Microstructure evolution and abnormal grain growth during copper wafer bonding

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Abstract: Evolution of microstructure morphologies and grain orientations of Cu-Cu bonded wafers during bonding and annealing were studied by means of transmission electron microscopy, electron diffraction and X-ray diffraction. The bonded Cu grain structure reaches steady state after post-bonding anneal. An abnormal (220) grain growth was observed during the initial bonding process. Upon annealing, the preferred grain orientation of the whole film shifts from (111) to (220). The effects of yielding and energy minimization may are possible reasons for the evolution of the preferred grain orientation.

Copper wafer bonding has been considered as a strong candidate for three-dimensional integrated circuit (3D IC) because of its low electrical resistivity and high electromigration resistance.[1] In order to achieve a reliable Cu-Cu bonding layer, the electrical, mechanical and material properties of the bonded layer should be considered.[2] In previous research, the qualitative bond strength of copper wafers was examined with the razor test.[2] Improving the bond quality by changing bonding parameters was also investigated[5]. In addition, different interfacial morphologies of the Cu bonded layer were observed and discussed.[3] For successful bonding, a post-bonding anneal is required.[2] However, the role of temperature during bonding and annealing was not well characterized, partially due to the lack of *in-situ* grain growth information. In this letter, the interfacial morphologies and grain orientation during bonding are analyzed. It is surprising that there is an abnormal grain growth during bonding and annealing. Since the resistance to electromigration is sensitive to the texture of Cu films, understanding and controlling Cu texture of the bonded layer is also essential.[4] Further discussion on the relation between this abnormal grain growth and the bonding process will be presented in this letter.

50 nm of Ta and 300 nm of Cu layers were successively deposited on N-type (100) 4” Si wafers. The wafers were bonded face to face in the Electronic Vision EV 450 Aligner and AB1-PV under a pressure of 4000 mbar for 30 min at 400°C. After bonding, some

bonded wafers were annealed at 400°C in N_2 ambient for 30 and 60 min. The morphology, diffraction pattern and grain size of bonded Cu-Cu layer were examined using a JEOL-2010 transmission electron microscope (TEM). During the TEM sample preparation, the samples were kept below 120 °C to avoid grain growth. The crystallography of the Cu film was analyzed by X-ray diffraction (XRD) [Rigagu D/Max].

Figures 1 (a)-(c) show the XTEM images of the bonded Cu-Cu layer and the major diffraction pattern of single grains from selected area: (a) before bonding, (b) after 30 min bonding, and (c) after 30 min bonding and 30 min annealing. As shown in Fig. 1(a), the Cu grains have an average size of 300 nm, and most grains show an (111) orientation which is similar to the results from other research.[5] In Fig. 1(b), the bonded interface is clearly observed but it is not straight. Different kinds of morphologies such as twins are present in the bonded Cu film. The distribution of grain size ranges from 300 nm to 700 nm. In addition, no major grain orientation but (111), (200) and (222) grain orientations are observed. However, after further annealing, Fig. 1(c) shows a well-developed grain texture with an 800 nm grain size profile and a major (220) orientation. In some area, there are even only single (220) grains existing in the bonded layer. It should be noted that these morphologies were observed in different wafers under the same bonding condition.

From these TEM images, it is evident that there is strong grain growth during bonding

and annealing. The jagged Cu-Cu interface shows that interdiffusion between two Cu layers takes place during bonding. However, the grain growth is incomplete and the bonded layer still contains defects such as twins in the individual layer, as shown in Fig. 1(b). The two original Cu layers can still be distinguished. During the post-bonding annealing, the bonded layer was provided enough energy to complete the grain growth and thus achieved a stable microstructure. As a result, the two Cu layers can no longer be distinguished in Fig. 1(c). Figure 2 shows the distribution of average grain size estimated from TEM images under different bonding conditions. There is a large distribution of grain sizes after 30 min bonding due to the incomplete grain growth. The grain growth then accelerated during the first 30 minutes of post-bond annealing. However, the grain size did not increase significantly beyond 30 minutes of annealing. This indicates that a certain amount of annealing time is required to complete the grain growth and to obtain an "intimate" Cu layer.

