Preliminary Hard X-ray Micro-spectroscopic Investigations on Thin-Film Ta-and-W Based Diffusion Barriers for Copper Interconnect Technology

James M. Ablett[1], Joseph C. Woicik[2] and Zsolt Tokei[3]

[1] *Brookhaven National Laboratory, National Synchrotron Light Source, Upton, New York, 11973, USA.*
[2] *National Institute of Standards and Technology, Gaithersburg, Maryland, USA.*
[3] *IMEC, Kapeldreef 75, B-3001 Leuven, Belgium.*

Abstract. Within the microelectronics industry, the requirement for reducing device dimensions for increased circuit performance and lower manufacturing costs has led to many avenues of research in advanced materials and fabrication processes. One of the most important challenges in ultra-large scale integrated technology is the fabrication of thin-film diffusion barriers that prevent copper interconnect lines diffusing through the barrier material and into the neighboring silicon layers. In this paper, we present preliminary synchrotron x-ray spectroscopy measurements as a tool for studying the properties of these buried barrier layers and consider the opportunity of applying the spatial resolution of an x-ray microbeam in probing different regions of the barrier material.

Keywords: Microelectronics, Diffusion Barrier, X-ray Spectroscopy, Synchrotron.
PACS: 82.80.Ej, 85.40.-e.

INTRODUCTION

As the width of Cu interconnect lines falls beyond 90 nm and is anticipated to reach 45 nm within the next few years, the need for forming ultra-thin highly-conformal barrier layers along the entire surface area of the interconnect trench is required for reliable device operation. Current research is focused on finding the optimal fabrication processes and barrier materials as devices continue to scale down in size. Barrier properties depend on a wide range of parameters such as growth processes, anneal temperatures, amorphous/polycrystalline structure, composition and thickness.[1-9] Barrier liners, comprising of refractory materials such as Ta, W, Ti and their nitrides, have been studied in their effectiveness for preventing Cu diffusion through the barrier layer and into the nearby silicon.[1-9] The major barrier reliability concern is that Cu impurity atoms are extremely mobile and therefore detrimental to active silicon transistor performance. Good adhesion of the barrier layer to the substrate and the copper interconnect is another important factor to consider. The ability to determine the local bonding environment of thin barrier layers using x-ray spectroscopy would be extremely beneficial as it could reveal important information governing the overall performance of the barrier material, which can be amorphous and therefore not measurable by x-ray diffraction. In this paper, we show through preliminary investigations, that x-ray micro-spectroscopy is a powerful analytical tool for studying these barrier systems.

EXPERIMENT

Feasibility studies were performed on tantalum and tungsten based barriers layers, grown by physical vapor deposition (PVD) and atomic layer deposition (ALD), with ALD identified as the likely candidate for growing highly conformal barrier films in sub 65 nm interconnect lines.[7] Both blanket barrier films, with and without Cu overlayers, and passivated Cu interconnect samples fabricated using damascene and chemical mechanical planarization processes, with varying line spacing, widths and barrier thicknesses were investigated. All samples were grown on Si(100) wafers, which had a SiO_2 insulating layer on the top. The experiments were performed at the

Downloaded 09 Feb 2007 to 130.199.3.130. Redistribution subject to AIP license or copyright, see http://proceedings.aip.org/proceedings/cpcr.jsp

National Synchrotron Light Source on the X27A micro-focus bending-magnet beamline, which has a 6 μm [vertical] x 17μm [horizontal] *fwhm* spot size.[10] The samples were mounted upright and at ~ 45° to the incident x-ray beam, on a motorized x-y-z stage for accurate positioning. A 13-element liquid nitrogen cooled germanium detector and associated digital processing electronics were used for collecting and analyzing the fluorescence x-rays emitted from the sample. For x-ray absorption near-edge structure (XANES) and extended x-ray fine structure (EXAFS) measurements, the incident x-ray energy was tuned across a particular absorption-edge of interest using a Si(111) channel-cut monochromator as the change in intensity of the various fluorescence line(s) of interest were recorded. More details on the X27A micro-focus experimental beamline and capabilities can be found in [10].

