Microstructure of 100 nm damascene copper overburden and lines

R.H. Geiss and D. T. Read

National Institute of Standards and Technology, 325 Broadway, Boulder, CO 80305

ABSTRACT

Keywords: copper, damascene, grain size, preferred orientation, texture, twinning

A detailed understanding of the crystallography of metallic conductors in modern interconnect systems is essential if we are to understand the influence of processing parameters on performance and reliability. In particular we must be able to evaluate the grain size, crystallographic orientation and residual elastic stress for interconnect lines having widths of tens of nm. Transmission electron microscopy might be the obvious choice, but sample preparation and small sample size make this technique unattractive. On the other hand, electron backscatter diffraction, EBSD, in a scanning electron microscope provides a very attractive tool. Sample preparation can be relatively simple, especially if one investigates the structures immediately after CMP; whole wafers may be measured if desired. One limitation to EBSD is that good diffraction patterns are obtained only from free surfaces and from a limited depth, say a few hundred nm in copper. Here EBSD will be used to compare structures for the pads and 100-nm lines in two variants of a commercial copper damascene interconnect structure. EBSD data collection will be discussed as optimized for characterizing differences in the texture, which were attributed to differences in the processing. By a unique approach to EBSD mapping we found that neither the texture nor the grain size of the overburden, as represented by the contact pads, propagated into the 100 nm lines, though they did propagate into some wider lines.

1. INTRODUCTION

Characterization and control of grain size and crystallographic texture of the narrow damascene copper interconnect (IC) lines is an essential requirement in manufacturing reliable advanced microelectronic devices. In this paper we demonstrate the use of electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM) for microstructural characterization of copper damascene interconnect lines. Many different methods for microstructural characterization of metal films have been reported in the literature; here we cite only a few relevant examples. The most widely-reported methods include: x-ray diffraction (XRD)¹, focused ion beam (FIB) imaging², transmission electron microscopy (TEM)³ and microbeam x-ray diffraction (MBXRD)⁴, along with scanning electron microscopy (SEM) and the associated diffraction technique, electron backscatter diffraction (EBSD)⁵. The key limitation of XRD is that with conventional laboratory XRD apparatus the analyzing spot size is at best 0.5 to 1 micrometer in diameter. For present day interconnect structural dimensions, this beam size implies that XRD is still a non-local technique. XRD can provide an average texture over a region, but not a metallurgical grain size. The variety of structures of different dimensions on a typical specimen chip implies that the x-ray beam would have to be directed at the structure of interest. This is difficult to accomplish with typical laboratory systems. FIB techniques are generally destructive and in any event implant Ga ions into the surface of the sample being studied. And unless FIB techniques are supplemented by EBSD they do not yield crystallographic information. TEM can surely give the highest spatial resolution, but sample preparation is definitely destructive and very labor intensive. MBXRD has an area resolution of 200-300 nm diameter and is continuing to improve, but the requirement of a high intensity photon source, *i.e.*, synchrotron, removes it from the realm of almost any laboratory. Finally, the combination SEM/EBSD enables the required analytical capability for IC line widths down to at least 65 nm and possibly down to the 22 nm lines projected for the near future. It is non-destructive other than a limitation of moderate sample size, associated with the physical size of standard SEM stages, and it is limited to obtaining data only from the top 100 nm, or so, of the specimen.

> Instrumentation, Metrology, and Standards for Nanomanufacturing, edited by Michael T. Postek, John A. Allgair, Proc. of SPIE Vol. 6648, 664808, (2007) · 0277-786X/07/\$18 · doi: 10.1117/12.735021

The purpose of this paper is to demonstrate some experimental requirements and data analysis techniques using SEM/EBSD for the determination of grain size and texture in 100 nm electroplated damascene lines and the associated pads in chemical mechanical polished (CMP) copper films.

