A video data-acquisition system for quantitative low-energy electron diffraction studies

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We have developed an easy to use and inexpensive low-energy electron diffractometer. The system is based on an Apple Macintosh II microcomputer and uses a high-resolution CCD video camera. The video interface is a single plug-in imaging board which digitizes the video signal in real time. No expensive auxiliary video processing devices are required. The system is of moderately high speed. A typical set of 16 IV curves with 125 data points in each curve can be generated from images summed over 16 frames in less than 4 min. Spot profile measurements are also routinely made. A description of the apparatus and the capabilities of the system are presented, illustrated using measurements of the epitaxial growth of bismuth films on III-V semiconductor surfaces.

INTRODUCTION

The measurement of low-energy electron diffraction (LEED) is one of the oldest experimental methods used in surface science. The LEED technique can be used to examine the symmetry of the surface unit cell, the surface atomic geometry, and the kinetics of surface adsorption and epitaxy.[1] Unfortunately, to study a surface beyond the determination of its symmetry has required laborious effort. Due to the continual progress made in experimental instrumentation and associated theoretical analysis methods, however, *quantitative* LEED studies are now becoming more numerous.

LEED information is obtained by measuring the intensity of the diffraction beams, the line shape of the diffraction beam, and, most recently, the background of the incoherently scattered electrons.[2,3] The most commonly used methods for measuring LEED intensities in the past have been the Faraday cup[4] and the optical spot photometer.[5] The Faraday cup is a movable electron collector that is mechanically positioned at the point in space where the intensity of the diffraction is to be measured; the spot photometer measures the brightness of light on a phosphor screen placed inside the LEED apparatus. The disadvantages of these techniques are that the data-collection times are long, usually several hours, and the mechanical tracking of the diffraction spots is tedious, prohibiting the routine collection of large amounts of data. Extended measurement times becomes a serious problem in systems where, e.g., adsorbates can decompose or desorb under prolonged bombardment of the incident electron beam.[6]

Since mid 1970s, new methods have been developed that decrease the time and effort required to perform quantitative LEED studies.[7-9] Perhaps, the most popular approaches rely on video technology, employing a video camera and computer image analysis processing. Although the data collection time can be reduced to mere minutes, the hardware for this approach has until recently been too expensive for most research groups.

In this article, we describe our development of an inexpensive video LEED system based on a modern personal computer and software system. It has considerable functionality despite its low cost and simplicity.

I. EXPERIMENTAL APPARATUS

Figure 1 is a schematic diagram of the basic units of the video LEED diffractometer we have developed for studies of epitaxially grown semiconductor surfaces. Although one has the choice of several manufacturers of LEED optics, we selected the Princeton Research Instruments Reverse View LEED unit (model RVL 10-120) with electronics from Physical Electronics Inc. The LEED optics has four nested hemispherical grids and a hemispherical glass collector screen coated with a transparent oxide coating and a layer of

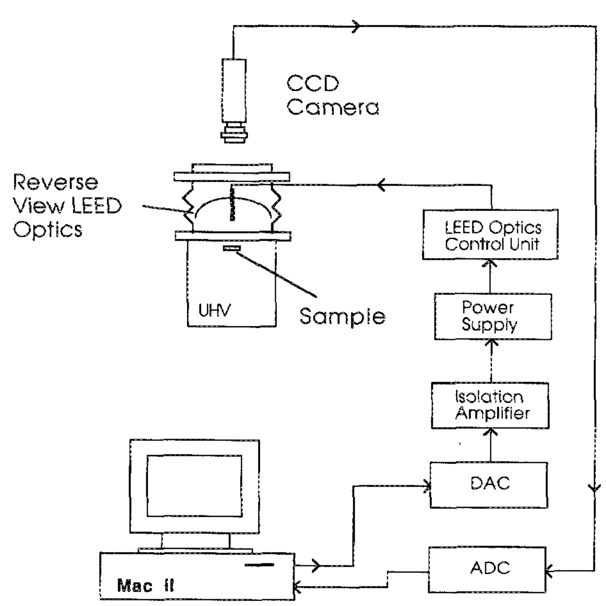

FIG. 1. Schematic diagram of the LEED experimental system.