RESULTS AND DISCUSSION

One of the difficulties in applying standard x-ray fluorescence spectroscopy to these barrier layers is the proximity of the core-level x-ray fluorescence energies of the associated elements at the L-edges of Ta and W, and at the K-edge of the Cu interconnect/overlayer. With a typical energy-dispersive detector, with an energy resolution of ~ 2%, i.e. 200 eV *fwhm* at 10 keV, peak overlap between the Ta/W Lα and Cu Kα lines is problematic. However, probing the L_2 absorption edge of Ta and W opens up additional decay channels that can be used to investigate these species of interest. To highlight this, Fig. 1. shows (plotted on a log scale) the MCA spectra taken above (diamond symbol) and below (straight line) the Ta L_2 absorption edge on a tantalum-based barrier film with a 1000 nm Cu overlayer. The barrier layer was prepared by PVD and the system was ordered as; Si(100) / SiO_2 / Ta_2N(5nm) / Ta(10nm) / Cu(1000 nm). As can be seen, the Ta $L\beta_1$ emission (L2-M4) is well separated from the neighboring fluorescence lines and the Compton/elastic scatter peaks.

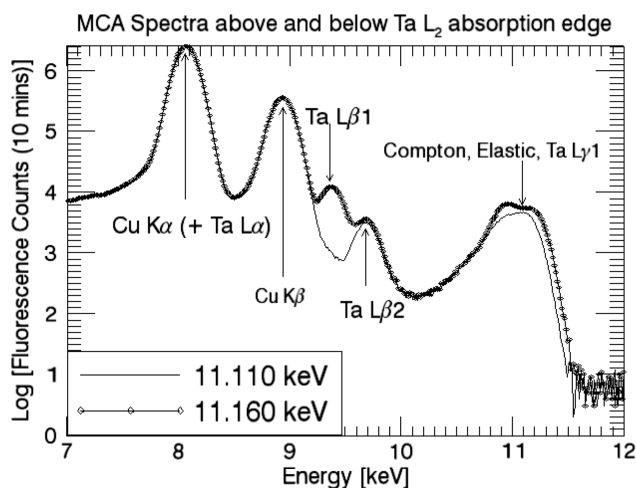

FIGURE 1. MCA Spectra from a Ta-based barrier liner with a Cu overlayer (blanket film), above and below the Ta L_2 absorption edge (11.136 keV). The peaks are labeled according to the main contributions from the various decay channels.

The ability of obtaining chemical and bonding information from the near- and- extended regions of the x-ray absorption spectra on Ta-based barrier systems is now realized. This is confirmed in Fig. 2., where Ta $L\beta_1$ fluorescence is plotted as a function of incident x-ray energy, across the Ta L_2 absorption edge (left), which is a combination of 40 scans, lasting ~ 40 hours in total. The corresponding EXAFS spectra is shown on the right, and although this preliminary data are somewhat noisy, better statistics will be available from real-world systems that have much lower copper coverage. We believe it is possible, through analyzing our other preliminary data, that good EXAFS spectra on < 5nm thin barrier films can be obtained under typical < 200 nm thick Cu interconnect lines within a reasonable amount of measurement time i.e. a few days.

Downloaded 09 Feb 2007 to 130.199.3.130. Redistribution subject to AIP license or copyright, see http://proceedings.aip.org/proceedings/cpcr.jsp

Furthermore, another diffusion barrier system candidate, WNC/Ta has been examined for potential x-ray spectroscopic analysis. These barrier layers were grown by ALD and the system was ordered as; Si(100) / SiO_2 / WNC(6nm) / Ta(10nm) / Cu(50-100 nm). Figure 3. (left and center) shows the MCA spectra taken above (diamond symbol) and below (straight line) the W L_2 absorption edge. The W Lβ1 fluorescence was identified as being a possible applicant for measuring XANES/EXAFS on these systems, and Fig. 3. (right) shows W L_2 XANES measurements on ALD 60 cycle (4nm) and 90 cycle (6nm) WNC/Ta- based barrier layers using this emission channel.

