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W. Ng, R.C.C. Perera, and J.H. Underwood

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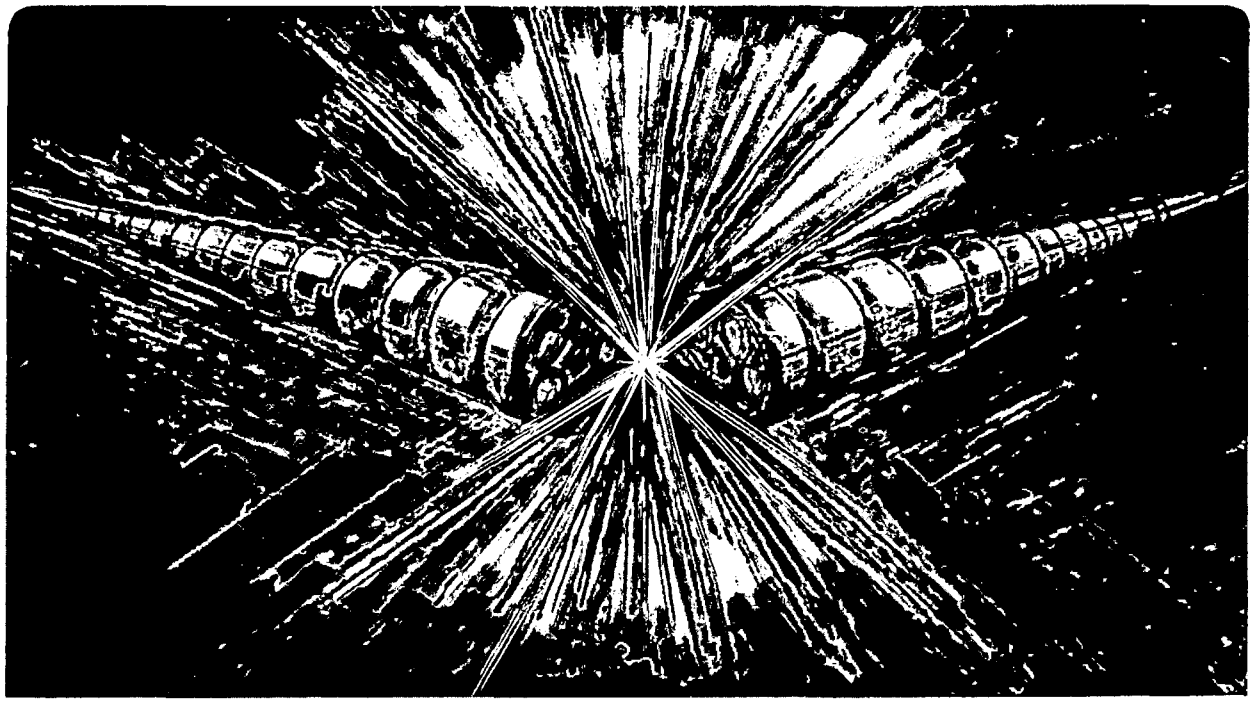

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INSTALLATION OF THE MAXIMUM MICROSCOPE AT THE ALS*

W. Ng, R.C.C. Perera, J.H. Underwood

Advanced Light Source
Accelerator and Fusion Research Division
Lawrence Berkeley National Laboratory
Berkeley, CA 94720, USA

Center for X-ray Optics
Materials Sciences Division
Lawrence Berkeley National Laboratory
Berkeley, CA 94720, USA

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Installation of the MAXIMUM Microscope at the ALS

W. Ng, R.C.C. Perera, J.H. Underwood
Lawrence Berkeley National Laboratory, Berkeley, CA 94720

S. Singh, H. Solak, and F. Cerrina
Center for X-ray Lithography, University of Wisconsin - Madison
Stoughton, WI, 53589

Abstract

The MAXIMUM scanning x-ray microscope, developed at the Synchrotron Radiation Center (SRC) at the University of Wisconsin - Madison was implemented on the Advanced Light Source in August of 1995. The microscope's initial operation at SRC successfully demonstrated the use of multilayer coated Schwarzschild objective for focusing 130 eV x-rays to a spot size of better than 0.1 micron with an electron energy resolution of 250meV. The performance of the microscope was severely limited, because of the relatively low brightness of SRC, which limits the available flux at the focus of the microscope. The high brightness of the ALS is expected to increase the usable flux at the sample by a factor of 1000. We will report on the installation of the microscope on bending magnet beamline 6.3.2 at the ALS and the initial measurement of optical performance on the new source, and preliminary experiments with surface chemistry of HF etched Si will be described.