It is interesting to compare the morphologies of the annealed sample (Fig. 1(c)) and the non-annealed ones (Figs. 1(b), 3(a) and 3(b)). In Fig. 1(b), a clear bonding interface can be observed and each layer seems to retain the similar morphologies before bonding, as shown in Fig. 1(a). In another sample, grains with different sizes, twins and defects are shown in Fig. 3(a). Both Fig. 1(b) and Fig. 3(a) show that the effects of bonding and

heating are not very significant in these two areas. However, in Fig. 3(b), a whole grain formed in the bonded layer. This suggests that the local grain growth in Fig. 3(b) was much stronger than those in Fig. 1(b) and Fig. 3(a). These three types of microstructures were observed randomly across the whole wafer and on different wafers.

These three images indicate that after 30 min of bonding, grain growth saturation for the whole bonded layer is not reached yet. At this stage, non-homogeneous bonding exists throughout the wafer, possibly due to local heating non-uniformity. To achieve grain growth saturation in the whole bonded layer, more energy is required. This explains the result that post-bonding annealing improves the bond strength in ref. 2: It is reasonable to assume that this incomplete grain growth area has weaker bond strength due to the existing interface. When a razor applies a force in the bonded layer, the crack would have the chance to grow along the "poor" (non-saturated grain growth) bonding interface. On the other hand, even with a huge force, the bonded layer cannot be delaminated if there is no interface existing in the layer. Therefore, through the second time grain growth from annealing, the microstructure of the whole bonded layer can be further arranged and then reaches a steady state with increased bond strength.

From the TEM diffraction patterns in Figs. 1(a) and (c), it is clear that there is a grain orientation transformation from (111) to (220). In order to make sure that this is not a local

phenomenon, bonded samples with one side substrate removed by grinding were analyzed by XRD. Grain orientations under different bonding conditions are shown in Fig. 4. It is evident that the (220) intensity increases while (111) and (200) intensities decrease after bonding and annealing. In addition, Table I shows the average fraction of grain orientations under different bonding conditions. The fraction of (220) increases upon bonding and further annealing, but does not increase significantly after 60 min annealing. The trend here is similar to the distribution of average grain size under different bonding conditions.

During the bonding and annealing processes, the intensity of (220) grains increases but that of (111) grains decreases. A preferred grain orientation (220) after bonding and annealing is observed while (111) is the preferred orientation before bonding. Since this is also identical to the results from TEM diffraction patterns, this phenomenon appears both in the local and the whole bonded layer.

The effect of yielding in grains is proposed to explain the (220) preferred orientation during bonding.[6] The Cu bonded layer is in a state of biaxial strain due to their attachment on both sides of the much thicker Si substrates. Since the in-plane stress in a grain is the product of the biaxial strain and biaxial modulus, which is a function of grain orientation factor C_{ijk}, the yield stress of the grain also varies depending on its orientation. It is reported that the orientation factor C_{ijk} of (111) has the largest value of 3.46 while that of (220) has

the smallest one of 1.42.[7] This means that the yield stress of (220) is much smaller than that of (111). Therefore, for grains of equal initial sizes, (220) grains will yield before (111) grains, thus the (220) grains have an energetic advantage for further growth.[6] This yielding process also leads to strain energy minimization.[6, 8] For further annealing process, the bonded wafer is heated again from room temperature to high temperature, so the in-plane strain also increases. Then, at high temperature, the secondary grain growth occurs; and at this time, (220) grains yield first and take this energetic advantage to minimize the strain energy in the bonded layer. This may explain why (220) grains grow faster than other grains and becomes the final preferred orientation. When the grain growth saturation is reached after 60 min annealing, the yielding process also stops.

It is reported that Cu with preferred (111) orientation has longer electromigration lifetime than that with preferred (220) orientation.[9] Therefore, the abnormal (220) grain growth during Cu wafer bonding is certainly not desirable in IC fabrication. It is imperative that methods to suppress (111) to (220) transformation while maintaining reasonable bonding strength during Cu wafer bonding should be developed.

