

## Development of different copper seed layers with respect to the copper electroplating process

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### Abstract

Two types of copper seed layers deposited by MOCVD and long throw sputtering (LTS) onto a tantalum barrier layer were used for electroplating (EP) of copper in the forward pulsed mode. MOCVD and PVD copper seed layers were compared with respect to step coverage, electrical resistivity, texture and adhesion behaviour. The different properties induce different electroplating fill attributes, including grain size and adhesion behaviour. MOCVD Cu seed layers show high step coverage, but do not adhere to the Ta barrier after the Cu EP. LTS Cu reveal strong (111) texture and excellent adhesion before and after Cu EP. Therefore, a CMP process could only be performed on patterned wafers with PVD/EP copper to obtain electrical data. The fabricated Cu lines show a high yield with respect to opens and shorts and standard deviations of the line resistance across the wafer. © 2000 Elsevier Science B.V. All rights reserved.

**Keywords:** MOCVD; PVD; Copper; Seed layer; Electroplating

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### 1. Introduction

The transition from aluminium to copper interconnects introduces new processes and materials into semiconductor manufacturing, e.g., dual damascene process flow, Cu electroplating and new diffusion barrier materials. Electroplating is an attractive deposition method for copper with the need for a conformal seed layer [1]. This seed layer can be provided by several deposition techniques. Particular properties including low thickness concurrent with coherence, low electrical resistivity, smooth surface and good adhesion to the barrier layer are required. In this paper the development of PVD and MOCVD copper seed layers is reported with respect to requirements of the electroplating process.

### 2. Experimental

150 mm substrates were used with 50 nm IMP PVD Ta and 20 nm PVD TiN as barrier layer.

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Patterned wafers have 1000 nm deep single damascene test structures in SiO<sub>2</sub>. Copper seed layers were deposited by MOCVD and long throw sputtering (LTS). The MOCVD process was performed ex situ on an Applied Materials Precision 5000 cluster tool using a precursor mixture of Cu(hfac)TMVS with the additives H(hfac) · 2 H<sub>2</sub>O (0.4 wt%) and TMVS (5 wt %). LTS Copper was deposited at 50°C in an Applied Materials Endura PVD tool in situ with the Ta barrier to achieve a smooth and conformal seed layer. Copper fill was done by electroplating with OMI Cubath SC chemistry and forward pulsed conditions using modified Semitool Equinox Radial equipment. The wafers were annealed afterwards at 250°C for 10 min in forming gas (5% H<sub>2</sub> in N<sub>2</sub>) to avoid self annealing effects and to provide stable layer conditions for CMP processing. Finally, copper and barrier CMP was performed on a IPEC Avanti 472 Single Head Polisher using a two-step polishing process and a touch up at the final platen. The first polishing step is the Cu removal with a high selectivity to Ta. The second polishing step removes the Ta with a high selectivity to Cu and less selectivity to the oxide to achieve a good degree of planarisation.

The characterisation of copper films was done by the determination of sheet resistance with a four-point probe method, film thickness with an alphastep profilometer, SEM, FIB, TEM, as well as by AFM and AES at selected samples. X cut tape tests with defined adhesion strength (10 N/25 mm) were performed to assess the adhesion.

### 3. Results

The MOCVD and PVD seed layer properties are summarised in Table 1.

#### 3.1. CVD Cu seed layer

The copper MOCVD seed layer was fabricated according to a three-step method. At first an Ar