2. EXPERIMENTAL

The test structures were manufactured using the conventional methods of preparing damascene copper for electronic interconnect structures. After having been electroplated, the specimen wafers were annealed and CMP processed, completely removing the overburden. The resulting structures consisted of serpentine arrays of 100 nm wide copper lines terminating in large test pads, along with other line structures of varying widths up to 250 nm wide. The spacing between the narrow lines in the serpentine structures was either 110 or 120nm. The film thickness after CMP was approximately 350 nm. The microstructure of the contact pads, approximately 100 micrometers square, was taken as representative of the overburden. Samples for SEM/EBSD analysis were prepared by cleaving approximately 1 x 1 square centimeter samples from the wafers and them mounting them on standard SEM stubs with conductive silver paint.

A commercially-obtained field emission SEM (FE-SEM), which provides a small, bright electron beam, was used. Images were taken with the sample at 0° tilt to confirm the 100 nm widths of the serpentine lines. The samples were then tilted to 70° for EBSD analysis (the conventional technique). All EBSD analysis was done at 20 kV using a \sim 5 nm probe with a probe current of about 600 pA. Individual EBSD data collection experiments, for the purpose of obtaining orientation maps, were taken using a variety of conditions, with the step size and the number of steps adjusted so that individual maps were collected in 60-90 minutes maximum. Maps acquired with longer times tended to show a large amount of drift of the specimen position relative to the probe beam, especially those from line structures which had alternating conducting metal and insulating oxide line geometry. Very large area maps were 100 x 90 micrometers with 200 nm steps, comprising 166610 points and requiring about 84 minutes. This protocol was used only on large continuous film areas, to provide the amount of data required for texture analysis.

It has been suggested that for accurate determination of grain size by EBSD, the average grain size must be large enough so that on average there are 10 sampled points per grain⁶. For a large area map of a line structure, 15×10 square micrometers, a step size of 30 nm was used, yielding approximately 180,000 points in about 60 minutes. For a 100 nm wide line, a 30 nm step gives an average of 3 pixels across a line on each scan. While this provides very good data for texture determination, it is unsatisfactory for grain size determination. To determine grain size and crystallography of the lines, a 20 nm step was used providing 5 to 6 pixels across each line per scan. For this step size a map of 1.0×1.5 square micrometers required about 30,000 points and took about 25 minutes. The effect of sample drift was minimized if the scan direction was across the lines rather then along them.

For grain size determination it is important that the mapping conditions satisfy the requirements set forth in ASTM E 1382, *Standard Test Methods for the Determination of Average Grain Size Using Semi-Automatic and Automatic Image Analysis*⁷. This document sets forth a guideline that the scanned area should contain at least 50 grains. Wright⁶ recommends that for EBSD measurements, a grain should have a minimum at least 10 pixels and the ensemble average should exceed 500 pixels per grain. An average of 500 pixels per grain might be a bit higher than needed for EBSD analysis. We have found that an average of about 100 pixels per grain gives consistent results, but the requirement of at least 50 grains per map is necessary⁸.

For texture determination by EBSD to be comparable to texture determination by XRD, a minimum of 10,000 grains are required for the analysis, again with a minimum of 10 pixels per grain⁹. This suggests that at least 100,000 points must be included in the map. This requirement was established considering moderately textured equiaxed fine grain material. In the instance of the line structure considered here, these requirements are relaxed since at least half of the pixels are from the dielectric between the lines, rather than from the metal lines, and the grains in these narrow lines are usually not equiaxed, but elongated along the line.

Finally, the analysis of grain size requires careful examination of the data. Annealed thin metal films do not usually have a normal distribution of grain sizes, but rather a lognormal distribution¹⁰⁻¹². In determining the grain size in the

pads this consideration was applied. In the analysis of the grain size in the lines, manual measurement was generally found to give the most satisfying results.

3. RESULTS

The results will be presented in two parts; (1) a comparison of texture in the contact pads with that of the serpentine thin metal lines and (2) a comparison of the grain size in the pads with that in the thin lines.

