) is a void or seamline free filling method. This method fills up the TSV trench from the bottom toward the top without a thick overburden. However, the bottom up filling time can take more than 10 times that of conformal filling methods, which increase the cost per wafer compared to conformal methods [5]. Also, it can easily cause impurities in the TSV. However, the findings in this study have shown that conformally filled TSV are prone to void growth due to the pre-existing void and seamline. Therefore, a study on how to increase the bottom up filling rate should be analyzed through the electroplating parameters such as applied current, chemicals, etc.

Figure 6.3: Conformal filling (a) and bottom up (b) filling method.
6.3.2 Electromigration

It is recommended that electromigration studies be done on bottom-up filled TSV. As described earlier, there are four driving forces which can cause the void growth phenomenon: 1) atomic concentration gradients, 2) electrical current (electromigration), 3) temperature gradients, and 4) mechanical stress gradients. Electromigration is a common failure in metallic interconnects and is the mass transport of a metal due to the momentum transfer between conducting electrons and metal atoms. Electromigration could cause failures in microelectronic devices by inducing voids, or hillocks (extrusions).

Void formation is strongly dependent on current direction, where it will appear near the cathode. A model explaining that as atoms migrate in a conductor, changes in mechanical stress occur [79]. Compressive stresses appear where the metal atoms accumulate and tensile stresses appear where the metal atoms deplete. Voids occur in the areas where atomic mass transport results in mass depletion, which eventually cause open circuits [79]. On the other hand, hillocks are formed in the areas of mass accumulation, which may be found near the anode. Hillocks (or extrusion) can cause short circuits depending on the metallization geometry and the proximity of metal lines to one another [80].

6.3.3 Effect of Anisotropic Cu

Understanding the impact of copper anisotropy on the “keep away zone” is necessary as the TSVs scale down to diameters smaller than 2µm, where strong elastic anisotropy of Cu has important implications on the thermo-mechanical stresses surrounding a TSV. As the copper grain size can easily grow from sub micron to more than 1µm. The TSVs below 2µm are dominated by the combination of few copper grains with different young modulus. This implies different orientations of grains (e.g. different elastic-plastic properties) could affect stresses around the TSV
differently. This causes impacts of copper anisotropy induced stress modulation on the charge carrier mobility (n-channel and p-channel) in active silicon [81]. Any stress measurement and simulation on the copper-filled TSV has to consider copper as elastic anisotropy. The stress measurement cannot rely on micro-Raman only, but including the copper stress measurement by micro-XRD and young modulus measurement of different copper grain by nano-indentation.

### 6.3.4 High Volume Liner Metrology

Improving the x-ray microscope as a high volume metrology technique to inspect the liner before copper filling is yet another future direction of this work. In chapter 3, the capability of 8keV laboratory based x-ray computed tomography microscope for resolving the TEOS liner can demonstrate where a liner image is a single 2D x-ray image with exposure times of less than 10s. The x-ray microscope is currently most capable of resolving the liner without physically cross sectioning the TSV arrays. However, there are some challenges on the tool which prevent it from handling 300mm wafers.

The x-ray microscope uses x-ray optical systems to improve its resolution, which limit the area for the 300mm wafer to rotate on the sample stage. Therefore, increasing the rotation area is needed. Furthermore, the wafer has to be thinned to $\sim 60\mu m$ to allow enough x-ray transmission, and at this thickness the wafer is very fragile. The commercially available motor hand used in all the in-line metrology is made to handle full thickness wafers ($\sim 775\mu m$). A new design of motor hand is needed to handle thinned wafers in the tool.
6.3.5 Wide Angle Transducer

Wide angle transducers operated at higher frequencies should be investigated as a new TSV void inspection technique. With the data shown in chapter 5, there is no doubt that wide angle transducer with 110MHz could easily detect the 5µm TSV arrays. However, its resolution is still limited to be used to resolve and inspect defects in copper-filled TSVs. Theoretically, if the operational frequency is higher than 110MHz, better resolution can be achieved, but changing the properties of the transducer could also easily induce noise. This suggests that further investigation should be continued using a wide angle transducer at different focal lengths and higher frequencies. The objective is to find the right transducer and optimize the application for copper-filled TSVs and bonded wafer.
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APPENDIX A
OVERLAY METROLOGY

A.1 Scanning Acoustic Microscope

SAM resolution limits its current capabilities for use as an overlay metrology tool for bonded wafer pairs used in 3D Integration. Thinning the top wafer allows the use of higher frequency, lower focal length transducers that do improve resolution, but not to the point where the SAM technique is useful for measuring overlay per the 2008 ITRS (minimum size of 1µm vias and 0.5µm overlay tolerance).

