TECHNICAL PAPER

# Kinetics of low temperature direct copper-copper bonding

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Abstract Wafer level metal bonding involving copper layers is a key technology for three-dimensional integration. An appropriate surface activation can be performed in order to obtain room temperature direct bonding with bonding strength to be compatible with microelectronic device integration. In this paper, we focus on bonding strengthening mechanisms in the low temperature range from 20 to 100 °C. In order to improve the bonding interface closure, several process parameters are studied: water amount available when surfaces are brought into contact and copper layers deposition technique have been identified to be predominant in this low temperature range. Based on metal oxidation theory, several mechanisms are proposed and some calculations on this bonding strengthening are performed. For the first time, activation energies of this phenomenon are obtained for copper layers deposited by different method in the specific environment of the bonding interface. This study gives guidelines and recommendations for the integration of this metal bonding technique at low temperature.

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#### 1 Introduction

Wafer bonding using metal thin films as intermediate conductive layer is a key technology for the development of three-dimensional integration. Since copper is widely used in CMOS interconnects for its low electrical resistivity and high electromigration resistance (Ryan et al. 1995), direct copper-copper bonding at low temperature is a promising option for implementation of three-dimension integrated circuits (3D-IC) or micro electrical mechanical systems (MEMS). Various bonding techniques exist to join copper surfaces in the temperature range below 400 °C involving for example a uniaxial compressive force (Chen et al. 2005), a surface activation by ion bombardment (Shigetou and Suga 2009), a surface passivation by an organic monolayer (Tan et al. 2009) or chemical mechanical polishing (CMP) steps (Di Cioccio et al. 2011). More specifically, the direct bonding technique involving CMP surface activation consists to put into contact two mirror-polished wafers which are held together by attractive forces at room temperature (RT) without any additional materials.

It has been already reported that copper–copper bonding strength performed at room temperature (20 °C) in cleanroom atmosphere increases with regards to storage time (Di Cioccio et al. 2011). In this paper, we show that this bonding strengthening is not always observed. Particularly, we will study the influence of bonding atmosphere and copper deposition method on this phenomenon. In this way, bonding will be performed in different atmosphere. Moreover, copper layers will be deposited by two different methods namely electrodeposition (ECD) and physical vapor deposition (PVD). In the light of these results and based on metal oxidation theory, bonding interface closure mechanisms in the low temperature range (from 20 to 100 °C) are then proposed. Some calculations are finally performed Fig. 1  $20 \times 20 \ \mu m^2$  AFM scan of copper surfaces obtained by PVD and ECD techniques. **a** After deposition. The RMS roughness is measured to 33 nm RMS. **b** After CMP. The RMS roughness is lowered down to 0.2 nm

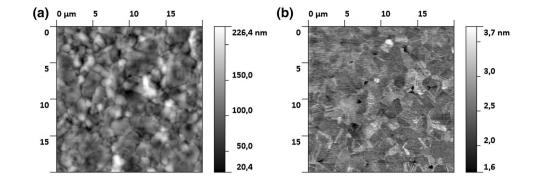


 Table 1
 Experimental studied conditions for copper-copper bonding in terms of deposition technique, bonding atmosphere and post-bonding annealing

| Copper layer deposition            | ECD copper layers       | PVD copper layers |   |                      |
|------------------------------------|-------------------------|-------------------|---|----------------------|
| Bonding atmospheres                | Cleanroom<br>atmosphere | Under<br>vacuum   | Under vacuum after a preannealing at 300 °C | Cleanroom atmosphere |
| Post-bonding annealing temperature | RT and 100 °C           | RT and<br>100 °C  | RT and 100 °C                               | RT, 75 and 100 °C    |

using Arrhenius plots to determine activation energies of this phenomenon for copper layers deposited by two different process which will be compared to free copper surfaces oxidation activation values.

#### 2 Sample preparation

200 mm thermally oxidized (100) silicon substrates are used in this study. Since silicon dioxide layer  $(SiO_2)$  is not an effective barrier, 1-µm-thick copper films are expressly deposited on a 20-nm-thick titanium nitride layer (TiN) which usually prevents copper diffusion in silicon substrate (Holloway et al. 1992). Copper layers deposition is performed by PVD or ECD. For ECD copper films, 200-nm-thick copper seed-layers obtained by PVD are used. An annealing of 400 °C under vacuum is then realized on the structure to improve the adhesion of copper and stabilize its microstructure (Kang et al. 2001).