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phosphorescent material. This reverse viewing instrument was selected, in part, to eliminate any unwanted visual interference from the sample holder. In our ultrahigh-vacuum experimental chamber, the optics are mounted vertically above the nominally horizontal sample. This orientation was selected to be compatible with our Perkin Elmer molecular-beam epitaxy growth system to which the LEED system will be attached in the future. The LEED optics are supported on an 8-in. formed bellows by three differential screws. When the sample is placed near the center of curvature of the hemispherical grids the orientation of the integral electron gun with respect to the sample surface normal can be finely adjusted by tilting the optics assembly supported on the bellows using the differential screws. The present sample holder is equipped with a liquid-nitrogen cooling system, capable of lowering the sample temperature to less than 130 K. The samples can also be heated in excess of 500 °C using a button heater placed within the holder.

The LEED optics are used to perform Auger measurements via the retarding field technique.[10] A modulation voltage of ~ 8 V peak to peak and an electron-beam current of approximately 10 μA are typically used. The modulation is coupled to the sample through a transformer, and a ramping retarding voltage is applied to the inner two grids of the LEED optics. A lock-in amplifier is used to detect the Auger current. The computer system drives the ramp voltage, and measures and plots the Auger signal. Without the use of any additional external filters,[10] we typically obtain a resolution of 8 eV, adequate for adlayer coverage studies and contamination checks.

The computer system controls the electron gun energy. A variable electron gun voltage is provided by a Kepco operational power supply over a range of 0–500 V which has an accuracy better than 0.2 V. This voltage is fed into the Physical Electronics LEED controller. The Kepco power supply has a slewing rate greater than 1 V per microsecond and a programming time constant less than 8 ms. During an energy ramp, a delay of 17 ms is automatically inserted by the software at every voltage step to ensure that the power supply output is stabilized.

A high-resolution CCD camera manufactured by Pulnix (model TM-840N) is used to collect the LEED images. An $f/0.85$ lens is used. The camera has a $\frac{2}{3}$-in. high-resolution CCD solid-state imager with 800×490 pixels. It operates either in the normal, direct read-out mode or in an integrating mode. In the integrating mode the camera inhibits the read out of the CCD for an integral number of complete video frames. During this time, the CCD continues to accumulate an optical signal until it is read out by the video circuitry. This is useful for low-light situations. The camera video signal is the commercial standard 1-$V_{p.p.}$ video signal with a 50-dB signal-to-noise ratio that provides one frame, or complete picture, every $\frac{1}{30}$ s. The camera could be directly connected to a monitor or a recording device. In our system it is fed directly into the computer hardware.

The experimental interface and control system is based on an Apple Macintosh II microcomputer. The computer has 5 Mbytes of RAM, 15.6672-MHz MC68020 processor with a MC68881 numerical coprocessor, color monitor, and 40 Mbytes of hard disk storage. Additional computer storage is provided by an Apple Tape Backup 40SC. The computer's internal NuBus expansion slots offer high-speed access to the required experimental interface cards. All analog I/O is performed using a MacADIOS II card.[11] This interface hardware provides an analog-conversion resolution of 12 bits for the high-voltage ramp. An external isolation amplifier is also used to buffer the computer from the high-voltage supply.

The second interface card is a Data Translation QuickCapture frame grabber.[12] It consists of an 8-bit video A/D converter with a 12.5-MHz conversion rate. The on-board 512 kbyte frame-store memory is integrated into the memory map of the Mac II expansion slot space and can be accessed at any time by using standard memory instructions. A frame is digitized and available in the on-board memory in $\frac{1}{30}$ s, essentially real time. Because image summation is achieved in software, no additional arithmetic logic unit is used, in contrast to other approaches.[9]

II. DATA ACQUISITION

An easy-to-use, powerful, yet versatile data-acquisition and data-analysis software package has been developed. The program, called MacLEED, is written in Macintosh Programmer's Workshop C and utilizes the standard Macintosh User Interface Toolbox routines. The pull-down-menu user interface provides a pleasing, easily learned operating environment. The digitized video images are displayed in a separate software window using a 7-bit grey scale on the computer color monitor. Using the standard implementation of the hardware, the computer is fast enough to transfer the digitized picture from the frame grabber memory to the video memory every eighth frame, yielding a choppy, yet satisfactory real-time representation of the LEED data. Hence, the computer's speed renders the use of an additional video monitor optional.

The software allows for three types of data acquisition. First, in normal operation the video signal is displayed continuously to a portion of the computer monitor to assist in adjusting such experimental parameters as the electron gun focus or the beam current. Snapshots of the diffraction pattern can be taken and the diffraction spot intensity evaluated on-screen to assist, for example, in the alignment of the sample and incident electron beam or in preparing for a current-versus-energy measurement.