3.1 Comparison of texture between pads and lines

As mentioned in the preceding section, for texture determination by EBSD to be comparable to that determined by XRD a minimum of 10,000 grains should be included in the analysis. To obtain such numbers in our analysis of the pads we merged 3 maps of 60 x 90 square micrometers, each comprising approximately 3500 grains, into a single dataset. The result included 10674 grains with a mean of 18 points per grain. An inverse pole figure (IPF) representation of one of the 60 x 90 square micrometer EBSD maps is shown in fig. 1. The colors represent the orientation of the grain normal to the film in accordance with the colors of the triangle in the corner of the figure. Each map consists of 155977 pixels of 200nm steps and took about 55 minutes. The data were adjusted to remove all subsets of pixels with less than 5 adjacent pixels of the same orientation. This treatment doesn't modify the data, it just removes the small groups of pixels which are incorrectly indexed, or not indexed at all, mostly due to surface irregularities on the sample. In effect it improves the signal to noise ratio of the analysis.



Fig. 1. A 90 x 60 square micrometer EBSD-IPF map showing the surface-normal orientation of the grains in one of the pads, according to the key in the stereographic triangle insert.



Fig. 2. (111) and (001) pole figures from the merged data set of 10674 grains in three different pads. Similar pole figures are obtained from each of the individual data sets. Note that the (111) and (001) pole densities are nearly equal.

The (111) and (001) pole figures from the merged data set for the pads are shown in fig. 2. Pole plots of the two pole figures show that the (111) pole is 24 times random and the (001) pole is 20 times random. Similar pole figures and pole plots were obtained from each of the 60 x 90 square micrometer maps comprising the merged data set. The pole figures show that there is a sizeable volume of grains with a (001) orientation. In fig. 1, notice that the grains colored red, which are those near (001) are much larger than the others.

The orientation of the grains in the pads was fairly uniform and even much smaller data sets from 10×15 square micrometer maps at 33 nm steps gave pole figures which were indistinguishable from those in fig. 2. Typically these maps had 150,000 points but contained less than 400 grains, which is too few for the recommended analysis. But the grains had an average of about 100 points per grain.

The mapping of large areas from the lines presented a problem since there was a great tendency for the sample to drift because of the alternating conductor/insulator structure. Nonetheless, we were able to acquire maps on 15×10 square micrometer scans with 33nm steps giving 159000 points. A typical map is shown in fig. 3.

Pole figures from this data set are given in fig. 4 and show a marked difference from those from the pads. Here the (111) pole figure shows a strong orientation, typically greater then 50 times random, while the (001) pole figure indicates the complete absence of grains oriented in that direction. This is in clear contrast to the pads where the volume of (111) orientated grains was the same as the volume of the (001) oriented grains.

3.2 Comparison of grain size in the pads and lines

According to ASTM Standard E1382, a digital image analyzed for grain size should contain at least 50 grains. As discussed for EBSD⁶, the average grain size is relatively constant for minimum grain diameters between 3 and 20 pixels. However to be in compliance with E1382 it was suggested that a minimum grain size of 10 pixels can be used but that the average number of pixels per grain, PPG, should be close to 500. Combining these two factors suggests that an EBSD map used to determine grain size should have a minimum of 25000 pixels, with the step size chosen so that there are about 25 steps across the diameter of an average grain.



Fig. 3. A 15 x 10 square micrometer EBSD-IPF map from part a 100 nm wide serpentine line structure. The grains appear in color while the oxide between the grains is black. The colors indicate the orientation of the normal to the plane of the image, according to the stereographic triangle insert.



Fig. 4. (111) and (001) pole figures from the 15 x 10 square micrometer EBSD map of a region of the 100 nm wide serpentine line structure. Note the extremely low population of (001) poles oriented normal to the surface.

We have reported previously⁸ that this indeed does seem to provide robust results if one analyzes the data considering that grain diameters, or PPG, are best described by a log normal probability distribution rather than the nominally used normal probability distribution. Both experimental and theoretical studies of annealed thin film behavior have found this to be the case. Implicit in this analysis is that the grains are circular so that grain diameters can be derived from the number of pixels per grain. If the grains diverge from circular shapes then it is almost mandatory that some type of manual measurement be used.

