A New Instrument for Submicron X-Ray Diffraction

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INTRODUCTION

The availability of high brightness 3rd generation rings allows the possibility of developing the more conventional synchrotron techniques into the microscopy realm. X-ray diffraction has been a technique used for 80 years in the study of materials usually on the mm length scale. Various new problems can be tackled if x-ray diffraction could be developed down to the micro-spatial length scale. As an example, extremely high mechanical stresses exist in modern microelectronic devices. These stresses are caused by thermal mismatch, by confinement, and by current flow. The effects of this high stress, particularly cyclic stress is to cause failure, from simple delamination to the more complicated case of electromigration where the high current flow results in material transport and the formation of voids in the metal conductor lines and subsequent chip failure (1). Although these problems are of great significance, to date no tool has been available to study stresses on the micron scale. Stress on the macro scale is routinely studied with x-ray diffraction. Here at the ALS we have instrumented beam line 7.3.3 with a new machine that is capable to studying stress on the micron scale by means of x-ray micro diffraction.

EXPERIMENTAL

Carrying out x-ray diffraction on the micron scale requires some different instrumental considerations compared to the more conventional macro x-ray diffraction. First there is the problem of finding the sample. Then there is the problem of how to measure rocking curves for such small samples without having to deal with 'sphere of confusion' issues associated with conventional goniometers. We have addressed the first problem by the use of white light to illuminate the sample – the Laue pattern generated by the micro crystal establishes its location and orientation. The second problem is addressed by keeping the sample fixed and scanning the wavelength.

The bend magnet beam line has a unity magnification toroidal mirror that produces a 50 by 200 micron focus just inside an x-ray hutch at the position of an adjustable x-y slit. The beam path in the hutch consists of the source defining x-y slits, a four bounce Ge or Si monochromator, followed by an elliptically bent Kirkpatrick- Baez mirror pair (2) which images the beam from the x-y slits on to the sample. The adjustable slits allow the ability to trade flux for spot size. The spot size can be varied on the sample from 0.8 x 0.8 microns to 15x5 micron (fwhm). The 4-crystal monochromator has the property of allowing the sub micron sample to be illuminated with either white or monochromatic x-rays (~6-14KeV). This is essential for characterizing crystals or crystal grains in the sub-micron range. Sample rotation is fixed and rocking curves are measured

