

Dielectric Materials Characterization for Hybrid Bonding

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Abstract— Wide range of choices are available for dielectric materials selection for hybrid bonding, which include: thermal oxide, PECVD TEOS SiO₂, SiN, SiCN and commercial polymer dielectrics. Among the polymer dielectrics, two types: high-temperature curable (350°C) and low-temperature curable (250°C) are investigated. Conclusion of bond strength characterization of different dielectric materials are as follows: Polymer > SiCN > TEOS SiO₂ > SiN. Bonding energy >2.5 J/m² and die shear strength ≥ 50 MPa, which is higher than the Si yield strength could only be achieved through polymer dielectrics. Dielectric delamination during post-bond annealing for enhancing Cu-Cu diffusion bonding is a major challenge frequently encountered for SiO₂ dielectric. Peeling stress generation is the root cause for such delamination. It is confirmed that polymer dielectrics with low Young's Modulus will mitigate such peeling stress generation. However, polymer dielectrics too have their own challenges. Fine scratches generation during chemical-mechanical polishing (CMP) is yet to be fully resolved. SiCN is identified to be the ideal candidate from CMP perspective. Replacing Cu with Cu/Sn solder was also explored for catering low T hybrid process flow. Pros and Cons of different dielectric materials in the process integration of Cu/dielectric hybrid bonding are discussed.

Keywords — Dielectrics, hybrid bonding, peeling stress, edge trimming, Cu damascene, annealing

I. INTRODUCTION

State of the art 3D memory, image sensors all involve Cu hybrid bonding, where the Cu dual damascene processes are integrated for interconnection purpose. This approach has humongous advantages over conventional μ -pillar and μ -bump based packaging technology [1]. Wide range of choices are available for dielectric materials selection, which include: thermal oxide, PECVD TEOS SiO₂, SiN, SiCN and commercial

polymers. Among the polymer dielectrics, two types: high-temperature curable (350°C) and low-temperature curable (250°C) are investigated [2]. All the above dielectric materials are evaluated in this work targeting fine-pitch hybrid bonding (6 μ m). Polymer dielectrics involve pre-curing and high-temperature bonding, while other commonly used dielectrics such as SiO₂, SiN and SiCN involve room temperature bonding (molecular bonding) followed by post-bond annealing. Bond strength is characterized by blade tests and die shear tests, while the bond quality is assessed through scanning acoustic microscopy using C-Mode (C-SAM) and scanning electron microscopy (SEM) cross-sections. Contact angle study was carried out on SiO₂, SiN and SiCN dielectrics to determine their respective wetting angle. Least wetting angle of 5° was achieved for SiCN surface. It was determined that bonding energy is inversely proportional to wetting angle.

Bonded wafers are thinned down to $\leq 50\mu\text{m}$ using edge trimming technique [3]. Both pre and post edge trim approaches are evaluated. Chemical-mechanical polishing (CMP) process is optimized to achieve targeted surface roughness (Ra) of around 2 nm for polymer dielectrics, while it is ≤ 0.5 nm for other dielectrics [4]. Major CMP challenges with respect to polymer dielectrics are generation of fine-scratches and μ -dents. It was determined that the lower post-bond annealing T of 250°C is sufficient for complete transformation to covalent bonding for SiCN dielectrics. Thus, SiCN and low-temperature curable polymers can cater for T constrained process integration flow. However, minimum post-bond annealing T of 300°C is required to facilitate grain boundary Cu-Cu diffusion at the interface. To overcome this temperature barrier Cu was replaced by Cu/Sn in the low T process flow. It was observed that the commonly encountered peeling stress generation resulting in the delamination of SiO₂ dielectrics in Cu/SiO₂ hybrid bonding

could be resolved by replacing the SiO₂ with polymer dielectrics [5].