A grain data set for an EBSD map is developed considering that a grain is defined by adjacent pixels having certain characteristics. As a minimum, a one degree angular mismatch is allowed, this being about the best that one can expect considering the reduced resolution of the patterns used in high speed mapping. More commonly a two to five degree mismatch is allowed. The minimum number of adjacent pixels satisfying this minimum angular mismatch is the other parameter commonly used to define a grain. Obviously one pixel alone should not be used to define a grain.

The EBSD-IPF map of a 15 x 10 square micrometer map is shown in fig. 5. The map was scanned in 33 nm steps which resulted in 159075 points. The histogram of the grain data set, allowing a minimum of 2 degrees misorientation between adjacent pixels in the grain definition is shown in fig. 6a. Obviously the grain sizes do not follow a normal distribution. The solid curve depicts a normal distribution curve for the data. Figure 6b is a plot of the distribution of the log of the same data and the solid curve a lognormal distribution curve fit to the data. The lognormal distribution is a far better fit to the data, as would be expected from the physics of annealed grain growth¹³, but the very large number of grains having a small number of points skews the data too strongly to the right even for a lognormal distribution. Inspection of the image suggests that the average grain diameter is closer to 1000 nm. Creating a grain data set by manually selecting the grains and excluding all grains with less than 50 pixels, gave an average grain diameter of 781 ± 630 nm, which is probably more consistent with the results that would

gave an average grain diameter of 781 ± 630 nm, which is probably more consistent with the results that would have been obtained using the classic manual intercept method. The average number of PPG determined from this analysis was 837.

Analysis of the lognormal distribution gives the average number of points per grain (PPG) for the map as 95, which represents a circular grain having a diameter of 315 nm. The average number of PPG of 95 is less than the desired 500. The only question would be that whether taking smaller steps, say 20 nm, which would give more PPG, would improve the values for the average grain size sufficiently to warrant the additional time. Figure 5 shows that the assumption that the grains are circular is not correct.

The analysis of the grain size of the line structure presents a unique problem since the grain width has a maximum dimension determined by the line width. Looking at fig. 7, which is a montage of 3 high magnification, 1.5 x 3 square micrometer maps, one can see that nearly all the grains span the line width, and have varying lengths. Obviously, they can not be classified as circular grains. Since standard EBSD analysis software does not have the capacity built in to deal with such structures, the measurement was done manually. The average grain length measured along the lines in this figure is 247 ± 103 nm with the minimum = 98 nm and the maximum = 545 nm.

4. DISCUSSION and CONCLUSIONS

The results shown above in section 3.1 show that the textures of the pads and the 100 nm lines are clearly different. The pads have an almost equal population of (111) and (001) orientations while the lines show a very strong (111) orientations and a complete absence of (001). The average grain diameter in the pads was manually determined to be 781 nm and the average long dimension of the grains in the lines was found by a similar analysis to be 247 nm. Therefore, it is clear that the overburden grain structure was not propagated into the narrow lines.



Fig. 5. Higher magnification, 15 x 10 square micrometer, EBSD-IPF map from a copper pad area. The colors show the orientation of the grains normal to the page following the triangle.



Fig. 6. a. Histogram showing the normal distribution of the grain file for the number of points per grain. The solid line represents the normal probability distribution fit to the data. b. Histogram of the distribution of the log of the number points for the same data. The solid curve shows the lognormal probability fit.



Fig. 7. Montage of three 1.5 x 3 square micrometer EBSD-IPF maps from a section of the 100 nm serpentine line structure. The color represents the orientation of the grains normal to the plane of the page according to the inserted stereographic triangle.

We have done pole figure analysis and grain size measurements for 250 nm wide lines in the same specimen chips; here we found that the differences in both texture and grain size between the wider lines and the pads were considerably less than for the 100 nm wide lines.

We used the available software to estimate the area fraction of coherent 60° (111) twins in the pads and 100 nm lines and found the pads to be heavily twinned, up to 40 %, while only 15 % of the lines displayed coherent twins. Here too the structure of the pads was apparently not propagated into the lines.

