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Facile non thermal plasma based desorption of self assembled monolayers for achieving low temperature and low pressure Cu–Cu thermo-compression bonding

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This paper reports Self Assembled Monolayer (SAM) of propanethiol desorption assisted low temperature, low pressure copper to copper (Cu–Cu) thermo-compression bonding, a technique which could potentially open up a whole new platform for developing next generation heterogeneous smart devices using 3D integrated circuit technology. Thiolated self assembled monolayers protect a freshly deposited copper surface from oxidation and other contamination. Removal of this SAM layer just prior to bonding would potentially bring down temperature and pressure thus rendering the process CMOS compatible. This paper focuses on using Non-Thermal Plasma (NTP) desorption, which is a simple, robust, room temperature technique for desorbing SAMs. The desorption was carried out in an indigenous chamber specifically designed for this purpose. Thermo-compression bonding post desorption resulted in a very good quality bonding with a bond strength of 132 MPa, performed at a relatively low temperature of 200 °C and a low pressure of 5 bar.

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1 Introduction

The past decade has witnessed a revolution in the communication and information technology fields owing to Complementary Metal Oxide Semiconductor (CMOS) technology scaling and its ability to integrate billions of transistors into a single chip. Chemistry has played a vital role in the development of this highly interdisciplinary technology. It is the backbone for critical processes like wafer cleaning, photolithography, wet and dry etching, oxidation, *etc.*^{1,2} The ever growing demand for accommodating more functionality within a single chip and the penchant to follow Moore's law has resulted in the scaling of device dimensions to such an extent that the device performance is no longer a constraint, and the performance of ICs is constrained by the interconnect technology which connects billions of transistors in the required manner.³ The semiconductor industry has successfully pushed the limits to achieve ultra-small and extremely fast transistors, so much so that the challenge and the focus has shifted to the delay caused by the interconnects rather than the transistor itself. 3D integration circuit (3D IC) technology is touted to be an alternative technology by the International Technology Roadmap for Semiconductors (ITRS) to overcome this

bottleneck. The long planar interconnects can potentially be replaced by short interconnects by stacking and integrating different modules vertically, resulting in a low RC time delay, a higher speed and an improved bandwidth. Apart from this, the major advantage of 3D IC technology is its ability to carry out heterogeneous integration.³ This opens up a new arena wherein CMOS compatibility is no more a constraint as different modules can be fabricated under different conditions and can finally be vertically interconnected or integrated using 3D IC technology.

Among the various 3D IC technologies proposed, wafer to wafer integration has received a lot of attention owing to its compatibility for precise alignment.⁴ Direct Cu–Cu bonding is preferred over other conventional ball grid⁵ and micro bump technologies⁶ as it is suitable for achieving simultaneous electrical, mechanical and hermetic binding, as well as the inherent advantages of Cu such as excellent electrical conductivity, better electromigration resistance *etc.*^{7,8} Several researchers have explored various possibilities of achieving Cu–Cu Wafer on Wafer (WoW) bonding with a focus on making the process CMOS compatible with a minimal thermal budget. Room temperature bonding at relatively low pressure has been reported using a Surface Activated Bonding (SAB) technique. But the stringent requirement of an atomically smooth surface in this technique, along with the requirement of an ultra high vacuum, makes the process complex and economically non-viable.^{9,10} Application of a high bonding pressure (3.28 GPa) has

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been reported for copper–copper bonding.¹¹ Recently Y. P. Huang *et al.* have reported the usage of Ti as a passivation layer but the pressure conditions used to achieve this are high.¹² However, such high pressures can have a detrimental effect on the underlying devices and may cause performance degradation. The use of a temperature of 400 °C and 4 kN contact force has been reported.¹³ But such a high temperature can be a potential cause of performance degradation; besides, the thermal budget is high as well.

Thermo-compression bonding is widely pursued as a potential method to achieve Cu–Cu bonding because this technique is more tolerant with regard to surface roughness, and consequently does not require a CMP processing step.¹⁴ The primary challenge that has to be addressed to make thermo-compression bonding viable and compatible with CMOS processing is depicted schematically in Fig. 1. Thermo-compression bonding was successfully demonstrated either at high pressure and room temperature (point A) or at low pressure and elevated temperature (point B). Both extreme conditions are not suitable for CMOS post processing as high pressure or high temperature degrades/destroys the performance of transistors beneath the Cu layers. The need of the hour is to pull the entire trend downward, as designated by the second curve, wherein both temperature and pressure are reduced (point C) to as low as possible. Temperature should ideally be near room temperature and pressure should be minimized to the extent possible.

