

Low Energy Electron Diffraction (LEED) Spectra: Aluminum

Abstract: The intensities of low energy electron beams specularly and nonspecularly diffracted from {100}, {110} and {111} surfaces of aluminum have been measured in a display-type LEED system as functions of electron energy, angle of incidence and azimuthal angle. Several of the measured and normalized spectra are presented, and the procedures followed in aligning the sample, reducing stray magnetic fields, and collecting and normalizing the data are described.

Introduction

Many theories have been proposed,¹⁻⁴⁴ most of them recently, to explain the diffraction of low energy electrons by crystalline surfaces. The purpose of these theories is to predict the distribution of back-scattered, or "reflected," electrons as a function of several parameters. For a given crystalline surface, with a given structure and potential function, the parameters are usually the energy of the incident electron beam, the angle θ between the incident beam and the normal to the surface, and the azimuthal angle ϕ between the projection of the incident beam on the surface and a specified crystallographic direction in the surface plane. There is thus a need for reliable, complete and accurately identified sets of intensity data with which to compare the results of theoretical calculations.

Some intensity data are indeed available in the literature for a number of surfaces, but these are often not wholly satisfactory: Most data are presented in the form of curves depicting the intensity of the specularly reflected (or 00) beam as a function of incident electron energy. The angle of incidence is often (though not always) given, but no statement is made about the procedure for and accuracy of its determination. The azimuthal angle is almost never specified. (The exceptions concern data for

LiF, NaF and graphite,²⁶ tungsten,^{11,38,39} and silver.³⁷) Very few, and rather incomplete, data are available about the dependence of the intensity curves on the angle θ , and hardly any about dependence on the angle ϕ . Even scarcer are data about the intensities of the nonspecularly reflected beams and their dependence on θ , ϕ and the electron energy.

The intensity data that are available have been collected predominantly with low energy electron diffraction (LEED) equipment of the display type,¹⁹ in which the intensity of a back-scattered beam is determined by measuring with a photometer the brightness of the spot produced on a fluorescent screen by the post-diffraction acceleration of the electrons. These data are usually reported in arbitrary units and few of them are reported quantitatively. In most cases the intensity of the incident beam, i.e., the incident current, is not known, nor is its usually strong dependence on electron energy.

The purpose of this paper is to present a reasonably complete set of LEED intensity data pertinent to three surfaces of aluminum: the {100}, the {110} and the {111} surfaces. Aluminum was chosen as the experimental material because, since it is the lightest face-centered-cubic metal and its electronic band structure has been the object of several investigations,^{45,46} it is a prime material for theoretical LEED calculations.^{7,23} In addition, previous studies⁴⁷⁻⁴⁹ have indicated that the {100}, {110} and {111} surfaces of aluminum can be cleaned sufficiently well for LEED observations and that these surfaces presumably

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have the "ideal" structure, which means that the atomic distribution in the plane of the surface is the same as that known to exist in any interior lattice plane parallel to the surface.

Our data were collected in display-type LEED equipment and are presented, as is customary, in the form of curves of beam intensity (in arbitrary but quantified units) versus electron energy. All curves have been normalized to constant incident current. The angular dependence of the diffracted electron beams was examined by varying the angle θ from 0 to 25° at constant, known values of the angle ϕ , and by varying the latter angle (for particular angles of incidence) in accordance with the symmetry of the surface. Thus, variations of the spectra with changes of the angles θ and ϕ should be accurate relative to each other, even if the absolute values of the angles were not known with high precision. Efforts were made, however, and are described, to control and determine precisely both θ and ϕ during each single run.

Bedair, Hofmann and Smith⁴⁸ published one spectrum up to 200 eV of the 10 beam (equivalent to the 11 beam in the notation used in the present paper) of Al {100}, probably for near-normal incidence (the angles θ and ϕ are not specified). Farrell and Somorjai⁴⁹ published one spectrum each for the 00, 20, 11 and 22 beams of Al {100} for $\theta = 1^\circ$ (ϕ is unspecified). The agreement between these curves and the corresponding ones in the present work (normal incidence, see Fig. 8) is very satisfactory.

Sample preparation

The single crystals of Al used in this work and the procedures followed for sample preparation were mostly the same as described in a previous paper.⁴⁷ The starting material was spectroscopically pure (total impurity content ≤ 0.1 ppm). The samples (16 mm $\times$ 6 mm $\times$ 0.6 mm) were spark-erosion cut; x-ray-oriented and lapped in the required crystallographic direction within less than 0.5°; mechanically polished on both major surfaces with, in sequence, 14- μ m, 8- μ m and 0.5- μ m diamond paste; electropolished (3 A, 30 V to 35 V) in a bath of 400 ml orthophosphoric acid, 380 ml ethanol and 250 ml distilled water at approximately 45°C for 2 min; and rf-sputtered (200 W, 4-in.-diameter cathode) under a pressure of 6×10^{-3} Torr of argon for approximately 2 hours. We estimate that the last step etched away a thickness of 300 Å to 400 Å. After the samples were put into the LEED system and baked routinely, and a base pressure of approximately 1×10^{-10} Torr was attained, the surfaces were cleaned with repeated sequences of argon ion bombardment (1 to 2×10^{-4} Torr of argon, $2 \mu\text{A}/\text{cm}^2$, 300 V) and annealing. The quality of the LEED patterns obtained, including the 00 spectrum at nearly normal incidence (see below), was identical to that obtained in the previous study of Al surfaces.⁴⁷

