CHARACTERIZATION OF STRAIN IN THIN-FILM STRUCTURES WITH MICRODIFFRACTION

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ABSTRACT

We have constructed a microdiffraction endstation for use at the monochromatic synchrotron beamline X20A of the National Synchrotron Light Source at Brookhaven National Laboratory. It consists of a tapered capillary for microfocusing and a limited four-circle diffractometer with high-precision sample positioning. It was designed for the characterization of strain, mosaic broadening, and orientation in samples that are interesting to the microelectronics industry, including thin metal film structures and semiconductor films. The unique combination of micron-scale diffraction imaging, fluorescence imaging, and high-precision lattice-parameter determination make this a valuable tool for both basic materials studies and applied research.

We illustrate the capabilities of the instrument with a recent study that characterizes thin-film/substrate interfaces. The quality of adhesion between 1mm diameter thin metal film “dots” and a silicon wafer substrate was assessed on a microscopic scale. The Shear-Lag model predicts maximum shear-stress at the film edge, smoothly decreasing toward the center of the dot. Using a microfocussed x-ray beam and recording the Si(004) reflection intensity, topographic images of the Si around and under an Al dot were constructed. Results show a striking difference between the stress transfer observed experimentally and that predicted by the model. The Al/Si interface is not fully coupled, and despite a uniform thickness profile and absence of surface cracks, shows piecewise disruptions in the stress transfer occurring at intervals from 60 to 250μm.

INTRODUCTION

As ever greater numbers of microelectronic devices are crowded onto chips in each generation to lower their cost and increase their speed, the areas of individual features in each device, such as contacts, gates, metal lines, and dielectric layers, continue to shrink. Moore’s Law, which predicts that computing power will double every 18 months, has held fast for the last two decades, primarily because of this “scale integration” (Very Large, Ultra Large, etc.). Experts predict that in the near future scaling will reach its practical limits, and that only the development of new materials and processes, or the invention of devices based on new paradigms, will keep the microelectronics industry on track. An invariant in all of this evolution has been a requirement, driven by economics, that no more than one chip fail for every thousand manufactured.
Each chip contains millions of transistors and an equal number of passive elements. In addition, hundreds of meters of sub-micron interconnection wiring (metallization) on multiple levels link these devices together and connect them to power, input, and output. Several million "vias" connect the various metallization levels. The smallest feature size of present-day transistors is 0.25 \( \mu \text{m} \). The Semiconductor Industry Roadmap\cite{1} predicts that by the year 2003 this will shrink by 50\% to 0.13\( \mu \text{m} \). One can surmise from the facts and predictions above that there is a great need to measure and understand materials and processes on a micron and sub-micron length-scale. The structure and behavior of materials constrained in size to these dimensions can be drastically different from that of the same materials in bulk. Likewise, even for systems that are macroscopic in size, homogeneity may break down on a local scale, and global materials constants and continuum mechanics might not accurately describe structure and mechanical behavior on a microscopic scale.

Many of the failures in microelectronic circuits are owed to stresses. These can arise from thermal or lattice mismatch of the disparate materials that must be coherently bonded to one another. They can also be caused by contamination, electromigration, etc. X-ray diffraction has proven to be an important tool for the direct determination of stress (or strain) and defect structures in solid crystalline materials. The availability of high-brightness synchrotron sources has allowed for this technique to be extended to the characterization of materials on a micron-sized scale in reasonable data-collection time. We have constructed a microdiffraction facility for use at a monochromatic bending-magnet synchrotron beamline. It has a tapered capillary for microfocusing and a limited four-circle diffractometer with high-precision sample positioning. It has been used during the past year to measure a number of samples interesting to the microelectronics industry. In this paper we will describe the instrumentation and discuss the characterization of the microfocussed beam, including divergence and beam size. Secondly, we will present results from a recent study: the measurement of interfacial stress/strain transfer between a silicon wafer substrate and 1mm diameter evaporated thin metal films that are under thermal residual stress at room temperature.

\section*{INSTRUMENTATION}

Figure 1 shows a schematic of the beamline and microdiffraction endstation. The components will be described under three subsections. A paper describing the instrumentation in greater detail is forthcoming\cite{2}.