A major bottleneck in achieving low temperature and low pressure thermo-compression bonding is the barrier for inter-diffusion created by the presence of native oxide and contaminants which doesn't allow two Cu layers to bond with each other. Cu has to be passivated just after deposition to protect it from oxidation and contamination, and the passivation mechanism should be robust enough to endure different ambient conditions. It is at this juncture a knowledge of chemistry comes in handy. Self Assembled Monolayers (SAM) of alkane thiols precisely help in preventing oxidation and contamination owing to the affinity of thiolated monolayers for metal layers. Formation of these organic monolayers is spontaneous and is non-toxic.^{15,16} Furthermore, SAMs have an excellent ability to remove traces of copper oxide from a copper surface. Once the protection is carried out, subsequent desorption of monolayers just prior to bonding creates a native oxide and contamination

free Cu surface which can be bonded using thermo-compression bonding under nominal temperature and pressure conditions.

Researchers have successfully demonstrated the protection of Cu layers using alkane thiols.^{4,7,12} The key process in this endeavor is the desorption of SAMs. Mechanical displacement of SAMs from a Cu surface with the application of high pressure (2.58 GPa) has been reported.¹⁷ Another method, with the application of heat (250 °C) to desorb these monolayers has also been reported.^{18,19} SAM desorption at higher temperature or its mechanical displacement using high pressure defeats the purpose of its utilization as a passivation layer. Hence it is imperative to explore an alternative low temperature process for removing SAMs just prior to bonding.

Non-thermal plasma (NTP) assisted desorption has the potential to overcome the above limitations. NTP desorption offers several advantages such as near room temperature operation, mild operating conditions, and multiple energetic species such as electrons, radicals, neutrals, *etc.*, that are capable of etching the surface.^{20,21} Depending on the input energy, the degree of ionization can be precisely controlled to desorb the SAMs and simultaneously ensure that the base material remains unaffected. Hence, it is possible to overcome the limitation of thermal desorption.

In the present study, non-thermal plasma (NTP) assisted room temperature thiolated SAM desorption was carried out to enable low temperature, low pressure thermo-compression Cu–Cu wafer bonding. Firstly, thiolated SAM desorption was carried out in an indigenous plasma reactor specifically designed for this purpose. This was followed by low temperature, low pressure Cu–Cu thermo-compression bonding. Thorough pre- and post-bonding characterizations were carried out to optimize the efficacy of SAM protection and room temperature desorption, and to analyze the quality of the interface. The critical step is to prove the low temperature and low pressure bonding technology which is typically done between two unpatterned full wafers. Once the technology is established, patterned bonding and electrical characterization can be carried out using well-established fabrication and characterization processes. We believe that this simple yet critical breakthrough will enable the development of next generation heterogeneous, smart systems.

2 Experimental

2.1 Plasma assisted thermo-compression bonding methodology

2.1.1 Sample preparation. 4", (100), n-type Si wafers were used for the entire operation. Standard RCA and piranha cleaning were performed. 10 nm of Ti and 100 nm of copper were sputter deposited. The Ti layer acts like an adhesive layer for copper. Before actual deposition onto the wafers, each target was pre-sputtered for at least 20 minutes. The sputtering system used throughout the work was the AJA Int. PHASE II system. Freshly deposited wafers were immediately immersed in a 1 mM propanethiol (C_3H_7S , Sigma-Aldrich) solution. It is reported that the binding energy of a SAM layer decreases with a lesser number of carbon atoms present in the chain.¹⁶ The

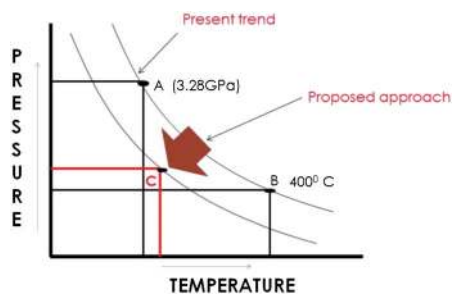


Fig. 1 Schematic diagram – relationship between pressure and temperature in thermo-compression bonding.

main reason for using propanethiol (C_3) in this present study is the expected quick removal of the SAM layer from the copper surface due to its low number of carbons.¹⁶ After SAM adsorption, the wafers were kept in a nitrogen purged dry vacuum box in order to avoid subsequent contamination.