Table 1  
Comparison of different seed layers and stack properties

	MOCVD seed layer	Long distance PVD seed layer
<i>Seed layer properties:</i>		
Thickness (nominal)	50–60 nm	100 nm
Step coverage Cu seed	75%	30%
Resistivity	4–6 μΩ cm	2.9 μΩ cm
Surface roughness ( $R_a$ by AFM)	5.5% of film thickness	2.6% of film thickness
Texture	No	Strong (111)
Adhesion (tape test)	Pass after annealing	Pass as-deposited
<i>Metal stack properties:</i>		
Step coverage electroplating	100%, no voids	100%, no voids
Resistivity	2.2 μΩ cm	2.0 μΩ cm
Grain size	Small	Large
Adhesion (tape test), as-deposited:	Do not pass	Pass
Adhesion (tape test), after annealing:	Do not pass	Pass

plasma pre-treatment (100 W, 120 s) was applied to ensure high nuclei density and very thin coherent seed layers, respectively [2]. Then the MOCVD at 150°C, 20 Torr, 650 mg/min precursor flow followed. Copper seed layers with a thickness of 37 nm and a uniformity of  $<5\%$  ( $1\sigma$  of sheet resistance) could be obtained by this method. But the adhesion of these CVD Cu seed layers is poor. Complete delamination occurred due to the tape test. This effect was noticed on TiN as well as on Ta as barrier material. An improvement of the adhesion could be obtained by a post-annealing at 350°C for 5 min being the third step. After annealing the copper films adhere excellently to the barrier layer. No copper was removable by the tape test. The MOCVD seed layer as well as the interface between seed and barrier layer was investigated with respect to the annealing effects.

AES depth profiles (Fig. 1) revealed the elements carbon and fluorine at this interface, which are assumed to originate from the precursor mixture. They were detected before and after annealing. A change in the amounts of carbon and fluorine at the interface Cu/TiN could not be detected. The small differences may base on lateral variations across the wafer. These findings correspond with TEM analysis (see Fig. 2). A 2–6 nm thin continuous layer between copper and barrier was detected by cross-section TEM. It still exists after annealing. However, the impurity level does not correlate with the noticed adhesion effects.

Furthermore texture of the seed layer on Ta was investigated by XRD. The measurements indicate no pronounced texture of the annealed Cu, which can be expressed by the peak intensity ratio  $I(200)/I(111)$ . It is 0.46 for randomly oriented powdered Cu samples. This factor reduces from 0.58 for as-deposited copper to 0.35 for annealed copper. A slight peak displacement relative to the theoretical position towards a higher angle was noticed. This displacement increases due to annealing and indicates stress within the copper.

A cross-section of a MOCVD Cu seed layer on Ta in a trench is shown in Fig. 3. A good step coverage of 75% was achieved. This metal stack was used for the Cu EP investigations.

