Electron Backscatter Diffraction Characterization of Inlaid Cu Lines for Interconnect Applications

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Summary: Automated electron backscatter diffraction (EBSD) techniques have been used to characterize the microstructures of thin films for the past decade or so. The recent change in strategy from an aluminum-based interconnect structure in integrated circuits to one based on copper has necessitated the development of new fabrication procedures. Along with new processes, complete characterization of the microstructures is imperative for improving manufacturability of the Cu interconnect lines and in-service reliability. Electron backscatter diffraction has been adopted as an important characterization tool in this effort. Cu microstructures vary dramatically as a function of processing conditions, including electroplating bath chemistry, sublayer material, stacking sequence of sublayers, annealing conditions, and line widths and depths. Crystallographic textures and grain size and grain boundary character distributions, all of which may influence manufacturability and reliability of interconnect lines, are ideally characterized using EBSD. The present discussion presents some results showing structural dependence upon processing parameters. In addition, the authors identify an in-plane orientation preference in inlaid Cu lines {111} normal to the line surface and <110> aligned with the line direction. This relationship tends to strengthen as the line width decreases.

Key words: thin films, copper, electron backscatter diffraction, texture

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Introduction

Aluminum metallization has been widely used for integrated circuit (IC) interconnects over the last 40 years, and a large knowledge base has been established on how to control the microstructure to expand manufacturing yield and reliability performance limits. It is well known that an aluminum alloy containing a small fraction of copper and a microstructure consisting of a uniform bamboo grain structure and strong {111} texture is best suited for withstanding electromigration under high current densities. It is also generally known that certain adhesion layers provide growth conditions for fine grained films that allow smooth sidewall formation during subsequent metal etch. The shift in strategy from Al to Cu interconnects, and from conventional metal etching to a damascene, or in-laid metal, fabrication technique presents new challenges to understand how manufacturing conditions control the microstructure of copper interconnect features. The final microstructure of inlaid copper interconnects forms within the confined space of the prepatterned dielectric, rather than within a two-dimensional thin film. Epitaxial substrate effects from the trench bottom and sidewalls influence the developing microstructure depending on the interface energy with the barrier or adhesion layer, the feature width and depth, and the thermally induced stress conditions in subsequent processing. Electron backscatter diffraction has been used in a number of investigations of thin Cu films and lines to determine grain size and crystallographic texture. The present discussion summarizes some of the information gleaned using EBSD about structural evolution in Cu films and lines. Also, the texture and grain size in single-level Cu lines as a function of line width are presented and compared with the structures previously observed in Al lines processed by the more conventional reactive ion etching (RIE) technique.

Materials and Methods

All EBSD measurements and analysis were performed using a TexSEM Laboratories (Draper, Ut., USA) orientation imaging microscopy (OIM 3.0) system attached to either a FEI XL-30 FE-SEM or an FEI XL-40 FE-SEM (FEI...
Company, Hillsboro, Ore., USA). It was previously demonstrated that the lateral spatial resolution of EBSD measurements using the column for this scanning electron microscope (SEM) is on the order of 10–20 nm depending on the material being analyzed (Field 1999, Humphreys et al. 1999, Nowell and Field 1998, Troost 1993) and is thus adequate for analysis of Cu thin film microstructures.

Structure evolution in inlaid Cu lines is generally accepted to be relatively independent of that observed in the Cu overburden film (Besser et al. 2001). Because of this, interrogation of line microstructure requires either cross sectioning to reveal the through-thickness structure, or plan-view analysis after over polishing to remove the remnant structure from the overburden. Electron backscatter diffraction analysis of the Cu lines discussed in the present work was performed on plan-view sections after a slight over polish during the chemical-mechanical planarization (CMP) process. The observed structures possess a distinct character that is a function of line width, indicating that the overburden microstructure did not significantly influence the observed structures.

Results

Previous investigations into the microstructure and texture of Cu films and lines by EBSD showed a strong dependence of texture and grain size distribution on plating bath chemistry and line height to width ratio (Besser et al. 2001, Field et al. 2001, Vanasupa et al. 1999). Copper lines were manufactured by the damascene technique in SiO2 using a 30 nm thick physical vapor deposited (PVD) aN barrier layer and a PVD copper seed layer. Copper films were deposited with three different electroplating chemistries (commercially available solutions, herein denoted A, B, and C) to form inlaid Cu line structures with 0.3 µm width and 0.5 µm height. All samples were allowed to self anneal at room temperature for 5 days before CMP. The resulting textures and grain sizes in the lines were radically distinct from one another.