Second, complete screen images can be accumulated by summing the video signal over a specified number of frames. The integrated image is stored in a 16-bit internal frame buffer data structure. Several image processing options are provided to allow the image to be displayed using the 256 colors or grey levels of the standard Mac II display. Plots of the image intensity for user selected regions and projected along arbitrary directions in the plane can then be plotted on the screen. This is useful, for example, when measuring beam profile widths. The angular resolution of the profile depends on the choice of lens magnification. In our standard configuration each pixel on the screen corresponds to 0.25°. The integrated images can be saved to disk in a raw format, re-

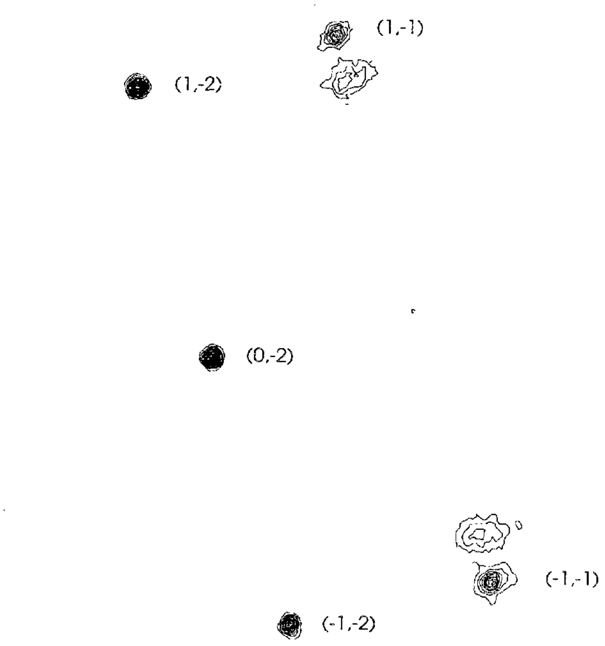

FIG. 2. LEED profiles at 130 eV for a 1-ML bismuth film on GaAs(110) at 150 K. The numbers indicate the diffraction indices of the integral order beams.

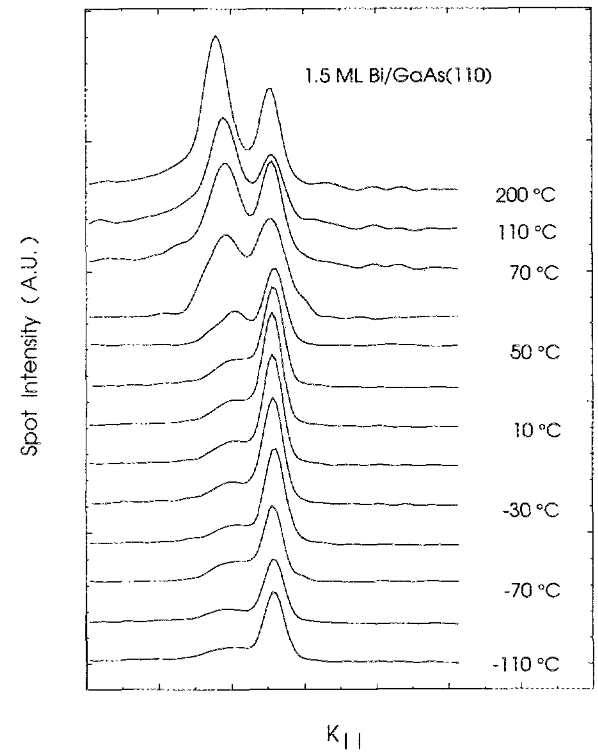

FIG. 4. LEED profiles of the $(1\bar{1})$ diffraction beam and the sixth-order spot along the long, or h, direction as a function of annealing temperature. The sample is a 1.5-ML bismuth film deposited at −110 °C.

quiring approximately 500 kbytes, or in a normalized 8-bit format, requiring only 250 kbytes. Due to these large storage requirements, an off-line tape unit is used to store a previous day's data for future analysis. The image files can be read directly into other software programs for additional rendering.