2.1.2 Plasma desorption. One of the key efforts in this endeavor is the room temperature desorption of monolayers using cold argon plasma. In this work an indigenously designed NTP corona discharge plasma reactor was used to create argon plasma for complete desorption of the thiol layer. This plasma was generated at room temperature and at atmospheric pressure. A schematic diagram of the setup is shown in Fig. 2. The reactor was a 1 cm thick glass tube with a 150 mm diameter and a length of 30 cm. The electrodes were made of stainless steel with a 2 mm thickness and a 100 mm diameter. The distance between the two electrodes was kept at 2 cm. A sample was placed vertically in between the two electrodes. An AC voltage was applied through a high voltage 0–40 kV AC source transformer (Jayanthi Transformers). The voltage level was kept at 3 kV with a 20 mA current and the frequency was 50 Hz, which corresponds to an input power of 60 W. Argon (Ar) gas was introduced in the glass reactor through a 1/8" Teflon tube. A mass flow controller (ALBORG) was used to regulate the gas flow rate. The argon flow rate was kept constant at 300 ml min⁻¹.

2.1.3 Bonding. Low temperature and low pressure bonding aided by SAM desorption is one of the key goals of this present study. 4", (100) Si wafers with 100 nm of copper were bonded just after plasma treatment in a wafer bonder system (AML, UK). The bonding conditions are tabulated in Table 1. The bonding operation was carried out at a reasonably low temperature of 200 °C and a relatively low pressure of 4 kN (5 bar) under vacuum conditions. Bonding carried out between SAM desorbed wafers yielded a very good bonding and its characterization is explained in detail in the Results and discussion section. As a control, as deposited and unprotected wafers were bonded using the same conditions. As expected, the control wafers did not bond at all owing to the diffusion barriers created by the native oxide layers.

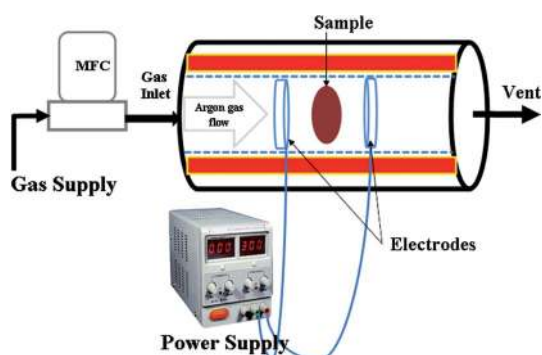


Fig. 2 Schematic diagram of the non-thermal plasma desorption setup. Argon plasma, generated at 3 kV, 50 Hz, 60 W, desorbs a self assembled monolayer of propanethiol from a Cu surface, without damaging the surface.

Table 1 Cu–Cu wafer bonding conditions

Bonding temperature	Bonding pressure	Time	Vacuum
200 °C	5 kN	90 min	5×10^{-6} mT

2.2 Pre-bonding and post bonding characterization

Contact angle analysis is one of the best non-destructive methods to examine surface conditions. In this study the impact of a DI water droplet on a copper surface was measured at different stages of the experiments. These measurements are used as one of the key methods to validate the presence and absence of SAMs on the copper surface. Any change in the surface morphology/composition leads to a change in the contact angle. A VCA Optima system was used for the study. For clearly identifying the surface composition, Secondary Ion Mass Spectroscopy (SIMS) was carried out as it is regarded as one of the best techniques for identifying components to a precision of parts per billion (ppb). It is a destructive test wherein simultaneous etching and depth profiling is carried out to extract the elemental details. In this present study, a depth profile of 30 nm from the surface was carried out. Atomic force microscopy (Bruker) was used to monitor the relative surface roughness of the copper surface at various stages of the operation. Surface roughness was tested on the freshly deposited copper surface, after SAM adsorption, and after plasma treatment. Cross-sectional transmission electron microscopy (XTEM, PHILIPS CM 200) was also carried out in order to gain insight about the interface at the atomic scale level. Superior bond strength is as important as electrical conductivity. Bond strength analysis indicates the mechanical stability of the bonded sample. If the mechanical stability of the bonded sample is poor then it cannot sustain subsequent processing steps. In this study, the bond strength of each sample was tested by using a Microtester (INSTRON). The load was provided by an actuator; speed was kept at 0.6 mm min⁻¹. The maximum limit of the system was at 950 N.