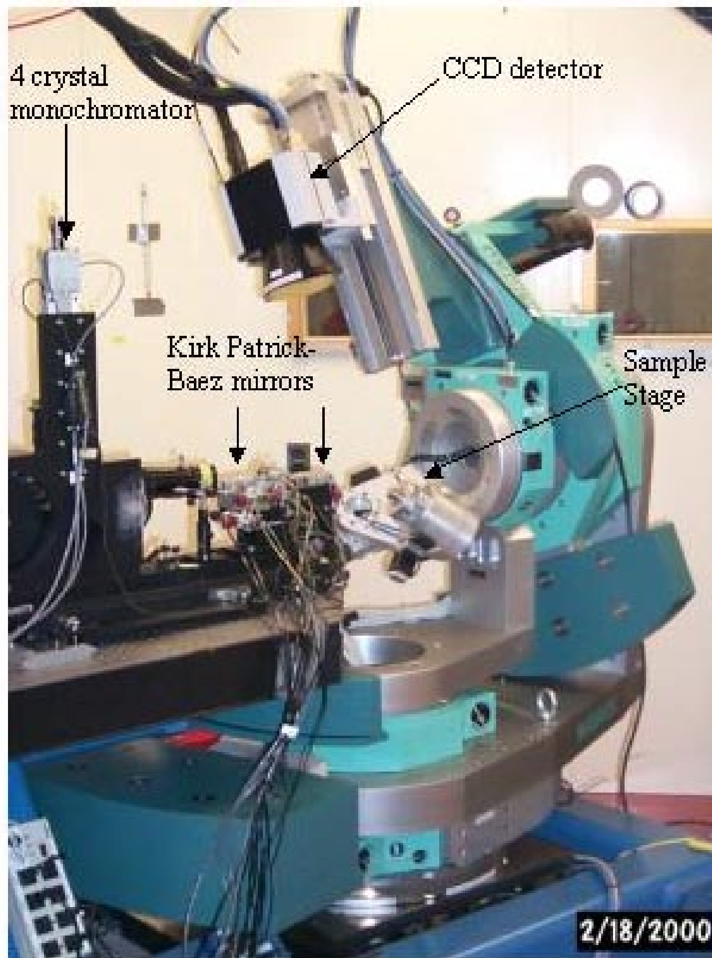

Figure 1. Photograph of the new X-ray micro diffraction facility on beamline 7.3.3

by scanning the wavelength of the 4-crystal monochromator. The sample is mounted on a precision translation stage to allow for characterization of different crystal regions or grains. The sample stage is supported on a state of the art six-circle diffractometer (Huber) equipped with encoders in the main rotation stages calibrated to a second of arc. The detector is a 4K x 4K CCD (Bruker) with a 9x9 cm view area mounted on a detector arm that can be positioned around the sample. The detector itself can be radially translated away from the sample along the detector arm. Figure 1. shows a photograph of the new instrument.

The micro diffraction technique that has been developed consists of illuminating the sample with the submicron broad bandpass (white) beam and collecting the Laue reflections using the large area CCD detector. The sample is orientated 45 degrees to the illumination beam and the CCD detector placed at 90 degrees as shown in figure 2. As the crystal structure of the sample is known, the Laue patterns yield the orientation of the micro-crystals. Slight displacements of the Laue spots from their “correct” (distortionally free) positions allows for the full deviatoric (distortional) strain tensor (3) of the micro-crystal to be directly derived. The dilatational component of the strain is obtained by switching to monochromatic beam and making energy scans on selected reflections. The sample is mounted on a precise positioning stage that allows the sample to be scanned.

INITIAL RESULTS

The machine was commissioned in December 99 and has been successfully applied to the study of residual strain in passivated (buried under an SiO_2 insulator) Al interconnect test structures as well as Cu blanket films. We have found that the microstructure at the sub-micron level in these materials is rather complex with grains divided into sub-grains and inter- as well as intra- granular variations of both strains and orientations, leading to the potential for some new insight on the understanding of failure in interconnects. The ability of the instrument to measure small changes in d spacing is

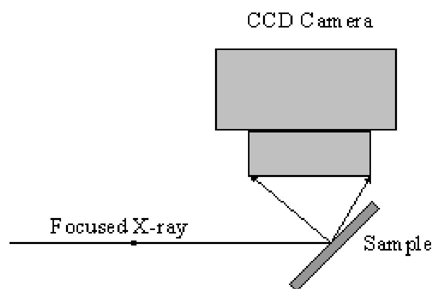

Figure 2. Schematic diagram of experimental arrangement.

crucial if the instrument is to measure the small strains in the micron sized samples. We have established that when measuring the deviational strain the crystal d spacing can be currently measured to an accuracy of 2x10-4, which corresponds to a stress accuracy of 20MPa for Al.

X-ray microdiffraction presents many advantages compared to alternative techniques for microtexture study such as backscattering electron microscopy/ microdiffraction. X-ray micro diffraction has much higher strain sensitivity and precision, the technique is non-destructive and can be applied to buried samples without need of special preparation. The technique is phase sensitive in the sense that it recognizes different crystalline structures, and with its high orientation sensitivity, allows the distinction of subgrains with angular misorientations of a fraction of a degree. One can take advantage of the penetrating property of X-rays to study the stress and microstructure within bulk samples. This technique is therefore suitable for a host of problems in materials science including the study of grain growth mechanism and plastic deformation.

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