\subsection*{Beamline}

The microdiffraction endstation was designed to be used at a bending-magnet beamline of the National Synchrotron Light Source (NSLS). Since one of the most important requirements was a system that can measure d-spacings of crystals accurately, it was decided that monochromatic radiation was preferable to white beam. White beam diffraction requires accurate determination of both the angle and energy of diffracted beams, and the standard practice of using a semiconductor energy-dispersive detector for energy measurement results in limited resolution. The endstation is
used at our IBM/MIT X20A Participating Research Team (PRT) beamline. One of the advantages of the PRT structure at the NSLS is the ability to modify or build beamlines to suit the needs of the team, and to have long periods of access for the commissioning and use of special instrumentation. The bending-magnet vertical source size is approximately 120μm, and the divergence is approximately 17μrad (0.001°)[3]. Beamline X20A has a horizontal acceptance of 4mrad (0.2°). The beam is focussed both vertically and horizontally by a toroidal Pt-coated silicon mirror at a 1:1 magnification.

This configuration folds some of the horizontal divergence into the vertical so that the focussed spot size at the sample position is about 1x1 mm. The mirror is followed by a double-crystal, fixed-exit Ge(111) monochromator. The total flux at the sample for 8-9 keV x-rays is on the order of 5x10\(^{11}\) photons/sec at a ring current of 250 mA.

**Capillary**

The x-ray beam is condensed further by total external reflection from the inner walls of a tapered glass capillary[4-8]. This is held in a gimbal mount which is rigidly attached to the diffractometer table and has three translational and two angular degrees of freedom for alignment. Our capillaries have entrance diameters of 100 or 50μm and exit diameters that range from 2 to 20μm. Because of the multiple bounces and angle for total external reflection, the convergence of the condensed beam is large. By performing scans of a knife edge across the beam at increasing distances from the tip of the capillary, and fitting the resultant sigmoidal curves to a Gaussian-broadened error function, we have measured this convergence to be \(-0.31°\). Using the same measurement we have determined the FWHM of the beam from our “5-μm” capillary, at a typical sample distance of 1.4mm from the tip, to be 10.6μm. The measured flux from the capillary at 8.5keV is \(-1.5x10^5\) cts/sec.

There are several other ways to define the size of the beam. One is to calculate the sinθ effect of sample tilt on the footprint of the beam. At a sample angle of 32° (θ for a Si(004) reflection at an x-ray energy of 8.5keV) the beam size in the diffraction plane direction (vertical) increases to
19.8μm. A third definition is the “dark field” size, or the actual size of the area on the sample that meets the diffraction condition. This is sample-dependent. For a silicon crystal at the Si(004) reflection angle, the diffracting area on the sample is 1.1μm in the diffraction plane direction and is 10.6μm in the horizontal plane (See Figure 2b). The integrated intensity from a Si(004) reflection is ~10^4-10^5 cts/sec., depending upon the capillary. Figure 2 shows the difference between the size of the undiffracted beam at the detector (875mm from the sample) and that of a beam diffracted from a silicon wafer.

**Figure 2a:** Incident beam image at detector (875mm from sample position).

**Figure 2b:** Si(004) reflection image at detector (875mm from sample position).

**Sample positioning and diffractometer**

The sample is mounted on a standard two-circle Huber diffractometer that has been modified slightly by the addition of partial χ and φ arcs, each mounted on an x-y translation stage. This allows for accurate alignment of the circle centers on the center of the diffractometer sphere. Interchangeable sample holders are mounted on high-resolution micro-translation drives (x, y, and z) having a step size of 0.5 μm and accuracy of 1μm. These drives in turn are mounted on hand-driven, lockable, coarse x and y translation drives. One of the sample stages has the capability for heating to 300°C, and attachment of a chip-carrier for sending current through samples. The sample is held in place by roughing-pump vacuum. High-magnification cameras are used for keeping track of sample placement with respect to the beam in a top-down view, and with respect to the capillary in a side view.