A.2 Confocal Infrared Laser Scanning Microscope

Since silicon is transparent to the infrared portion of the electromagnetic spectrum, IR microscopy is an appropriate metrology technique for measuring the overlay of bonded wafer pairs. The Olympus Confocal IR Laser Scanning Microscope uses near infrared (1310nm) wavelengths to see through the silicon wafer at the top of a bonded wafer pair. Sufficient resolution allows the IR microscope to be used to analyze the overlay alignment fiducials of bonded wafer pairs.

Shown in Figure A.1 are the overlay alignment fiducials used to align and bond a wafer pair. The alignment structure is a combination of the box on the top wafer (785 level) and the cross on the bottom wafer (750 level). The distance between the box and cross fiducials is used to calculate overlay error. Overlay error in X and Y can be measured directly, and rotation can be calculated by comparing the overlay alignment fiducials on the left and right sides of the bonded wafer pair. Figure A.1
shows almost perfect overlay of the 785 level (Mirrored Metal 2) to the 750 level (Metal 1 and Via) in this specific bonded wafer pair.

Figure A.1: Overlay Alignment Fiducials.

The difference in X and Y offset in the overlay alignment fiducials can be used to calculate rotation in the bonded wafer pair. Figure A.2 shows a wafer pair that is grossly misaligned in Y (+26.5\,\mu m), but a difference in Y offset on a left and right side die, separated by 112.08mm in the center row also reveals a slight rotational error. The ability of the IR confocal microscope to see and measure the overlay accuracy in a bonded wafer pair makes it particularly well-suited for post bond inspection. At better than 0.5\,\mu m resolution, it is already capable of measuring overlay at the resolution defined by the ITRS.
In Figure A.3, measurements can be taken relative to the intensity of reflected signal as shown for measuring the overlay offset in the X and Y direction using the overlay alignment fiducials. Notice that the edge of the four square boxes of the overlay alignment fiducial are black. This is an artifact of the IR Confocal microscope technique, and is assumed to be from the confocal system pinhole that does not receive the reflected signal from the edge of the overlay alignment fiducial boxes. This is proposed for further study in 2010, characterization of the black edge is planned to help improve the overlay algorithms Olympus is developing for automating X, Y and rotational offset measurement.
Figure A.3: Measured X and Y Offset in Bonded Wafer Pair using IR Confocal Microscope.

Figure A.4 shows line/space pairs that are the smallest features printed on SEMATECH’s bonded wafer pairs. These seal lines are designed to be separated by a 0.5\(\mu\)m space, and the line/space pairs are clearly resolved by the Olympus Confocal IR microscope.
Figure A.4: IR scope Measurement Taken at 0.5\(\mu\)m Line/Space Pairs.
APPENDIX B
DEFECT METROLOGY

B.1 Scanning Acoustic Microscope

Improper bonding can leave a trapped pocket or void of highly reflective material that SAM can easily detect with its ultrasonic transducer. The void does not transmit any ultrasound, and the reflected peak is easily distinguished as a defect at the interface of bonded wafer pairs. SAM is somewhat limited by resolution to be totally useful as a patterned wafer defect detection tool, but it is particularly useful when looking at blanket films bonded together.