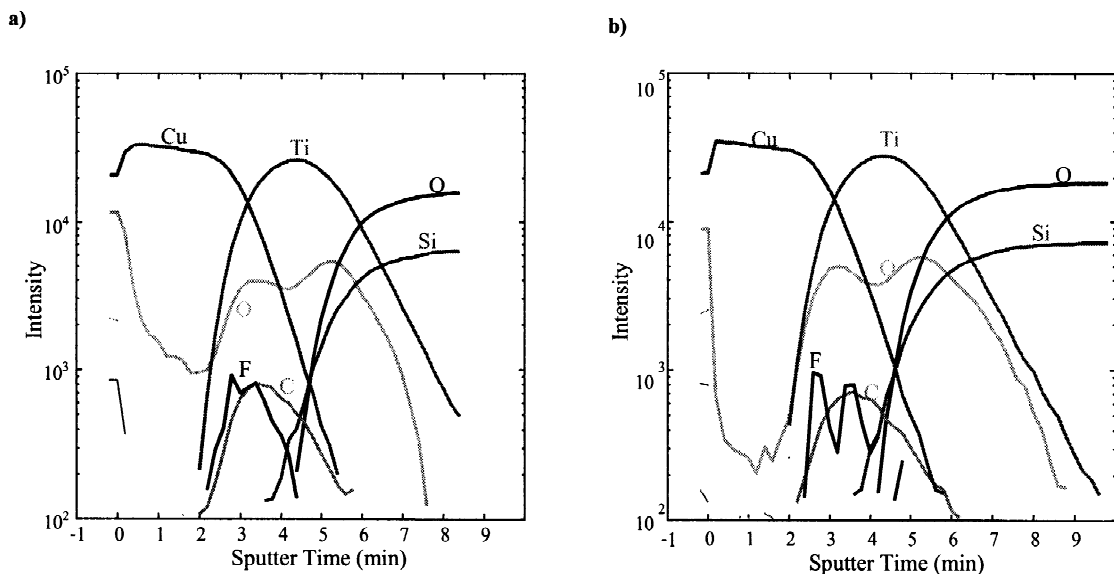


Fig. 1. AES depth profiles of the layer stack Cu/TiN/SiO<sub>2</sub>: (a) as-deposited; (b) after annealing (350°C, 10 min, vacuum, in situ).

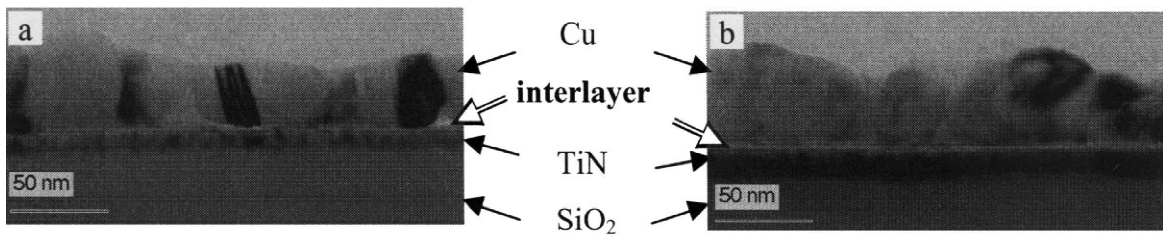


Fig. 2. Cross-section TEM reveals a thin interlayer between copper and plasma pre-treated barrier layer (a) before and (b) after annealing (350°C, 10 min, vacuum, in situ).

### 3.2. PVD Cu seed layer

A 100 nm thick PVD Cu seed layer deposited in a high aspect ratio trench (AR = 4) is depicted in Fig. 4. Higher nominal film thicknesses are needed due to the low step coverage of the PVD seed layer. The surface roughness assessed by AFM was lower compared to MOCVD Cu. The half mean roughness value was determined with 2.6% of the film thickness, indicating a very smooth surface. A further difference between PVD and CVD copper seed layer concerns textures. Whereas for the CVD copper after annealing a random orientation of the Cu grains was noticed, the PVD copper has a strong (111) texture as-deposited. No peaks beside the (111) were detected. The adhesion behaviour

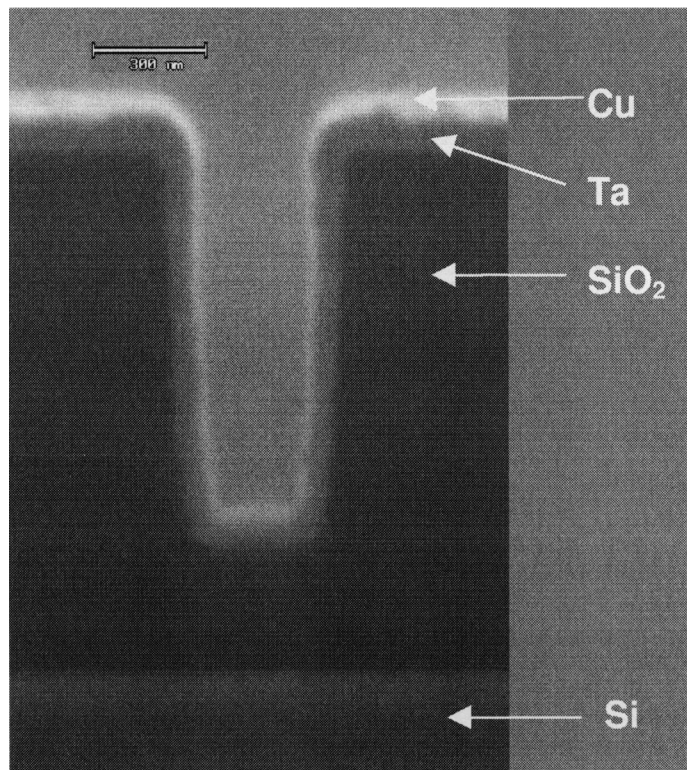


Fig. 3. CVD copper seed layer in a trench (AR = 4, annealed at 350°C, 10 min).

