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X-ray Microscopy: An Emerging Technique for Semiconductor Microstructure Characterization

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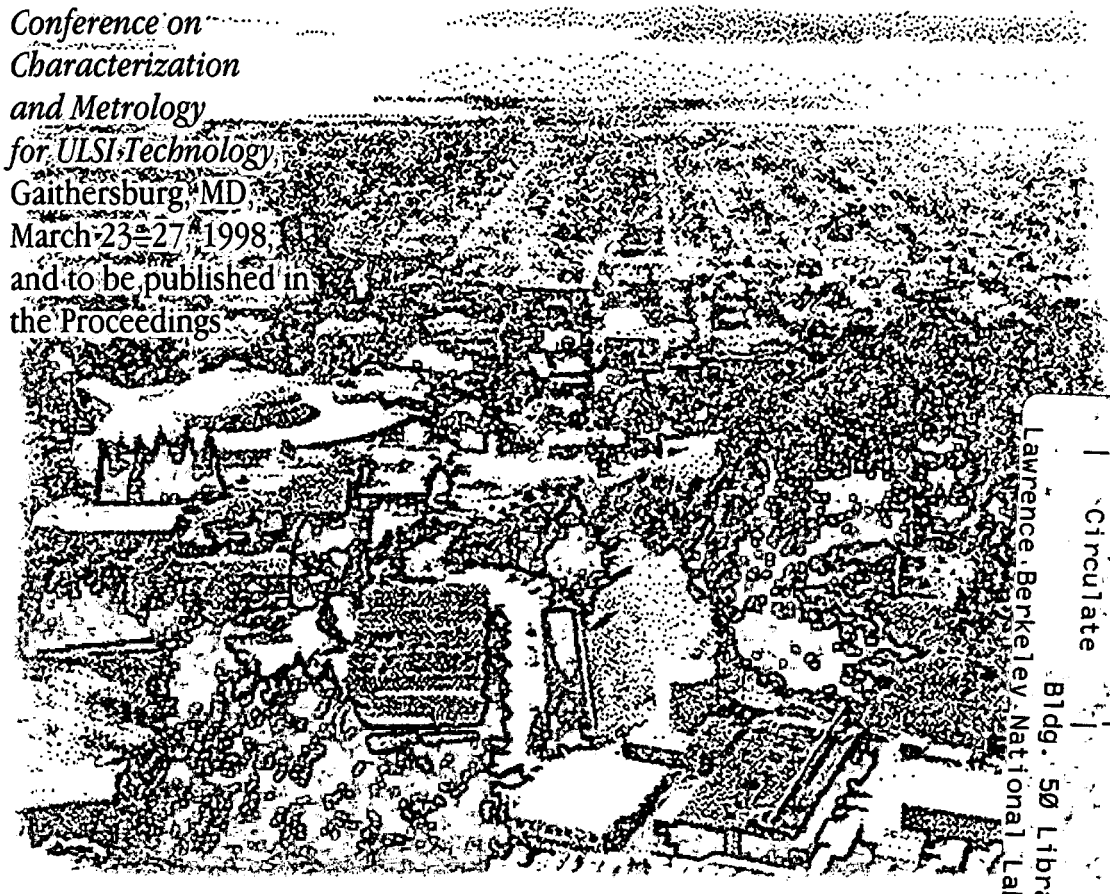

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The advent of third generation synchrotron radiation x-ray sources, such as the Advanced Light Source (ALS) at Berkeley have enabled the practical realization of a wide range of new techniques in which mature chemical or structural probes such as x-ray photoelectron spectroscopy (XPS) and x-ray diffraction are used in conjunction with microfocused x-ray beams. In this paper the characteristics of some of these new microscopes are described, particularly in reference to their applicability to the characterization of semiconductor microstructures.

