Microstructure Examination of Copper Wafer Bonding

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The microstructure morphologies and oxide distribution of copper bonded wafers were examined by means of transmission electron microscopy (TEM) and energy dispersion spectrometer (EDS). Cu wafers exhibit good bond properties when wafer contact occurs at 400°C/4000 mbar for 30 min, followed by an anneal at 400°C for 30 min in N_2 ambient atmosphere. The distribution of different defects showed that the bonded layer became a homogeneous layer under theses bonding conditions. The oxidation distribution in the bonded layer is uniform and sparse. Possible bonding mechanisms are discussed.

Key words: Wafer bonding, copper, TEM, EDS, oxidation

INTRODUCTION

Wafer bonding between layers of silicon and SiO_2 have been studied widely and applied successfully to various applications such as SOI,[1] sensors and actuators.[2,3] Moreover, the possibility of joining heterogeneous materials by wafer bonding has recently attracted attention in the micro fabrication community because of its design flexibility and process compatibility with existing CMOS technology. [2,3,4] In particular, research on copper wafer bonding appears attractive, as Cu has low electrical resistively, high electro migration resistance, and has already been integrated into the main stream interconnect technology.[5] With these benefits, copper wafer bonding is definitely an interesting option for implementation of 3D integrated circuits.[6]

The bond strength of copper wafers were previously examined with the razor test. [7] Silicon wafers, coated with 300 nm evaporated copper, were successfully bonded at 450°C for 30 min with a post-bonding anneal in N_2 for 30 min. For successful bonding, a post-bonding anneal is required. Different bonding conditions, such as bonding temperature, anneal temperature, anneal time, and copper layer thickness, were also discussed. [7]

This paper reports the morphologies in different areas of copper bonded wafers. Microstructures at the interfaces were examined by TEM and oxide distribution in the

bonded layer was investigated with EDS. The relationship between defects and the formation of the homogeneous bonded layer will be discussed. In a "good" bond wafer, the bonded layer became a homogeneous layer, and the oxide concentration was low but uniformly distributed.

EXPERIMENTAL

N-type (100) Si wafers were used in this study. All wafers were cleaned with 3:1 (by volume) H_2SO_4:H_2O_2 solutions before metal deposition process. 300 nm Cu and 50 nm Ta films were deposited with an electron beam deposition. The Ta layer acts as a Cu diffusion barrier up to 550°C.[8] Prior to bonding, wafers were dipped in 1:1 (by volume) H_2O:HCl for 30 s followed by a DI water rinse and spin dry. Any native oxide on the Cu surface should be removed through this process.

The wafers were ready to be bonded in the Electronic Vision EV 450 Aligner and AB1-PV bonder after the Hal treatment. The bonding procedure starts by clamping two Cu wafers together face-face, with the wafers separated by three 30-μm metal flaps on the bond chunk. Afterwards, the wafers and the chuck were placed in the EV bonder. N_2 was purged before the chamber was evacuated to 10^{-3} torr. A 300-mbar force was then applied at the center of the top wafer while the flaps were pulled. When the wafers were in full

contact, a 1000-mbar force was applied while both wafers were being heated at a rate of 40°C/min. Once the temperature reached 400°C, a 4000-mbar force was applied for 30 min. Afterward, it required 2 hours to cool the wafers to room temperature. Bonded wafers were then annealed at 400°C in a diffusion furnace in N_2 ambient for 30 min.

The morphologies of the Cu-Cu bonded wafer interface were examined with a JEOL-200CX scanning transmission electron microscope (STEM) and JEOL-2010 transmission electron microscope. The TEM examinations were performed at 200kV, with a point-to-point resolution of 0.23 nm. Most of the XTEM micrographs were taken along the [110] zone axis of the Si substrate. Energy Dispersion Spectrometer (EDS) Analysis, a Link ISIS energy dispersive analysis of the X-ray instrument attached to the JEOL-2010 TEM, was used to determine the compositional distribution of oxidation in local areas of samples in the Cu bonded layers. Various beam sizes and a process time of 100 sec were chosen for EDS analysis for the samples.