II. TEST VEHICLE FABRICATION

Fig. 1 schematically shows the process flow employed for fabricating 12" wafers used for demonstrating Cu/non-polymer hybrid bonding. SiO₂/ SiN/SiCN dielectrics are deposited using plasma enhanced chemical vapor deposition process (PECVD), where the deposition temperature plays a critical role in determining the % of porosities in thin film. % of carbon in SiCN also plays a deciding role [6]. Total dielectric thickness varied between 1-1.5µm. Densification annealing is carried out at a temperature regime between 250°C-350°C [4]. It was ensured that the maximum Cu density does not exceed 25%. Cu pad size and pitch employed are 3µm and 6µm respectively. Uniform dielectric etching was carried out. Ti/Cu thin film was used as barrier cum seed layer. It is deposited using physical vapor deposition (PVD) technique. Electroplated Cu was annealed at 300°C for 2h. In this process flow, daunting task is placed on the CMP process, where the dielectric Ra is preferred ≤ 0.5nm and the Cu roughness ≤ 1 nm [4]. Cu dishing is preferred to be kept as low as 5nm. Higher Cu dishing needs to be compensated by higher post bond annealing T [5]. Post bonding annealing T depends upon the corresponding Cu dishing value and it varies between 250°C -350°C.

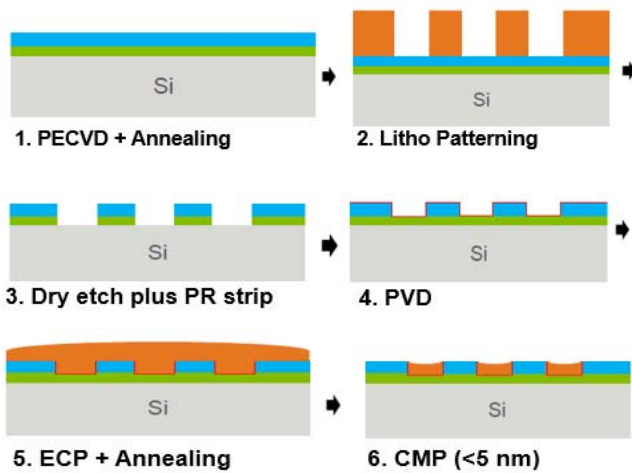


Fig. 1. Schematic process flow employed for fabricating Cu/non-polymer dielectric hybrid bonding test vehicle.

Fig. 2. schematically shows the process flow employed for fabricating 12" wafers used for the demonstration of Cu/polymer hybrid bonding. Alternate process flow is employed here compared to the conventional, where the polymer coating and subsequent curing are done first. This process flow is recommended from the CMP perspective. Targeted polymer coating thickness varied between 5-10µm. Further optimization is possible by controlling the spin speed and spin time. Lower spin speed results in thicker film [2]. Pre-baking condition is typically ≤ 150°C for 2 min. Curing profile needs to be optimized. Max curing T varies between 250°C-350°C for commercially available polymer dielectrics. Typical curing t varies between 1-2h. Polymer dielectrics significantly shrink

during curing. Typical reduction in thickness varies between 30-50%. Percentage of thickness reduction is proportional to the % of solvents employed during synthesis. Similar Cu hybrid bonding pad layout was used. Major challenge encountered in the Cu/polymer CMP is the generation of fine cracks and micro-defects. Typical Ra achieved for polymer dielectric post-CMP is around 2nm. Advantage of polymer dielectric is, it is not very sensitive to Ra. This could be attributed to the thermo-compression bonding.

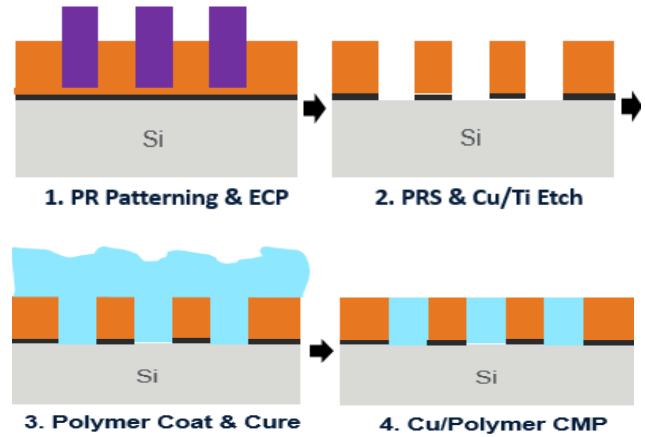


Fig. 2. Alternate schematic process flow employed for fabricating Cu/polymer dielectric hybrid bonding test vehicle.