3 Results and discussion

3.1 SAM adsorption

Contact angle measurements were carried out to optimize the time of immersion of the wafers in the thiol solution. As shown in Fig. 3, the contact angle of a DI water droplet on the freshly deposited copper surface is 18°. This low angle is attributed to the hydrophilic nature of Cu.²² Post thiol immersion, the surface turned hydrophobic owing to the hydrophobic nature of the SAM. Periodic contact angle measurements revealed a significant change in the first hour of immersion but a relatively low change in the following hours. After 3 hours of immersion the variation in the contact angle was minimal, suggesting that the chemisorption of the SAM onto the Cu surface was complete. For the subsequent experiments, Cu wafers were immersed in 1 mM propanethiol for three hours.

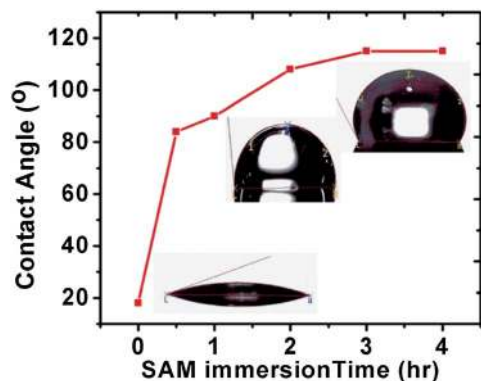


Fig. 3 Contact angle of a DI water droplet versus immersion time in SAM solution. The hydrophilic nature of the freshly deposited Cu inverts to hydrophobic after 2 hours of immersion in propanethiol solution. After 3 hours of immersion the contact angle becomes saturated as the entire area is covered with the SAM.

3.2 Cold Ar plasma desorption

Cold argon plasma desorption was carried out to desorb the SAM just prior to bonding. The density of plasma needs to be optimized in order to ensure complete removal of the SAM layer from the Cu surface without affecting the Cu film beneath it. The density of plasma depends on the applied bias and time. A constant bias of 3 kV is used throughout the experiments. Higher voltages would create a denser plasma which could potentially destroy the Cu surface. Hence it is prudent to play around with the time of desorption. This is a common practice in the IC industry. Oxygen plasma is typically used at a fixed power for a small period of time to remove contaminants post photolithography and this process is often referred to as descumming.²³ A relatively small time exposure of 3 minutes could not effectively remove the SAM. This can be concluded from the contact angle measurement performed post desorption. As shown in Fig. 4, the contact angle is 115° which suggests the hydrophobic nature of the substrate, thus indicating the presence of the non-desorbed SAM. Multiple

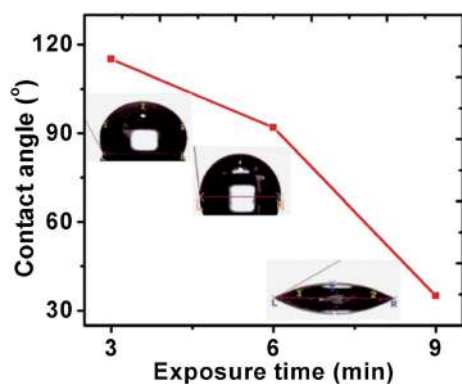


Fig. 4 Contact angle of DI water droplet versus duration of exposure to argon non-thermal plasma. The Cu surface transforms from hydrophobic (115°) to hydrophilic (35°) after desorption of propanethiol at 3 kV.

iterations were carried to optimize and figure out the right time of exposure. Samples exposed at 3 kV and for 9 minutes to cold plasma were found to be effective at carrying out complete desorption of the SAM layer without damaging the Cu film. This was corroborated by the sharp decrement in contact angle after 3 kV of plasma treatment, evident from Fig. 4, to a value of 35° indicating the hydrophilic nature of the surface which would not appear until the SAM is completely desorbed. Thus it is evident that the SAM layer is completely desorbed from the surface. The slight difference between the freshly deposited copper contact angle and plasma treated copper contact angle may be attributed to presence of a nominal amount of carbon on the copper surface which is natural after prolonged exposure of the SAM in ambience. In order to reaffirm the complete removal of SAM, SIMS analysis was carried out. Prior to performing SAM desorption utilizing argon, we reported the utility of helium plasma in carrying out SAM desorption.²⁴ Though helium plasma is efficient in achieving SAM desorption, argon is a preferable choice as it is cheap and is regularly used in CMOS process flow.