INTRODUCTION

The latest generation of synchrotron radiation sources is characterized by having extremely high brightness. As the flux in a small focus is directly proportional to brightness, this type of source is optimized for microscopy, and at the ALS we have developed a range of such instruments with spatial resolutions from a few microns to less than 100 nm. For chemical identification and mapping, we have instruments based on traditional laboratory techniques such as infrared vibrational spectroscopy and x-ray photoelectron spectroscopy, as well as the complementary technique of x-ray absorption spectroscopy in which tunable synchrotron radiation has to be used. For structural characterization we have combined traditional single crystal and powder x-ray diffraction with a microfocused probe with micron dimensions. Although most of the systems developed so far can be classified as research tools, a system for micro-focused x-ray photoelectron spectroscopy (μ-XPS) of microstructured wafer samples has been developed in collaboration with Intel Components Research division and Applied Materials. This system has many of the tools found on commercial systems dedicated to the examination of wafers, including optical fiducialization, rapid sample introduction and high precision wafer motion systems. The intent of this paper is to give a brief overview of the capabilities of these systems.

MICROSCOPE CHARACTERISTICS

a) Microfocus XPS

A simple grating monochromator is used to produce tunable radiation from 250 - 1300 eV. The monochromator produces a vertical image of the synchrotron source, and a spherical mirror produces a horizontal stigmatic image at an adjustable pinhole aperture. This aperture of nominal size 20 x 40 μm provides the object for a pair of crossed elliptical mirrors to focus at 20 and 40:1 demagnification to a 1 μm focus. Experimental measurements have verified that a focus of approximately this size is formed. The mirror system is situated close to the sample, inside the measurement chamber, and is fully bakeable. Photoelectrons are energy analyzed using a commercial XPS analyzer (Phi Omni). A particular feature of the design is that the sample can be viewed using a high aperture optical microscope, and this is used to re-fiducialize the sample after transfer so that a coordinate reference system can be re-established. Together with an in-situ optical interferometer and an ultra-stable x-y sample stage, sub-micron absolute positioning and tracking can be achieved. The optical microscope can also read Tencor light scattering detection coordinate files, and move the sample stage to intercept selected particles. The sample transfer system and sample x-y stage are designed to work with sections of wafers up to 50 x 50 mm. The system was developed in collaboration with Intel Components Research Division, and Applied Materials. Technical aspects of the system are described in [1,2] and applications and quantification of performance is reported in [3,4].

All aspects of the optical and x-ray microscopes are computer controlled. The system is used for the identification of surface chemical species, such as those produced after plasma processing, and for particle identification. One of the great advantages of synchrotron radiation over laboratory sources is that the photon energy can be tuned over wide ranges. In this way the cross section of a particular core level can be optimized, and the surface sensitivity can be 'tuned' by variation of the kinetic energy and hence the mean free path of electrons in the solid. For example, the cross section for carbon 1s photoemission increases by approximately a factor of 50 in going from Al K radiation (1486 eV) to 340 eV (approximately 50 eV above threshold). In addition, the kinetic energy of C 1s electrons at this energy would be in the minimum of the mean free path, and so surface features would be emphasized over the bulk. As many of the processes of interest relate to surface properties, for example adhesion, the ability to 'tune in' to the surface through an appropriate choice of photon energy is of key importance. Of equal importance for materials that are easily damaged such as polymers is the ability to increase the cross section of a required core level, and hence minimize the radiation dose. This effect can also be used to reduce the cross section of an unwanted component. One important feature of the μ-XPS system is that it is permanently installed on beamline 7.3.1.2, and dedicated to the characterization of microstructures. The synchrotron source runs 24 hrs/day, 5 days/week, and the monochromatized beam is shared equally between μ-XPS and another photoemission microscope (PEEM). This arrangement ensures that samples can be handled on relatively short notice.

b) Scanning Photo-Electron Microscopy (SPEM)

This system is similar to the μ-XPS apparatus described above, but uses an ultra-bright undulator source rather than a bending magnet radiation source, and uses zone plate focusing. The system is located on beamline 7.0.1 [5]. The detection system is the same as above, but the higher incident flux combined with the superb focusing characteristics of a zone plate allow a much better energy and spatial resolution. Zone plates are simply circular diffraction gratings whose period decreases towards the outer rings. They are made by electron beam lithography and patterned in gold, nickel or other metals depending on the photon energy range. If the zone plate is correctly made, the ultimate resolution is approximately given by the spacing of the outer most zone. In this case for reasons of flux we use a relatively coarse zone plate, and achieve a spatial resolution of around 0.2 μm. For transmission microscopy where lower signals can be tolerated, much finer zone plates can be used, and ones with 20 nm outer zones have been made, and a resolution of 30 nm has been reported. The SPEM produces spatially resolved maps by raster scanning the x-ray beam across a fixed sample, while recording a particular photoelectron peak intensity. This is done by moving the zone plate on a piezo driven stage in areas up to 100 by 100 microns, and allows the sample to have all the normal features of an ultra-high vacuum surface science system, such as sample heating and cooling. For examining larger areas, the sample stage itself can be moved over 20 x 20 mm. The zone plate system can be lowered out of the beam to allow direct observation of the sample using an optical microscope. This allows specific parts of the sample to be examined with reference to optical fiducial marks. Another characteristic of this system is that very high energy resolution can be achieved, better than 0.1 eV over the whole working range of photon energies (200 - 900 eV).