RESULTS

Interface morphology of the bonded wafer

Figures 1 and 2 show the cross sectional transmission electron (TEM) images of the Cu-Cu bonded layer. This wafer was bonded at 400°C and 4000 mbar for 30 min. and

then annealed at 400°C for 30 min in N_2 ambient atmosphere. In Fig.1, the TEM sample was taken from the center of the wafer, which shows a well-bonded layer. Random interfaces, grains, and defects are observed but no voids or vacancies are found. In Fig. 2, however, the TEM sample was taken from near the edge of the wafer. A mixture of good bond and unbonded areas is observed in this Cu-Cu region. The actual sizes of the voids and unbonded vacancy area should theoretically be smaller. Enlargement of vacancy sites were probably caused by ion-milling of the TEM sample during sample thinning.

Morphologies of "good bond" area

The microstructures of the well-bonded layer were examined by TEM, as shown in Figures 3(a) and (b). The condition of the samples is the same as that in Fig. 1. Different kinds of defects were observed: stacking faults, twins and dislocations were formed in the bonded layer with different directions. Two groups of defects with different directions are observed in Fig. 3(b). One group is parallel to the metal layer formed along the Cu-Cu interface. The other group are continuous twins with 75 degrees respect to Si substrate that penetrates the bonded layer.

Oxidation examination

After HCl treatment and bonding under vacuum, the oxidation distribution in various areas in the bonded wafer were examined by EDS, as shown in Table I. The

tested areas are shown in Fig. 4.

In the ”good bond” layer, the quantity of oxygen in the layer is lower than 3 wt% when the beam size of X-ray is 500 nm. Furthermore, the local oxygen quantity, both in the middle of the bonded layer and near the edge of the Ta layer, is also lower than 3 wt% when the X-ray beam sizes are 25 and 5 nm. In the partially bonded areas, the oxygen content at the surface, interfaces, and bonded regions were all below the detection threshold of EDS.

DISCUSSION

Previous Results

A recapitulation of our previous results,[6,7] with various bonding and annealing conditions, are listed in Table II. The razor test was used to qualitatively determine the bond strength. A “good bond” means that a razor cannot penetrate the bonding interface. A “partial bond” is where the wafers separate from the bonding strength when considerable force is applied in to the razor. Finally, a “poor bond” occurs when a razor separates the two wafers with relative ease or the wafers did not bond at all. These results indicate that both at 400 and 450°C for 30 min, Cu wafers were bonded successfully with a post-bonding anneal at 400 and 450°C in N_2 for 30 min, respectively. The anneal is a

necessary process to obtain a good wafer bond.

For IC technology, the processing temperature is a key point of cost and yield. The lower the process temperature, the lower the cost and the higher the yield. Therefore, the acceptance of the wafer bonding technology partially depends on one's ability to lower the bond temperature. To examine the microstructure and the bonding mechanism, copper wafers bonded at 400°C (the lowest temperature with reliable bonding) and 4000 mbar for 30 min with a 30 min N_2 anneal at 400°C for 30 was chosen for discussion.

Morphologies of bonded wafer

Comparing Figures 1 and 2, a good bond area with no vacancies could be obtained at the center of the bonded wafer. A partial bond area, where vacancies and good bond areas mix, occurs near the wafer edge. The different results come from the pressure profile distribution across the wafer, which produces uneven stress points during bonding. In our bonding apparatus, the full load pressure of 4000 mbar was applied to a metal plate by a piston located at the center of the wafers. The piston then creates a pressure wave emanating from the wafer center. The metal plate transfers the pressure profile to the wafers, and wafer- to-wafer contact occurs. It is reasonable to assume that the pressure applied on the edge of the wafer was less than the 4000 mbar applied at the center. Under the lower pressure, this area does not receive enough pressure to bond. Therefore the

Cu-Cu area of the edge of the wafer only could form a partial bond.