Important information can be obtained from a semiquantitative analysis of the screen images. By way of example, we display in Fig. 2 a contour plot that has been created using Image Tool[13] from a measured diffraction image. The image represents the diffraction of electrons at 130 eV from a surface having one monolayer of bismuth deposited on GaAs(110).[14] A (1×1) reciprocal space surface net that matches that of the underlying substrate is the primary feature. The contour map also clearly indicates the existence of additional diffraction spots. The addtional spots are sixth-order spots arising due to the formation of a (6×1) bismuth superlattice on the GaAs(110) surface. The spots are broader in the k direction than the integral order spots indicating more disorder in the superlattice in that direction. This direction is the [001] direction of the substrate crystal and is transverse to the zigzag chains that form on the substrate.[15] A thermal anneal of the as-deposited film can be expected to improve the long-range order of the surface. We demonstrate the annealing effect of temperature on the as-grown film in Fig. 3, where a contour plot of the (01) beam from a 0.7-ML film is displayed before (a) and after (b) an annealing cycle. Panel (a) corresponds to diffraction from a bismuth overlayer prepared without subsequent annealing. The spot is somewhat diffuse. Panel (b) shows the same diffraction spot after heating to 200 °C. The sharper, more compact spot indicates an improved surface ordering. The elongation of the spot is consistent with the picture that the two-dimensional bismuth overlayer forms islands at submonolayer coverages that exhibit greater order transverse to the zigzag chain direction.[14] Annealing also affects the character of the sixth-order spots. This is demonstrated in Fig. 4. In this figure the integral $(\bar{1}\bar{1})$ spot and neighboring sixth-order spot are profiled using MacLEED along the h direction of reciprocal space as a function of annealing temperature. The 1.5-ML bismuth film was initially deposited at low temperature and then annealed to the temperatures indicated.[16] Clearly, the anneal promotes an improved order in the superlattice, transforming the sixth-order spot from a weak shoulder to a clearly distinguished peak that is actually more intense than the corresponding integral order spot.

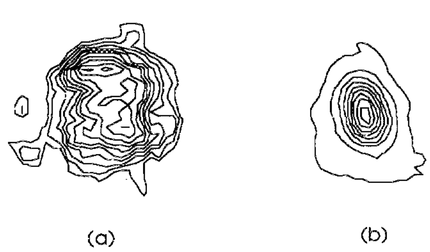

FIG. 3. Effect of annealing to 200 °C on the (01) diffraction beam at 89 eV from a 0.7-ML bismuth film on GaAs(110).

In the third mode of data collection, the diffraction beam intensities can be measured as a function of beam energy to form what are called IV curves. The MacLEED system is able to collect IV data in a relatively short period of time, thereby minimizing the risk of electron-beam damage. A single IV curve consisting of 125 data points can be measured from images integrated over 16 frames in approximately 140 s, while a set of 16 beams can be measured in approximately 230 s.

The procedure of measuring an IV curve can be summarized as follows: (1) The trajectories of selected diffraction spots are determined, (2) a desired number of frames are accumulated, (3) the positions of the spots on the screen are determined, (4) the background intensities are subtracted, and (5) the integrated intensities are measured. The beam energy is then incremented by one step, typically 2 eV, and steps (2)–(5) are repeated.

To determine the diffraction spot trajectories in a normal incidence geometry, a set of measurement windows are drawn on the screen around the selected diffraction spots at two different beam energies. The size of each window can be adjusted by the experimenter to best match the size of the spots in relation to the size of reciprocal space unit cell of the LEED pattern. A set of straight line trajectories are then computed internally that logically link the pairs of windows at the two energies. During the measurement, the positions of the spots along these trajectories at each beam energy are estimated using the Bragg diffraction condition. To refine this estimate, MacLEED searches for a local maximum in the region and centers the measurement window on the maximum automatically. The search is limited to the immediate vicinity of the initial estimate. This procedure best positions the measurement window around a diffraction spot and is well behaved at energies where the beam intensity vanishes.

The intensities are calculated from the measurement windows after the video signal is integrated over a given number of frames, usually 16. However, to reduce IV data-acquisition time, the images are integrated differently than when collecting an entire image as described above. At any given beam energy during the energy scan, a LEED pattern is digitized by the image grabber and stored into its on-board memory. MacLEED only copies to the program memory a portion of the image defined by the measurement windows. For example, if a 20×20 pixel measurement window is selected, a 40×40 memory block is copied to the main memory. The larger size memory block is necessary to accommodate the search algorithm. Therefore, for a typical IV scan containing 14 beams, only a total of $40\times40\times14=22$ kbytes are being transmitted through NuBus to the main memory, instead of the entire 512 kbytes. At each energy this process is repeated to accumulate spot intensities over several video frames. Because displaying a video image on the screen takes $\sim$267 ms, the computer screen showing the LEED pattern and moving measurement windows is updated only once at every energy while spot intensities are being summed.

Once the position of a spot is located, MacLEED computes the average intensity of the perimeter of the measurement window. This value is used as a measure of the background intensity due to incoherent scattering. It is subtracted from the integrated intensity for that measurement window. The result constitutes one IV data point. Several other background subtraction methods were tried yielding essentially equivalent results.

