AIP | Proceedings

TEM Measurements of Grain Orientation in Nanoscale Cu Interconnects using ACT

Jinho An, Jeff Pickering, K. J. Ganesh, and P. J. Ferreira

Articles you may be interested in

Grain structure analysis and effect on electromigration reliability in nanoscale Cu interconnects
Appl. Phys. Lett. **102**, 131907 (2013); 10.1063/1.4799484

Synchrotron measurement of the effect of linewidth scaling on stress in advanced Cu/Low- k interconnects
J. Appl. Phys. **106**, 053524 (2009); 10.1063/1.3212572

Line Scaling Effect on Grain Structure for Cu Interconnects
AIP Conf. Proc. **1143**, 151 (2009); 10.1063/1.3169254

Reduction effect of line edge roughness on time-dependent dielectric breakdown lifetime of Cu/low- k interconnects by using C F 3 I etching
J. Vac. Sci. Technol. B **27**, 649 (2009); 10.1116/1.3100268

Dependence of the electromigration flux on the crystallographic orientations of different grains in polycrystalline copper interconnects
Appl. Phys. Lett. **90**, 241913 (2007); 10.1063/1.2742285

TEM Measurements of Grain Orientation in Nanoscale Cu Interconnects using ACT

Jinho An[1,2], Jeff Pickering[2], K.J. Ganesh[1], P. J. Ferreira[1]

[1] Materials Science and Engineering Program, The University of Texas at Austin, Austin, TX 78712, USA
[2] Department of Mechanical Engineering, The University of Texas at Austin, Austin, TX 78712, USA (Now at Schlumberger Ltd.)

ABSTRACT. Transmission electron microscope (TEM) is used in conjunction with an Automated Crystallography for TEM (ACT) to index the crystal orientation of 180 nm wide Cu interconnects using the Nano Beam Diffraction (NBD) mode in the TEM. An FEM software, OOF2, was used to simulate the local quasi-hydrostatic stresses in the interconnect lines based on the local orientation data, and was compared results obtained from stress induced void (SIV) formation in 180nm Cu interconnects studied through *in-situ* TEM (Transmission Electron Microscope) heating. SIV were induced at temperatures of around 230°C. Correlation between the stress simulations and the experimental results show that point of high local stresses and high stress gradients seem to influence the formation of the SIV in the Cu interconnect lines. A description of a new technique, called D-STEM that allows for obtaining diffraction patterns from crystals only a few nanometers in size is also given, that will allow for characterizing even smaller width Cu interconnects for future generation microelectronic devices.

Keywords: Cu interconnect, Transmission electron microscope, Automated Crystallography for TEM, in-situ heating grain orientation, stress induced voids, OOF2

INTRODUCTION

The existence of stresses in Cu interconnects is a key issue, as it affects the reliability of micro and nanoelectronic devices, particularly electromigration and stress induced voids (SIV) [1-7]. However, there is still a lack of fundamental understanding of the relationship between local stresses and grain orientation. One of the main reasons for this is that the determination of crystal orientation at the nanoscale is not a trivial task. So far, the community has relied strongly on the use of X-ray diffraction (XRD) and Electron Backscattered Diffraction (EBSD) for determining crystal orientation. XRD can be used to obtain the overall texture of nanocrystalline materials, but it does not provide local crystal orientation. EBSD, on the other hand, has been a vital tool to acquire precise crystal orientation information from individual grains over a large area. However, even with the use of a Field Emission Gun (FEG) Scanning Electron Microscope (SEM) it is currently extremely challenging, if not impossible, to experimentally gather results from grains with sizes below 50 nm [8]. In addition, the resolution of the EBSD technique is a function of the material (e.g. highly conductive materials will give better results), which ultimately limits the range of materials that can be studied. Furthermore, sample preparation is critical for accurate EBSD analysis, which can again hinder its applications. Due to the inherent limitation of XRD and EBSD, there is a crucial need for obtaining the orientation of individual grains with sizes below 50 nm. Such methodology will have impact not only on Cu interconnect technology, but also on the general field of nanotechnology.