III. DIELECTRIC MATERIALS CHARACTERIZATION: SURFACE ROUGHNESS AND CONTACT ANGLE

Tetraethyl orthosilicate (TEOS) SiO₂ deposited at two different T regimes were evaluated. Low T TEOS was deposited at 180°C. High T TEOS was deposited at 350°C. Dielectrics were subjected to densification/curing prior to Ra and contact angle measurements (Table I). Reported Ra are measured post-CMP. Contact angle was measured prior to plasma treatment. Lower contact angle is required for room temperature bonding only, since the bonding mechanism is molecular bonding. Therefore, the surface needs to be hydrophilic in nature. However, for polymer dielectric, it is thermo-compression bonding and hence, the high contact angle can be ignored.

Table I: Ra and contact angle measurements.

Dielectrics	Surface Roughness Ra (nm)	Avg. Contact Angle (°)
Low T TEOS – SiO ₂	0.6 – 0.8	11
High T TEOS – SiO ₂	0.6 – 0.8	9
SiN	1.1 – 1.25	15
SiCN (High Carbon)	0.45 – 0.6	5
High T curable Polymer	28 – 32	≥ 45
Low T curable Polymer	1.9 – 2.2	23

For PECVD deposition Ra non-uniformity was observed between the wafer center and edges. Lower contact angle is preferred for molecular bonding indicating SiCN is the prospective dielectric candidate among non-polymer dielectrics. Highest contact angle was measured for SiN surface. Selection of appropriate plasma condition is critical in order to enhance the hydrophilic nature of the surface. N₂ plasma is identified to be the ideal plasma for SiO₂ surface. Ar plasma for SiO₂ surface results in surface roughening. This is applicable to both low and high T TEOS. O₂ plasma is identified to be the appropriate plasma for SiN surface. Avg. contact angle of SiN can be reduced to 9°. However, it will result in the oxidation of the Cu surface and hence, it is not applicable. It was not possible to measure the contact angle for SiCN surface after plasma treatment irrespective of the plasma gas since it was almost flat and fall within tool limitation. Anything below 5° cannot be precisely measured. No major difference in Ra was observed between the low T and high T TEOS. Good correlation in results was observed between contact angle and surface roughness. Only exception was TEOS SiO₂.

IV. CMP OPTIMIZATION

The most critical process in the hybrid bonding process flow is the CMP process [4]. In this work, the wafers were polished on Applied Reflexion® LK platform. Very low Ra for both the dielectrics and Cu needs to be achieved. Cu dishing always need to be controlled ≤ 10 nm. CMP process needs to be optimized such that the uniformity between the center and the edges are improved and the range is minimized. Table II lists the Ra of different dielectric materials. Usual process parameters that are fine-tuned are slurry composition, slurry flow rate, pad pressure and time. Different slurry composition is used for polymer and non-polymer dielectrics. Uniform removal rate was achieved, when the pad pressure was maintained as low as 1psi. Removal rate is sensitive to the slurry concentration variation. Non-uniformity within wafer was always controlled ≤ 10%. Least Ra was achieved for SiCN. Significant reduction in Ra during CMP was achieved for high T curable polymer. Similar Ra was achieved for both high T and low T deposited TEOS SiO₂.

Table II. Post CMP optimization – achieved Ra value

Dielectrics	Surface Roughness (nm)	
	Pre-CMP	Post CMP
Low T TEOS – SiO ₂	0.6 – 0.8	0.3 – 0.4
High T TEOS – SiO ₂	0.6 – 0.8	0.3 – 0.4
SiN	1.1 – 1.25	0.6 – 0.75
SiCN (High Carbon)	0.45 – 0.6	0.2 – 0.3
High T curable Polymer	28 – 32	1.5 – 2
Low T curable Polymer	1.9 – 2.2	1 – 1.2

Targeted post CMP Ra for non-polymer dielectrics is 0.5 nm and this could not be achieved for SiN dielectric. Achieved Ra value for Cu post-CMP is 1nm. It was observed that the pre-CMP Ra value for high T curable polymer is way higher than the low T curable polymer. However, one order-of-magnitude reduction in the Ra value could be achieved for high T curable polymer after CMP (Fig. 3), resulting in comparable Ra to the low T counterpart (Fig. 4). Ra is a critical metric for bonding and blanket polymer bonding was carried out to validate this, which will be discussed in Section IV.

