

# Influence of metal annealing deposited on oxide layer

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**Abstract:** We investigated the influence of RTP annealing of multi-layered metal films deposited on oxides layer. Two types of oxides, BPSG and P-TEOS, were used as a bottom layer under multi-layered metal film. The bonding was not good in metal/BPSG/Si samples because adhesion between metal layer and BPSG oxide layer was poor by interfacial reaction during RTP annealing above 650°C. On the other hand bonding was always good in metal/ P-TEOS /Si samples regardless of annealing temperature. We observed the interface between oxide and metal layers using AES and TEM. The phosphorus and oxygen profile in interface between metal and oxide layers were different in metal/BPSG/Si and metal/P-TEOS/Si samples. We have known that the properties of interface was improved in metal/BPSG/Si samples when the sample was annealed below 650°C.

## 1. Introduction

Recently semiconductor process technology has been rapidly developed and IC chips with hundreds of millions transistors are manufactured by sub-quarter micrometer technology. As the feature size of devices reduces, many new materials are developed and device structure is changed to improve the characteristics and reliability of devices. In addition, the processing of manufacturing is more complicated and the processing steps for IC fabrication are more increasing as device size become smaller and device integrity become higher. As the wafer size and manufacturing steps are increasing the condition of processing for IC fabrication is more severe. Therefore, scientists and engineers have been developing the new manufacturing technologies to overcome this problem.

In device processing, however, some phenomena are generated during the device processing and they have a bad effect on device properties. Such phenomena were mainly generated at the interface between two thin films and the study on the interfacial reaction is one of the important technologies for improving device characteristics. Oxide film and metal film were widely used as a thin films in

microelectronics devices. Oxide films were used as gate oxide, IMD (intermetal dielectric) and CMP processing for planarization, and dielectric materials for capacitor[1-4]. LOCOS for field oxide are used to isolate neighboring devices. However, STI (shallow trench isolation) replaced LOCOS processing as the feature size continue to decrease because there are two problems, bird's beak and decreasing of active area, in LOCOS[5]. Multi-layered metal films composed of Al, Ti, TiN film were used to improve the metal line reliability[6,7]. Ti film has been used to reduce the contact resistance to Si and TiN film has been used as a diffusion barrier layer and anti-reflection layer. In the last three years, copper will replace the aluminium to improve the device performance because copper has low resistivity and high resistance to electromigration [8]. In this paper, we investigated the effects of metal annealing deposited on oxide layer. Particularly, the interface effect between oxide layer and metal layer is characterized as a function of metal annealing temperature.

## 2. Sample Fabrication

We used BPSG (borophosphosilicate glass) and P-TEOS (plasma enhanced chemical vapor deposition TEOS) oxide films as a bottom layer under multi-layered metal films. Each of oxide films was deposited on the (100) P-type bare silicon wafer. BPSG oxide film was deposited on the Si substrate by APCVD using TEOS [tetraethyl-orthosilicate,  $\text{Si}(\text{OC}_2\text{H}_5)_4$ ], TMOP [trimethylorthophosphite,  $\text{PO}(\text{OCH}_3)_3$ ], and TEB [triethyl-borate,  $\text{B}(\text{OC}_2\text{H}_5)_3$ ]. The thickness of BPSG oxide film was 9000 Å and the concentrations of boron and phosphorus in BPSG were 14 mol % and 6 mol %, respectively. Then, BPSG oxide film on silicon substrate was densified at 850 °C, 30 min in  $\text{N}_2$  ambient in the furnace. P-TEOS oxide film with thickness of 9000 Å was deposited on the Si substrate by PECVD only using TEOS. 600 Å Ti thin film and 600 Å TiN thin film were successively deposited by sputter on the BPSG/Si substrate and the P-TEOS/Si substrate without breaking the vacuum. Two samples, metal/BPSG/Si and metal/P-TEOS/Si, were

RTP annealed at 550 °C, 650 °C, 700 °C, and 800 °C, 30 sec in N<sub>2</sub> ambient. Then, Al film (5000 Å) and TiN (400 Å) film were successively deposited by sputter. The pad with 110 μm × 110 μm was formed by photolithography and bonding test was carried out to evaluate the interface effect on metal annealing temperature.

### 3. Results and Discussion

The samples RTP annealed at 650 °C, 30 sec in N<sub>2</sub> ambient after deposition of Ti(600 Å) and TiN(600 Å) films on the oxide film were bonding tested. The results of bonding test are shown in Table 1.

Table 1. The results of bonding test in metal/BPSG/Si sample and metal/P-TEOS/Si sample. All the samples were annealed at 650 °C, 30 sec in N<sub>2</sub> ambient.