Many different defects can be formed during the wafer bonding process. Figure B.1a is a good bonded interface from a pair of wafers, which bonded with BCB material. In Figure B.1b, small voids can be found over the entire bonded wafer pair interface, and were attributed to bubbles out gassing during the adhesive curing process. The SAM image can tell if a coating defect from insufficient bonding adhesive created the pie-slice defect as shown in Figure B.1c. Figure B.1d shows the interface of copper-bonded wafer pairs where voids are shown in bright contrast. The bonded wafer pair shown in Figure B.2a also shows an interesting defect call dendritic structure, which is formed during the heating and cooling steps for the BCB adhesive process. These crystalline dendritic structure formations clearly point to an out of control bonder process. When the bonded wafer pair is physically touching, without the bonding material in between the wafers at the interface, it causes the kissing bond defect as shown in Figure B.2b.
Some patterned wafer defects are easily shown by SAM. Poor bonding at the edges of a bonded wafer pair using copper to copper bonds revealed an interesting defect subsequently termed “liquid intrusion.” Wafers with die patterns stepped to the edge had insufficient bonded areas to prevent water from the SAM technique from wicking along the interface of the patterned wafer bonds. Water from the transducer coupling liquid was observed to be migrating towards the center of the wafer (dark band along the wafer edge in Figure B.3, filling the voids in the pattern with liquid. As a result, the liquid fills the voids in the pattern, reflecting less SAM energy back to the transducer. This could be misinterpreted as improved bonding over time, but is clearly a result of the defective interface bonds on the wafer pair – gaps that allow the liquid intrusion to occur. Even after the bonded wafer pair is left to dry, the effects of the liquid incursion can still be seen 24-hours later.
Figure B.2: Dendritic Structure Grows During Solidification (a) and Kissing Defect (b) is found in BCB Bonded Wafer Pairs.
One attempt at preventing the liquid incursion at the interface of patterned bonded wafer pairs was to only print complete die, and leave an edge exclusion area for oxide bonding to seal the bonded wafer pair (Figure B.4). Only printing whole die to help control liquid incursion in a bonded wafer pair is effective, but poor bonds in the wafer edge exclusion area still allow liquid incursion to occur. Figure B.5 shows two spots on a patterned wafer pair where poor bond quality in the edge exclusion area has allowed liquid incursion to occur during SAM metrology. A good seal at the interface of bonded wafer pairs is absolutely essential to prevent liquid incursion happening during SAM metrology, and any other wet process step that is required.
Figure B.4: Bonded Patterned Wafer Pair with Whole Die Only, With Edge Exclusion for Oxide Bonding to Prevent Liquid Intrusion.

Figure B.5: Bonded Patterned Wafer Pair With Whole Die Only, Some Liquid Incursion Observed with SAM Metrology.
Using SAM in bonded wafer pair metrology is limited by SAM’s inability to resolve less than 100µm defects at the interface of bonded wafer pairs (Figure B.6). Wafer thinning and higher frequency/shorter focal length transducers can be used to improve SAM resolution, but they are work around. IR confocal microscopy is better suited to defect review for less than 100 micron defects. SAM is useful for blanket wafer defects, and patterned wafer defects that are large enough to be resolved by SAM to make them distinct from the pattern.

The Sonix Visions SAM has a coordinate system for identifying defects that provides information on size and location. It references all defects from the wafer notch, and it is possible to navigate a defect review tool (e.g. IR Confocal Microscope) to the center of the identified defect. It is not KLARF (KLA-Tencor’s file format) compatible, but has the beginnings of such compatibility. A project to convert Sonix defect map coordinates into KLARF format will be initiated in 2010.

Figure B.6: Dendritic structure defect not shown by SAM.

B.2 Confocal Infrared Laser Scanning Microscope

The confocal infrared laser scanning microscope that’s installed in the Olympus FR3220-IR microscope has provided 0.5µm resolution for overlay and defect metrology. Better resolution has enabled the microscope to look at smaller (vs. SAM)
bonded wafer pair defects, and as it supports KLARF file format, defect review is possible. Infrared is useful for looking through silicon at the interface of bonded wafer pairs, as silicon is transparent to the 1310nm IR used in the IR Confocal microscope.

Figure B.7: Incomplete Bond Adhesive Coverage (a), Over-Etched Alignment Structure (b), Dendritic structure at Probe Pads (c), and Exposed, Over Polished TSV (Copper Smear) After Thinning (d).
IR micrographs show incomplete bonding material coverage over topography as seen in Figure B.7a. In Figure B.7b, the bonding material flowed into an over-etched alignment structure causing the defect pattern around the structure. Dendritic structure as seen in IR are shown in Figure B.7c. Exposed TSVs after thinning in the polish/grinder reveal an over polish condition in Figure B.7d. De-wetting pattern around alignment structure (Figure B.8a) and its 3D reconstruction reveals Unevenness etching, which indicated by red arrows in Figure B.8b. Embedded particle is found below the patterned layer by focus at the defect plane, which shown in Figure B.9. All of the defects can be attributed to a process tool being out of control, and allows feedback on corrective action indicated to tweak the process tool back into control.