3.3 SIMS analysis

Compositional analysis confirms the presence of elements on the surface. Fig. 5 depicts the SIMS results before and after plasma treatment. Depth profiling was carried out up to a thickness of 30 nm. This test is performed to doubly ensure the removal of the SAM. Profiling to 30 nm thickness from the top of the surface is sufficient as the SAM that is present on the surface is a few nm thick. The concentration profiles of various ions present within the profiled 30 nm thickness in the SAM protected sample and plasma desorbed sample are shown in Fig. 5(a) and (b) respectively. The element of interest is sulphur whose concentration is reduced to minimal trace levels in the sample where plasma desorption was carried out compared to the SAM protected sample. This indicates that the NTP plasma desorbed surface is devoid of the SAM. These minimal traces are unavoidable owing to the very high affinity of sulphur for metals.

3.4 Surface analysis

One of the major criteria of successful thermo-compression bonding is the smoothness of the Cu surface. As roughness

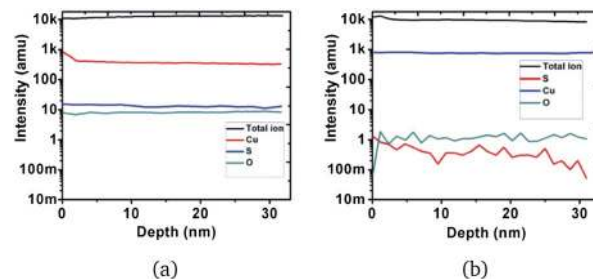


Fig. 5 (a) SIMS result of the Cu surface with the SAM layer before performing NTP desorption. (b) SIMS result of the Cu surface after NTP desorption.

increases, the pressure required for achieving thermo-compression bonding increases owing to a reduction in the area of contact between the two copper layers. AFM analysis was performed to study the effect of plasma on the roughness of the Cu surface. Fig. 6 shows the AFM images of the (a) as deposited Cu surface, (b) SAM modified Cu surface and (c) SAM desorbed Cu surface. The average roughness of the as deposited film is 0.8 nm. Upon SAM adsorption, the average roughness increases to 1.6 nm. This can be attributed to the chain length of the SAM. The roughness after plasma treatment is 1.2 nm. The slight difference in surface roughness between the pure Cu and the Cu after plasma exposure might be due to the presence of a small amount of carbon particles on the surface, which remain even after plasma treatment. As the difference is nominal it is evident that plasma exposure did not affect the copper surface planarity.

3.5 Cross sectional TEM analysis

The quality of the interface was studied using cross sectional TEM analysis which is shown in Fig. 7. In Fig. 7(a), the magnification is chosen in such a way that all the sputter layers are clearly visible. Two inferences can be drawn from this image. Firstly, there is no clear distinguishable interface between the two Cu layers which is an indication that Cu has interdiffused from one layer to another. Secondly, the image indicates growth of polycrystalline grains of copper. This is further corroborated by the XTEM image taken at a higher magnification shown in Fig. 7(b).

3.6 Bond strength analysis

Bond strength is the figure of merit of good bonding. To compare the bond strength with other reported values the 4" bonded sample was cut into 1 cm × 1 cm pieces using a diamond cutter. A shear test was carried out on these diced pieces to examine the resultant mechanical bond strength using an Instron microtester instrument. In this test, a thin blade is aligned at the cross-section of the bonded interface of the chips. The load on the blade is progressively increased until destruction of the bond occurs. The load that is applied to achieve this

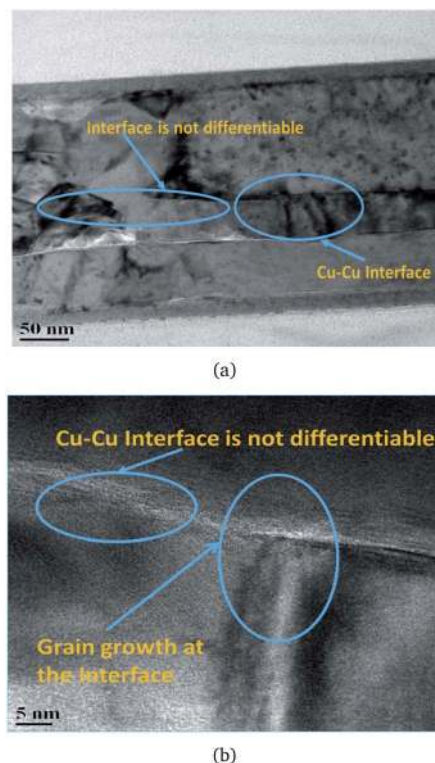


Fig. 7 (a) Cross-section transmission electron microscope (XTEM) image of the Cu–Cu bond interface, and (b) at a higher magnification. Grain growths and interdiffusion of Cu atoms from one wafer to the other wafer suggest excellent bonding.

