ELEVENTH INTERNATIONAL CONFERENCE ON INTERGRANULAR AND INTERPHASE BOUNDARIES 2004

JOURNAL OF MATERIALS SCIENCE 40 (2005) 3149–3154

Direct bonding of CMP-Cu films by surface activated bonding (SAB) method

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The chemical mechanical polishing (CMP) process is indispensable to the fabrication of Cu wiring layers in the large-scale integration (LSI). Recently, a direct bonding method with low bonding temperature is required for the CMP-Cu surface in order to obtain a narrow bonding pitch. In this study, we realized a direct bonding between CMP-Cu films by means of the surface activated bonding (SAB) method at room temperature. The critical vacuum pressure to obtain large bonding strength was estimated at about 4×10^{-3} Pa from the growth rate of oxide on an active surface measured by the X-ray photoelectron spectroscope (XPS). The films were bonded successfully at the vacuum pressure better than around 3×10^{-3} Pa with the shear strength larger than 50 MPa. The transmission electron microscope (TEM) observation showed that the polycrystalline films with the mean surface roughness of 0.3 nm were bonded directly between Cu grains in atomic level. Moreover, the adhesion between the films was improved due to the stress relaxation at the interface during the thermal aging test conducted at 200 and 300°C in the vacuum condition.
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1. Introduction

For the system LSI, a narrow bonding pitch is one of the most important requirements to achieve high electrical performance. However, the electro-migration (EM) and RC delay become serious in the conventional Al wiring as the design rule shrinks and the working temperature increases [1]. The wiring material is shifting from Al to Cu since Cu has low electrical resistivity and high activation energy as compared with Al [2]. Whereas, the conventional dry etching process cannot be used for Cu since the sublimation temperature of Cu compound generated in the process is high [3]. Therefore, the damascene process is employed to fabricate Cu wiring layers. In the damascene process, the CMP process is used to flatten the surface of Cu wiring in order to ensure the focal accuracy in the photolithography because this is the only method to achieve the surface roughness of less than a few nm. Recently, a direct bonding method for the CMP-Cu surface with low bonding temperature is demanded in order to shrink the bonding pitch by preventing the thermal expansion mismatch between the chips. Hence, we applied the surface activated bonding (SAB) method to the interconnection between the CMP-Cu surfaces.

Fig. 1 shows the schematic representation of the SAB method. The principle of the SAB method is based on the attractive force between atomically clean surfaces. The clean surface is obtained by the dry process such as the bombardment of Ar fast atom beam (FAB), ion beam, and the plasma irradiation in a certain clean atmosphere like high vacuum condition. Then the atomic bond is generated between the surfaces to decrease the surface energy when they approach within interatomic distances [4, 5]. Consequently, there is no need for the heating temperature in this method. Moreover, different materials such as ceramic, metal, semiconductor, and even plastic can be bonded without adhesive layers between samples [6, 7]. In the SAB method, the vacuum pressure is very important to obtain high quality of bonding such as large bonding strength since the cleanness of the surface is degraded as the oxide grows on the active surface. Concretely, the vacuum pressure at which an inactive oxide film terminates the active surface is considered as the threshold value. However, the optimum vacuum condition for the CMP-Cu surface is not clarified unlike the simple single crystalline sample because the oxidization behavior of the CMP-Cu surface has not been elucidated [8, 9]. In addition, the interfacial structure and reliability of the interface are still unknown.

In this study, we measured the growth rate of oxide on a clean CMP-Cu surface by the XPS in order to estimate the critical vacuum pressure at which a continuous oxide film was generated. Then the bonding experiment was carried out and the direct interface was evaluated from the die shear test and the TEM observation. Furthermore, the thermal reliability was investigated by the thermal aging test conducted in the vacuum and atmospheric condition.

2. Oxidization behavior of CMP-Cu surface

2.1. Experimental procedure

The description of the test vehicle is presented in Fig. 2a. The CMP-Cu thin films with the size of 9.4×3.4 mm^2 and 22×22 mm^2 were prepared on

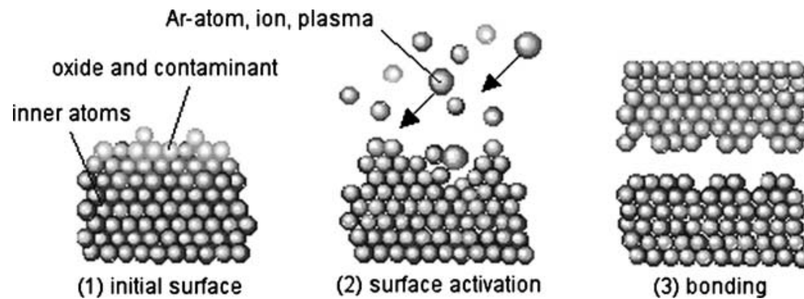

Figure 1 The illustration of the principle of the surface activated bonding (SAB) method.