In conclusion, an abnormal Cu (220) grain growth during bonding and annealing was observed by TEM, electron diffraction and XRD. It is suggested that the growth of (220) grains is easier than that of (111) and (002) grains since (220) grains have a lower yield

stress and the growth has minimized the surface or strain energy. The preferred orientation becomes (220) after further annealing. Both grain sizes and (220) grain intensity reach a saturated state after 30 min of annealing. In addition, further annealing after bonding can effectively improve bond strength and the grain structure is saturated at this time.

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Figure and Table Captions

Figures 1(a)-(c) XEM images of the Cu-Cu bonded layer and the major diffraction pattern of single grains for selective area: (a) before bonding (b) after 30 min bonding (c) after 30 min bonding and annealing

Figure 2 Average grain sizes as a function of different bonding conditions

Figures 3(a) and (b) Various of XEM images of the Cu-Cu bonded layer after 30 min bonding

Figure 4 XRD patterns for different bonding conditions

Table I Variation of (220)/(111) intensity ratio of the bonded layer for different bonding conditions

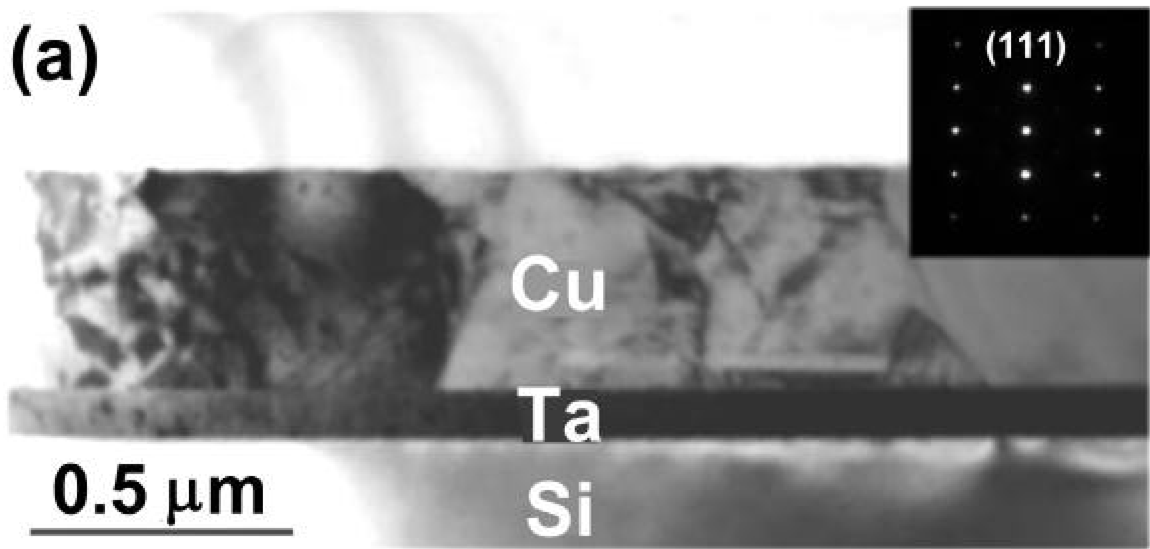

Figure 1(a)