Type of oxide	metal thickness (Å)	Error rate (%)	The number of samples
P-TEOS	5000Å	0	5548
P-TEOS	5000Å	0	5472
BPSG	5000Å	2.2	2774
BPSG	5000Å	1.3	1292
BPSG	5000Å	1.4	3040

Bonding failure was occurred in the pad of metal/BPSG/Si substrate sample. Multi-layered metal films on the BPSG oxide layer came off during the bonding test because adhesion was not good between metal layer and BPSG oxide layer. But no bonding failure was occurred in metal/P-TEOS/Si substrate samples. The pad of failed sample and cross section of failed sample were observed by SEM, which is shown in Fig. 1. Fig. 1 (b) illustrates multi-layered metal films on the BPSG oxide layer were peeled off. Fig. 2 shows the AES depth profile of bonding failed sample. The analysis was performed from the BPSG surface because the multi-layered metal film was removed by bonding test. The BPSG surface of bonding failed sample was surveyed before AES depth profile. Ti, B, P, O, and Si peak was observed in the surface and C peak was observed, too because of contamination. A phosphorus piled up in metal and BPSG interface as shown in Fig. 2. The concentration of phosphorus in metal and BPSG interface is higher than that of deeper region inside BPSG. The thickness of phosphorus rich layer was about 200 Å regarding as the etch rate of oxide was 100 Å/min by Ar in AES system. It was assumed that this phosphorus rich layer was formed according as phosphorus in BPSG is moving to interface when metal film was annealed at 650 °C. Fig. 3 shows the the AES depth profile of the normal pad of metal/BPSG/Si substrate sample. The analysis was performed after the upper multi-layered metal films on the BPSG film was sputter etched by Ar. Phosphorus rich layer could not be observed in metal and BPSG interface. The difference Fig. 2 and Fig. 3 is piled up phosphorus region in

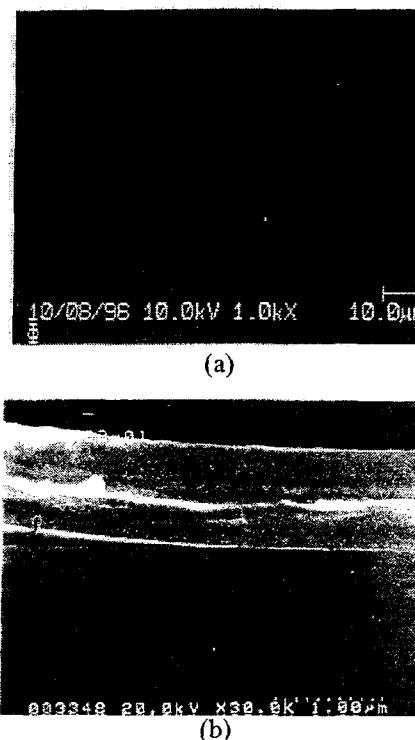


Fig.1. The photograph of the pad of failed sample (a) and the cross section of bonding failed sample at interface between metal and oxide films (b).

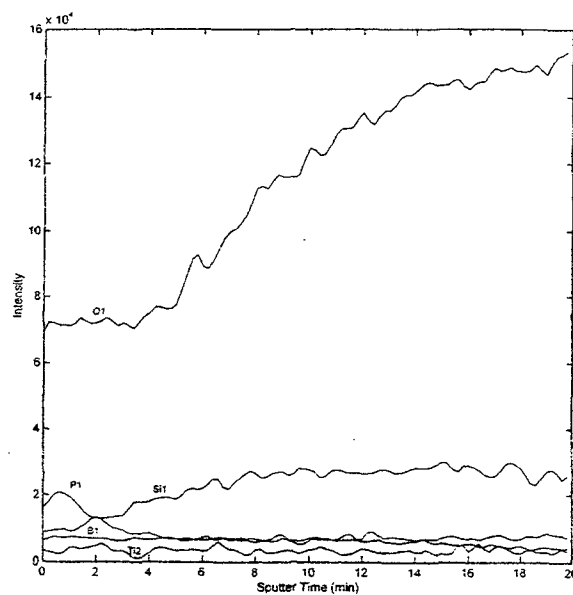


Fig. 2. AES depth profile of bonding failed pad of metal/BPSG/Si substrate structure.

the metal and BPSG oxide interface. The other difference was oxygen profile in the interface between metal and Oxide layers. Oxygen concentration in the failed sample decreased near the metal and BPSG interface. The variation of oxygen concentration, however, in the normal pad of the

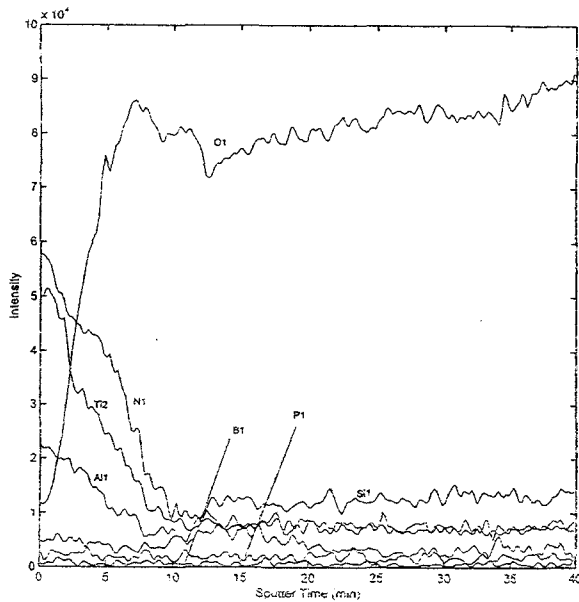


Fig. 3. AES depth profile of normal pad of metal/BPSG/Si substrate structure.