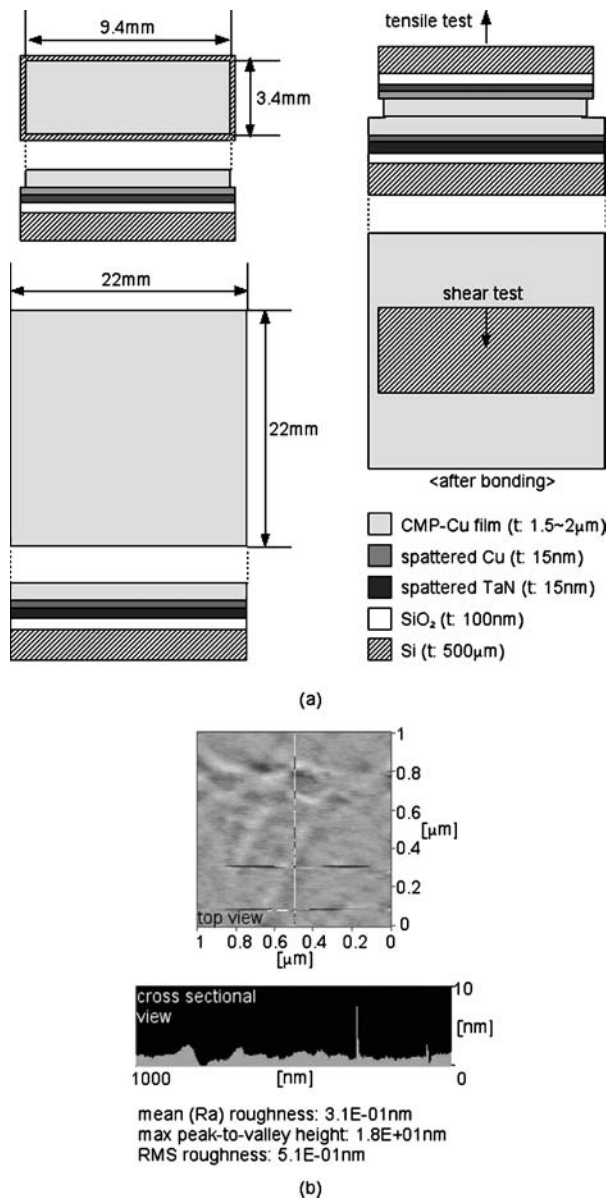

Figure 2 The description of (a) the structure of test vehicle, and (b) the surface roughness measured by the AFM.

Si chips as follows. A Cu film was electroplated on a Si chip after the sputter deposition of SiO_2, TaN and Cu, and then flattened by the CMP process. Then a CuO capping layer was formed by the citric acid in order to maintain the initial flatness of the CMP-Cu surface since it is known that the surface after the planarization generates the uneven oxide and contamination. The atomic force microscope (AFM) image provided in Fig. 2b showed that the mean surface roughness of the fabricated CMP-Cu film was about 0.3 nm. In the previous study about the bonding between Si chips, the surfaces with the roughness of around 1 nm were successfully bonded [10]. Since a large deformation of the surface is expected on Cu compared with Si, it is considered that the fabricated CMP-Cu sample is flat enough to obtain the contact on the whole area of the surface. Then the fabricated CMP-Cu sample was introduced to the XPS observation. The XPS apparatus equips a vacuum chamber in which an Ar radio frequency (RF) plasma generator with the power source of 50 W is installed. The etching rate for the Cu surface was measured beforehand as about 50 nm/min. The oxygen gas with the purity of 99.999% can be introduced into the chamber to oxidize the active surface. For the surface analysis, an Al-Kα monochrometer with the power source of 10 kV and 30 mA is installed in the analysis chamber. The surface resolution of the monochrometer for a CMP-Cu surface is about 2 nm. This chamber also includes an Ar ion beam source with the power source of 1000 eV and 20 mA for the depth profiling. In the XPS analysis, we measured the thickness of the initial inactive layer to determine the optimum etching depth for the surface activation. After that, the depth profiling was carried out on the surface oxidized at the gas pressure of 10, 100 and 1000 Pa in order to estimate the critical vacuum pressure from the growth rate of oxide on an activated CMP-Cu surface.