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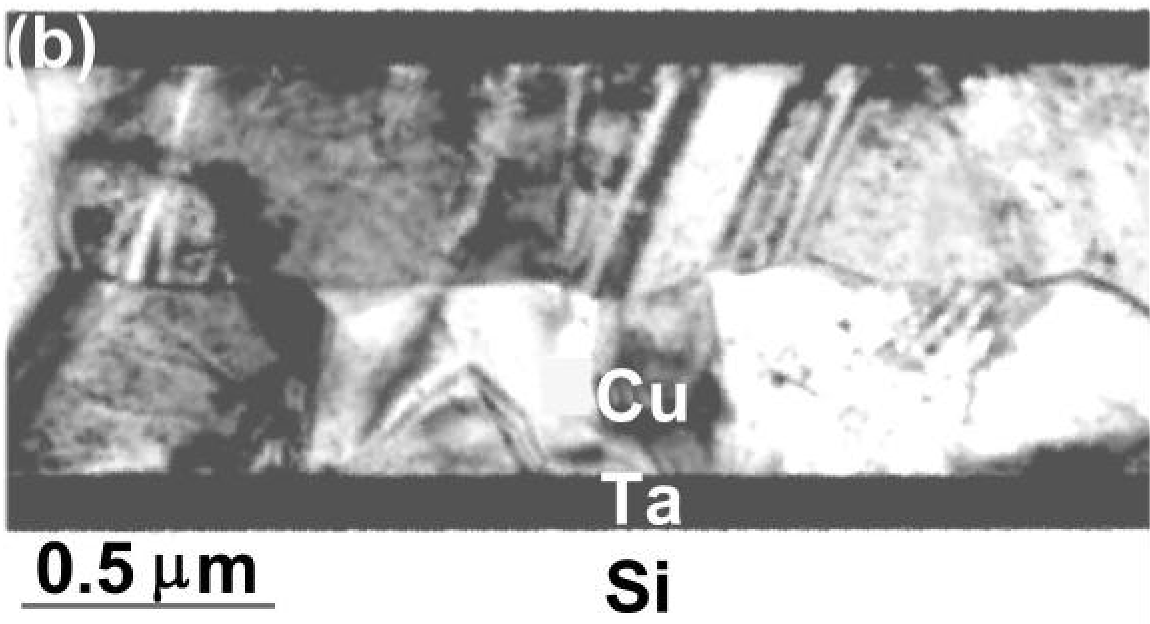

Figure 1(b)

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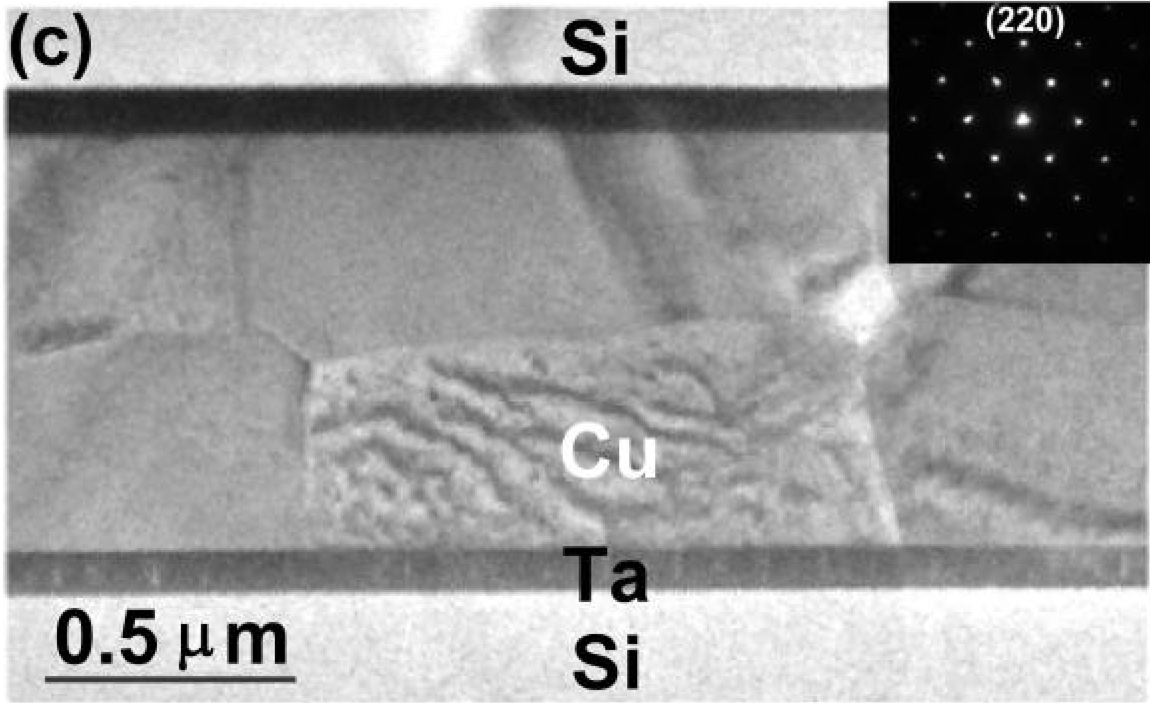