Finally, the beam current is measured once for each run and is used to normalize the raw IV curves. This is necessary

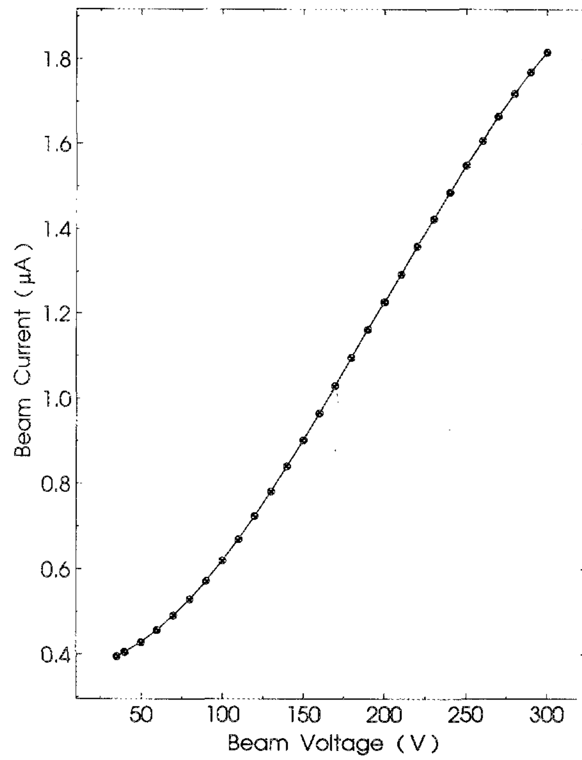

FIG. 5. Measured nonlinearity of the electron-beam current for the Perkin-Elmer/Princeton Research Instruments LEED electron gun and controller.

because the Perkin-Elmer electron gun controller is unable to regulate the beam current. A typical beam current versus electron energy curve is presented in Fig. 5. The curve deviates from linearity at the low- and high-energy portions of the range. The deviation is possibly large enough to affect the results of the modeling calculations that use the IV curves as input.

III. IV PERFORMANCE

Our research program primarily involves the collection of IV curves for epitaxial overlayers on semiconductor surfaces. Because a precise knowledge of the incident angle is crucial for the multiple scattering calculation analysis of the IV data, a very important first step in our IV measurement is the electron-beam alignment. We favor a normal-incidence geometry in our measurements because it is the most convenient one to obtain experimentally. Using MacLEED, a beam alignment method has been developed which allows us to align the beam normal to the sample surface to within $\sim0.5°$.

The center of the shadow of the LEED electron gun assembly provides a natural reference point because of the coaxial design of the gun. Because in the normal incidence geometry the (00) beam is obscured by the electron gun, the computer projects the position of the (00) beam estimated by the intersection of the diagonal lines connecting selected spots. The choice of spots can be made interactively and different choices can be made to check the procedure. The LEED optics is then tilted using the differential screw sup-

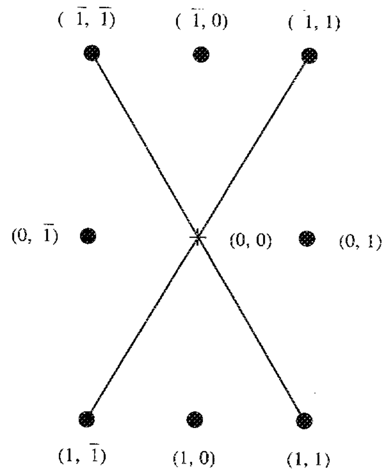

FIG. 6. Schematic diagram showing beam-alignment method. Dots represent the LEED pattern of GaAs(110) surface. The intersection of lines connecting selected diffraction spots is the projected position of (00) beam.

ports so that the position of (00) beam coincides with reference point. (In other experimental chambers, the sample orientation might be adjusted, instead.) The resulting alignment is schematically illustrated in Fig. 6.