1. Introduction

The photoemission microscope MAXIMUM was designed and built by the University of Wisconsin and it was installed at the Synchrotron Radiation Center[1]. The instrument was developed in three phases, which started in 1988 and spanned a period of 6 years. We have successfully demonstrated the use of reflective optics for focusing soft-X-rays near 100 eV to a spot size of 95 nm, and we also show the possibility of spectromicroscopy at this resolution with high energy resolution. Over the last 2 years, the microscope was used to study a range of surface physics problems of both fundamental and applied interests[2,3,4]. During the operation of the microscope, it became apparent that the microscope's performance is severely hampered by the relatively low brightness of Aladdin, which limits the available flux at the microscope's focus, and

this in turn limits the achievable spatial resolution because of signal-to-noise considerations. The MAXIMUM experiments performed at SRC were often forced to operate at a reduced resolution, in order to achieved realistic counting rates. The development of third generation light sources like the Advance Light Source (ALS) and Elettra (Trieste) with their increased brightness has provided the possibility of tremendous improvement in achievable resolution. A Participating Research Team (PRT) was formed to move MAXIMUM from Wisconsin for installation and operation at ALS. The microscope was moved to the ALS in April of 1995. It was installed on bend magnet beamline 6.3.2 and preliminary tests and experiments were performed at the ALS. We will discuss the installation and characterization of the microscope, and we will report on the first results from this test experiment. We will also discuss plans for moving to an undulator source and the expected performance.

1.1 Microscope Design

The detailed design and implementation of the microscope have been published previously[1], and it will be briefly reviewed here. The basic layout of the microscope is as follows: radiation from the synchrotron source is monochromatized and focused to illuminate a pinhole, which serves as the source for the microscope optics. A multilayer-coated Schwarzschild Objective (S.O.) produces an image of the pinhole with a 20X demagnification. When a sample is placed at the focus, photoelectrons emitted from the irradiation are collected by an electron spectrometer. Information regarding the band structure of the irradiated area can be obtained from the Energy Distribution Curves (EDC). The sample is rastered to produce a 2-D image, and by using different parts of the EDC as the input signal, different contrast mechanisms can be produced. One of the most important strength of photoemission microscopy is the large number of well-understood contrast mechanisms that can be used for imaging. The contrast mechanisms that we have explored are: a) Topography, b) Elemental distribution, c) Chemical shifts, d) Band bending and Fermi level, and e) Valence band lineup. f) Electrostatics.

The installation of MAXIMUM at the ALS will have two phases. We first moved the existing microscope, the electron energy analyzer and the sample preparation chamber (a fully integrated system) to the ALS for installation on BL 6.3.2. In the second phase, the microscope will be installed on undulator beamline BL 12.0, and we will also extend the performances of the microscope by upgrading the existing optics and the electron energy analyzer. The spatial resolution is expected to reach below 500Å with high count rate. We will report on the progress of the first phase of the installation.

2. Installation at BL 6.3.2 at the ALS

MAXIMUM was disassembled and shipped from SRC in May of 1995. Modification to the support structures was necessary due to seismic requirements at ALS. The upgrade involved the replacement of the old kinematicly mounted support by a standard six struts support system. The sample transfer system was also rebuilt with a six struts support system. The upgrade was completed in August of 1995. The microscope was installed on BL 6.3.2 at the ALS for preliminary testing. BL 6.3.2 is a bending magnet beamline, operating in the range of 50-1000eV. This beamline was designed to be used principally for calibration and characterization of x-ray optical components at the ALS. The bend magnet source and the beamline's optical characteristic did not fill the pinhole and the microscope's objective effectively. Nevertheless, it was adequate for preliminary testing of the microscope. The main objective of this experimental run was to characterize the reassembled microscope with its new support stand and sample transfer system. Preliminary experiments were also carried out at a photon energy of 129.3 eV to test and characterize the performance of MAXIMUM at ALS Beamline 6.3.2. The measured flux at the microscope focus was $2x10^8$ phs/sec. The measured flux was 50X less than expected; this discrepancy was caused by the large figure errors in the final refocussing mirror of the bemline, which produced multiple vertical images at the pinhole. For these preliminary experiments, we chose a pinhole which operates at a modest spatial resolution of 1.25μm. After the microscope was characterized, we used MAXIMUM's ability to image local differences in surface chemistry of a sample, in this case a Si wafer patterned with 92 nm thick silicon