In order to address this challenge, the use of Transmission Electron Microscopy (TEM) is required. Conventionally, by using Kikuchi mapping, one can index the orientation of individual grains [9]. The beam is converged using a large C2 aperture to produce incoherent scattering. This allows the user to obtain the correct crystallographic information. However, this method is slow and the use of a large convergence angle limits the use on smaller grains. In addition, thinner samples are difficult to characterize because of the possible absence of Kikuchi bands. Furthermore, many TEMs now use an

ultrahigh resolution pole-piece, which limit sample tilting to only 15 degrees and make difficult to determine the crystal orientation.
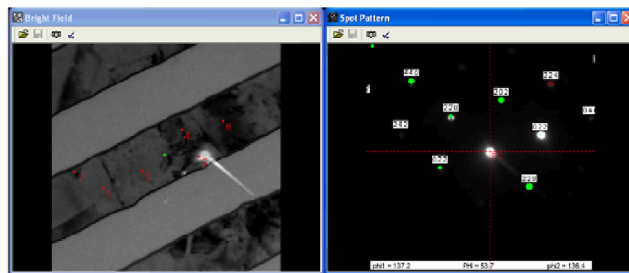
In this regard, a new method called "Automated Crystallography for TEM" (ACT) was used in this paper. In the past, the ACT software, made by EDAX (Materials Analysis Division of AMETEK Inc.), has been used to automatically index diffraction patterns (DP), including off-zone axes, using a selected area aperture [10]. However, this approach was only limited to large grains, due to the aperture sizes. In this work, we have used the ACT software to index Cu grains, as small as 20-30 nm, in 180nm wide interconnect structures, using Convergent Beam Electron Diffraction (CBED) or Nano Beam Diffraction (NBD) mode in the TEM. The ACT software allowed us to index the crystal orientation relatively quickly. In this fashion, nanoscale Cu grains were individually indexed, and the local texture identified. This information is essential for understanding the fundamental mechanisms of stress-induced voids and electromigration in copper interconnects. In general, this method is an important tool for obtaining crystallographic information from nanocrystalline materials whose grain sizes are too small for EBSD.

EXPERIMENTAL PROCEDURE

The Cu interconnects used in this research were provided by Freescale Semiconductors. The Cu interconnects were electroplated and annealed at 250 °C for 30 minutes. The dielectric layer used was F-TEOS. The 10-20 nm thick Ta diffusion barrier was physically deposited, and the 50 nm SiNx passivation layer fabricated by chemical vapor deposition (CVD). An additional layer of F-TOES has been deposited on top of the passivation layer [11].

The crystal orientation of the plan view TEM samples were obtained using the ACT software provided by EDAX. In order to obtain diffraction patterns from individual grains, the TEM was used in CBED or NBD mode using a 10 micron C2 aperture. The CBED mode was used for larger grains, while NBD was used for smaller grains (e.g. grains smaller than 100nm).

In order to obtain the crystal orientation, the area of interest was first tilted over a wide range (± 10 degrees) to identify all the grain boundaries in the viewing area, as typically grain boundaries may appear out of contrast under different tilting conditions. The image was then recorded on the ACT software. The composite imaging function allows the user to overlap the electron probe with the bright field image (Fig. 1). This function is very helpful in allowing the user to place the probe over the individual grains of interest, and ensure that the probe is not overlapping two adjacent grains. Once the probe is placed over the grain, the diffraction pattern (DP) is obtained. The DP is then automatically indexed. Details of DP indexing are described elsewhere [10].

FIGURE 1. Captured image of the ACT software window. (a) shows overlap of interconnect lines and spot of beam (white dot indicated by arrow). Red numbers in (a) are where previous DP patterns were obtained. (b) shows the DP pattern from grain overlapping with beam in (a) and its crystal orientation indexed with ACT.

167

In order to distinguish between kinematical and dynamical diffraction spots, and to ensure that the indexing is accurate, off-zone axis patterns can be tilted to the closest zone-axis, and indexed. The simulation function is used again and the tilt conditions are simulated, allowing the user to match the simulated DP with the obtained DP. Based on these results, the kinematical (forbidden) diffraction spots are identified, and omitted from the analysis.