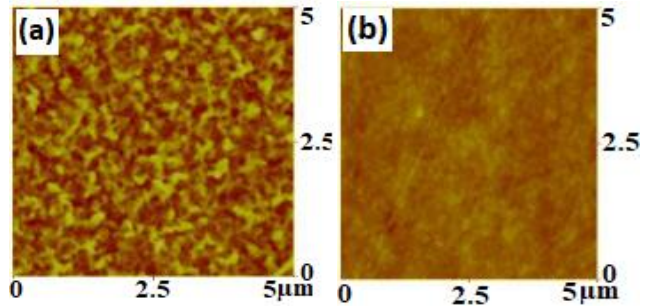


Fig. 3. AFM micrographs of high T curable polymer (a) pre-CMP (b) post-CMP.

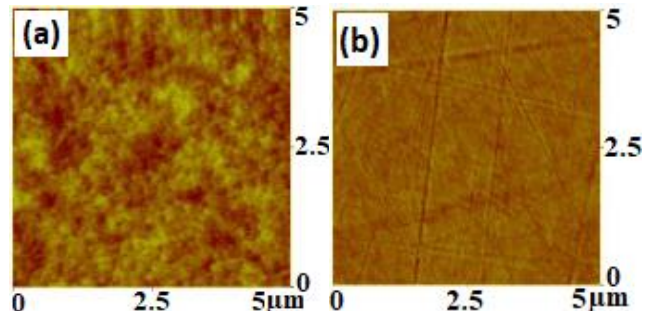


Fig.4. AFM micrographs of low T curable polymer (a) pre-CMP (b) post-CMP.

It was determined that polymer CMP flow is prone to Cu pad sidewall delamination (Fig. 5a). FullVision® endpoint technique was used to achieve accurate remaining polymer thickness with reduced stress imposed on the Cu sidewall after Cu reveal. The result is intact polymer adhesion on Cu sidewall as shown in Fig. 5b.

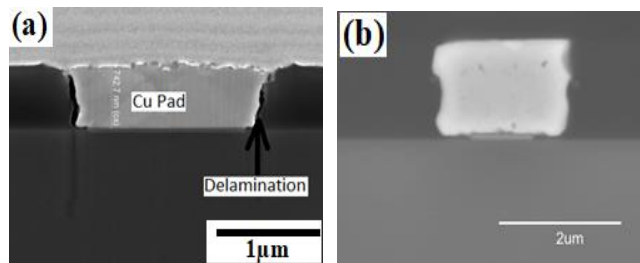


Fig. 5. Polymer CMP (a) Cu pad sidewall delamination (b) No sidewall delamination with improved CMP process.

Least Cu dishing around 2nm could only be achieved for SiCN dielectric. Lower Cu dishing value facilitates reduction in the required post-bond annealing T.

IV. DIELECTRIC MATERIALS: BOND STRENGTH CHARACTERIZATION

Bond strength was characterized by both blade test and die shear. Blade test was carried out at wafer level, while die shear was carried out at die level. Bonded wafer was diced into 5*5 mm² die size for the purpose of die shear. 3* 3 mm² dies were used for high T curable polymer, since the shear strength was extremely high and the max shearing force limitation is 100 kgf. Blade was inserted inside the bonded wafer and immediately scanned using C-SAM. Bonding energy is analytically calculated using the Maszara model, where the crack length is determined from the corresponding C-SAM image [4]. Similar trend was observed in bonding energy and shear strength for all dielectric materials. Highest bond strength was achieved for high T curable polymer while the least bond strength was observed for SiN. Although no difference in Ra was observed between the low T and high T TEOS SiO₂, while in bond strength high T TEOS was clearly ahead. Max. bonding energy that can be determined using blade test is 2.5 J/m², which is limited by Si yield strength

Table III. Bond strength of different dielectric materials.

Dielectrics	Bond Strength	
	Bonding Energy (J/m ²)	Shear Strength (MPa)
Low T TEOS – SiO ₂	0.9 – 1.1	12 – 15
High T TEOS – SiO ₂	1.2 – 1.4	18 – 20
SiN	0.6 – 0.8	7 – 10
SiCN (High Carbon)	1.6 – 1.8	26 – 30
High T curable Polymer	> 2.5	45 – 50
Low T curable Polymer	> 2.5	35 – 40

Highest bond strength was achieved for SiCN dielectric in the non-polymer category. It was observed that the carbon content in the SiCN thin film plays a detrimental role in influencing the bond strength [6]. Carbon content used in this film varied between 25-30 atomic %. Above bond strength for SiCN was achieved using post-bond annealing T of 350°C (Fig. 6). No any major difference in bond strength was observed, when the post-bond annealing T was reduced to 250°C. This could be attributed to the higher dangling bond defects on the SiCN surface, when compared to TEOS SiO₂ or SiN surfaces [6]. Highest bond strength could be only achieved for polymer in the fully cured state. This phenomenon was common for both high and low T curable polymers. Skipping the hard cure process step or partial curing significantly compromises the

bond strength (Fig. 6). Thus, the bond strength could be directly linked to the cross-linking density.