is the bond strength. In the present case, the bond quality was so good that extension occurred not because of the shearing of the interface, but due to the chiseling of one of the bonded wafers. Beyond the maximum load, cleavage of the interface occurred. Fig. 8 shows the data depicting the applied load vs. extension. The point where the load suddenly drops abruptly is the point where the bond was cleaved. The load at that point is the maximum load that the bonded wafer could withstand before failure. This destructive test was repeated on 10 samples which are cut from different locations in the wafer to account

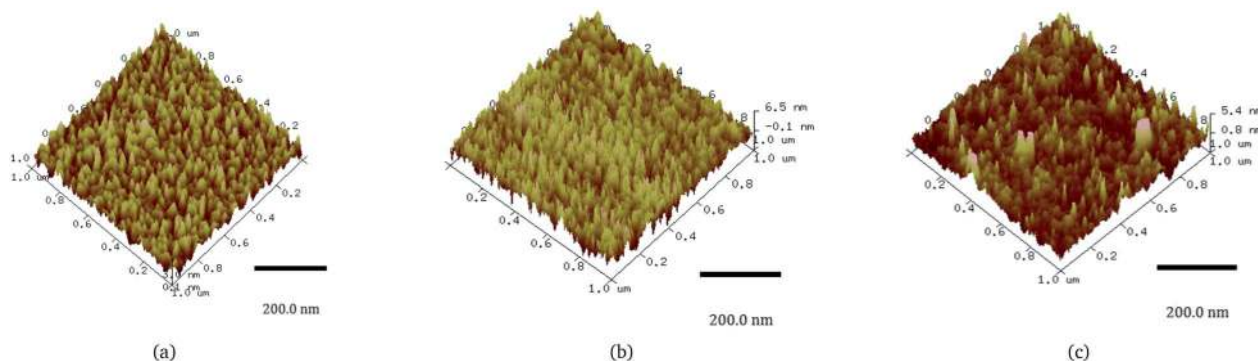


Fig. 6 (a) AFM surface roughness scan of freshly deposited pure Cu – roughness 0.8 nm; (b) AFM surface roughness scan of the Cu surface after 3 h of immersion in propanethiol solution – roughness 1.6 nm; (c) AFM surface roughness scan of the Cu surface after 9 minutes of non-thermal argon plasma desorption – roughness 1.2 nm.

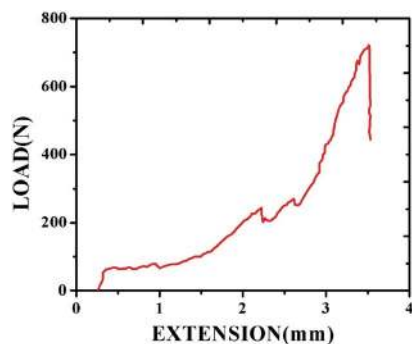


Fig. 8 Load vs. extension graph for a 1 cm × 1 cm bonded sample – bond strength: 726 N.

for the variability in bonding over the entire wafer area. The average bond strength was found to be 726 N, or equivalently 132 MPa, after normalization with the cross-sectional area, with a standard deviation of <3%. The results are very much comparable to results reported in the literature.

4 Conclusions

Room temperature desorption of propanethiol from a copper surface by cold argon plasma has been demonstrated. Contact angles of DI water on the copper surface before and after plasma exposure indicate the complete removal of the SAM layer from the surface. SIMS results further corroborate the complete removal of the SAM. Atomic force microscopy analysis suggests that after desorption the copper surface roughness remains approximately the same as that of the pure copper surface. This room temperature desorption results in not only achieving SAM desorption at ambient conditions, but also enabling successful carrying out of thermo-compression Cu–Cu wafer to wafer bonding at a relatively low temperature of 200 °C, and a relatively low pressure of 4 kN. A high quality, defect free XTEM image and a bond strength of 132 MPa indicate a very good quality of bonding. The proposed method could potentially open up a whole new platform for carrying out low temperature bonding for a variety of 3D IC applications.

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