2.2. Results and discussion

Fig. 3 shows the composition of the initial surface of the CMP-Cu film including the inactive layer. The spectra of C1s and Cu2p3/2 are also provided to elucidate the chemical binding condition of C and Cu at the depth of around 3 and 8 nm respectively. The depth profiling showed that the inactive layer denoted as C and O disappeared at the depth of 10–15 nm. As seen in the spectra, the topmost layer with the thickness of 7–8 nm consisted of organic molecules such as C─H and C═O and the lower layer to the depth of around 15 nm was composed of Cu-organic complex, CuO, Cu_2O and Cu. It turned out that the optimum etching depth to activate the CMP-Cu surface was around 15 nm. Since the etching rate of Ar RF plasma beam for the CMP-Cu surface was about 50 nm, we set the irradiation time at 20 s for the following XPS observation.

Fig. 4 shows the relationship between the exposure time to the oxygen gas and the thickness of oxide. In addition, the spectra of Cu LMM at the exposure time of 600 and 3600 s were presented to show the

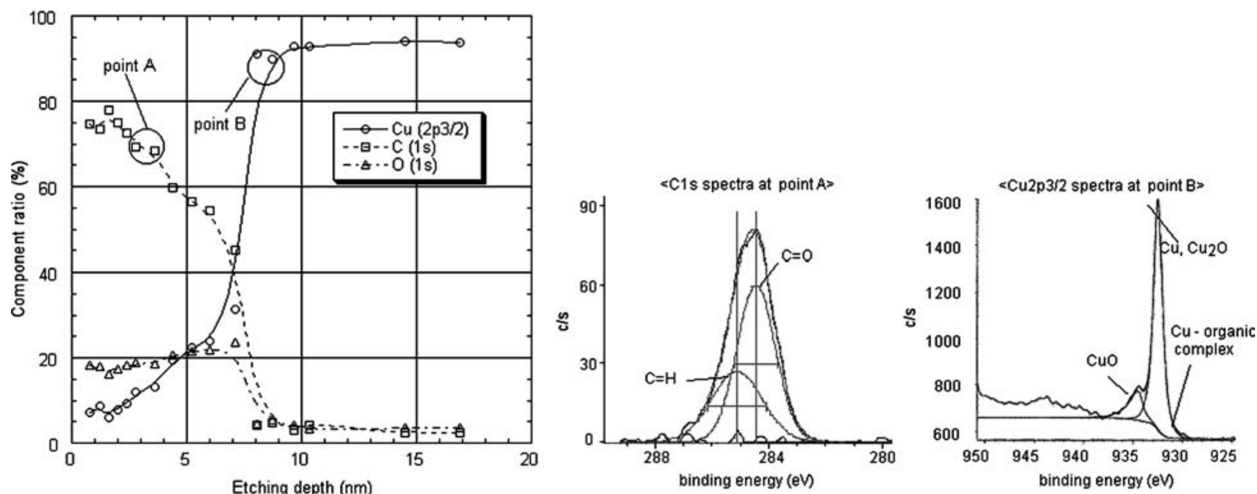

Figure 3 The composition of the surface of CMP-Cu film corresponding to the etching depth. The XPS spectra of C1s and Cu2p3/2 are shown together.