Figure 1 contains representative orientation images from self-annealed 0.3 µm lines for each of the deposition conditions investigated. The images were taken from specimens manufactured using plating processes A, B, and C from left to right across Figure 1. These orientation images are shaded with crystallite lattice poles aligned with the specimen normal orientation according to the key presented in the unit triangle as part of Figure 1. (All orientation images shown in this paper follow this convention.) It is apparent that individual grains from the structure obtained using process A span the entire width of the trench, while those in the process C structure generally span only half the width, leaving a grain boundary seam down the center of the line. The majority of the grains in the process B line span the width of the trench, but a significant fraction of those observed do not. Scanning electron microscopy cross-sectional analysis of these lines shows that the trenches were completely filled in all cases, with no egregious voids near the trench floors.

Grain size analysis was performed by orientation imaging for all specimens. In the orientation imaging study, a grain is defined as a set of contiguous measurements bounded by a region of misorientation >2° from point to point over the area scanned. Electromigration is influenced by grain size because the grain boundaries offer a path of rapid diffusion to accommodate migrating atoms, in comparison with the bulk lattice. Coherent twin boundaries do not offer a path of rapid diffusion similar to that expected from a random, high angle boundary and should therefore not be included in grain size determinations for this purpose. For those 0.3 µm wide lines shown in Figure 1, the average length of grain along the lines were 0.98, 0.26, and 0.16 µm when twin boundaries were not included as grain boundaries, for the A, B, and C processes, respectively.

Crystallographic textures were measured from all lines using the data obtained during orientation imaging. The present study included four to six scans of the 0.3 µm lines that were 15–30 µm in length, giving between 60 and 200 grains for each condition. To obtain a statistically reliable measure of such textures from individual measurements would require on the order of 5,000 individual crystallite lattice orientations (Engler et al. 1999). Obviously, the statistics of the determination are poor for such weakly textured films, but the results point to important conclusions. Figure 2a–c contains {111} pole figures showing the in-line Cu textures from the A, B, and C bath chemistries, respectively. The directions identified on the pole figures are

![Fig. 1 Test structures for 0.3 µm wide A, B, and C bath chemistries from left to right. The orientation color key is also shown.](image-url)
SW for trench sidewall and LD indicating the line direction. The intensity scale of the pole figures shows a maximum (black) at five times random. Processes A and B result in very weak textures. Process A shows no identifiable trend in texture, while process B contains a small fraction of {111} planes aligned with the trench sidewalls. Process C results in a texture of near six times random, with the preferred orientation being the {111} plane aligned with the trench sidewalls.

One potentially important piece of information obtained from spatially specific orientation measurements is that of misorientations between neighboring crystallites. Specifically, the fraction of twin boundaries in a Cu line may be of some importance in characterizing the microstructure for interconnect applications. All specimens analyzed contain a relatively high fraction of Σ3 boundaries as determined by misorientation. These are the annealing twin boundaries prevalent throughout all structures and typical of face-centered cubic (FCC) metals with low-stacking fault energies. The fraction of grain boundaries classified as twin boundaries by orientation imaging for these structures are 0.85, 0.49, and 0.27 for the self-anneal processes after deposition using A, B, and C bath chemistries, respectively.

In a separate experiment, Cu lines were prepared from damascene trenches in TEOS-based oxide consisting of a number of 180 × 180 (µm)² sections, each with a different line width ranging from 0.20 to 10 µm and 0.5 µm deep. The metallization process consisted of 300 Å PVD Ta barrier layer + 800 Å PVD Cu seed layer and electroplated Cu fill. The spacing between the lines was not the same in all sections and varied with the width of the lines and the section in use; however, this is assumed to have no effect in our present study. The wafers were annealed at 400ºC and the overburden was removed by chemical mechanical polishing (CMP) to reveal the Cu line structure. The cross-sectional SEM images indicated no voids or observable defects in the lines along the thickness.