For most samples the surface symmetry can be used to refine the alignment to greater accuracy. MacLEED allows one to integrate the intensity of any spot on the LEED image interactively. Thus, the intensity of the symmetry related spots are measured and used to fine tune the position of the LEED optics. To test this procedure, a set of IV curves for

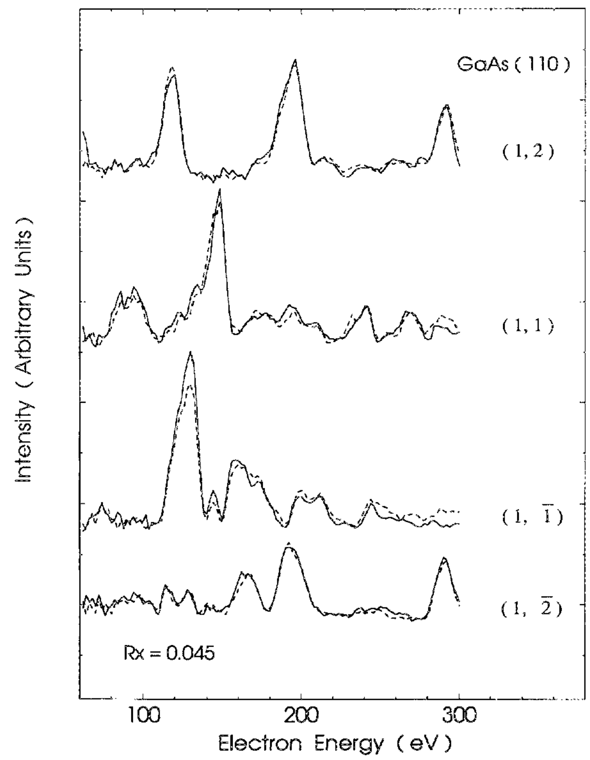

FIG. 7. IV curves of selected symmetry-related diffraction beams for the best alignment; i.e., the projected (00) beam coincides with the reference point. Dashed and solid lines represent $(\bar{h},k)$ and (h,k) beams, respectively.

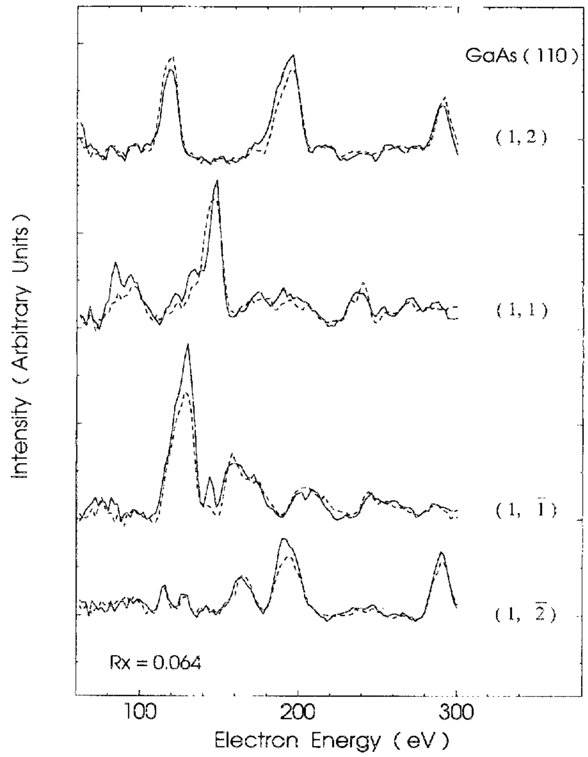

FIG. 8. Same as Fig. 7, except the alignment is off normal by $\frac{3}{4}°$ in the h direction.

symmetry related beams can be collected. In Figs. 7–11 the IV curves of two pairs of the symmetry-related beams on GaAs(110) are plotted for various incident angles. For this surface, the unit cell symmetry introduces an $(hk) = (\bar{h}k)$

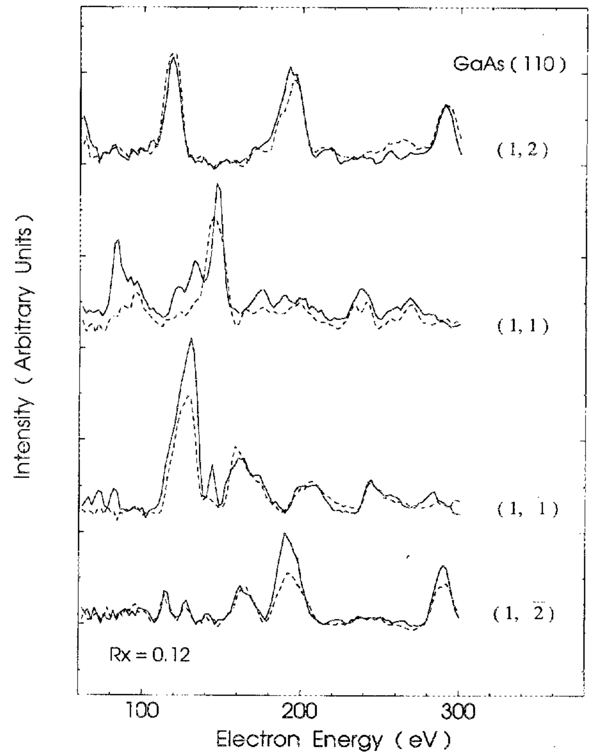

FIG. 9. Same as Fig. 7, except the alignment is off normal by 2° in the h direction.