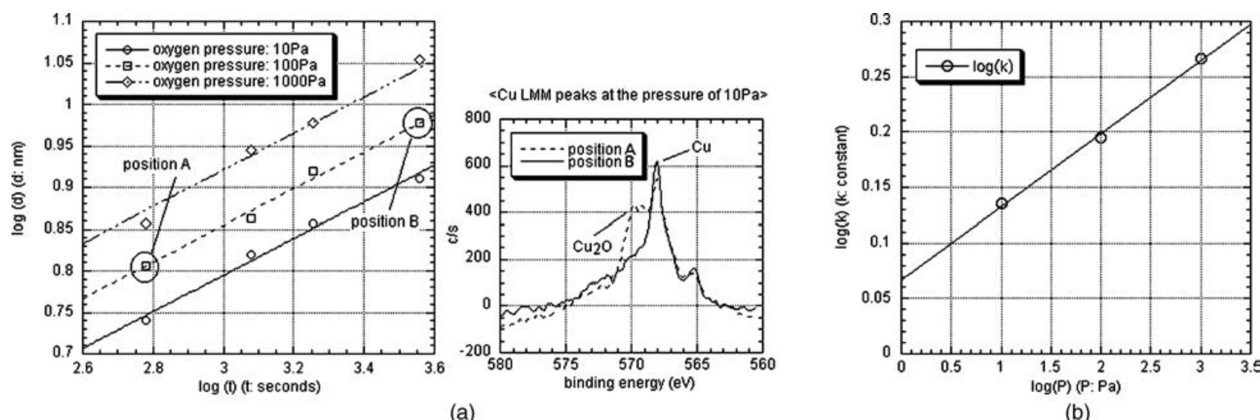

Figure 4 The growth rate of the oxide described from (a) the relationship between the exposure time and the thickness, and (b) the rate constant that depends on the gas pressure.

chemical binding condition of the oxide because the adjacent peaks of Cu and Cu_2O cannot be separated in the Cu2p3/2 spectrum. The spectra showed that Cu_2O layer was generated dominantly on the active surface at room temperature. Here, we consider the growth rate of oxide. When assuming that the growth rate of oxide is described by the following expression

$$d = kt^n \tag{1}$$

where d is the thickness, k is the rate constant that depends on the gas pressure and t is the exposure time [11], the values of n and k can be obtained by fitting the measured thickness of oxide to this expression as shown in the dotted lines in Fig. 4a. The rate constant k was derived as about 1.36, 1.57, and 1.87 respectively at the gas pressure of 10, 100 and 1000 Pa, and the value of n was obtained as about 0.22. Fig. 4b shows the relationship between the rate constant and the gas pressure. It turned out that the rate constant k for the CMP-Cu surface was described approximately by the following expression

$$k = CP^n \tag{2}$$

where the values of C and n are respectively about 1.17 and 0.066. Since it is considered that a continuous oxide film is as thin as several molecule layers [12], we estimated the vacuum pressure at which five Cu_2O molecule layers with the thickness of about 2 nm were generated. The SAB apparatus that is mentioned later requires 60 s until the activated surface contacted each other. Therefore, substituting 2 nm and 60 s for d and t respectively in the expression (1), the rate constant k was calculated as about 0.81. From the expression (2) substituting 0.81 for k, the vacuum pressure P was figured out as around 4×10^{-3} Pa.

3. SAB for CMP-Cu films

3.1. Experimental procedure

Fig. 5 provides the schematic representation of the SAB apparatus that is composed of a high vacuum chamber in which a pair of Ar fast atom beam (FAB) generators with the power source of 15 mA and 1.5 kV are installed to activate the surfaces of the samples. Since the etching rate of Ar-FAB for the CMP-Cu surface was measured as 2.5 nm/min, the irradiation time was set at 5 min. The bonding head can sway around the sphere fulcrum to compensate the tilt of upper chip and to follow the surface of lower sample. As a preliminary examination, the optimum bonding pressure required to contact the whole area of the surface of CMP-Cu sample was measured as about 10 MPa.