The lines from these different sections were analyzed using orientation imaging to determine the texture and grain size. The analytical conditions were 25 keV accelerating voltage using a step size of 50 nm. The sections scanned were 20 × 15 (µm)² to obtain about 100,000 data points for each region. These data were used to calculate the grain size and the pole figures to determine the microstructure and preferred orientation of the grain structure in the lines.

The orientation maps for some the Cu lines used in the experiment are shown in Figure 3, along with the orientation color key. The results of the average grain diameter, excluding and including twins as separate grains, and the twin boundary fraction for the various line widths is shown in Table I. The maps and the grain size values clearly show a distinct difference between the wider and the narrower lines. The narrower lines, that is, those <1 µm wide, were completely bamboo structured with the grains spanning through the line width, while the lines with the larger widths had a multicrystalline nature. The average grain size (diameter), as is expected in this case, increases with increasing line width. The average grain diameter value in the wider lines was in the order of the grain sizes for the blanket films with a similar thickness. The constraint imposed by the sidewall on the grain growth is likely the reason for the decrease in the average grain sizes in the narrow lines.

Crystallographic textures were calculated from the orientation measurements using a discrete binning procedure, as described by Matthies and Vinel (1999) with a bin size of 5º. The {111} and {110} pole figures were calculated and compared for the different line widths. Figure 4 contains representative {111} and {110} pole figures for lines of 0.20 µm and 2.8 µm width. For all line widths, the grains predominantly had a {111} out of plane orientation, as generally observed in electroplated blanket films. The {111} out of plane component is explained by the surface energy minimization criteria in FCC blanket films. In lines, if the plating of the Cu takes place from the bottom of the

<table>
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<tr>
<th>Line width (µm)</th>
<th>Average grain dia (µm), with twins</th>
<th>Average grain dia (µm), without twins</th>
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<tbody>
<tr>
<td>0.20</td>
<td>0.21</td>
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<td>0.26</td>
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<td>0.70</td>
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<td>2.80</td>
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<td>3.64</td>
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<td>5.04</td>
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Fig. 2 (111) pole figures from the 0.3 µm lines shown in Figure 1 for room temperature processes A, B, and C. The gray scale shading (white to black) indicates 0–5 times random for all plots. LD=line direction, SW=side wall.
trench and not on the sidewall, then the argument of surface and interface energy is still valid giving a (111) out of plane texture in the grains. The {111} orientation intensities varied with the line width, showing that the side wall and stress differences become more of a factor with the decreasing width. A plot showing the maximum {111} intensity as a function of line width is shown in Figure 5. The chart shows a U-shaped curve with a minimum obtained around 1 µm.

The {110} pole figure of the 0.2 µm lines shows a nonuniform in-plane intensity distribution and a maximum {110} orientation along the line length. Figure 6 contains inverse pole figures giving the pole orientation intensity aligned with the line directions. These texture plots show a {110} maximum along the line length, showing the preferred in-plane texture component. These texture values increased with the decreasing line width, and the highest value of 4× random intensity was observed in the case of the narrowest lines, 0.2 µm wide. This result confirms the increasing effect of the sidewall on texture evolution as line width decreases and height to width aspect ratio increases.

Discussion

Orientation dependence in thin films is controlled by processing conditions including sublayer material, thickness, and stacking sequence. Preferred orientation develop-
opong during deposition, recrystallization, and grain growth is generally considered to be either surface energy dominated for thin films or strain energy dominated for thicker films (Thompson and Carel 1995). When line microstructures are considered, additional energetic and physical constraints control texture evolution within the line. The inlaid Cu line is surrounded on three sides by a barrier layer material. The constraint associated with energy minimization for these interfaces could be more dominant than surface energy considerations for the free surface. In addition, grain boundary energy minimization could play a role in texture development during grain growth since the grain boundary area will tend to minimize, creating a bamboo-type grain morphology. Sanchez and Besser (1998) and Hau-Riege and Thompson (1999) discussed structure evolution and the energetics associated with grain growth for free-standing and inlaid line microstructures. Two line width-dependent phenomena were observed in the textures of the Cu lines. First, the \{111\} texture strength decreased with line width down to about 1 \(\mu\)m, then began to increase as line width further decreased to 0.2 \(\mu\)m. Second, an in-plane orientation preference was observed for the most narrow lines.