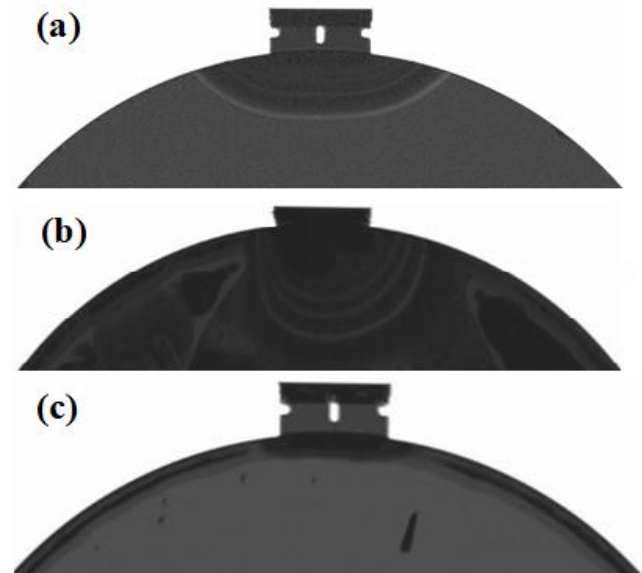


Fig. 6. C-SAM micrographs depicting crack propagation area after blade insertion (a) SiCN (b) High T polymer- partially cured (c) High T polymer – fully cured.

In the case of polymer, it was determined that the resulting bond strength is > 2.5 J/m², which is greater than any known typical inorganic dielectric strength. This may provide a pathway for higher bonding reliability in future packaging requirements. The role of CMP is critical because unpolished polymer surface is too rough. Bonding is not feasible without CMP since the bonding wafers delaminate right away after any bonding attempt. It was determined that the highest bond strength could only be achieved for polymer in the fully cured state. This phenomenon was common for both the high and low T curable polymers. Skipping the hard cure process step or partial curing significantly compromises the bond strength (Fig. 6). Thus, selection of the right polymer is critical especially, when low-temperature bonding is required.

V. Hybrid Bonding

For TEOS SiO₂, SiN and SiCN dielectrics bonding is carried out at room temperature. Prior to room-temperature bonding, the wafer is subjected to in-situ plasma clean and in-situ DI water clean. This is followed by post-bonding annealing. Post-bond annealing T varies between 250°C - 350°C. Post-bond annealing serves two purposes: (a) It facilitates Cu-Cu diffusion bonding (b) It converts weak molecular bonding into strong covalent bonds. Selection of appropriate post-bond annealing T depends on multiple factors (a) corresponding average Cu dishing value (b) density of dangling bond defects on the surface. Selection of insufficient annealing T may result in no diffusion across the Cu-Cu interface, resulting in macro-voids generation (Fig. 7). It was determined that a minimum post-

bond annealing T of 300°C is required for TEOS SiO₂ irrespective of the Cu dishing value. Thus, the required post-bond annealing T for SiO₂ varies between 300-350°C. Lower post bond annealing T for TEOS SiO₂ compromised the bond strength. Unlike TEOS SiO₂, no any significant difference in bond strength was observed for SiCN, when subjected to post bond annealing at 250°C. This could be attributed to the higher dangling defect density in the SiCN surface, which is reflected in contact angle measurements too. Another major challenge, encountered in the TEOS SiO₂ dielectric, when subjected to high post-bond annealing T in excess of 300°C is the generation of peeling stress resulting in the delamination of TEOS SiO₂ dielectric bonding (Fig.8). Hence, it is essential that lower Cu dishing value around 5 nm is achieved for TEOS SiO₂ so that it could be subjected to the lower side of the temperature regime. Fig. 9 illustrates fine-pitch Cu/SiO₂ hybrid bonding using post-bonding annealing T of 300°C. Average Cu dishing value is 5 nm and this is achieved by using a combined Real Time Process Control (RTPC™) and laser endpoint technology on Applied Reflexion® LK.