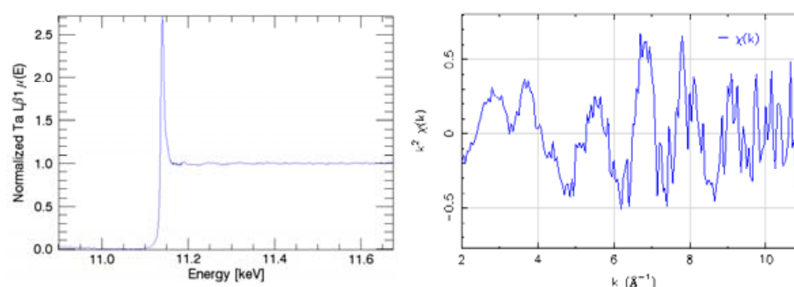

FIGURE 2. Ta L_2 absorption edge scan, obtained by recording the Ta $L\beta_1$ fluorescence intensity and corresponding EXAFS data taken on a PVD Ta_2N (5nm) / Ta (10 nm) barrier with a 1000 nm Cu overlayer.

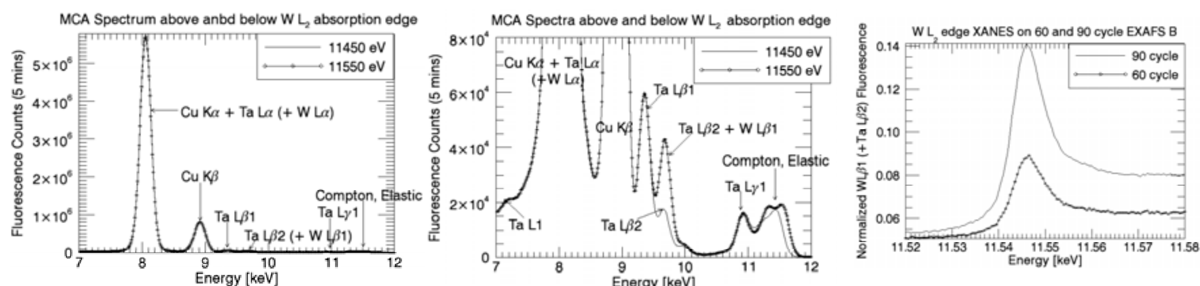

FIGURE 3. Left and Center: MCA Spectra from a WNC/Ta-based barrier layer with a Cu overlayer (blanket film), above and below the W L_2 absorption edge (at 11.544 keV). The peaks are labeled according to the main contributions from the various decay channels. Right: W L_2 absorption-edge XANES scan on 60 (4nm) and 90 (6nm) cycle WNC/Ta based barrier layers with a Cu overlayer.

The spatial resolution offered by x-ray micro/nano probes allows for the possibility of examining the chemistry/bonding properties of these buried barrier layers at various locations around and beneath the Cu interconnect lines. Preliminary measurements on passivated 10 μm wide 120 nm thick Cu interconnects, separated by 40 μm and 90 μm 'field' regions, and with a 4 nm thin ALD Ta_3N_4 barrier layer were performed at X27A. Cu Kα fluorescence recorded at an incident x-ray energy of 9.5 keV, and Ta $L\beta_1$ fluorescence recorded at the peak of the Ta white-line at 11.142 keV, are shown in Fig. 4a. as the 10 μm wide interconnect lines, spaced by 40 μm, are scanned across the x-ray microbeam. The Cu and Ta fluorescence increases across the interconnect lines, but do not fall to zero off a line. The reason for this was that sub-micron copper line 'dummies' were inserted within the 'field' regions for these particular test structures. Figure 4b. similarly shows the variation in Cu fluorescence across 10 μm wide, 90 μm spaced interconnect lines and Fig. 4c. shows a Cu fluorescence map across an area that contains two copper pad structures. Clearly, the application of x-ray microprobes for exploring the chemistry and bonding of the barrier layers at various locations in device structures is evident. Figure 5. shows the Ta L_2 XANES on a PVD Ta_2N(5nm)/Ta (20nm) blanket film (straight line A14) and a 4nm ALD Ta_3N_4 barrier used in a copper interconnect test structure (diamond and asterisk symbols). As is shown, the nitrogen content within the Ta_3N_4 barriers is clearly evidenced by the spectral signature of the near-edge region and the shift in the absorption edge towards higher x-ray energies, compared to the Ta_2N(5nm)/Ta(20nm) blanket film which is of predominantly Ta character.