In-situ TEM heating experiments were conducted in a FEI TECNAI X-twin TEM, equipped with double tilt Gatan heating stage capable of heating up to 1100°C. Initial *in-situ* TEM heating of the Cu interconnect samples was conducted from room temperature to 500 °C at a rate of 10 °C/min. Based on previous results [11], the temperature was generally maintained at around 230°C, at where void formation was most readily observed. In order to minimize the oxidation of the exposed Cu interconnects, in-situ TEM heating was performed immediately following the TEM sample preparation, and a plasma cleaner was also used to remove any possible oxidation of the Cu post TEM sample preparation.

The local hydrostatic stresses present in Cu interconnects were simulated using OOF2, based on the local crystal orientation information obtained through ACT. OOF2 is an FEM software developed by the National Institute of Standard and Technology (NIST). The OOF2 stress simulations were run in a Dell Precision 530 Workstation with a SuSE Linux operating system.

Based on the TEM images, the software "Adobe Photoshop" was first used to identify and illustrate the copper grains along the Cu interconnect. Each grain was first designated an arbitrary color. Every pixel in each individual grain must be of a uniform color, as the OOF2 program recognizes each pixel group as an independent object (in this case, as an independent grain). Subsequently, the crystal orientation of each grain, obtained using ACT, and the mechanical properties of copper and TEOS [12-13] were used as input data in the OOF2 program and assigned to each individual grain.

In simulating the hydrostatic stresses, the following assumptions were used: 1) A biaxial stress model (i.e. $\sigma_{z} = 0$) was considered. Despite the fact that the Cu interconnect lines were subjected to 3D quasi-hydrostatic stresses $(\sigma_x \rangle \sigma_y \rangle \sigma_z)$, σ_x and σ_y are the major normal stresses and thus used in the simulations, 2) the diffusion barrier was removed from the calculations, as the DB is very thin (10~20 nm) and plays a minimal role in affecting the local stresses, and 3) the Young's modulus of SiO_2 was used for F-TEOS.

For the FEM analysis, quad meshes were used for the microstructure. A relatively coarse mesh was first constructed, and then refined by creating finer meshes where needed, especially where fine grains were present. Displacements of the Cu interconnect lines due to normal stresses present were used as boundary conditions for the stress simulations. The normal stresses used were previously reported for Cu interconnect lines with similar dimensions [1]. The resulting hydrostatic stresses will be represented using a thermal spectrum, and the same hydrostatic stress legend will be used throughout the paper. Since the main interest lies in analyzing the effects of local hydrostatic stresses on Cu interconnects, and most of the discussion will be based on the hydrostatic stress analysis, the term 'stress' used in subsequent chapters will be that of the hydrostatic stress, unless otherwise noted.

RESULTS AND DISCUSSIONS

The ACT results obtained from the crystal orientation analysis of 85 Cu grains from 9 different lines covering an area of approximately 9 microns by 180 nm is given in Figure 4. As shown from the inverse pole in fig. 2, while many (111) oriented grains are evident, many (110) and (115) grains (twins), as well as other grain orientations exist in the Cu lines. In comparison with the EBSD technique, the ACT software allows the identification of very fine grains (as small as 20-30 nm) that were present in the Cu lines. Moreover, the removal of the top passivation layer is not necessary, as long as the thickness of the passivation layer does not interfere with the TEM observations.

The local hydrostatic stress along the Cu interconnect lines was then simulated. The crystal orientation obtained from ACT was used as input data in the OOF2 software, which takes into account the strong anisotropy of Cu, namely a variation in the elastic modulus from 67 GPa along the [001] direction to 190 GPa along the [111] direction. Since the ultimate purpose of the OOF2 stress simulations is to correlate

the local stress ('stress' refers to hydrostatic stress unless otherwise noted) along the Cu interconnects lines with void formation, the boundary conditions should be selected for the temperature at which voids were observed to form. As void formation occurred in the range 220-250 $^\circ$C, an average temperature of 230 $^\circ$C was used for the calculations. The normal stresses at this temperature, namely σ_x=262 MPa and σ_y=191 MPa, were obtained from Gan *et. al.* [1] and used as boundary conditions.
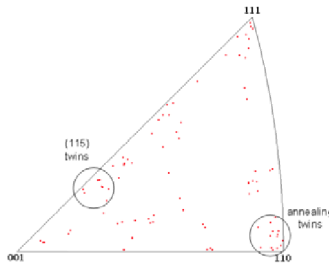
FIGURE 2. Inverse pole figure based on 85 grains measured at the top layer of 180nm Cu lines.