Figure 1(c)

Figures 1(a)-(c) XEM images of the Cu-Cu bonded layer and the major diffraction pattern of single grains for selective area: (a) before bonding (b) after 30 min bonding (c) after 30 min bonding and annealing

K. N. Chen *et al.*

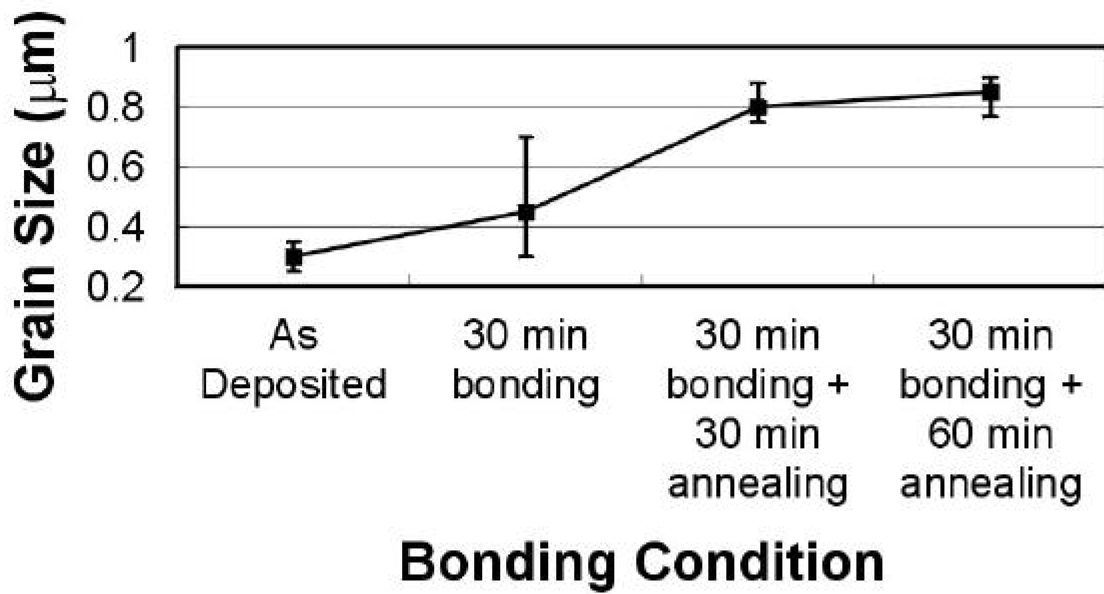

Figure 2 Average grain sizes as a function of different bonding conditions

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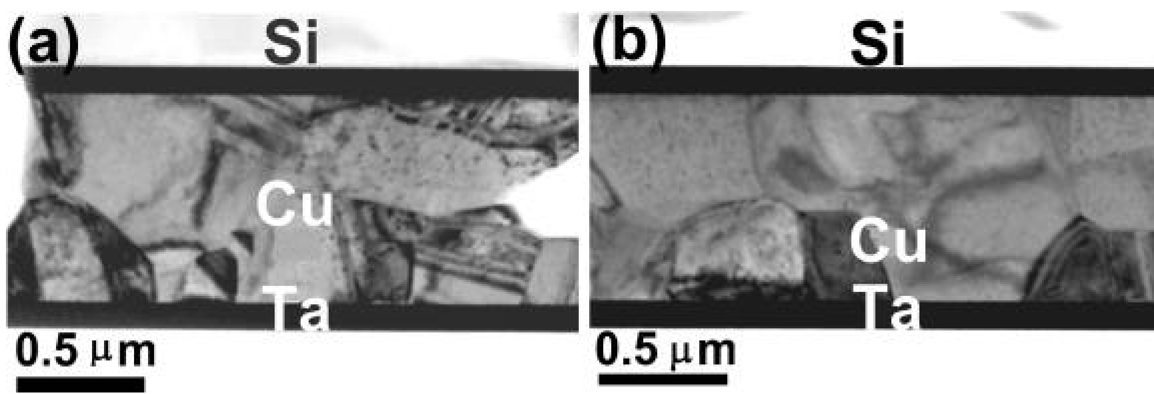