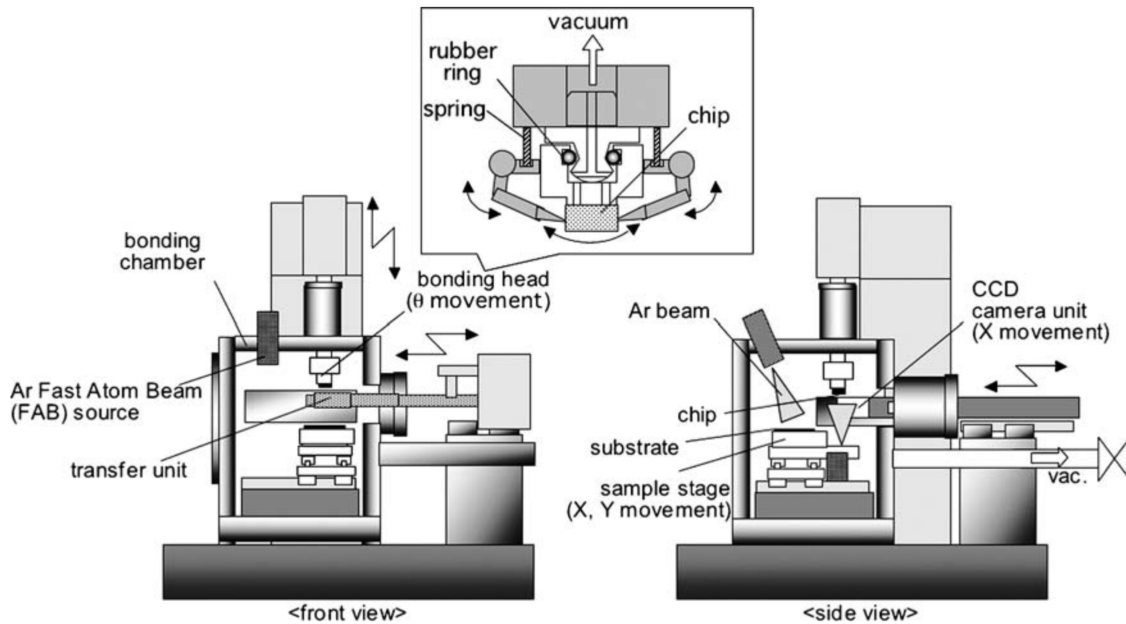

Figure 5 The schematic representation of the structure of the SAB bonder.

The sample with the size of $22 \times 22\ \mathrm{mm}^2$ was placed on the lower sample stage and the other sample with the size of $9.4 \times 3.4\ \mathrm{mm}^2$ was attached on the bonding head. After the surface activation by the Ar-FAB irradiation was conducted, the samples were bonded at the vacuum pressure of 2.0×10^{-5} Pa, 6.0×10^{-4} Pa, 1.7×10^{-3} Pa, 2.5×10^{-3} Pa, 4×10^{-3} Pa, and 6.3×10^{-3} Pa. Then the die shear test was carried out to evaluate the bonded area recognized as the fracture area on the debonded surface.

3.2. Results and discussion

Fig. 6 shows the relationship between the background vacuum pressure and the bonded area. Almost all area of the surface was interconnected until the vacuum pressure increased to around 3×10^{-3} Pa. This value is almost equal to the calculated critical pressure that is required to form a continuous oxide film on an active surface. Also, the successfully bonded samples failed at the shear strength around 50 MPa, which is sufficiently large for the practical use. The actual shear strength at the interface was considered larger than the obtained value since the fracture occurred at the interface between CMP-Cu film and the chip.

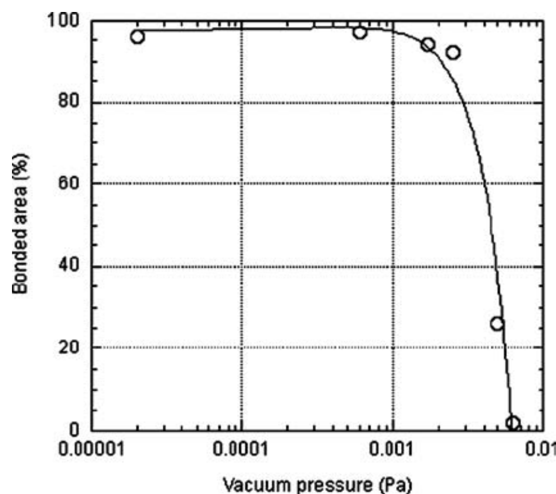

Figure 6 The change in the bonded area at various vacuum pressures.

4. TEM observation

4.1. Experimental procedure

The direct interface between CMP-Cu films was observed by the TEM with the acceleration voltage of 200 kV. The sample used for the observation was the one that was bonded at the vacuum pressure of 2.0×10^{-5} Pa. Then the thermal aging test was carried out in the TEM vacuum column at 200 and 300°C. In addition, the interface that aged at 250°C for 500 h in the atmospheric condition was investigated.

4.2. Results and discussion

The TEM observation images of the direct interface between the CMP-Cu films are presented in Fig. 7. The low-magnification images in (a) showed that the polycrystalline Cu films were bonded directly between Cu grains. In the image shown in (b), the contrast of the strain due to the deformation of the surface on the moment of contact was observed on the interface. It is considered that the surface with the roughness of 0.3 nm deformed enough to obtain the tight adhesion between surfaces. The high-magnification images in (c) showed that the CMP-Cu surfaces were successfully bonded in atomic level. The interface contained very few void and the oxide layer was not seen obviously. Although the active surfaces might be oxidized even in the high vacuum condition before they were contacted, the oxide generated at the vacuum pressure of 2.0×10^{-5} Pa was considered too thin and unstable to remain at the interface.