Downloaded 09 Feb 2007 to 130.199.3.130. Redistribution subject to AIP license or copyright, see http://proceedings.aip.org/proceedings/cpcr.jsp

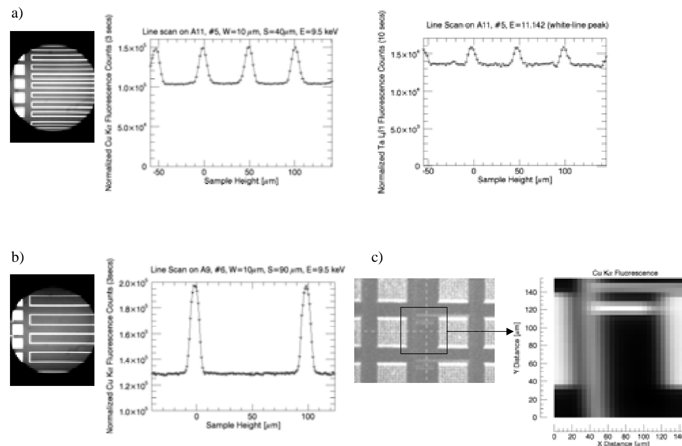

FIGURE 4. X-ray fluorescence scans across copper interconnect a) & b) and pad structures c).

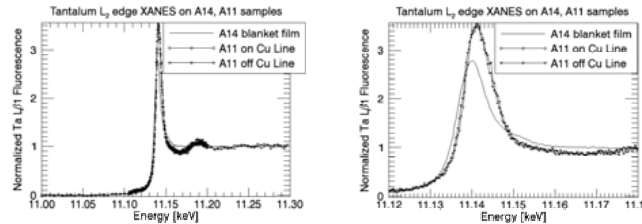

FIGURE 5. Ta L_2 absorption-edge XANES on Ta_2N (5nm)/Ta (20nm) blanket film (straight line A14) and the Ta_3N_4 barrier on a Cu interconnect patterned sample, both on and off the interconnect lines (diamond and asterisk symbols A11).

CONCLUSION

In this paper, we have shown the prospective use of x-ray spectroscopy for studying buried thin-film diffusion barriers for copper interconnect technology. By accessing the L_2 absorption edges of Ta and W, these barrier layer systems can be investigated, with the potential of revealing the chemistry and bonding of these species through XANES and EXAFS measurements. In addition, measurements on test structure devices can be performed using x-ray micro-spectroscopic techniques to probe different regions of the barrier material, such as the sidewalls and trench bottoms of Cu interconnect lines. Furthermore, with the increasing availability of synchrotron x-ray nano-probes with spatial resolutions below 100 nm, these instruments open up new opportunities for studying sub-100 nm device structures for real-world microelectronics applications.

ACKNOWLEDGMENTS

Research carried out at the National Synchrotron Light Source, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Office of Basic Energy Sciences and Division of Materials Sciences under Contract No. DE-AC02-98CH10886.

REFERENCES

1. Chung, H.C. and C.P. Liu, Surface and Coatings Technology **200**, 3122 (2006).
2. Hübner, R., et al., Thin Solid Films **500**, 259 (2006)
3. Jiang, L., et al., Jap. Journ. Appl. Phys. **41**, 6525 (2002).
4. K.-L., O., et al., Journ. Vac. Sci. Techn. B **23**(1), 229 (2005).
5. Kim, H. and S.M. Rossnagel, Thin Solid Films **441**, 311 (2003).
6. Kim, H., et al., Journ. Appl. Phys. **98**, 014308-1 (2005).
7. Kim, H., Surface and Coatings Technology **200**, 3104 (2006).
8. Li, N., D.N. Ruzic, and R.A. Powell, Journ. Vac. Sci. Techn. B **22**(6), 2734 (2004).
9. Wang, H., et al., Appl. Phys. Lett. **81**(8), 1453 (2002).
10. Ablett, J.M., et al., Nucl. Instrum. and Meth. in Phys. Res. A **562**, 487 2006.

Downloaded 09 Feb 2007 to 130.199.3.130. Redistribution subject to AIP license or copyright, see http://proceedings.aip.org/proceedings/cpcr.jsp