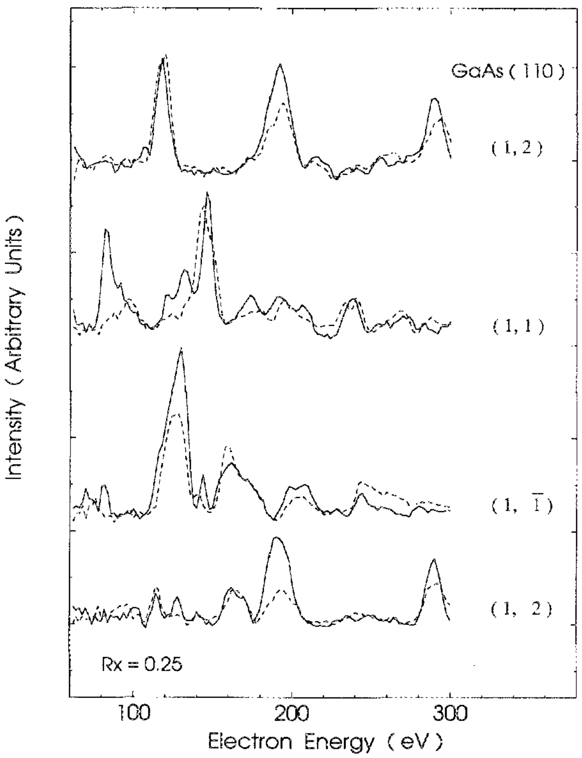

FIG. 10. Same as Fig. 7, except the alignment is off normal by 3° in the h direction.

condition on the diffraction intensities. For each beam in the figures the solid line and the dashed line represent the symmetry-related IV data. Figures 7–10 show the changes in the IV curves with angle for the incident beam in the $k = 0$ plane. Estimating from these figures, we believe that our alignment procedure along this direction is accurate to ~0.5°.

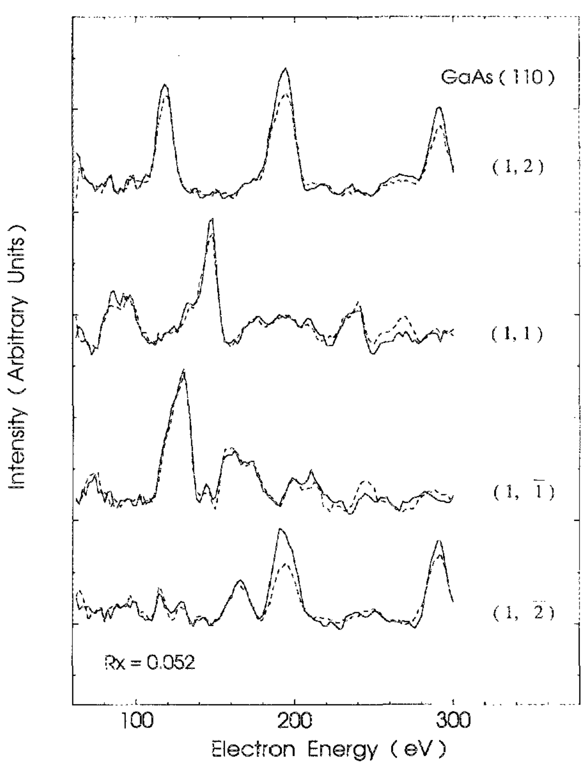

FIG. 11. The resulting IV curves for the alignment in the k direction with sample rotated azimuthally by 90°. The incident beam was aligned initially $\frac{1}{4}$° off normal.

The alignment procedure can be repeated in other symmetry planes for the general case. However, GaAs(110) only has one mirror plane. To judge the alignment in the other direction, the k direction, the sample can be rotated azimuthally by 90° and the IV curves collected again. Figure 11 shows the results of one such measurement with the electron beam aligned initially $\frac{1}{4}$° off normal with respect to the sample. The mirror plane symmetry is retained, verifying that the original alignment is quite good.