Decreasing \{111\} texture strength as the line width narrows can be attributed to the increasing influence of the sidewalls. For wide lines, the metal is essentially in a state of biaxial stress, and the free-surface and bottom interface energies of the lines dominate grain growth and texture development. The low \{111\} surface energy dominates growth and results in a preferred \{111\} fiber texture with little influence from sidewall nucleated growth. As the line width decreases, there is increased likelihood that the sidewalls will contribute significantly. When the height-to-width ratio of the lines becomes >1, the interface energy associated with the sidewalls could become a dominant factor in structure evolution and a \{111\} sidewall texture can develop (Besser et al. 2001, Lingk et al. 1999). There was

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**Fig. 5** Chart showing the change in the (111) peak intensity with varying line width.

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**Fig. 6** The inverse pole figure texture plots with line length as the reference direction showing a preferred orientation of (110) along the line length.
no evidence of sidewall-oriented textures in any of the lines investigated in this study, indicating again that additional factors influence texture evolution and grain growth in electroplated inlaid Cu lines (such as bath chemistry and sublayer material and thickness). The observed increase in texture strength at the most narrow line widths is also an indication that sidewall nucleation and grain growth influenced by the sidewall interface energy is not a dominant mechanism of texture development.

In addition to potential sidewall nucleation, the stress-state in the Cu line changes to one of tri-axial stress when the height-to-width ratio is near ≥1.0. Because of the physical constraints on three sides of the lines, this effect continues to increase as the line width narrows. Another structural feature observed from the EBSD imaging of the Cu lines is the relatively high frequency of twin boundaries in lines having widths near 1 µm. Figure 7 shows the fraction of twin boundaries as a function of line width. In this case, the twins were identified as those having misorientations within 2° of the ideal twin misorientation (60° about a <111> axis). It is apparent by comparison of Figures 5 and 7 that the high twin fractions exist in line widths where the weaker textures were present. It should be pointed out that this observation was not an anomaly that could be attributed to inhomogeneity of the microstructure. The weaker textures and higher fractions of twin boundaries were consistently present in lines of approximately 1 µm in width as observed over several different and widely separated regions. When a large number of twin boundaries exists in a microstructure, there is a concomitant weakening of the texture. A single grain twinned repeatedly over the various twin variants, and including twins of twins, results in a relatively random distribution of orientation components after only three twinning generations (Humphreys and Hatherly 1995). Therefore, the weaker texture in the regions where a large number of twin boundaries exists is not surprising. The growth of annealing twin is dependent on several factors, including stress in the material, but is not generally well understood. It is apparent from the data presented herein that, for the given processing conditions, twin boundaries are most likely to develop in lines having widths near one micron. Since these lines were side by side with those of other widths, it is reasonable to conclude that the differences in stress state are responsible for the higher fraction of twin boundaries; these boundaries, in turn, are responsible for the weaker textures observed.

The observed in-plane orientation preference for the narrow inlaid Cu lines shows that the annealed structure in the line, for the processing conditions used, tends to a {111} surface normal and a [110] direction along the line length. The {111} surface orientation is the low-energy surface in FCC materials and provides evidence of bottom-up growth of the Cu during electroplating. Side-wall growth would result in a preferred orientation having the {111} pole aligned with the trench side wall. The [110] in-plane orientation component must be a result of either interface or grain boundary energy minimization. Interface energy minimization would require that the {211} plane aligned with the trench sidewalls is the low-energy orientation plane for Cu grains in contact with the Ta barrier (for planes normal to the {111} surface orientation). Grain boundary energy minimization would require that the {110} plane aligned with the grain boundary plane is the low-energy position for [111] tilt boundaries. While interface energy data are not available, the energy of free surfaces shows that the {112} plane is the lowest energy plane normal to the {111} surface orientation (Sundquist 1964).

Perhaps not coincidentally, a similar preferred structure was observed in heavily annealed, free-standing, narrow Al lines. Figure 8 is adapted from Field and Wang (1998), showing the preferred structure in the Al lines. The narrow line Cu microstructures discussed in this paper tend to the same orientation preference as shown for the narrow Al lines. This comparison suggests that the {211} plane in the {111} oriented Cu grains creates a low energy interface with the Ta barrier in a manner similar to the Al grains where it is presumed that free surface energy minimization results in the observed texture.
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