The good bond area

Figures 3(a) and 3(b) show a good bond area in the bonded wafer. Unlike the partial bond, no vacancies are observed. On the other hand, random interfaces and grains are still observed in some areas. Also, some defects, stacking faults, twins and dislocations are seen in the layer.

The bonding interface should exist in the middle of the layer, but none were observed in Figs 3(a) and 3(b). Interatomic bonds formed at the interface between two surface layers is speculated to be similar to the bond in the bulk in this bonding condition. It is quite difficult to identify the bonding interface after the strong bond formation. At this time, the layers contain a high density of defects and the bonding interface can be regarded as a kind of crystal defect.[9] Therefore, it is suggested that defects parallel to the metal layer were formed along the Cu-Cu interface, as shown in Fig. 3(b).

Most important and interesting of all, twins with 75 degree with respect to Si substrate were observed in the bonded layer. These twins are continuous and penetrate the bonded layer. If there were still a distinct interface or defect areas between the two original copper layers, these twins could not have been formed and would have stopped at the interface instead. However, since there is no evidence of truncated twins in this area,

it implies that twins were formed after the uniform bonded layer was formed.

These results suggest that several steps occurred during bonding and annealing. First, two copper layers were bonded into one layer at this condition. At this time, the interface between two layers should still exist but not quite defined. The copper atoms near the surface of different layers may have mixed together because the distance between surfaces was very close to an atomic scale under such high pressure. At the same time, some defects were formed at the interface because the temperature was raised. During post-bonding anneal, atomic diffusion across the Cu-Cu interface was enhanced. Grain growth and recrystallization initiated, and the Cu-Cu interface in some regions started to disappear. This is the onset of a homogeneous Cu-Cu bond. After a homogeneous layer was formed, continuous twins could form and then penetrate the bond layer.

The oxidation distribution in bonded wafer

Any oxide in the metal line would raise the resistance and decrease the performance of the line. Since copper is easily oxidized in atmosphere at elevated temperatures, preventing oxides on the surfaces of copper layers is an important issue.

From the results in Table I, the bonded wafer exhibits a low level of oxygen (less than 3% wt), and its concentration is uniform across the wafer. Therefore, successful bonding persists despite the presence of copper oxide; these results imply two

possibilities. First: Oxides on the original copper layer were effectively removed through HCl treatment, and the remaining of the oxides may be considered negligible during bonding. Second: Although the oxides on the original copper layer were only partially removed through HCl treatment (or new oxides formed between the HCl treatment and bonding), the oxygen atoms diffused into the layer easily and quickly and then distributed everywhere in the bonded layer under this high temperature and pressure. The result shows that under this condition and HCl treatment, the quantity of oxide in the bonded layer could be effectively controlled for bonding to proceed.

CONCLUSION

In this work, the morphologies of copper wafers bonded at 400°C and 400 mbar for 30 min. and then annealed at 400°C for 30 min in N_2 ambient atmosphere were examined by TEM. TEM observations showed that theses conditions lead to a good bond area in the center of the wafer. A partial bond area was formed in the edge of the wafer, probably due to a reduction of the pressure applied. Moreover, microstructures in the good bond area were studied. Different types of defects formed along the interface of the two original copper layers. Continuous twins penetrating the whole bonded layer showed that the layers became homogeneous and the bonding interface disappeared under these bonding

conditions. EDS examination shows few oxidized regions in the bonded layer which are distributed uniformly.