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K. N. Chen *et al.*

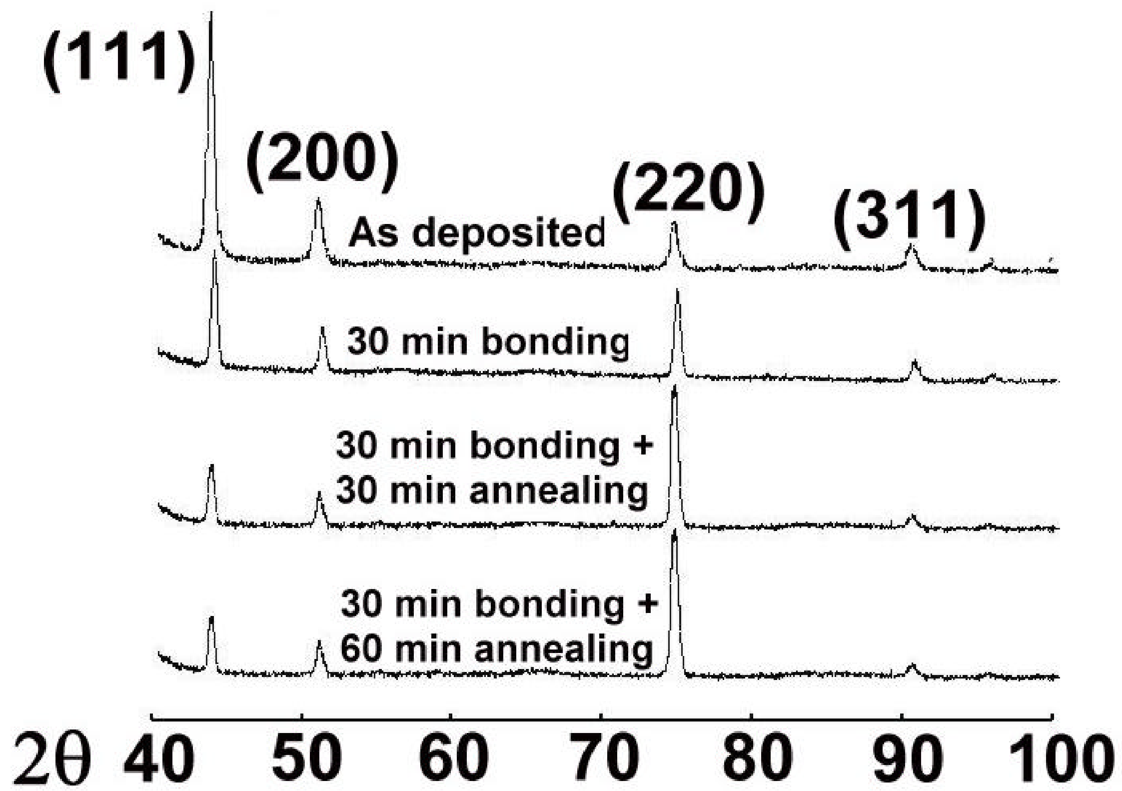

Figure 4 XRD patterns for different bonding conditions

K. N. Chen *et al.*

	(111)	(220)	Other orientations
As deposited	0.62	0.13	0.25
30 min bonding	0.42	0.33	0.25
30 min bonding +30 min annealing	0.23	0.57	0.20
30 min bonding +60 min annealing	0.23	0.60	0.17

Table 1 The average fraction of grain orientations under different bonding conditions

K. N. Chen *et al.*