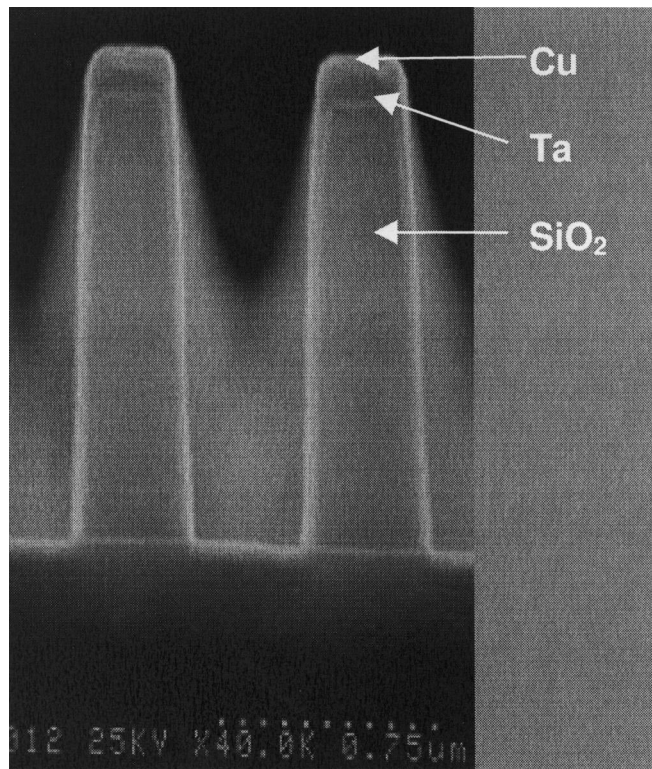


Fig. 4. As-deposited PVD copper seed layer in a trench (AR = 4, PVD at 50°C).

of PVD copper is excellent. It passes the tape test at the as-deposited stage. A remarkably lower specific electrical resistivity was determined for PVD copper than for MOCVD copper due to the different film thickness and surface roughness.

### 3.3. Cu EP fill

The trench fill by electroplating is very good, no voids could be found for either type of seed layer. But different seed layer properties induce different electroplating fill process attributes (Figs. 5 and 6). For PVD seed layer processing an epitaxial growth of the electroplated film on the seed layer is detectable, while for the MOCVD process flow a boundary formation between the seed layer and the electroplated film occurs. A slightly lower electrical resistivity was measured for EP Cu on a PVD seed, and the cross-section of this copper film reveals a bigger grain size than that of EP Cu on MOCVD seed. The adhesion behaviour of the Cu films after electroplating is completely different for the two types of seed layers. After the annealing before CMP PVD/EP Cu films passed the tape test but MOCVD/EP Cu films did totally fail (delamination at the CVD Cu/Ta interface). The same result was noticed for as-deposited MOCVD/EP copper. Due to this matter no CMP processing was possible on this metal stack. A further optimisation of the MOCVD Cu deposition with respect to adhesion is required.