metal/BPSG/Si sample very low in the metal and BPSG interface. It was assumed that the pile up effect of phosphorus and the difference of oxygen profile were generated during the RTP anneal process. We observed the metal and BPSG oxide interface of bonding failed metal/BPSG/Si sample using TEM. Fig. 4 shows TEM photograph of bonding failed sample. From Fig. 4, we can see phosphorus rich layer in interface between metal and BPSG oxide, which is different image as compared with those of BPSG and metal film. This layer could be found in the limited area, not all area in metal and the BPSG oxide interface. This result was consistent with the results of AES analysis. Its thickness was about 200 Å and it was good agreement with the phosphorus rich layer thickness of bonding failed pad of metal/BPSG structure observed by AES depth profile. In TEM analysis the interface between

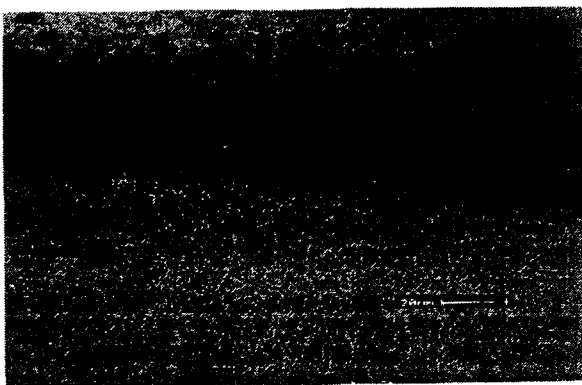


Fig. 4. TEM photograph of bonding failed sample in metal and BPSG oxide interface.

metal and P-TEOS oxide of metal/P-TEOS/Si samples was very clean. We examined the effect on RTP annealing temperature in metal/BPSG/Si and metal/P-TEOS/Si samples. The samples were annealed at different RTP temperatures in N<sub>2</sub> ambient. The metal/BPSG/Si samples were RTP annealed at 550 °C, 650 °C, 700 °C, and 800 °C after depositing Ti and TiN on BPSG oxide film. The metal/P-TEOS/Si samples were RTP annealed at 650 °C, 800 °C and no annealed after depositing Ti and TiN on P-TEOS oxide film. The results of the bonding test of these samples are shown in Table 2.

Table 2. The effect RTP annealing temperature of the metal/BPSG/Si substrate and metal/P-TEOS/Si samples.

Types of oxide	RTP anneal temperature	Error rate (%)	The number of samples
P-TEOS	no annealed	0	1872
P-TEOS	650 °C	0	1872
P-TEOS	800 °C	0	1872
BPSG	550 °C	0	1872
BPSG	650 °C	3.8	1872
BPSG	700 °C	2.6	1872
BPSG	800 °C	2.6	1872

In the case of metal/BPSG/Si samples bonding failure was occurred when the samples were RTP annealed only above 650 °C. On the other hand, bonding failure was not occurred in the metal/P-TEOS/Si samples regardless of RTP annealing temperature.

If RTP annealing temperature was below 650 °C, bonding failure was not found even though the samples were fabricated using BPSG oxide as a bottom layer under multi-layered metal films. It was considered that the interfaces reaction forming phosphorus rich layer was not activated in metal/BPSG/Si samples. From these results, we confirmed that in metal/BPSG structure bonding failure resulted from interfacial reaction piling up phosphorus in the interface between metal film and BPSG oxide film during RTP annealing process above 650 °C.

#### 4. Conclusions

We observed the interface between metal layer and oxide layer to evaluate the effect of metal annealing. The samples were fabricated by depositing multi-layered metal films on oxide film. BPSG and P-TEOS oxide films were used as a bottom layer under multi-layered metal films. The fabricated samples were RTP annealed at 550 °C, 650 °C, 700 °C, and 800 °C in N<sub>2</sub> ambient and no annealed after deposition of Ti and TiN layers.

Bonding failure was occurred in metal/BPSG/Si samples annealed above 650 °C and bonding was always good in metal/P-TEOS/Si samples. Observing the failed samples by SEM, AES, and TEM, phosphorus piled up and oxide

concentration decreased in metal and BPSG oxide interface. It was considered to be generated from interfacial reaction during RTP annealing process.

Conclusively, we have known that multi-layered metal films deposited on BPSG oxide must be annealed below 650°C to improve the adhesion multi-layered metal films and BPSG oxide and the samples with metal/PETEOS/Si are independent of annealing temperature.

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