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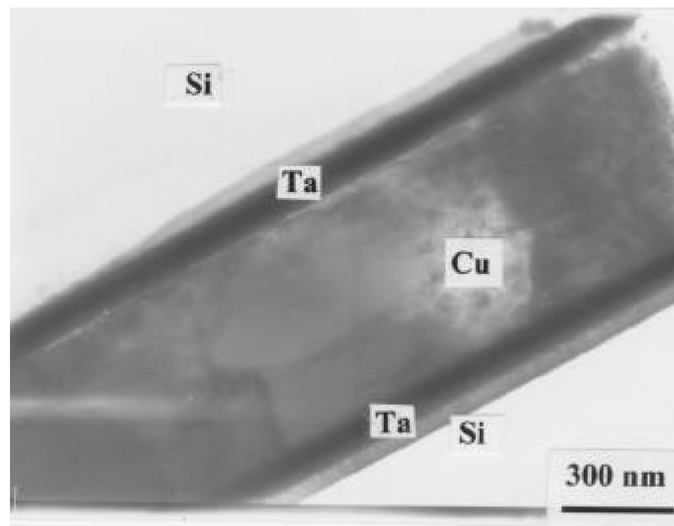
Figure Captions

Figure 1 XTEM image of the Cu-Cu bonded layer. This wafer was bonded at 400°C and 4000 mbar for 30 min. and then annealed at 400°C for 30 min in N_2 ambient atmosphere. Sample was taken from the center of the wafer.

Figure 2 XTEM image of the Cu-Cu bonded layer. This wafer was bonded at 400°C and 4000 mbar for 30 min. and then annealed at 400°C for 30 min in N_2 ambient atmosphere. Sample was taken from near the edge of the wafer.

Figures 3(a) and (b) XTEM images of the microstructures of the well-bonded layer.

Figure 4 XTEM image for EDS tested areas.



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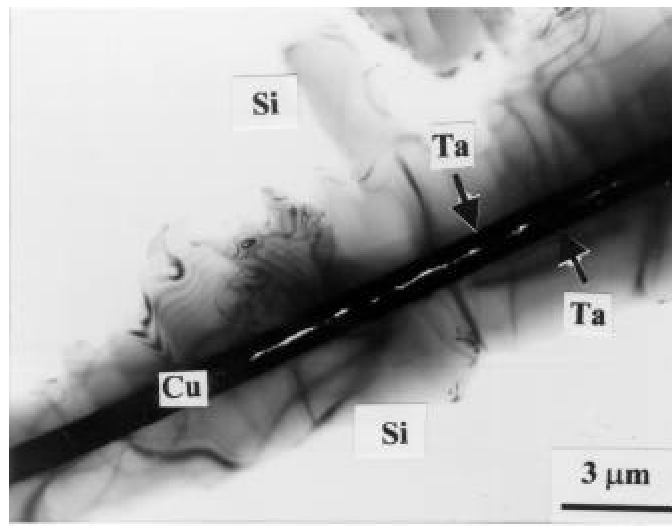
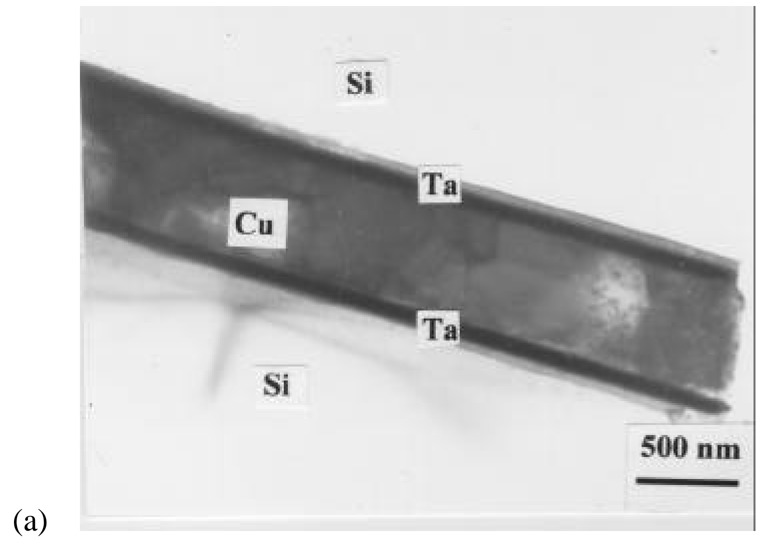
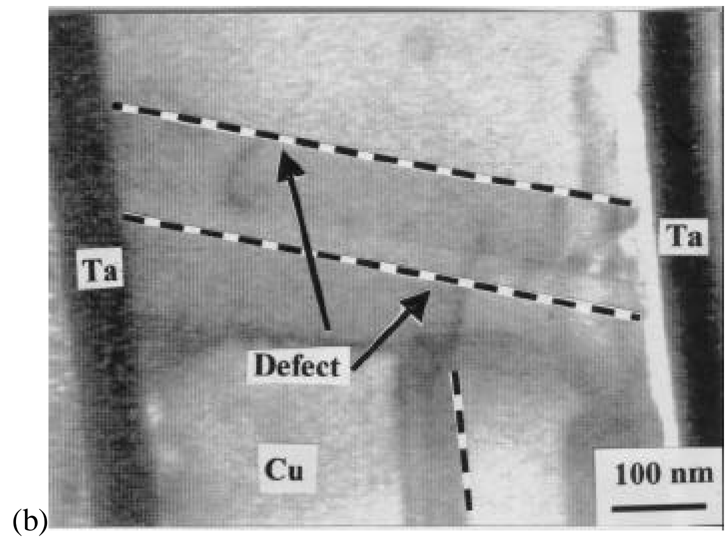