Results of electrical measurements on non-passivated double track meander (DTM) structures with

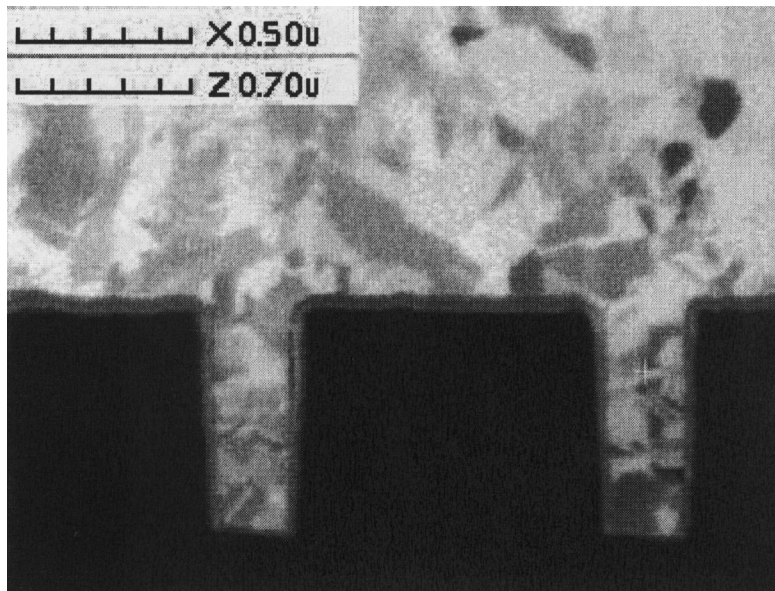


Fig. 5. Copper EP fill on MOCVD Cu seed layer (tilted FIB image).

0.4  $\mu\text{m}$  line width and 0.4/3.6  $\mu\text{m}$  spacings are shown in Figs. 7 and 8 (metal stack: Ta barrier/PVD Cu seed/EP Cu). A high yield (about 98%) of the DTM structures was achieved. The within-wafer and wafer-to-wafer variation of the line resistance values are below 10% (min–max). The goal value

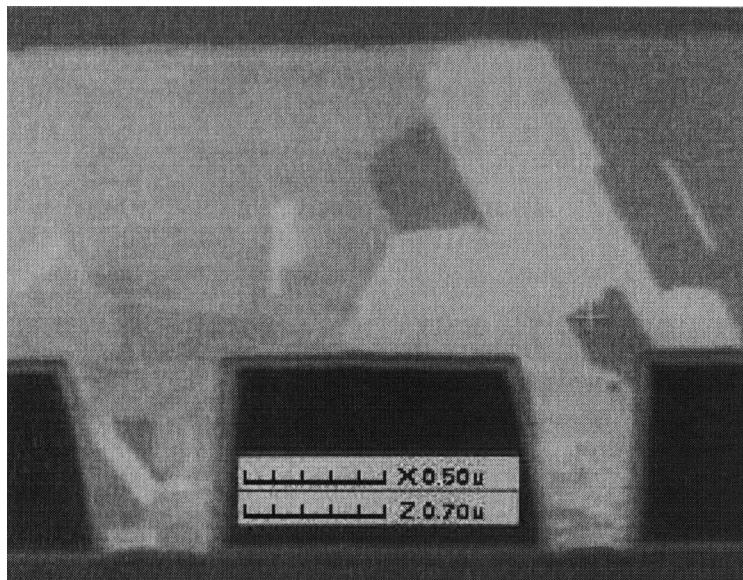


Fig. 6. Copper EP fill on PVD Cu seed layer (tilted FIB image).

