# Orientation and Stress Mapping on Beamline 7.3.3.: a New Tool to Study Material Properties at Submicron Scale

N. Tamura<sup>1</sup>, R. Spolenak<sup>2</sup>, B.C. Valek<sup>3</sup>, R.S. Celestre<sup>1</sup>, A.A. MacDowell<sup>1</sup>, H.A. Padmore<sup>1</sup> and J.R. Patel<sup>1,4</sup>

<sup>1</sup>Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA
<sup>2</sup>Bell Laboratories, Lucent Technologies, Murray Hill NJ 07974 USA
<sup>3</sup> Dept. Materials Science & Engineering, Stanford University, Stanford CA 94305 USA
<sup>4</sup>SSRL/SLAC, Stanford University, P.O.BOX 43459, Stanford CA 94309 USA

## **INTRODUCTION**

Mechanical properties of materials (resistance to applied stress, fatigue, temperature cycling,...) are highly dependant on their microstructure and initial local stress state. We have demonstrated a synchrotron technique capable to resolve grain orientation and stress in polycrystalline samples with a spatial resolution compatible with the length scale of their microstructures (submicron to a few microns) and initial results have been presented [1,2]. Further progress in the hardware and software design on the X-ray microdiffraction beamline (7.3.3.) include the ability to completely map in-situ the orientation and stress of a given material with the submicron X-ray beam spot size provided by state-of-the art achromatic beam focusing technique [3].

## **EXPERIMENTAL SETTING**

The experimental setting of the beamline is described in Fig 1. The X-ray synchrotron beam from a bent magnet source is focused via a pair of bendable Kirkpatrick-Baez mirrors to a submicron size (0.7x0.8 µm FWHM). A 4-crystal Si monochromator is used to easily switch between white and monochromatic beams while the same area on the sample is illuminated. The sample is on reflective geometry, the surface making an angle of 45° relatively to the incoming beam. The outcoming Bragg reflections are collected using a large area CCD detector (Bruker, active area of 9x9 cm) placed at about 3 cm above the sample. Illuminating an area of interest with submicron white beam provides a Laue pattern which can be image-treated and automatically indexed. The indexing yields at the same time the crystal orientation and deviatoric (distortional) strain tensor of the illuminated area. By putting the sample on an X-Y piezo stage, it can be scanned under the focused beam with a submicron step size. This allows obtaining orientation and strain/stress maps of the material. The complete strain tensor (6 components) can also be computed by additionally determining the dilatational ("hydrostatic") component. This is achieved by measuring the energy of at least one reflection using the monochromator.

### ANALYSIS AND RESULTS

A software package developed at the ALS (X-MAS for X-Ray Microdiffraction Analysis Software) is used for data collection, Laue patterns indexing, strain refinement and monochromatic beam scans analysis. An orientation map "smoothing" algorithm also allows for the automatic determination of grain boundaries by fitting the intensity profile of each individual crystal grain and intersecting the resulting normalized profiles. The technique was applied to test samples fabricated to study electromigration failure mechanism in thin metal lines, which interconnect millions of transistors in modern integrated circuits. They consist of patterned Al(Cu) line (length:  $30 \mu$ m, thickness:  $0.7 \mu$ m, width: 4.1 and 0.7  $\mu$ m) vapor deposited on an Si

wafer and buried under a glass passivation layer. As a comparison, data has been also taken on unpassivated Al(Cu) pads (blanket films).



Fig. 1.- Experimental setting of the X-ray Microdiffraction end station on beamline 7.3.3.

Fig 2 shows orientation and deviatoric stress maps on a  $5x5 \ \mu m$  region in the pad and on the 4.1  $\mu m$  and 0.7  $\mu m$  wide lines. The stress in the pad appears to be biaxially tensile in average, which is consistent with macroscopic stress measurements using conventional X-ray techniques. However, at microscopic scale, stress is actually far from being homogeneous. It is triaxial rather than biaxial, with local differences reaching 60-80 MPa.



Fig. 2.- In-plane orientation and deviatoric stress components along x, y and z for the Al(Cu) blanket film (top), 4.1  $\mu$ m wide passivated line (bottom left) and 0.7  $\mu$ m wide passivated line (bottom right).

Similarly, lines displayed local variations of 60-80 MPa in stress for the 4.1  $\mu$ m line and up to 140 MPa for the 0.7  $\mu$ m line. As the line gets narrower, the level of stress gets higher and, in average, from biaxial becomes triaxial. Orientation maps also show the change in the microstructure from polycrystalline in the pad and in the 4.1  $\mu$ m line to "bamboo"-type for the 0.7  $\mu$ m line. Electromigration-induced failure in interconnect metal lines are highly dependant on the microstructure and initial stress state of the samples. The capability demonstrated by the instrument to probe non-destructively local grain structure as well as stress becomes particularly relevant to understand microstructure-related failure mechanisms and to predict where the line is likely to fail during service. The X-ray Microdiffraction Laue technique is extremely promising for this kind of study and initial electromigration in Cu [4] and Al [2] lines have been presented elsewhere.

## REFERENCES

- 1. MacDowell, A.A., Celestre, R.S., Tamura, N., Spolenak, R., Valek, B.C., Brown, W.L., Bravman, J.C., Padmore, H.A., Batterman, B.W. & Patel, J.R., *Proceeding of the* 7<sup>th</sup> *International Conference on Synchrotron Radiation Instrumentation*, in press (2000)
- Tamura, N., Valek, B.C., Spolenak, R., MacDowell, A.A., Celestre, R.S., Padmore, H.A., Brown, W.L., Marieb, T., Bravman, J.C., Batterman, B.W., and Patel, J.R., *Mater. Res. Society Symp.*, 612, in press (2000)
- 3. MacDowell A.A., Celestre R., Chang C.H., Franck K., Howells M.R., Locklin S., Padmore H.A., Patel J.R., and Sandler R., *SPIE Proceedings* **3152**, pp. 126-133 (1998)
- Spolenak R., Barr, D.L., Gross, M.E., Evans-Lutterodt, K., Brown, W.L., Tamura, N., Macdowell, A.A., Celestre, R.S., Padmore, H.A., Valek, B.C., Bravman, J.C., Flinn, P., Marieb, T., Keller, R.R., Batterman, B.W., and Patel, J.R., *Mater. Res. Society Symp.*, 612, in press (2000)

This work was initiated with support from the LBNL Laboratory Director's Research and Development Fund. Additional support was provided by NIH grant GM51487 and by the US Department of Energy, Office of Basic Energy Sciences, under contract # DOE-AC03-765F00098. We thank Intel Corp. for partial funding of the beamline.

Principal investigator: Nobumichi Tamura, Advanced Light Source, Lawrence Berkeley National Laboratory. Email: ntamura@lbl.gov. Telephone: 510-486-6189.