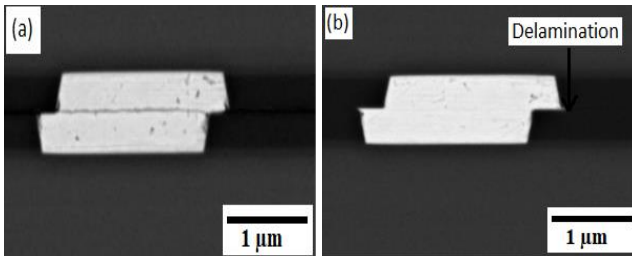


Fig. 8. Cu/SiO₂ hybrid bonding (a) insufficient post-bond annealing T (b) Excess post-bond annealing T.

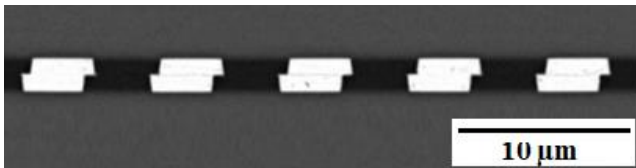


Fig. 9. Fine-pitch Cu/SiO₂ hybrid bonding using post-bond annealing T of 300°C.

Certain specific applications such as memory device packaging require low T hybrid bonding process flow, where the peak process T is constrained at 250°C. This is only feasible by using either SiCN dielectric or low T curable polymer. Many literatures claim that the Cu-Cu diffusion bonding can be achieved as low as 200°C. However, it was observed that good Cu-Cu diffusion at the interface through grain boundary diffusion mechanism could only be achieved at 300°C. This issue was resolved by plating a thin layer of Sn over Cu. Now, the metallic bonding mechanism has been changed from the diffusion bonding mechanism to transient liquid phase (TLP) bonding. Here, the minimum required post-bond annealing T should be higher than the melting point of the low-melting interlayer, which is Sn. Melting point of Sn is around 232°C and hence, 250°C is sufficient [7]. Cu-Sn intermetallic was observed at the interface (Fig. 10). Thus, lower T hybrid

bonding process flow is feasible by selecting either Cu-Sn/SiCN or Cu-Sn/low T curable polymer. The misalignment issue was resolved by designing Cu hybrid bonding pads of different dimensions and intentional offset during bonding.

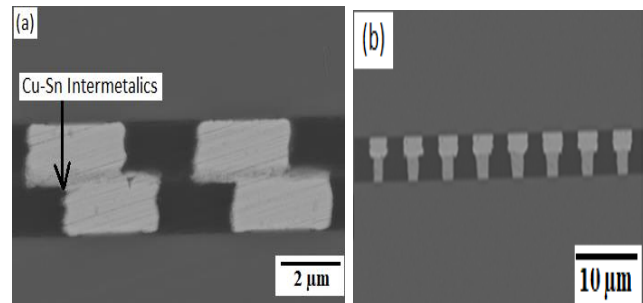


Fig. 10. Low T hybrid bonding: (a) Cu-Sn/polymer dielectric (b) misalignment offset by different Cu pad design.

For Cu-Sn/polymer hybrid bonding, no post-bond annealing is required, since it would be a thermo-compression bonding. Bonding is carried out inside a bond chamber. Bonding T is similar to the curing T. It was determined that a bonding t of 5 min is sufficient. Diffusion kinetics is much faster for Cu-Sn TLP bonding, when compared to Cu-Cu diffusion bonding [7]. Bonding force of 20 KN was applied throughout thermo-compression bonding. Wafer to be bonded are first aligned and separated by flags, prior to bonding. In-situ baking of 5 min at 150°C was carried out in a low vacuum of 10⁻² mbar, prior to thermo-compression bonding. In the case of Cu-Sn/SiCN hybrid bonding, post-bond annealing is carried out in furnace. Although 5 min is sufficient for Cu-Sn TLP bonding, it is sufficient for SiCN dielectric. It was determined that minimum post bond annealing t of 1h is required for complete transformation of molecular bonds to covalent bonds for SiCN surface.