Figure B.8: De-wetting pattern around metal structure (a) and unevenness etching effect (b).
Figure B.9: Focus at defect plane reveals blurry (a) is caused by embedded particle below pattern layer (b-c).
APPENDIX C

THICKNESS METROLOGY

C.1 Scanning Acoustic Microscope

One of the capabilities of SAM is measuring wafer thickness. Whenever there is a change in interface, a reflected peak will show in the SAM amplitude vs. time graph. The peak to peak time can be converted to thickness as the speed of sound is known through silicon. The five pairs of bonded wafer pairs that were used for evaluating wafer thinning and its contribution to improved resolution in SAM and IR microscopy were used for thickness measurements by SAM, and compared to a capacitance based wafer thickness/bow/warp tool (MTII). The measurement is taken at the center of each pair of wafers and the result is shown in Table C.1).

<table>
<thead>
<tr>
<th>Top Wafer Thickness</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTII (µm)</td>
<td>113.3</td>
<td>181.84</td>
<td>313.88</td>
<td>384.41</td>
<td>778.17</td>
</tr>
<tr>
<td>SAM (µm)</td>
<td>98</td>
<td>212</td>
<td>310</td>
<td>384</td>
<td>792</td>
</tr>
<tr>
<td>Nominal (µm)</td>
<td>100</td>
<td>200</td>
<td>300</td>
<td>400</td>
<td>775</td>
</tr>
</tbody>
</table>

Figure C.1: SAM and MTII Measurements.

Figure C.1 is plotted based on the measurements from Table C.1. The measurements taken by MTII and SAM are represented in blue and red line respectively. While the black line is represent the nominal thickness of the top wafer for the five bonded wafer pairs. SAM can provide some thickness information, but is not accurate enough to measure the exact thickness of wafer. A properly designed ultrasonic transducer for remaining silicon thickness of interest will improve the accuracy of
SAM wafer thickness metrology.

Figure C.2: SAM and MTII Measurements.
APPENDIX D
EFFECT OF WAFER THINNING ON RESOLUTION

Figure D.1: Preparation of Bonded Wafer Pair.

Figure D.1 gives a simple picture on the preparation of bonded wafer by combining two wafers. The top wafer is MM2 which consists of mirrored metal 2 structures, while the bottom wafer is M1V1 and has metal 1 structures and vias. After surface inspection, the wafers are aligned using overlay fiducials, and then bonded together using a set of parameters (temperature, force, time) in the bonder. The bonded wafer is then thinned from the back of the MM2 wafer, from 775 µm to the desired thick-
ness. A cross section of how the Metal 1 layer of bottom wafer contacts with mirrored metal 2 of the top wafer through a via is shown in Figure D.2).

For the resolution evaluation, five pairs of bonded wafers were prepared with different thicknesses. Four of those wafers were thinned from the top wafer to 100µm, 200µm, 300µm, and 400µm silicon remaining, while one of the wafers maintained the original thickness of 775µm (not thinned). Wafers were bonded using a copper to copper bond process. Filled via depth is 400nm (not TSV).

![Figure D.2: Cross Section of a Bonded Wafer with Thinned Top Wafer.](image)

### D.1 Wafer Thinning Effect on SAM Resolution

SAM is considered to be a very good void metrology tool due to its high sensitivity to voids or air pockets. The acoustic wave used by SAM propagates through any elastic material, allowing it to have a fairly large depth of focus. It can easily penetrate a pair of bonded (∼1550µm) wafers and identify defects at the interface. SAM is a useful void metrology method that has been chosen to inspect void related defects at the interface of bonded wafers. In the resolution section that follows, it’s shown that higher frequency transducers can give better resolution. Thus, if the wafer is thinned down to a certain thickness, a higher frequency transducer can be used to get better resolution in SAM imaging. In this thinned wafer evaluation, resolution is observed to change with different frequency transducers on various thicknesses of bonded wafers.
Figure D.3: SAM Micrograph of a Patterned 300mm Wafer.

