Copper Bonded Layers Analysis and Effects of Copper Surface Conditions on Bonding Quality for Three-Dimensional Integration

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In order to achieve copper wafer bonding with good quality, surface conditions of copper films are important factors. In this work, the effects of surface conditions, such as surface roughness and oxide formation on the bond strength, were investigated under different bonding conditions. Prior to bonding, copper film surfaces were kept in the atmosphere for less than 1 min, 3 days, and 7 days, respectively, to form different thicknesses of oxide on the surface. Some copper wafers were cleaned using HCl before bonding in order to remove the surface oxide. Surface roughness of copper films with and without HCl cleaning was examined. Since surface cleaning before bonding removes oxides but creates surface roughness, it is important to study the corresponding bond strength under different bonding conditions. These results offer the required information for the process design of copper wafer bonding in three-dimensional integration applications.

Key words: Wafer bonding, copper, oxide, surface roughness

INTRODUCTION

The concept of three-dimensional integration was first proposed in the 1980s.¹⁻³ Three-dimensional integration provides more functionality within a smaller footprint, and might further reduce chip size.⁴ This advantage is especially attractive for wireless and military applications. In addition, performances such as signal delay and clock skew can be effectively improved for integrated circuits with a reduced total interconnect length in threedimensional integration.⁵⁻⁸ Thus, wire length between microprocessor and memory can be greatly reduced by using three-dimensional integration. Flexibility in device technology and materials selection is also provided through the parallel processing of separate layers or wafers, which are finally stacked together using three-dimensional integration fabrication. Therefore, it is possible to stack analog and digital designs on different layers together, or optoelectronic and electronic devices on different material substrates together.

Wafer bonding is a key technology for the development of three-dimensional integration.^{9,10} In addi-

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tion, wafer bonding has been used in silicon on insulator (SOI) and microelectromechanical systems (MEMS) applications.^{9,11} The ability of wafer bonding to create good bonding quality across the wafer enables the possibility of heterogeneous integration of devices.

The MIT's three-dimensional integration is based on direct copper wafer bonding at low temperature.¹⁰ Device wafers, with copper interconnects for electrical connectivity and copper pads for structural support, are sequentially bonded to each other in a face-to-back fashion using copperto-copper low temperature thermo-compression bonding.

Before copper wafer bonding becomes a reliable technology in three-dimensional integration, fundamental studies on bonding properties are necessary. Fundamental studies should be started from copper blanket films bonding without patterns.^{12–15} Bonding parameters for excellent bonding results of copper blanket films bonding can then be applied to copper interconnects bonding. This paper studies the effects of copper surface conditions on bonding quality. Results in this paper can be useful references for three-dimensional integration applications. Copper Bonded Layers Analysis and Effects of Copper Surface Conditions on Bonding Quality for Three-Dimensional Integration

EXPERIMENTAL PROCEDURES

Depositions of 300 nm copper and 50 nm tantalum films were performed on 4 in. silicon wafers with an electron beam deposition in a vacuum of 1×10^{-7} Torr. The deposition rate was about 0.3 nm/s. The tantalum layer acts as a copper diffusion barrier up to 550°C.¹⁶

After metal deposition, wafers were divided into two groups. One group underwent surface cleaning prior to bonding while the other group did not. Wafers that did not undergo the cleaning process were kept in the atmosphere for less than 1 min, 3 days, and 7 days, respectively, to form different thicknesses of oxide on the surface prior to bonding.

Wafers on which surface oxides were removed after copper deposition were dipped in 1:1 (by volume) of H_2O : HCl for 30 sec followed by a DI water rinse and spin dry. Any native oxide on the copper surface should be removed through this process. Reactions of HCl and surface oxides are shown below:¹⁷

$$\begin{array}{l} Cu_{2}O(s) + 8Cl^{-} + 2H^{+}(aq) \rightarrow \\ 2CuCl_{4}^{2-}(aq) + H_{2}O(l) + 2e^{-} \end{array} \tag{1}$$

$$\begin{array}{l} CuO(s) + 4Cl^{-} + 2H^{+}(aq) \rightarrow \\ CuCl_{4}^{\ 2^{-}}(aq) + H_{2}O(l) \end{array} \tag{2}$$

Surface morphologies of wafers with and without HCl clean before bonding were investigated by an Autoprobe CP atomic force microscope (AFM).

The bonding was carried out in an Electronic Vision (EV) AB1-PV bonder. The chuck with the wafers was placed in the EV bonder. Nitrogen 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 out. 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 the desired bonding temperature, a 4000-mbar force was applied for the desired bonding duration. The duration of 2 h is required to cool the wafers to room temperature after bonding. Some bonded wafers were further annealed in a diffusion furnace in nitrogen ambient at the desired anneal temperatures and durations.

Dicing was performed in order to examine the bonding strength of copper bonded wafers. Bonded wafers were cut into 5 mm \times 5 mm square dies at a speed of 0.69 mm/sec by a DAD-2H/6T Automatic Dicing Saw. By monitoring the failure percentage of the bonded pieces due to dicing stress, the bond strength was qualitatively estimated. Failure is defined as die debonding during or after dicing.