Figure 2 XTEM image of the Cu-Cu bonded layer. This wafer was bonded at 400°C and 4000 mbar for 30 min. and then annealed at 400°C for 30 min in N_2 ambient atmosphere. Sample was taken from near the edge of the wafer.

(a)



(b)



Figures 3(a) and (b) XTEM images of the microstructures of the well-bonded layer.

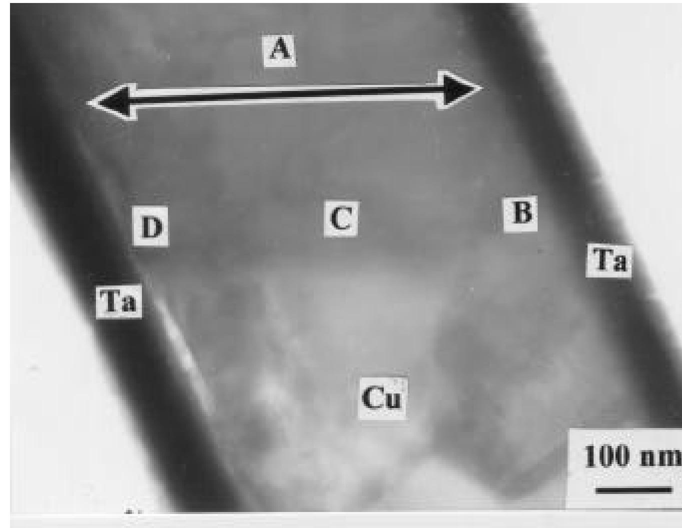


Figure 4 XTEM image for EDS tested areas.

Table I EDS examination for oxygen in different areas of the bonded copper layer

	Beam Size (nm)	Oxygen (wt %)	Location Description
A	500	2.67	whole bonded layer
B	25	2.13	near Ta layer
	5	2.22	
C	25	2.53	near bonded interface
	5	2.78	
D	25	2.98	near Ta layer
	5	2.89	

Table II The results of razor test for the bond strength under various bonding and annealing conditions[7]

Cu film (nm)	Bonding Temperature(°C)	Bonding Time(min)	Anneal Temperature(°C)	Anneal Time(min)	Bond Quality
300	400	30	400	30	Good
300	400	30	No	No	Partial
300	450	30	450	30	Good
300	450	30	No	No	Partial