c) Photoemission microscopy (MAXIMUM)

The Multiple Application X-ray Imaging Undulator Microscope (MAXIMUM) is a scanning x-ray microscope like the μ-XPS and SPEM systems previously described, but achieves a microfocus using a Schwarzschild objective to demagnify a pinhole object at 20:1 demagnification [6]. This objective consists of a convex - concave mirror pair, with light entering at approximately normal incidence through a hole in the center of the concave mirror, with subsequent reflections from the convex and concave surfaces. The great advantage of this arrangement is that it can collect a large aperture while maintaining diffraction limited performance. In order to work at soft x-ray energies and so access a significant number of core levels, the optical surfaces have to be coated with a multilayer mirror. This consists of alternating ruthenium and boron carbide layers,

and so the periodicity of this system defines the photon energy. In this case, an energy of 130 eV is used, and so means that the photoemission features seen are highly surface sensitive. This system was originally installed at the Synchrotron Radiation Center in Wisconsin and moved to beamline 12.0.1 in 1995. The 4 orders of magnitude higher brightness of the ALS translates directly into detected signal, and so the system can now work at its optimum performance. It achieves <100 nm spatial resolution, and has a total energy resolution of around 0.2 eV. The system is fully UHV compatible and has been used for a wide range of experimental studies on semiconductor materials [7,8, 9].

d) Photo-Emission Electron Microscopy (PEEM)

Photoemission electron microscopy using x-ray excitation (X-PEEM) combines two established techniques, x-ray absorption spectroscopy (XAS) and full field electron imaging to give a powerful tool for high-resolution chemical characterization of surfaces. In an X-PEEM system, monochromatic tunable radiation is condensed to illuminate a small field of view at the sample, typically 30 μm in diameter. Electrons photoemitted from this area are first accelerated in a high field region between the sample and the front of the first electron lens, and then subsequently imaged by an objective and several projector lenses at high magnification. The final image is then recorded on a phosphor screen - CCD system. This type of system has been used for many years using ultra-violet excitation, and by the use of appropriate filters, excellent work function contrast can be obtained. Such systems have achieved < 10 nm resolution (10). PEEM can also be combined with tunable synchrotron radiation however to give spectroscopic information [11]. If the energy of a photon beam is increased through the region of an x-ray absorption edge, the total yield of electrons from the sample will usually exhibit a series of sharp features within 10 eV of the edge. The Near Edge X-ray Absorption Fine Structure (NEXAFS) contains information on elemental composition, chemical bonding, bond orientation as well as magnetic state, and can be used as a unique fingerprinting tool. In order to record spectroscopic information with a PEEM, images are recorded at increments of increasing energy through an absorption edge. The integrated intensity in areas that show some feature of interest can be obtained by summing the intensities in individual pixels, and this intensity can then be plotted out through the series of images. The local NEXAFS spectrum can therefore be obtained. Because of the parallel nature of full field imaging, this type of microscopy can be fast, with whole volume data sets (a succession of images at increasing photon energy) recorded in a few minutes. Penetration depths for soft x-rays in solids are much greater than the range of low energy electrons, and this means that most primary photoelectrons are scattered to lower energies by the time they emerge from the surface. The energy spectrum imaged by the PEEM is therefore centered at a few eV and is around 10 eV wide. This combined with the chromatic nature of electron lenses gives a lower resolution limit of around 20 nm for a microscope operating at 10 KV/mm extraction potential. The mean free path of low energy electrons in solids is typically 2 - 5 nm and this sets the effective probing depth of the technique. Further advances in electron optics in which a chromatic aberration compensation system is used is expected to reduce the resolution limit to around 2 nm [12].