Assuming a 2D model and the aforementioned boundary conditions, the stress analysis performed by OOF2 in the 180 nm lines revealed a range of quasi-hydrostatic stresses from 270 MPa to 420 MPa. In particular, high local hydrostatic stresses are frequently observed at triple junctions, where a Cu grain boundary meets the DB interface.

In order to observe the effect of local stresses on void formation, *in-situ* TEM heating experiments were performed on 180 nm lines previously analyzed by ACT to determine the local grain orientation. Subsequently, the local stresses were simulated to correlate the sites where void formation occurred with the calculated stresses (Fig. 3). As voids were previously observed to form at the triple junctions, these triple junctions are especially of interest. Of the nine available triple junctions present in the Cu interconnect line in Fig. 3(d), void formation occurred at the triple junction between grains *a* and *b* (Fig. 3b). Grain 'a' has an orientation of $\{11\,5\,1\}<\bar{3}\,8\,\bar{7}>//TD$ while grain 'b' has a crystal orientation of $\{0\,2\,3\}<13\,\overline{12}\,8>//TD$. The grains are 7.13° and 5.18° away from $\{012\}$, respectively. As shown in Fig. 3c-d, the junction between grains *a* and *b* is characterized by a high local stress. In addition, a careful observation of the stress distribution in the Cu interconnect line shown in, along the left edge of the Cu line, shows that the highest stress exists at the site where void formation occurred [14]. This is in similar agreement with results previously reported for larger Cu grains on Cu films [15]. The stress values at the very bottom and top of the Cu interconnect line should be disregarded, as these values are artifacts caused by the truncation of material.

More recently, a novel electron diffraction technique, called D-STEM has been developed using a JEOL 2010F TEM/STEM instrument to enable automated analysis of orientations in nanocrystalline Cu grains. The D-STEM mode consists of a 1-2 nm parallel probe scanned over the specimen to obtain a bright-field or dark-field STEM image. The beam is controlled, translated and subsequently positioned on the image at the feature of interest, while the diffraction pattern is recorded on a CCD camera. This technique has been successfully employed to obtain spot diffraction patterns from nanoparticles as small as 3 nm [16-17]. With constant downscaling of line widths, this technique would particularly be useful for accurately obtaining diffraction information from very small nanocrystalline Cu grains; a task currently challenging using existing techniques. Results obtained by using D-STEM on Cu lines corresponding to 120 nm wide lines are depicted in Fig. 4.
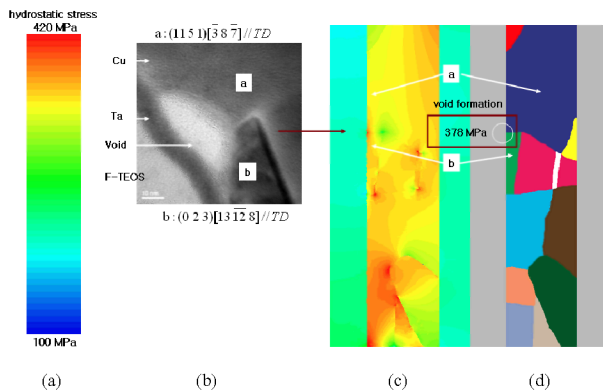
(a)   (b)   (c)   (d)