The SAM micrograph in Figure D.3 shows the interface for a pair of 300mm bonded wafer with a total thickness of 1550\(\mu\)m. The bright area is caused by an air pocket (void) and the dark area represents bonding (without voids). The wafer notch can be seen at the bottom of the wafer. SAM has the capability to display the bond quality for the entire wafer, or it can be focused on a reduced area of the wafer to concentrate on a single die of the bonded wafer pair.

Figure D.4 Figure D.6 demonstrates the change in resolution when the SAM scans on a die with three different thickness of top Silicon remaining on a bonded wafer pair. These bonded wafers are 1550\(\mu\)m (Figure D.4), 1175\(\mu\)m (Figure D.5), and 1075\(\mu\)m (Figure D.6) thick, with top wafer thickness of 775\(\mu\)m, 400\(\mu\)m, and 300\(\mu\)m, respectively. Thinner top silicon thickness enables different transducers to be used, the frequency used to do inspection can increase with thinner silicon remaining. The 1550\(\mu\)m bonded wafer is scanned with 110 MHz while 1175\(\mu\)m and 1075\(\mu\)m
bonded wafers are inspected with 230 MHz and 300 MHz, respectively. The red boxes in Figure D.4–Figure D.6 show the resolution getting better when a higher frequency transducer is used on the thinned wafer. The probe pads (in black) within the highlighted red boxes are more clearly seen in the wafer with the least amount of silicon remaining on the top wafer (Figure D.6 – 300 µm). These probe pads are 120 µm squares, and are seen to be more poorly resolved in the wafers with 400 µm and 775 µm silicon remaining on the top wafer.

Figure D.4: Single Die on a Bonded Wafer Pair with 775 µm Top Silicon Remaining.
Figure D.5: Bonded Wafer Pair with 400\(\mu\text{m}\) Top Silicon Remaining.

Figure D.6: Bonded Wafer Pair with 300\(\mu\text{m}\) Top Silicon Remaining.
Looking at even smaller features than the 120µm$^2$ probe pads enables a further comparison for changes in resolution enabled by looking through thinner top silicon remaining on the bonded wafer pair. The wafer overlay alignment fiducials are a 70µm x 70µm cross, and the alignment box fiducial is a 20µm square as shown in Figure D.7.

The change in resolution for thinned wafers is apparent on the 70µm x 70µm overlay alignment fiducials. Figure D.8 shows the .gds for the overlay alignment fiducials. In most 3D integrations, TSVs will be used to connect metal 1 and metal 2 together. These overlay marks are etched at the same time as the TSVs to about 25–50µm deep. For this evaluation, the vias used were only 400 nm thick. Wafers with TSVs were not available at the time for evaluating resolution effects.
Figure D.8: gds Image Showing the Overlay Alignment Fiducials.

Figure D.9: Bonded Wafer Scanned with 230 MHz Transducer; SAM Images Show the Alignment Fiducials for 100µm (a), 200µm (b), 300µm (c), and 400µm (d) Silicon Remaining on Top Wafer.

Figure 9a–Figure D.9d show a set of SAM images on bonded wafers scanned with a 230 MHz transducer. These bonded wafers are 100, 200, 300, and 400µm thick silicon remaining on the bonded wafer pair’s top wafer. Better resolution of the overlay fiducials occur when there is only 200µm and 300µm silicon remaining on the top wafer. The 230 MHz transducer has a focal length of 5.9mm, versus 8.0mm for the 110 MHz. An assumption is made that the wafer with 100µm silicon remaining
on the top wafer is too thin, while the wafer with 400\(\mu\)m silicon remaining on the top wafer is too thick for the 230 MHz transducer that was available for this study. They are out of focus, and the overlay alignment fiducials cannot be seen in the SAM image. Further studies are needed to verify the results. A repeated experiment with more properly selected transducers with focal lengths optimized for expected wafer thickness is planned for 2010. The 230 MHz transducers do provide better resolution, but can barely resolve 70\(\mu\)m features.