nitride features[1]. This experiment is part of an ongoing project with the microscope. Fig. 1 shows a partial yield image of the sample. The image is 80 μm x 80 μm and it was acquired with a step size of 1 μm at a photoelectron energy of 0 eV. The bright raised square corresponds to the nitride feature and the surrounding area corresponds to the silicon substrate. EDCs taken on Si and SiNH regions of the sample show a dramatic chemical difference. These are shown in Fig.2 and Fig. 3, respectively. The sample was subjected to a light chemical cleaning by immersion in 1% HF for 4 min. followed by a 2 min. rinse with deionized H_2O. It was then put into the experimental chamber and pumped down within 15 minutes. When a pressure of 1 x 10^{-7} torr was reached (after 1 hour), the focused x-ray spot was positioned at the corner of a SiNH feature on the Si substrate. A cylindrical mirror analyzer (CMA) with a workfunction of 5 eV was used to acquire EDC's at both locations. An EDC acquired on the Si region is shown in Fig. 2 after background removal and curve fitting with Gaussian functions. Gaussian functions were used since instrumental broadening is expected to dominate in our spectra. Fig.3 shows the EDC taken on the SiNH region. In both EDC's Peak A corresponds to the 2p core level of Si. The $2p_{3/2}$ and $2p_{1/2}$ components were not resolved; due to the modest energy resolution chosen, the peak is found to be centered between the two at a kinetic energy of 24.8 eV (binding energy 99.5 eV). Fig.2 shows that two other peaks, B_1 at 22.0 eV and C at 21.1 eV, are found in addition to A_1 on the chemically cleaned Si. Notwithstanding the noise in the data and the difficulty in obtaining a perfect fit, these peaks can be assigned, tentatively, to the Si^{+3} and Si^{+4} oxidation states of Si, since 0.8 eV - 0.9 eV of chemical shift can be attributed to each bond a Si atom has with an oxygen atom, with the shift for SiO_2 ranging from 3.6 eV to 4.4 eV[5]. The SiO_2 peak is also broadened as expected. The intensity of the clean Si is lower than that of the oxide peaks since the chemical clean was not sufficiently thorough. It is well known that the oxidation of HF etched Si not only proceeds more slowly than that of annealed Si but also results in the preferential formation of the lower oxidation state oxides SiO_2, SiO, and Si_2O_3 over SiO_2[6,7]. The lower oxidation state oxides form first also on annealed Si[8]. This is consistent with the lowered rate of oxidation of HF-cleaned Si. The presence of SiO_2 on our sample can partially be explained by the incomplete removal of the native oxide present on the

[1] Stoichiometrically, silicon nitride can be represented by Si_3N_4. However, the silicon nitride formed in practice does not conform to this formulation. The nitride is often also in a hydrogenated state, regardless of the method of formation. In this paper the expression SiNH will be used to refer to silicon nitride