FIGURE 3. (a) Stress range (b) TEM image of void forming between grain **a** and **b** (c) Stress contour map of interconnect line (d) Schematic diagram of the 180 nm Cu interconnect. The void formed with the white circle at a triple junction.
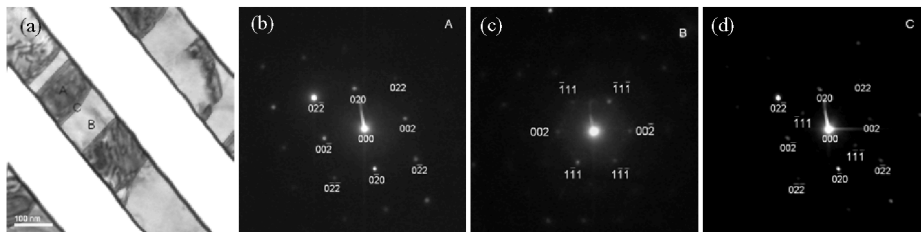

FIGURE 4. (a) Bright-field STEM image of 120 nm wide copper lines (b) Diffraction pattern obtained from grain labeled 'A' in fig. 4(a), representing crystal orientation close to [100] (c) Diffraction pattern obtained from grain labeled B in fig. 4(a), representing a near [110] crystal orientation (d) Diffraction pattern from the grain boundary region labeled C in fig. 4(a), clearly depicting diffraction information from both grains, A and B.

CONCLUSIONS

Individual crystal orientation of nanoscale Cu grains, as small as 20-30 nm, in 180 nm Cu interconnects were obtained by TEM using ACT. The local crystal orientation information was used to simulate the local hydrostatic stresses in the Cu interconnects, and correlated with *in-situ* TEM heating experiments and consequent void formation. The local stress simulations and the in-situ TEM results seem to indicate that the presence of high local stresses and stress gradients are a key factor in void formation. With the use of the novel D-STEM technique, such analysis can be conducted in an automated fashion an on even finer Cu interconnects.

ACKNOWLEDGEMENTS

REFERENCES

[1] D. Gan, G. Wang, P. Ho, *Proc. of the IITC 2002* 271-273
[2] A. Wikstrom, P. Gudmundson, S. Suresh, *Journal of Applied Physics* **86** (1999), 6088-6095
[3] Conal E. Murray, Paul R. Besser, Christian Witt, Jean L. Jordan-Sweet, *Applied Physics Letters* **93** (2008) 221901
[4] H. Okabayashi, *Materials Science and Engineering* **R11** (1993) 191-241
[5] A. Budiman, W. Nix, N. Tamura, B. Valek, K. Gadre, J. Maiz, R. Spolenak, J. Patel, *Applied Physics Letters* **88** (2006) 233515
[6] R. Gleixner, B. Clemens, W. Nix, *Journal of Materials Research* **12** (1997) 2081-2090
[7] H. Zhang, G.S. Cargill III, Y. Ge, A.M. Maniatty, W. Liu, *Journal of Applied Physics* **104** (2008) 123533
[8] F.J. Humphreys, Scripta Materialia **51** (2004) 771-776
[9] D. Williams, C. Carter, *Transmission Electron Microscopy*, Plenum Press (1996)
[10] Chunfei Li, David B. Williams, *Micron* **34** (2003) 199-209
[11] J.H. An, P.J. Ferreira, *Applied Physics Letters* **89** (2006) 151919
[12] T. Courtney, *Mechanical Behavior of Materials*, Mcgraw-Hill (2000)
[13] Haixia Mei, J.H. An, Rui Huang, P.J. Ferreira, *Journal of Materials Research* **22** (2007) 2737-2741
[14] J. An, J. Pickering, P.J. Ferreira, (to be submitted to *Journal of Materials Research)*
[15] A. Sekiguchi, J. Koike, S Kamiya, M. Saka, and K. Maruyama, *Applied Physics Letter* **79** (2001) 1264-1266
[16] K.J. Ganesh, M. Kawasaki, J.P. Zhou, P.J. Ferreira, *Proc. Microscopy and Microanalysis*, (2009) (in press)
[17] K.J. Ganesh, M. Kawasaki, J.P. Zhou, P.J. Ferreira, (to be submitted to *Microscopy Research and Technique*)