RESULTS AND DISCUSSION

Figure 1 shows a typical cross-sectional transmission electron (TEM) image of the bonded copper layer. This wafer was bonded at 400°C under a pressure of 4000 mbar for 30 min followed by an anneal at 400°C for 30 min in atmospheric nitrogen



Si

Fig. 1. A typical cross-sectional TEM image of the bonded copper layer. This wafer was bonded at 400°C with a pressure of 4000 mbar for 30 min followed by an anneal at 400°C for 30 min in atmospheric N_2 ambient.

ambient. In Fig. 1, there is no obvious interface observed in the bonded copper layer after strong bond formation between the two layers. In a previous study, we proposed that several steps occurred during the bonding and annealing processes.¹² Two copper layers were first bonded together after bonding, but the interface between two layers still existed. Under the bonding pressure and temperature, copper atoms of different layers may have mixed together from the diffusion process. During further nitrogen anneal, atomic diffusion, grain growth, and recrystallization occur, and copper-copper interface began to disappear until a homogeneous layer is formed.

Figure 2a–c shows the corresponding failure percentages of bonded wafers with different bonding conditions after dicing when the wafers were (a) bonded at once, (b) kept in atmosphere for 3 days before bonding, and (c) kept in atmosphere for 7 days before bonding, respectively. All results are the average of failed percentages from several wafers under the same bonding condition. Figure 2a shows that, when bonding and anneal temperatures were 350°C or 400°C, the bonded pieces did not fail. The failure percentages of wafers increased to values above 20% when wafers were bonded and annealed at 250°C. However, when the bonding and anneal temperatures were at 200°C, the failure percentages were much higher. Almost all of the bonded pieces failed for wafers bonded at 200°C followed by nitrogen anneal.

In Figure 2a, the bonding quality improves with the increase of bonding temperature. If the process temperature was below 300°C, further anneal deteriorated the bonding quality. The possible



Fig. 2. Corresponding failure percentages of bonded wafers with different bonding conditions after dicing when the wafers were (a) bonded at once, (b) kept in atmosphere for 3 days before bonding, and (c) kept in atmosphere for 7 days before bonding. All of the results are the average of failed pieces from several wafers under the same bonding condition.

mechanism has been discussed in the previous study. 15

Figure 2b and c shows the dicing results of wafers that were kept in atmosphere for 3 days and 7 days before bonding. With the same bonding condition, the failure percentages in these two figures are generally higher than those in Fig. 2a. In addition, the failure percentages in Fig. 2c are higher than those in Fig. 2b. Figure 2b and c shows similar trends to Fig. 2a as described above: the bonding qualities were improved with the increase of bonding and anneal temperature. The effect of annealing is also observed in these figures.

In order to compare the effect of duration in atmosphere before bonding, Fig. 3a-c shows the corresponding failure percentages of bonded wafers when the bonding conditions were (a) 30 min bonding, (b) 30 min bonding and 30 min anneal, and (c) 30 min bonding and 60 min anneal. Before bonding, the wafers were kept in atmosphere within 1 min for



Fig. 3. Corresponding failure percentages of bonded wafers when the bonding conditions were (a) bonding 30 min, (b) bonding 30 min and anneal 30 min, and (c) bonding 30 min and anneal 60 min. Before bonding, the wafers were kept in atmosphere within 1 min, for 3 days or 7 days. The bonding temperatures ranged from 200°C to 400°C.

3 days or 7 days. The bonding temperatures ranged from 200°C to 400°C. It can be seen from these figures that, at low bonding temperatures, the failure percentages increase with the increase of the duration of which wafers were kept in atmosphere before bonding. At higher bonding temperatures this trend is not obvious since these failure percentages are close to zero.

Oxide formation on copper film surface is the likely reason for the above observation. Oxides formed on the copper film surface after the wafers were unloaded from the deposition chamber. The thickness of oxide increases with the duration of which wafers are kept in the atmosphere. Therefore, wafers with longer storage duration before bonding showed greater surface oxide thickness. At low bonding temperatures, these thick oxide films become barriers when copper atoms diffuse from one layer to another. In addition, copper atoms at low bonding temperatures do not achieve high kinetic energies compared to those at high bonding temperatures. Therefore, if the thickness of oxide is great, copper atoms might not be able to diffuse across the bonding interface. However, at high temperatures, copper atoms can achieve enough energy to complete the bonding process during bonding. This explains why the failure percentages of bonded wafers at high bonding temperatures such as 400°C are zero.

With HCl surface cleaning prior to bonding, the results showed that there was no obvious difference between failure percentages of bonded wafers with and without HCl clean when bonding temperature was above 300°C. After HCl cleaning, as described above, the oxide on the copper film surface should be removed. However, at the same time, the surface roughness was increased from 1.15 nm to 1.55 nm. According to the previous study, the surface roughness is a factor that affects the bonding quality when the bonding temperature is low.¹⁸ It should be noted that oxide formed on the wafer surfaces again between HCl clean and loading the wafer into the bonding chamber. The thickness of newly formed oxide, however, is small compared to the thickness of oxide before cleaning.

At high bonding temperatures, both surface oxide and surface roughness are not dominant factors that affect the bonding quality. Therefore, the failure percentages of bonded wafers with and without HCl cleaning prior bonding are both zero when the bonding temperatures are high enough, such as 400°C.

CONCLUSIONS

In conclusion, we have studied the bond strength of bonded copper wafers with different surface conditions, such as surface roughness and oxide formation, under different bonding conditions and have shown that bonding quality improves with increased bonding temperature. If copper-coated wafers are kept for longer durations between metal deposition and bonding, bonding quality degrades because of the increase of surface oxide thickness. At high bonding temperatures, HCl cleaning does not play a significant role in improving the bonding quality. These results offer the required information for the process design of copper wafer bonding in various applications.

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