Si wafer by the light HF etch used. A stronger HF etch for 20-30 minutes would have been more effective. Cleaner equipment and purer solutions are also essential in avoiding contamination of the wafer during cleaning[9]. However, the fact that the SiO_2 chemical shift is at the lower end of the range at -3.6 eV rather than the -4.0 eV typical of the native oxide, may indicate that strain at the Si/SiO_2 interface may be lowering Si-O bonding energies[7,10]. In this case, incomplete removal of the native oxide is not implicated. Fig.3 shows the two shifted peaks B_2 and D in addition to the clean Si peak A_2 on the EDC taken on the SiNH feature. Peak D at 19.3 eV is not present in the EDC acquired on the Si substrate and has been observed on SiNH in past experiments on SiNH as well. It can be assigned to SiNH although the nominal chemical shift for stoichiometric SiNH is only 2.7eV[5] because of local charging of the almost 0.1μm thick nitride layer and the fact that the exact chemical composition of the SiNH in this sample is not known. Peak B_2 at 21.7eV may arise from Si_2O_3, although its chemical shift of -3.0 eV is 0.2 eV off the expected shift of Si_2O_3 on Si. This oxide of silicon may have resulted from a contamination layer formed on the surface of the SiNH during ion-implantation of the sample after nitride patterning which was not completely removed during the chemical cleaning, which is designed for Si, not SiNH. Another evidence that this is indeed the case is that Peak B_2 was not present on SiNH in an experiment on an identical sample cleaned by heating to 1200°C in UHV, which results in the removal of material from the entire surface of the sample. In the current experiment, both peaks are widened as expected for chemically shifted components. The fraction of signal from clean Si is expectedly lower for a SiNH sample, but the fraction of SiNH signal is lower than that of the silicon oxide, because of the incomplete chemical clean.

3. Discussions

The first test of the MAXIMUM microscope at the ALS was successful. The performance of the microscope was close to the expected level for a bending magnet beamline. We expect to enjoy the improved signal very soon at the ALS, when the mirrors in the refocussing mirror box of BL6.3.2 are replaced with lower figure error mirrors and the flux previously lost to the multiple vertical images is regained. The full potential of the MAXIMUM microscope operating at close to the diffraction limit will be realized when it is moved to the undulator beamline BL 12.0. The preliminary experiments on chemical

etching of the Si/SiNH system demonstrated the feasibility of such experiments. Clearly, more detailed studies need to be done to better understand and resolve some of the issues in this complex system. HF etched Si is a very important system for technological applications in the IC industry and exhibits interesting physics as well. We plan further higher resolution experiments on various chemically cleaned H-terminated samples. With improved sample preparation, we expect to conduct spectromicroscopy of stable, nominally unpinned Si (100) and (111) with core level spectra containing varying ratios of surface shifted components to study local variations in the surface chemistry of chemically cleaned Si.

Acknowledgments

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Figure Captions

Fig. 1 Secondary electron 80 μm x 80 μm image of a pad of SiNH on Si. The raised square is a SiNH feature and the surrounding area is the Si substrate.

Fig. 2 EDC of the Si 2p acquired on the Si area of the sample depicted above. The peaks A_1, B_1, and C correspond, respectively, to clean Si, Si_2O_3, and SiO2.

Fig. 3 EDC of the Si 2p acquired on the SiNH feature on the sample depicted above. Peaks A_2, B_2, and D are assigned to clean Si, Si_2O_3 (tentatively), and SiNH, respectively.