Use of the θ circle and φ and χ arcs is necessary for alignment of single crystal samples and small crystalline grains, in order to get an accurate measurement of the lattice spacing. In addition, a long sample-to-detector distance of ~875mm is used for the same reason. For a silicon wafer at the
Si(004) reflection angle of 32° θ (8.5 keV) our measured Δd/d is 0.0003. For measurements of several reflections from the same grain or feature, one can compensate for the sphere-of-confusion by a measurement and transform calculation technique that is described in a paper now in preparation[9].

Our microdiffraction facility has the capability for three modes of operation. One is diffraction imaging (micro-topography), where the detector is tuned to a reflection of interest, and the diffracted intensity is collected as a function of position of the sample in the beam. This mode is used for grain mapping, defect imaging, strain contrast imaging, etc. Once a "mesh" has been made across a submillimeter-sized area, one can return easily to regions of high intensity and "tweak" them up, in preparation for more detailed measurement of tilt, mosaic broadening, or lattice spacing. This is the second mode — standard diffraction scanning. The third mode of operation is fluorescence mapping, where the sample is again translated in the beam and the fluorescence signal of interest is collected by a Si(Li) energy-dispersive detector. This is useful for mapping fluorescence markers or other features for alignment of the sample in the x-ray beam, and for measuring the sphere-of-confusion of the diffractometer.

INTERFACIAL STRAIN IN THIN METAL FILMS ON SILICON

An important problem in microelectronics manufacturing is the failure (fracture, delamination, etc.) of metalization. Strong and continuous adhesion between metal and substrate is required under all processing and operating conditions. In order to understand and predict behavior, continuum mechanics are used to model the strain at the interface between two adhering materials having different lattice constants and/or residual thermal stresses. One model that describes the transfer of stress/strain between a substrate and a discrete film section is the Shear-Lag Model[10]. It assumes a perfect interface and elastic deformations. The stress transfer occurs through shear stresses generated between the film and substrate. In the Shear-Lag model, these shear stresses

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\text{Stress} = \sigma_{\text{film}}, \sigma_{\text{substrate}}
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\text{Position} = \text{A, B, C, D}
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\text{Film Interface}
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\[
\text{Substrate}
\]

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\text{Figure 3: Schematic of the Shear Lag model. The shear stresses in the interface are a maximum at each end of the film and zero near the center, provided that the film dimension is greater than the critical length, } \ell. \text{ Between points B and C, the normal strain (due to an applied stress } \sigma_a) \text{ in the film, the interface, and the substrate are equal, and the ratio } \sigma_{\text{film}}/\sigma_a \text{ is equal to the ratio of the elastic moduli of the film and the substrate.}
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are a maximum at the outer edges of the film and decay smoothly toward the center. The normal stress along the loading direction (in the film plane) is zero near the edges and increases towards the center. Figure 3 shows a schematic of the stress transfer predicted by the Shear-Lag model. The maximum stress that can be transferred depends upon the length of the film and the elastic moduli of the film and the substrate. In order for this model to be applied with meaning to small metal lines and features, it must describe the mechanical behavior of the film on a µ-sized length-scale.

We used the strain-contrast intensity measured from the silicon substrate under 1-mm diameter metal film “dots” to investigate the quality of adhesion on this length-scale and the applicability of the Shear-Lag model. Results are presented here for 850Å-thick film structures of aluminum evaporated onto Si(001) wafer substrates[11]. Mesh scans were taken of the Si(004) peak intensity of the substrates, in a ~2x2mm area around and under a dot. These were taken in 20µm steps. High-resolution scans were taken subsequently in 2µm steps across the dots at several places. Since silicon is a nearly “perfect” crystal, x-rays scattered from it exhibit dynamical diffraction, where the intensity is proportional to the structure factor, F, owing to primary and secondary extinction effects[12,13]. If, however, the crystal is distorted by a strain gradient, bending, or defects, extinction does not occur and kinematical diffraction theory describes the scattering. In this case, the diffracted intensity is proportional to F². If these films conform to the Shear-Lag model, with ideal strain transfer between the silicon and the metal, one would then expect that the integrated Si(004) intensity measured across one of these dots would be a fixed value outside the dot, would increase sharply at the edge, decrease smoothly towards the center, and show the mirror image behavior on the other side of the dot. If the “critical length” of the shear-stress region is less than the radius of the dot, the intensity at the center should be equal to that of the bare wafer, minus absorption due to the overlying metal film. Figure 4 shows a schematic of the expected behavior.