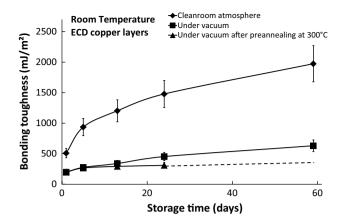
After deposition and annealing, RMS (Root Mean Square) roughness value of 33 nm measured by atomic force microscopy (AFM) on a 20  $\times$  20  $\mu$ m<sup>2</sup> area is too high to allow direct bonding for both copper deposition techniques (Fig. 1a). Thanks to an appropriate chemical mechanical polishing (CMP) step, a final copper layer thickness of 500 nm and a RMS surface roughness value of 0.2 nm on a 20  $\times$  20  $\mu$ m<sup>2</sup> area are obtained (Fig. 1b). Copper surface exhibits after these CMP steps a typical highly hydrophilic behavior (water droplet contact angle below 7°) which stays stable during 1 h (Gueguen et al. 2009). The bonding is performed under three different atmospheres: at room temperature under cleanroom atmosphere (20 °C



Fig. 2 Acoustic microscopy scan of a 200 mm copper-copper stack bonded at room temperature. *Black areas* are bonded

and 40 % of relative humidity), under vacuum (surfaces are brought into contact in a bonding chamber pumped down to  $10^{-3}$  Pa) and under vacuum after a preannealing step (surfaces are heated to 300 °C, cooled down and brought into contact in a bonding chamber pumped down to  $10^{-3}$ Pa). Since a annealing at 400 °C is performed after deposition in order to stabilize copper microstructure, we check that the pre-bonding annealing step at 300 °C in the third case does not change its microstructure (e.g. grain size). After bonding, assemblies are stored at RT in cleanroom atmosphere or annealed at temperature below 100 °C under nitrogen atmosphere. All experimental conditions are summarized in Table 1.

In all cases, defect-free assemblies are characterized by scanning acoustic microscopy (SAM) (Fig. 2) (Khuri-Yakub 1993). No defectivity is observed after post-bonding annealing. Bonding toughness measurements are performed



**Fig. 3** Bonding toughness evolution of copper–copper bonded structures involving ECD copper layers as a function of storage time for three different bonding atmospheres. For sample bonded under vacuum after a preannealing step, the bonding toughness after 24 days of storage is extrapolated (*dash line*)

by the razor blade insertion method (Maszara et al. 1988). This technique was initially developed for brittle materials fracture. Since copper layers are ductile, the measured toughness contains an additional plasticity term (Bertholet et al. 2007). In this work, qualitative comparisons are possible because the thickness of copper films and therefore at first order the mechanical properties of all studied samples are considered equivalent. Relative variation on these measurements carried out by this technique is in range of 15 %. In some cases, copper oxide thickness measurements at the bonding interface are performed by ellipsometry on each side of the assembly just after interface opening.

#### **3** Results

#### 3.1 Bonding atmosphere influence

In this first experimental part, involving only structures with ECD copper layers, bonding toughness behavior is studied depending on various bonding atmospheres: bonding at room temperature under vacuum and bonding at room temperature under vacuum after a preannealing at 300 °C. Figure 3 shows bonding toughness evolution as a function of storage time at room temperature for these three different bonding atmospheres. Bonding toughness value after 60 days of storage for sample bonded under vacuum after a preannealing at 300 °C has not been obtained yet but its future behavior has been extrapolated (dash line on Fig. 3). Toughness of assembly which is bonded in cleanroom atmosphere exhibit the highest value after one day of storage and increases more rapidly than toughness of samples

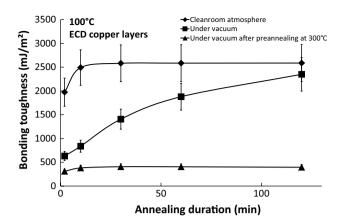


Fig. 4 Bonding toughness measurements performed on copper-copper bonding involving ECD copper layers after post-bonding annealing at 100 °C as a function of annealing duration for three different bonding atmospheres. All samples are opened at Cu–Cu bonding interface

bonded under vacuum. After 60 days of storage, assembly which is performed in cleanroom atmosphere exhibits the thickest copper oxide layer at the bonding interface than sample bonded under vacuum (Table 1).