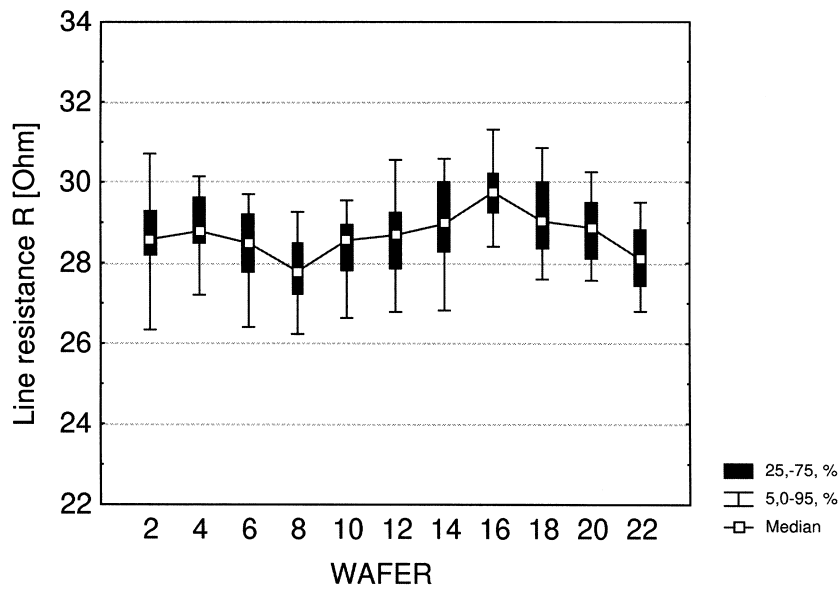


Fig. 7. Line resistance of DTM structures with 0.4  $\mu\text{m}$  width (Ta/LTS Cu/EP Cu).

of 29  $\Omega$  for the line resistance, which was calculated from the trench geometry and the resistivity of the blanket barrier and Cu films, was very well achieved. This can be attributed to good control of the CMP as well as to no degradation of the Cu resistivity in the trenches during processing. The leakage current between the unpassivated Cu meander structures is comparable to a standard passivated AlCu metallisation.

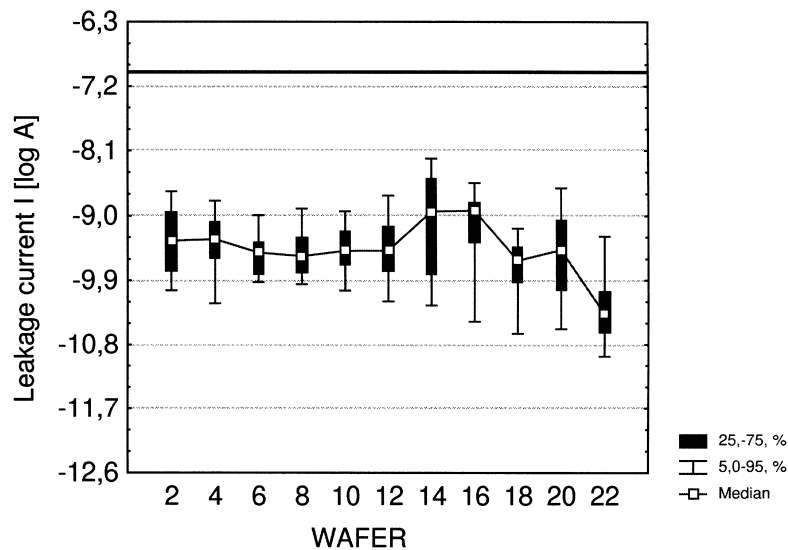


Fig. 8. Leakage current at DTM structures with 0.4  $\mu\text{m}$  width and 0.4/3.6  $\mu\text{m}$  spacings (Ta/LTS Cu/EP Cu).

#### 4. Conclusion

A comparison of MOCVD and PVD copper seed layers revealed different properties. For the same side wall thickness PVD seed layers have to be two times thicker than MOCVD seed layers because of the lower step coverage. Due to this matter and a smoother surface the electrical resistivity measured at blanket films is lower. Whereas the MOCVD copper shows no pronounced texture the PVD copper has a strong (111) texture. Both types of seed layers did pass the adhesion tape test on Ta, but the MOCVD Cu had to be annealed to achieve good adhesion.

The EP copper films have no voids for both types of seed layers. Due to bigger grain sizes the EP/PVD copper has a slightly lower electrical resistivity of  $2.0 \mu\Omega \text{ cm}$ . The adhesion behaviour was found to be completely different for the two types of seed layers. Only PVD/EP copper films passed the tape test. Therefore, only the Ta/PVD Cu/EP Cu metal stack could be polished and electrically measured. Electrical data show excellent yield, line resistance control, excellent leakage current between unpassivated Cu lines and a tight distribution of the measured values.

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