The degree of alignment can be made quantitative using a reliability factor calculation. The x-ray reliability factor R_x, defined in Ref. 17, is generally used by our group to measure the extent to which a set of theoretically calculated diffraction intensities computed for a trial geometry matches that measured experimentally. It also provides a convenient estimate of the reproducibility of the experimental data. In Figs. 7–11, R_x values are listed which were computed between two sets of four beams. The beams selected were related by the $(hk) = (\bar{h}k)$ symmetry property of the surface. The R_x values vary considerably as the sample alignment is degraded. For the best case, the R_x value is 0.045; for a 3° misalignment the R_x value increases to 0.25. In general, IV data is collected for 18 or more diffraction beams and for several different samples. The reproducibility of the data measured by the R_x values computed between data sets is approximately 0.04, a value that is consistent with that obtained using other experimental techniques.[18]

Linearity of the system is another important issue. We tested the linearity of the system against several parameters. Figure 12 demonstrates the linearity of the optical system. Plotted as circles is the intensity of a strong diffraction beam as a function of f number. Because the f number is inversely proportional to the aperture and the measured intensity should be proportional to the square of the aperture, the reciprocal of the square root of the measured intensity should be proportional to the f number. This proportionality, indicated by the triangles in Fig. 12, demonstrates the linear response of the optical system over a range of approximately three orders of magnitude in intensity. The overall response of the system as a function of incident beam current was also tested and shown to be linear within the usual experimental range of currents, 0.4–2 μA.

The instrumentation response function can be estimated using data from a cleaved GaAs(110) surface. First, the ratio of the FWHM of the sharpest diffraction spot observed at a 130-eV incident beam energy divided by the reciprocal lattice constant was determined to be 0.047. Using the development of Lu and Lagally,[19] the FWHM of the instrument response function for this ratio is 0.53°. The corresponding transfer width of the system, defined by Park, Houston, and Schreiner,[20] is 119 Å. From the FWHM of the instrument response function, we estimate the resolving capacity, or minimum angle of resolution,[19] of our instrument to be 0.33°, where a measurement uncertainty of 10% is assumed. Since the cleaved GaAs(110) surface is by no means perfect and the measurement error can be reduced, e.g., by integrat-

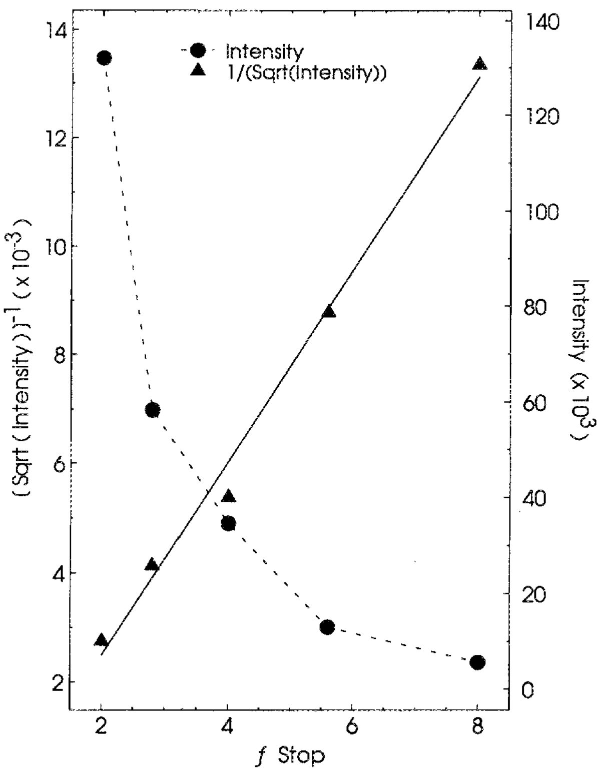

FIG. 12. The measured intensity and its reciprocal square root plotted as a function of the camera lens *f* stop. The intensity of a typical IV curve is nominally 100 000 (arbitrary units).

ing the picture over more frames, the actual FWHM of the response function and the minimum angle of resolution should be smaller than these estimates.

We have checked data collected by this system against previously reported measurements for clean GaAs(110)[21] and GaAs(110)-$p(1\times1)$ Sb-1 ML[22] where a spot photometer had been used. In each case we have found excellent agreement. Our system has been successfully used to study for the first time using LEED the atomic structure of the Bi/GaAs(110) interface.[14,16,23] A study of molecular-beam epitaxially grown structures is also currently under way.

In conclusion, a new LEED data-acquisition system has been developed. The system uses relatively simple components, yet has a rapid data-collecting speed and is very versatile. Because the hardware cost of the data-acquisition system is less than $10 000, it provides an affordable approach for research groups interested in quantitative LEED studies.

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