Fig. 8a–d and e provide the result of the aging test conducted in the vacuum and atmospheric condition

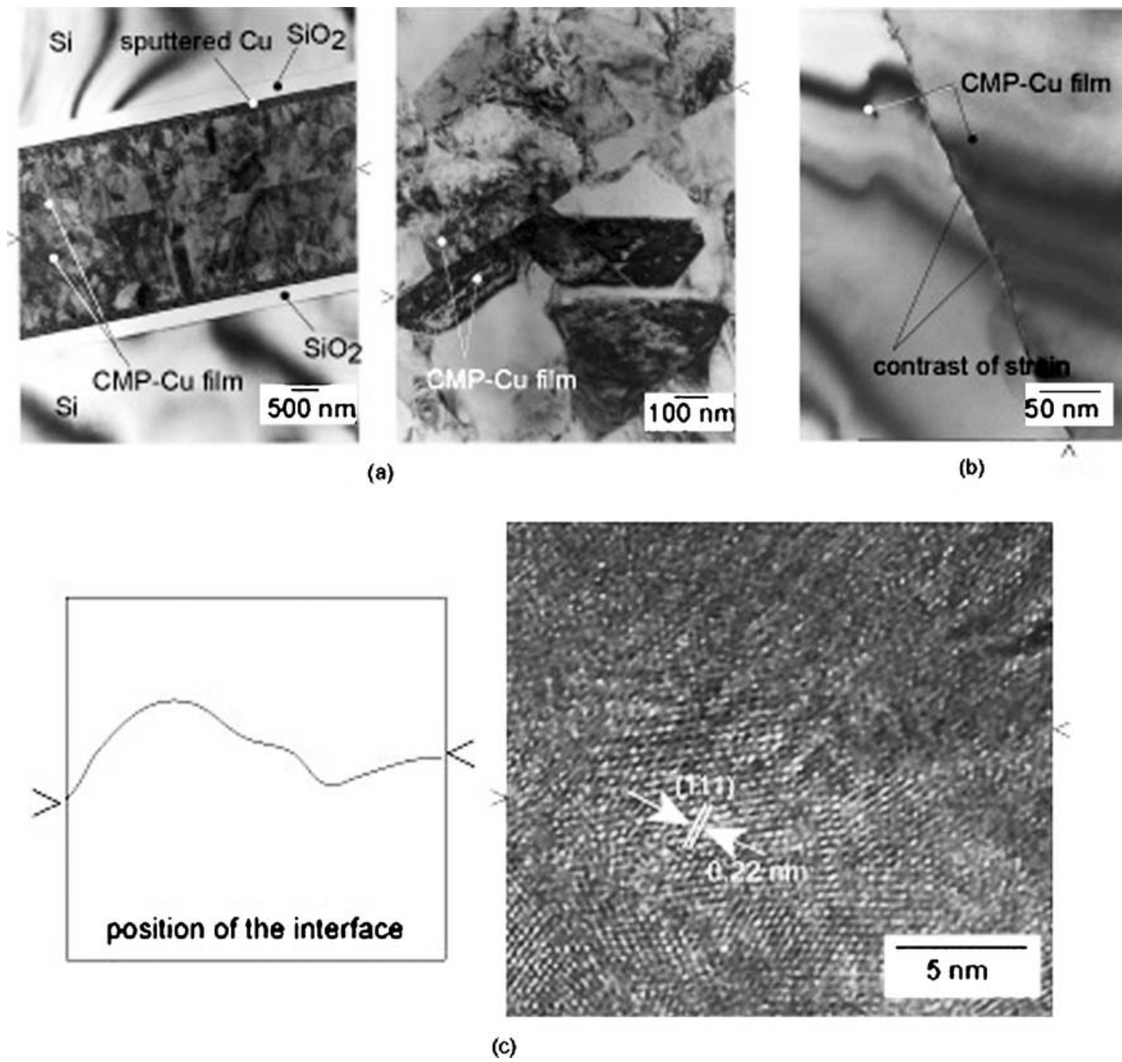

Figure 7 The TEM images of the interface between the CMP-Cu films (a) in the low magnification, (b) with the contrast of the strain due to the deformation of the surface, and (c) in the high magnification.