In Figure D.10, the bonded wafer pair with 300\(\mu\)m remaining silicon on the top wafer is scanned with a 300 MHz transducer. The SAM image of the overlay alignment fiducials shows better resolution imaging than using either 110 or 230 MHz transducer. This is expected from previous discussions explaining that higher frequency gives better resolution. The SAM images clearly show the improvement in resolution when higher frequencies are applied to thinner wafers, but still not reaching useful resolution (e.g., gauging overlay). Even when using the 300 MHz frequency transducer, the 70\(\mu\)m x 70\(\mu\)m overlay alignment fiducial is still blurry in the SAM image. Part of the blurriness is related to the weak contrast between copper and silicon. This weak contrast is further exacerbated by the relatively shallow vias; only 400nm thick copper features (vias) were available for this evaluation.

The bonded wafers are not connected with a TSV (typ. 25–50\(\mu\)m deep); it is a 400nm deep copper via. When the initial SAM signal reaches the interface at the top of the 400nm copper metal layer, a reflected signal will propagate in the opposite direction back towards the transducer. Once the detector receives a reflected signal...
signal from the copper interface, the first signal in this case, an amplitude peak will be shown in a SAM graph based on intensity versus time. Another reflected signal from the interface between copper and silicon, a second signal, is then received by the SAM detector. An amplitude peak for the second signal is shown in the graph later than the first peak if the time between the first and second signals that reach the detector is long enough to allow the detector to distinguish between those reflected signals. Unfortunately, the copper in the bonded wafer pair vias used for this study is not thick enough to allow the detector enough time to distinguish between the reflected silicon and copper peaks. Most of the reflected signal from these two interfaces overlaps with each other before reaching the detector as shown in Figure D.10. Only a small amount of these two reflected signals is resolved by the detector, and the signal will be reduced by attenuation before it reaches the detector. This causes weak contrast between the silicon and copper materials, and contributes to a loss in SAM resolution. As a result, the overlay alignment fiducial in the SAM image appears blurred. A repeated experiment using TSV (25–50µm thick vias) is planned for 2010, and a resultant improvement in SAM resolution is anticipated.

The SEM bars present on the bonded wafer pairs have even smaller geometries available (1–5µm features) to assess SAM resolution improvement through wafer thinning, but were not resolved with any combination of transducer frequency, focal length or silicon remaining on the top wafer.

Figure D.11: Reflection Signal from 400nm Thick Copper Vias and Overlap with Silicon Signal and are Difficult to Separate.
D.2 Effect of Non-Filled Via in SAM

An evaluation was added to the experiment for wafer thinning to improve SAM resolution, incorporating unfilled vias (air). Features with large differences in acoustical impedances are more easily resolved (e.g., air and copper). Silicon and copper have similar acoustic impedance, so contrast is reduced. To study of the effect of a non-filled via in SAM, a pair of wafers were bonded. The MM2/top wafer was bonded to an etched via only/bottom wafer (not copper filled, and no metal 1 features). The top wafer was not thinned; it remained as a full thickness wafer at 775 µm. Figure D.11 compares this non-filled via bonded wafer pair with the filled via and metal 1 bonded wafer pair.

![Normal Bonded wafer with filled Via and Metal](image)

**Figure D.12:** Cross Section of the Non-Filled Bonded Wafer Pair.

![Bonded wafer with un-filled Via and Metal 1](image)

**Figure D.13:** Cross Section at Alignment Structure.
A .gds cross section of the overlay alignment fiducials is shown in Figure D.2. The structures in blue are on the via level of the bottom wafer, while the orange structure is on the top MM2 wafer. With the 110 MHz transducer, a SAM scan on the overlay alignment fiducials shows the effect of non-filled via (Figure D.13). The micrographs show that the contrast increased significantly, and resolution improved. Usually a non-filled via would (usually) not be desirable in a bonded wafer pair, but the idea of intentionally incorporating “air” into overlay alignment fiducials to improve contrast in SAM could increase resolution to a point where SAM could be considered useful for overlay metrology. SAM experiments in 2010 will further explore how adding “air” to these features can improve SAM resolution.

Figure D.14: Effect of Non-Filled Via, Air Improves Contrast and Resolution.