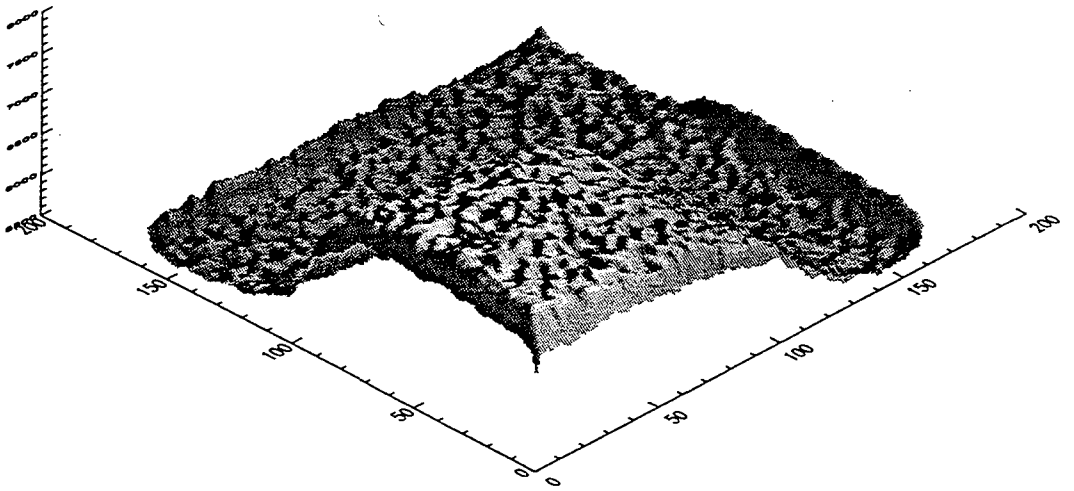

Fig.1

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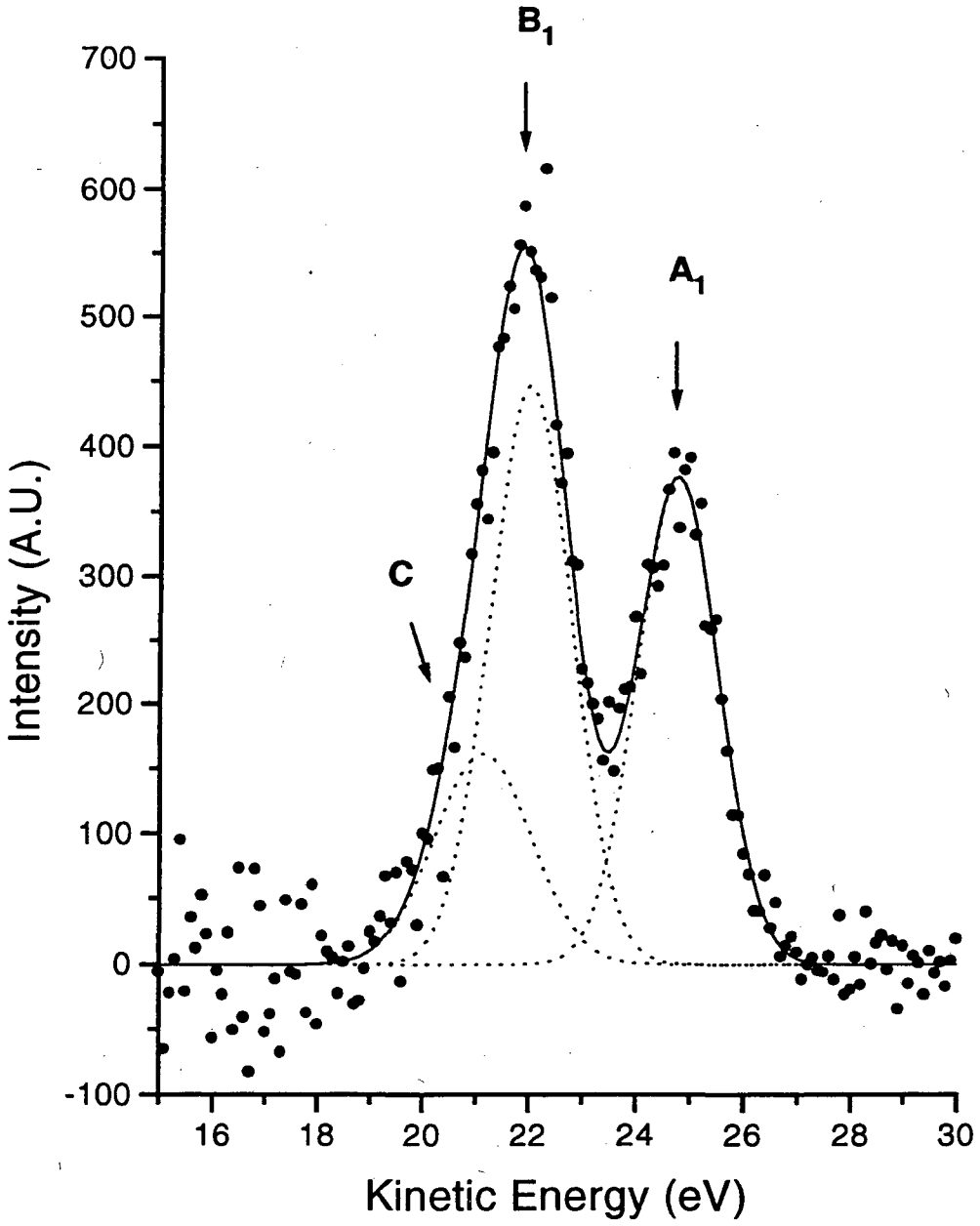