Figure 4: Top figure shows schematic for scanning sample while recording Si(004) diffraction intensity. Bottom figure shows expected intensity across the Al dot if the interface behavior follows the Shear-Lag model.
Figure 5 shows a Si(004) intensity contour plot from one Al dot interface, and a high-resolution profile scan across the dot at a Y Position of 2.54. Diffraction from the bare silicon substrate is around 13000 counts per second. The circular region contains nonuniform variations in intensity ranging from 15000-25000 cps and corresponds to the Si directly below the Al dot. The profile scan shows an abrupt increase in Si(004) intensity at the edges of the dot, and many minima and maxima across the dot. Although the intensity is highest near the edges, it also remains quite high in the middle. These intensity variations imply that there are many disruptions in strain transfer across this interface, with small “coherent” sections about 60-250μm long. These coherent sections are much larger than the grain size of the aluminum, which is approximately equal to the thickness of the film. The many local intensity maxima indicate that local shear maxima exist within the interior region of the interface, despite the fact that there are no visible surface cracks in the aluminum. Profilometer traces across the dots also showed very uniform thickness, with surface height variations below 10nm. Our diffraction results show clearly that this film has an adhesive interface that is piecewise discontinuous, and cannot be described by macroscopic models for mechanical behavior[11]. Mechanisms for imperfect adhesion might be contaminants at the interface, or stress concentrators such as micro voids or triple grain boundary junctions that would induce semiperiodic local stress-yielding. Contour plots and line profiles for tungsten and copper dots more closely resemble the ideal trace in Fig. 4, and will be reported elsewhere[13].
CONCLUSIONS

We have described the instrumentation and presented specifications for our microbeam diffraction facility, which has been designed for high-resolution measurements at a monochromatic bending-magnet synchrotron beamline. A tapered glass capillary condenses the beam down to a spot size varying from 2 to 20\( \mu \)m. Both the microbeam and instrument are extremely stable. An advantage of the tapered capillary is that it defines the incident diffraction vector, \( \mathbf{q}_i \), extremely well, and \( \mathbf{q}_i \) remains unvarying during an experiment, except for small changes in intensity. The sample positioning is reproducible over a long time period. A grain found in an overnight mesh scan can be located with ease the following day. Although no special mechanical measures have been taken to reduce the sphere-of-confusion, an algorithm has been developed for measurement and compensation of it at different diffraction angles. The endstation can operate in three modes: diffraction-mapping, fluorescence-mapping, or standard diffraction scans (radial and transverse).

To illustrate the capabilities of the instrument, we have described the use of Si(004) strain-contrast imaging to investigate the interfacial strain in metal films deposited on silicon wafers. It was found that evaporated 1\( \mu \)m diameter Al dots on Si(001) wafers are not well described by the Shear-Lag model, and have disrupted adhesion on the scale of microns.

This is only one example of materials and problems of interest to the microelectronics industry that can be investigated effectively with microbeam diffraction. This result, unfortunately, does not show the capability of the instrument for performing diffraction scans at local regions of interest. We have, however, done such measurements on a variety of samples, and usually consider the “topography” mode as simply a tool for finding features on which we home in and take high-resolution radial or rocking-curve scans. Problems we are currently studying include behavior and structure of relaxed SiGe/Si thin films, stress mechanics of W/Si films, stress in thin metallization lines, and stress/strain transfer in Cu, W, and Ni features on Si. Analyses of results are in progress and publications will be forthcoming.

Although we cannot produce sub-micron beam sizes at a bending-magnet source, we have an instrument that is capable of answering a great many questions of interest to the microelectronics industry. With the advent of in-vacuum small-gap undulators at the NSLS, and changes in our microfocusing optics, we will have the capability for building beamlines for sub-micron diffraction that rival those of third-generation synchrotrons.

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