We had two significant problems in obtaining the grain sizes for these samples: (1) The geometry of the lines does not allow the standard analysis techniques to be used. (2) The large number of small groups of neighboring pixels that appear to be grains drives the average grain size to an unrealistically small value. Both problems were dealt with here by doing manual analysis. Future work is needed to address these problems.

In summary, a comprehensive study has been conducted comparing the texture and grain structure of the overburden, represented by larges area pads, and the thin, 100 nm lines in damascene copper IC chips after CMP. The three properties measured; texture, grain size and twinning; were sufficiently different between the two structures to conclude that the crystallographic properties of the overburden were not propagated into the 100 nm damascene copper lines.

ACKNOWLEDGEMENT

The financial support of the NIST Office of Microelectronics Programs is gratefully acknowledged. This work is a contribution of the U.S. National Institute of Standards and Technology. Not subject to copyright in the United States.

REFERENCES

- Tracy, D. P.; Knorr, D. B. "Texture and Microstructure of Thin Copper-Films," *Journal of Electronic Materials* 22 (6), 611-616, 1993.
- Besser, P. R.; Zschech, E.; Blum, W.; Winter, D.; Ortega, R.; Rose, S.; Herrick, M.; Gall, M.; Thrasher, S.; Tiner, M.; Baker, B.; Braeckelmann, G.; Zhao, L.; Simpson, C.; Capasso, C.; Kawasaki, H.; Weitzman, E., "Microstructural characterization of inlaid copper interconnect lines," *Journal of Electronic Materials* 30 (4), 320-330, 2001.
- 3. Kelly, J. J.; Tian, C. Y.; West, A. C. "Leveling and microstructural effects of additives for copper electrodeposition," *Journal of the Electrochemical Society* **146** (7), 2540-2545, 1999.
- 4. Tamura, N.; MacDowell, A. A.; Spolenak, R.; Valek, B. C.; Bravman, J. C.; Brown, W. L.; Celestre, R. S.; Padmore, H. A.; Batterman, B. W.; Patel, J. R. "Scanning X-ray microdiffraction with submicrometer white beam for strain/stress and orientation mapping in thin films," *Journal of Synchrotron Radiation* **10** 137-143, 2003.
- 5. Humphreys, F. J. "Review Grain and subgrain characterisation by electron backscatter diffraction," *Journal of Materials Science* **36** (16), 3833-3854, 2001.
- 6. Wright, S., Personal communication, Notes on grain size determination, 2007
- 7. ASTM E 1382, "Standard Test Methods for Determining Average Grain Size Using Semi Automatic and Automatic Image Analysis", ASTM International, West Conshohocken, PA.
- 8. Geiss, R. H.; Read, D. T. "Need for Standardization of EBSD Measurements for Microstructural Characterization of Thin Film Structures," in *Proceedings of Frontiers of Characterization and Metrology for Nanoelectronics;* American Institute of Physics Press: New York, 2008.
- 9. Wright, S., Bingert, J., and Nowell, M., "A Comparison of Textures Measured using X-Ray & Electron Backscatter Diffraction," submitted to *Metallurgical Transactions*, 2007.
- Sanchez-Bajo, F.; Ortiz, A. L.; Cumbrera, F. L. "Novel analytical model for the determination of grain size distributions in nanocrystalline materials with low lattice microstrains by X-ray diffractometry," *Acta Materialia* 54 (1), 1-10, 2006.
- 11. Tracy, D. P.; Knorr, D. B.; Rodbell, K. P. "Texture in Multilayer Metallization Structures," *Journal of Applied Physics* **76** (5), 2671-2680, 1994.

- 12. Vaz, M. F.; Fortes, M. A. "Grain-Size Distribution the Lognormal and the Gamma-Distribution Functions," *Scripta Metallurgica* **22** (1), 35-40, 1988.
- 13. Fayad, W.; Thompson, C. V.; Frost, H. J. "Steady-state grain-size distributions resulting from grain growth in two dimensions," *Scripta Materialia* **40** (10), 1199-1204, 1999.