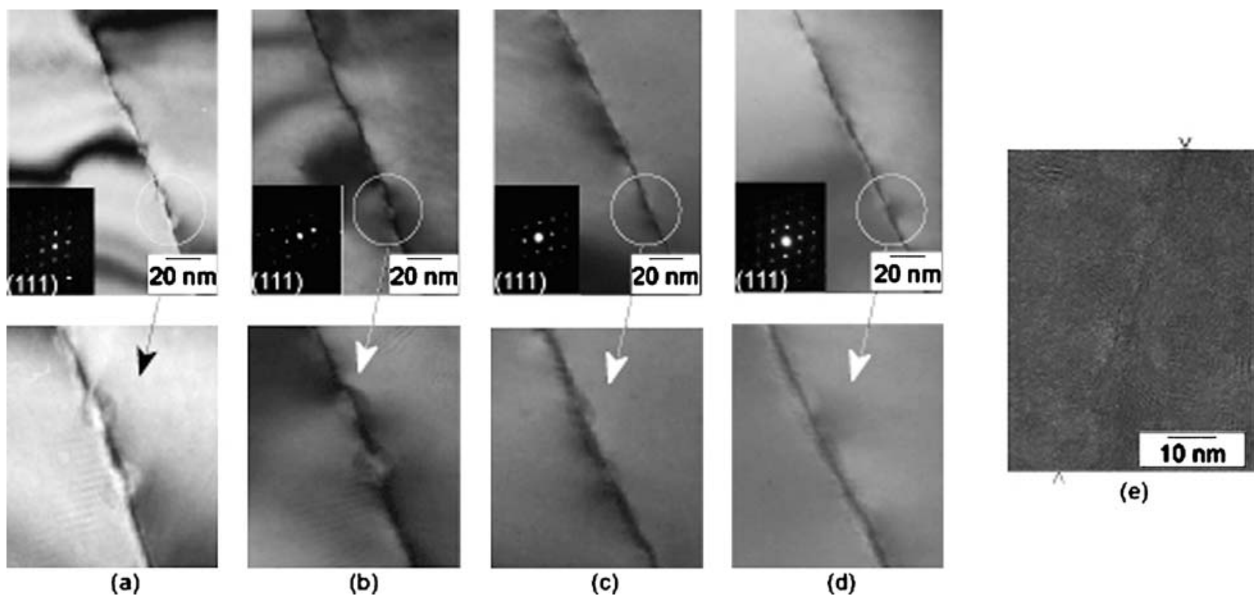

Figure 8 The TEM images of the interface aged in the condition of (a) the vacuum at 200°C for 120 min, (b) the vacuum at 300°C for 120 min, (c) the vacuum at 300°C for 150 min, (c) the vacuum at 300°C for 180 min, (d) the vacuum at 300°C for 210 min, and (e) the air at 250°C for 500 h.

respectively. The pictures in (a)–(d) showed that the contrast of the strain at the interface disappeared and the interface became equable as the aging time increased since the interfacial stress was relaxed by the heating temperature. Moreover, the high thermal reliability of the direct interface between CMP-Cu films generated by the SAB method was suggested since neither the oxide nor the gap were generated at the interface aged at 250°C for 500 h in the atmospheric condition as shown in the image (e).

5. Conclusions

We realized a direct bonding between the CMP-Cu films by means of the SAB method at room temperature, and obtained an atomic-level interconnection with large bonding strength and the high thermal reliability. We estimated the critical vacuum pressure at which a continuous oxide film was generated to be about 4×10^{-3} Pa from the growth rate of oxide on an active surface measured by the XPS. The whole area of the surface was bonded at the vacuum pressure better than about 3×10^{-3} Pa with the shear strength larger than 50 MPa. The TEM observation showed that the interconnection was successfully obtained between Cu grains without visible void and the oxide layer at the interface. In the thermal aging test conducted at 200 and 300°C in the TEM column, the contrast of the strain due to the deformation of the surface on the moment of the contact disappeared and the interface became equable as the heating time increased. Moreover, no visible degradation of the interface was observed in the sample aged at 250°C for 500 h in the atmospheric condition.

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Received 8 September 2004
and accepted 31 January 2005