After 60 days of storage at room temperature or 24 days for sample bonded under vacuum after a preannealing at 300 °C, an annealing at 100 °C for different durations in nitrogen atmosphere is carried out. Figure 4 shows bonding toughness with regards to the 100 °C annealing duration for the three different bonding atmospheres. Toughness of assembly which is performed in cleanroom atmosphere increases until 2.5 J/m<sup>2</sup> and stays quite constant with additional thermal budget. For bonding under vacuum, an increase of bonding toughness is measured which finally reaches about 2.5 J/m<sup>2</sup> after 2 h at 100 °C. However, for the preannealed sample before bonding under vacuum atmosphere, bonding toughness slightly increases and then remains roughly constant at 0.4 J/m<sup>2</sup> at 100 °C for the maximal postbonding annealing durations performed here. Oxide thicknesses at bonding interface are equivalent to 4 nm for samples bonded under cleanroom and vacuum atmospheres, whereas no oxide is detected by ellipsometry for sample bonded under vacuum after the preannealing step (Table 2).

In this section, we showed the effect of various bonding atmospheres for samples involving copper layer deposited with the same deposition technique. Since the cleanroom bonding atmosphere is more favorable for bonding strengthening at room temperature in the following we will keep this process parameter and vary the deposition methods for copper.

## 3.2 Copper deposition influence

ECD and PVD copper layers are bonded in a cleanroom atmosphere. After CMP surface activation, roughness and

 Table 2
 Oxide copper thickness measured by ellipsometry after mechanical opening right after a storage time at room temperature of 60 days

| Bonding atmosphere                                    | Cleanroom atmosphere (nm) | Under vacuum (nm) | Under vacuum after preannealing at 300 °C |
|---|---------------------------|-------------------|---|
| Copper oxide thickness after 60 days of storage at RT | $3.23 \pm 0.43$           | $0.00 \pm 0.24$   | Not measured                              |
| Copper oxide thickness after 120 min<br>at 100 °C     | $4.31\pm0.49$             | $4.29\pm0.49$     | $0.00 \pm 0.24$ nm                        |

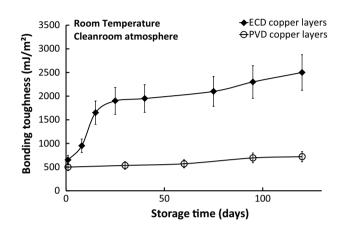


Fig. 5 Bonding toughness evolution with storage time at room temperature for copper-copper bonding performed with copper layers deposited by two different techniques and under cleanroom atmosphere

hydrophilic surface behavior are equivalent for the two copper layers deposited by the two different techniques (as measured by respectively, AFM and water drop contact angle). Figure 5 shows bonding toughness evolution with storage time at room temperature for copper layer deposited by ECD and PVD technique. Assembly performed with ECD copper layers exhibit an increase of bonding toughness until 2.5 J/m<sup>2</sup> after 120 days of storage in good agreement with literature results (Di Cioccio et al. 2011). Samples which are performed with PVD copper layers exhibit low bonding toughness evolution from 0.5 to 0.7 J/m<sup>2</sup> after 120 days of storage. A bonding toughness measurement performed after 10 months of storage shows only an additional improvement to reach values of 0.9 J/m<sup>2</sup> (not shown on Fig. 5).

A low temperature post-bonding annealing is then applied on both structures just after bonding. Figure 6 shows bonding toughness evolution as a function of annealing duration for both copper depositions with regards to different thermal budgets. Different bonding strengthening kinetics can be observed: samples with ECD copper layers have a fast bonding strengthening kinetics compared to those with PVD copper layers. Bonding toughness values of 2.5 J/m<sup>2</sup> can be obtained at 100 °C during half an hour for ECD samples whereas 10 h are necessary for PVD samples.

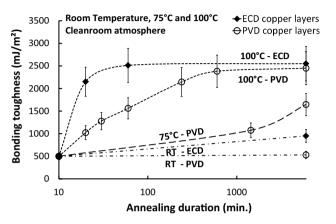


Fig. 6 Bonding toughness as a function of post-bonding annealing duration (with a logarithmic scale). Effect of low temperature annealing for two different copper deposition process (bond closure under cleanroom atmosphere). Bonding toughness measurements were not performed for ECD deposited sample annealed at 75  $^{\circ}$ C

For samples with PVD copper layers, copper oxide thickness at bonding interface are compared after 6,000 min of storage at room temperature or annealing duration at 100 °C (Table 3). A relationship between bonding toughness value of 2.5 J/m<sup>2</sup> and a 4-nm-thick copper oxide layer presence at the bonding interface is observed.