VI. EDGE TRIMMING

In recent years, the thickness of memory device and the controller chips has been reduced due to the drive for miniaturization and to serve the purpose of capacity enlargement of smart devices [3]. Hence, the hybrid bonded wafers are thinned down to a thickness as low as ≤ 50μm. During this process, edge chipping is common and may result in crack propagation leading to wafer breakage. Hence, edge trimming is necessary to remove the rounded shape on the outer edge of the wafer, which causes edge chipping [8]. Edge trimming is not necessary, if the thickness of the wafer to be reduced is ≥ 200μm. Typically used pre-edge trimming depth varies between 150-200 μm. Edge trimming can be carried out prior to hybrid bonding or post-hybrid bonding. For hybrid bonding involving polymer dielectric, both approaches are suitable, since polymer dielectric is not very sensitive to particles. However, for hybrid bonding involving non-polymer dielectrics, post-bond edge trimming is preferred. This is because it is very sensitive to particles generated during the edge trimming process. Further, edge trimming approach is

usually carried out in class 1000 and above cleanroom ambience. Pre-bond edge trimming approach can only be considered, when protective layer is used. Thin photoresist is used as temporary protective layer. Fig. 11 illustrates the particles generated in non-polymer dielectric, when protective layer is not used. Another risk of using temporary protective layer is that the achieved targeted Ra value in CMP will be slightly altered during the removal of photoresist, which is usually a wet clean process.

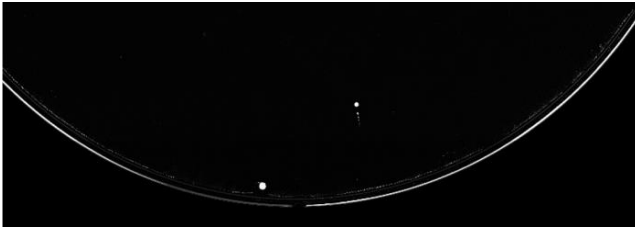


Fig. 11. Infrared (IR) micrograph showing particles generated in the pre-bond edge trimming process, when protective layer is not used.

Edge trimming width varies between 0.5 mm to 3mm. 0.5 mm is sufficient for polymer dielectrics, while 3 mm is required for non-polymer dielectrics. This is because generally uniform bonding is achieved for polymer dielectrics while wafer edges are not bonded well for non-polymer dielectrics (Fig. 12). Typically edge trimming width ≥ 3 mm is not preferred, since it will compromise the throughput, as it is a time-consuming process. For a 300 mm wafer, 0.5 mm edge trimming width consumes about 15 min. However, it is quite common that higher edge trimming width may be necessary in post-bond edge trimming approach to overcome the edge voids issue in non-polymer dielectrics.

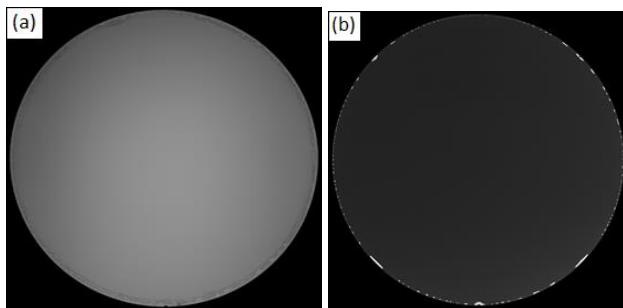


Fig. 12. Edge trimming width is determined by edge bond quality (a) polymer dielectrics (b) non-polymer dielectrics.

VII. CONCLUSION

All the possible dielectric materials for hybrid bonding have been evaluated and the following conclusions have been attained.

- (a) For memory stacking application, where low T hybrid bonding process flow is preferred, recommended dielectrics are SiCN with high carbon content and low T curable polymer.

- (b) SiCN is preferred from the CMP perspective. Least Ra and Cu dishing could only be attained for the same.
- (c) Highest bond strength was achieved for high T curable polymer followed by SiCN. Max. bond strength of polymer can be only realized in fully cured state.
- (d) SiCN is identified to be the most hydrophilic in nature and post-bond annealing T of 250°C will suffice.
- (e) Replacing Cu with Cu/Sn solder will ensure thermo-compression bonding involving polymer dielectric material can be reduced from 1-2 h to just 5 min.
- (f) Peeling stress generation resulting in SiO₂ delamination can be mitigated by SiCN, where the post-bond annealing T requirement is only 250°C. This issue can be overcome by polymer dielectrics due to inherent low Young's Modulus.
- (g) Polymer dielectrics is preferred from wafer thinning perspective. Edge trimming width of 0.5 mm is sufficient.

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