The ALS has two PEEM systems. The first PRISM [13] consists of an electrostatic objective lens typically working at an extraction potential of 10 KV, a movable aperture in the back focal plane, a projector lens and a channel plate-phosphor-CCD imaging system. The system has a spatial resolution of around 200 nm and is located on undulator beamline 8.0. This beamline is shared between several end stations, and gives a photon energy range of 70 - 1300 eV. A dedicated system, PEEM2, has been built on bending magnet beamline 7.3.1.1 [14]. This beamline was specifically optimized for PEEM for magnetic materials, and covers an energy range of 250 - 1300 eV [15]. The microscope operates at 30 KV, and has in addition to PRISM a stigmator-deflector at the objective back focal plane, an additional projector lens, and a set of adjustable back focal plane apertures. The system is currently being commissioned and is expected to give a resolution of 20 nm. The system is equipped with in-situ evaporation, and a sample preparation facility.

(e) Infrared microscopy

Synchrotron-based infrared (IR) beamlines provide a brightness advantages over conventional IR sources of 100 - 1000 in the near to mid IR region. This means a similar factor in intensity advantage for microscopy. At the ALS, bending magnet beamline 1.4 is dedicated to IR studies, and is equipped with a commercial IR interferometer and microscope for the 2 - 30 μm wavelength range [16]. The beamline extracts 40 x 10 mrads of light from the storage ring, passes it through a diamond isolation window, and uses parabolic collimating optics to produce a parallel beam for the interferometer and microscope. The 2 - 30 μm range is commonly used for the chemical fingerprinting due to the presence of many vibrational bands in this region. The system is being commissioned, but has so far been used to study a huge diversity of samples, from organic particles to bacteria used in bio-remediation. The whole system is located in a semi-clean room environment.

(f) Micro-diffraction

This system was originally developed to apply to the problems of thin film strain, particularly with reference to electromigration in integrated circuit interconnect structures. Although this problem has been widely studied using electron microscopy [17], and with focused x-ray probes [18], an understanding of stress at the spatial scale of single grains in Al-Cu lines is still lacking. The apparatus we have built for this task consists of a pair of elliptically bent mirrors to produce a 'white beam' micro-focus of 4 - 13 keV x-rays [19], a 4 crystal monochromator to monochromatize the incoming beam, a sample scanning stage to manipulate specific parts of a structure into the x-ray beam, and an x-ray CCD camera to record the diffraction patterns. A unique feature of the system is that the monochromator has co-linear input and output beams, and so by rotating to zero angle, the white x-ray beam can be directly focused on the sample. Rotating the crystals into the beam to provide monochromatic light causes no apparent beam motion at the micron level. White beam mode is used to record Laue diffraction patterns, from which we extract orientation information. Knowing orientation and the index of each diffracted beam, we also know the energy of each beam in the case of an un-strained grain. To measure strain then simply means that we have to insert the 4 crystal monochromator and measure the energy of a set of reflections. The energy differences to the un-strained case directly give strain information. We have obtained 0.8 x 1.5 μm resolution, and measured single grain orientation and strain in Al-Cu lines at this resolution. The ultimate strain resolution is simply set by the precision with which we can measure the energy of reflections, and this should be near 10^{-5}. Initial results on this system are reported in this conference [20]. A dedicated beamline for micro-diffraction, 7.3.3, has now been constructed and is undergoing commissioning. Commissioning of the end station will start in November this year following delivery of a new CCD x-ray detector and goniometer system [21].

(g) Micro-X-ray absorption spectroscopy

The system described above for micro-diffraction can also be used for micro-x-ray absorption spectroscopy (μ-XAS) in the 4 - 13 keV energy range. Using white beam, very rapid identification of the elements of interest can be made using fluorescence detection. Once these areas have been found, the monochromator can be scanned through an elemental absorption edge, and the NEXAFS features near the edge can be used as a unique chemical fingerprint. Most of the initial work using this technique has centered on analysis of environmental samples, one such example being the speciation of Zn as an oxalate form in a fungus [22]. It has also been employed to speciate the chemical form of Fe in an inclusion in solar cell silicon [23]. The ability to perform high spatial resolution chemical speciation in air, with samples in their native state make this technique particularly powerful.

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The work reported here represents that of many people working at the ALS and in university groups in the field of microscopy using x-rays.

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