Fig.2

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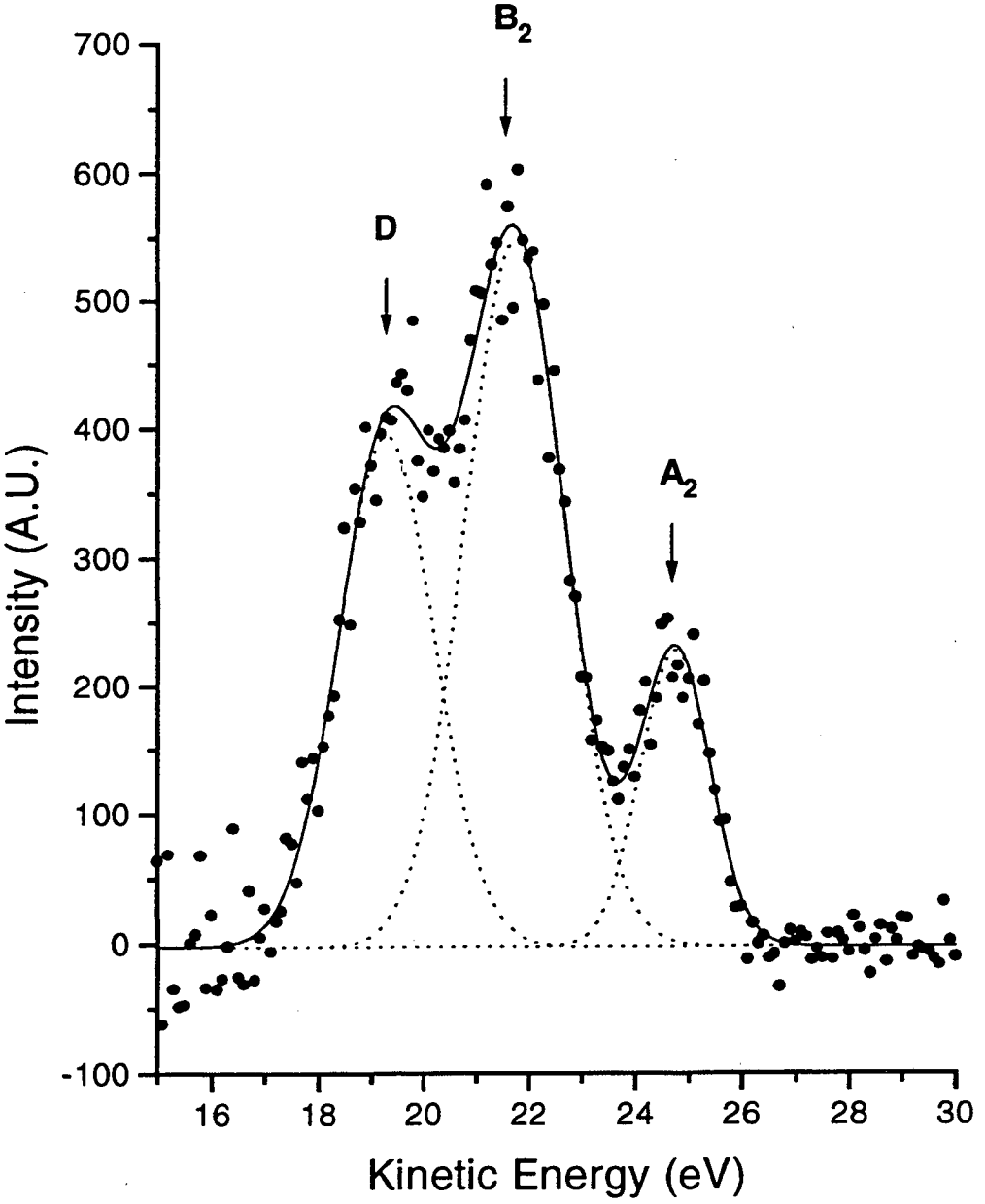

Fig.3

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