#### 4 Discussion

#### 4.1 Water role on bonding strengthening

The mechanism of the bonding strengthening in the (RT, 100 °C) temperature range is already reported as a metal oxidation at the bonding interface during storage or annealing at low temperature (Di Cioccio et al. 2011). At room temperature, they show that oxidation leads especially to a bonding toughness increase until 2.5 J/m<sup>2</sup> which is correlated to the formation of a 4-nm-thick copper oxide layer of at the bonding interface (observed by High Resolution Transmission Electron Microscopy—HRTEM). X-Ray Reflectivity (XRR) measurements also confirm this assumption since the measured drop of electron density at the bonding interface during storage at room temperature

Table 3 Oxide copper thickness measured by ellipsometry after mechanical opening after the application of a thermal budget on samples bonded with PVD copper layers

| Thermal budget applied on bonded samples              | 6,000 min at 20 °C | 6,000 min at 100 °C |
|---|--------------------|---------------------|
| Copper oxide thickness just after sample opening (nm) | $0.56 \pm 0.32$    | $4.78\pm0.53$       |

correspond to the creation of an interfacial layer of cuprous copper oxide (Cu<sub>2</sub>O) (Di Cioccio et al. 2011).

Hydrophilic bonding interface is a confined environment with a specific amount of water and gazes encapsulated. At the direct metal bonding interface, adsorbed water monolayers, brought into contact during bonding, interact with metal layers through an oxidation reaction. Two behavior of water molecules adsorbed on hydrophilic surfaces can be distinguished: molecules linked to metal atoms and superficial water molecules weakly linked to each other (Danielson 2014). Copper surfaces placed under vacuum condition down to  $10^{-3}$  Pa before bonding can lose a part of adsorbed water especially the weakly linked superficial monolayers. A thermal preannealing at 300 °C under vacuum can also promote full adsorbed water molecules to desorb (Roth 2012). Our experimental results realized with different bonding atmospheres highlight the role of the amount of encapsulated water during copper-copper bonding to be a key parameter to control bonding strengthening (Figs. 3, 4). Indeed, a faster bonding strengthening is observed for bonding performed under a higher relative humidity atmosphere (cleanroom atmosphere here at 40 %) for copper surfaces sensitive to water oxidation mechanisms.

Metal oxidation theory at our low temperature range and for very thin films determines that oxidation rate is limited by chemical species diffusion, especially by metallic anion diffusion through oxide layer (Lawless 1974). In order to activate faster oxidation kinetics, an additional thermal budget at 100 °C is applied on bonded structures (Fig. 4). A bonding toughness evolution is observed for two samples which reach a value of 2.5 J/m<sup>2</sup>: bonding performed in cleanroom atmosphere (with a fast kinetics less than 30 min) and bonding performed under vacuum (with a thermal budget of 2 h at 100 °C). This may confirm that copper surfaces placed in vacuum down to  $10^{-3}$  Pa do not desorb the whole of adsorbed water monolayers since an oxidation takes places at 100 °C. Annealing under vacuum above 100 °C result in total surface water desorption and also a partial surface carbon contaminants cracking (Roth 2012). Partial surface passivation may induce a slower bonding toughness strengthening due to oxide growth and the limitation of bonding toughness to an asymptotic value of 0.4 J/m<sup>2</sup> even after 120 min at 100 °C.

#### 4.2 Impact of copper layers deposition technique

Since ECD technique is often used in industrial context, most of copper-copper direct bonding studies are performed on copper layers deposited by this technique (Di Cioccio et al. 2011; Gueguen et al. 2009; Martinez et al. 2013). However, some applications could need other copper layer deposition technique such as PVD. The aim of this paper is a comparison of the strengthening behavior at low temperature of copper layers deposited by ECD and PVD. Surface preparation steps on copper layers deposited by these two different techniques just before bonding lead to equivalent surface properties in terms of roughness and hydrophilic behavior. So the difference of bonding toughness strengthening kinetics observed at room temperature between samples deposited by different techniques (Fig. 5) should be linked to different chemical parameters.

Metal oxidation theory describes a specific mechanism for very thin oxide film formation (below 10 nm) in the low temperature range (from 20 to 200 °C) (Cabrera and Mott 1949). In ambient atmosphere, oxygen and water are readily adsorbed on metal surface and form the first oxide monolayers. A strong electric field exists between the adsorbed oxygen layer–oxide interface and the metal–oxide interface. Under the action of this field, ions anions diffuse through the oxide layer until reaching adsorbed oxygen. This matter transport leads to the thickening of the oxide layer. For the temperatures and oxide thickness considered in our study, the rate of oxidation is only determined by metal anion diffusion through the oxide layer (Cabrera and Mott 1949; Lawless 1974).

ECD and PVD techniques mainly differ by impurities amount incorporated during deposition. On the one hand, PVD consists in sputtering of a pure copper target by argon plasma in vacuum leading to a copper layer with a pure composition. On the other hand, ECD uses an electrodeposition bath containing additives such as suppressors, inhibitors and accelerators. Chlorides, sulfurs and carbon compounds are incorporated into copper layers deposited by this technique (Kondo et al. 2002).

Impurities incorporation in copper layers could explain the different results with regards to storage at room temperature and low temperature annealing. The presence of chemical species in the subsurface of copper layers could modify the field value between interfaces and change the oxidation kinetics. This may explain bonding strengthening kinetics difference between structures bonded with PVD and ECD copper layers.

# 4.3 Modelisation of oxidation at bonding interface at low temperature

In this part, bonding toughness strengthening will be treated by standard chemical rate theory (thermal activation and diffusion kinetic: we assume a direct relation between interfacial oxide growth and bonding toughness). Bonding toughness is measured for three different temperatures for structures involving PVD copper layers (20, 75 and 100 °C) and two temperatures for structures involving ECD copper layers (20 and 100 °C) (Fig. 6).

Bonding toughness and copper oxide thickness measurements show that the asymptotic bonding toughness value of 2.5 J/m<sup>2</sup> can be correlated to the formation of 4 nm of cuprous oxide at the bonding interface (Di Cioccio et al. 2011). Cabrera and Mott predicts that copper oxidation in the considered temperature range is a phenomenon limited by copper anion diffusion which follows a power law (Eq. 1).

$$d_{\rm Cu_2O} = a.t^b \tag{1}$$

where  $d_{\text{Cu}_2\text{O}}$  is the copper oxide thickness at the bonding interface, *t* represents the time (e.g. storage time or annealing duration) and *a*, *b* are constants. Nika and Hall established a time dependence of  $t^{1/2}$  for the oxidation of copper films in ambient air in free surface configuration from 100 to 300 °C (Nika and Hall 1979). We consider that metallic anion diffusion mean free path of diffusion equation:

$$d_{\rm Cu_2O} = \sqrt{2Dt} \tag{2}$$

Based on Eq. 1 and bonding toughness measurements, durations required to reach 2.5  $J/m^2$  (which means formation of 4 nm of copper oxide at bonding interface) are extrapolated in the case of assemblies involving PVD copper layers at RT and 75 °C. Then, the diffusion coefficient of metal anions through the oxide layer is determined for each temperature which can be described by Eq. 3.

$$D(T) = D_0 e^{\frac{-E_a}{kT}} \tag{3}$$

where D is the diffusion coefficient of copper through the oxide layer,  $D_0$  is a pre-exponential factor,  $E_a$  is the activation energy of the phenomenon, k is the Boltzmann constant and T is the absolute temperature. From Arrhenius plots (Fig. 7), apparent activation energies of 0.9 and 2.1 eV respectively, for ECD and PVD copper layers have been determined for this oxide layer growth in this experimental configuration. High incertitude is considered for the value corresponding to storage at 20 °C of structures bonded with PVD copper layers since kinetics is very slow.

Apparent activation energy value difference between ECD and PVD process is consistent with literature results: Indeed, Nika and Hall calculated values from 0.87 to 1.89 eV in free copper surface configuration in ambient air.

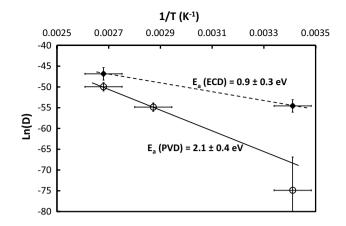


Fig. 7 Arrhenius plot associated to the temperature-activated phenomenon of bonding toughness strengthening at low temperature

They also showed that copper layers obtained by evaporation deposition technique (which have impurities concentration quite close to PVD) show higher apparent activation energies values compared to ECD copper layers.

### 5 Conclusion

Copper-copper direct bonding using CMP surface activation has been studied in the low temperature range between room temperature and 100 °C. Since bonding toughness strengthening at room temperature is not always measured, we have determined several factors which could affect this phenomenon. Firstly, it clearly appears that water presence at the bonding interface is necessary to obtain a good bonding strengthening at room temperature. Secondly, copper layers deposition technique have also an impact on bonding strengthening kinetics: copper layers deposited by electrodeposition exhibit faster bonding strengthening. Impurities presence seems to be a key parameter of this strengthening phenomenon. Activation energy for toughness strengthening give energies of 0.9 and 2.1 eV respectively, for structures bonded with ECD and PVD copper layers in cleanroom atmosphere. Finally, this study gives guidelines and recommendations to optimize bonding at very low temperature.

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