Experimental procedures

• Equipment

The LEED system used is a display-type Varian unit⁵⁰ with a 4-in. fluorescent screen that allows observation of the specularly reflected beam up to an angle of incidence θ of about 25°. The samples were mounted in a holder⁵¹ that allows continuous and independent variation of both θ and ϕ , the latter angle for approximately 300°. The brightness of the diffraction spots on the fluorescent screen (i.e., the intensities of the corresponding diffracted beams) was measured with a spot photometer⁵² with an acceptance angle of 0.5° that was aimed at the desired spot and maintained manually in the correct position. The output of the photometer was connected to the y axis and the voltage of the electron gun (proportional to the energy of the incident electrons) to the x axis of an x-y recorder, the gun voltage being swept by means of a motor-driven potentiometer from 300 V to 0 V in approximately 3.5 minutes.

• Determination of the incident current

To determine the intensity of the electron beam incident on the surface to be studied, the sample's potential was raised 90 V above ground (to reduce to a minimum the loss of electrons due to secondary emission) and the current to ground was measured with an electrometer as a function of the gun voltage. The incident current depends rather strongly on the gun voltage (Fig. 1). Variation of the positive bias voltage on the crystal from 90 V to 180 V was found to have little or no effect on the dependence of the incident current on voltage. This dependence remained unaltered during each single run, i.e., as long as the gun cathode was not exposed to poisoning gases, but it changed considerably from run to run, as the system was opened to change the sample. Figure 1 serves as an example of the different shapes that the curve of incident current versus voltage can assume in different runs.

• Effect of magnetic fields

Residual magnetic fields in the LEED chamber (due to the earth's magnetic field and to imperfect shielding of the sputter-ion-pump magnets) affect the paths of the incident electrons in inverse proportion to the electron energy. Hence, the angles θ and ϕ vary continuously as the electron energy is decreased. Baker⁵³ has shown that for a residual field of 0.1 Oe the change in θ is 4° when the electron voltage varies from 300 V to 10 V. The magnetic-field effect can be detected easily by observing the motion of the 00 spot on the screen with varying electron energy. In the present work three sets of coils were wound directly on the flanges of the LEED chamber and the dc currents through them were adjusted separately for minimum motion of the 00 spot on the screen (as viewed

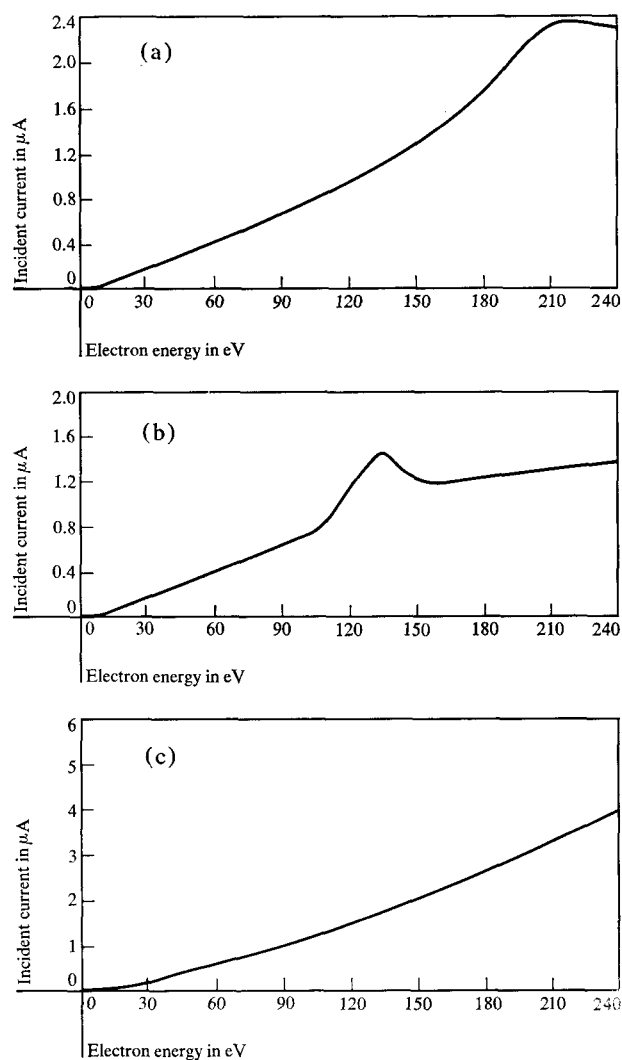


Figure 1 Example of the dependence of the incident electron current on voltage. Only one cathode was used to obtain curves (a), (b) and (c), but between the runs the cathode was exposed to standard atmospheric pressure and then rejuvenated.

through the photometer telescope) in the range 20 V to 300 V. Below approximately 20 V the spot moved rapidly off the screen with decreasing voltage, and was noticeably defocused. The data, if taken below 20 V, are therefore to be considered only qualitative.

• *Determination of sample position for normal incidence*

To collect reliable intensity data it is imperative that the orientation of the sample for normal incidence of the electron beam be established precisely. This allows not only the determination of the spectra of nonspecular beams for normal incidence but also the calibration of the zero point on the goniometer scale (of the sample

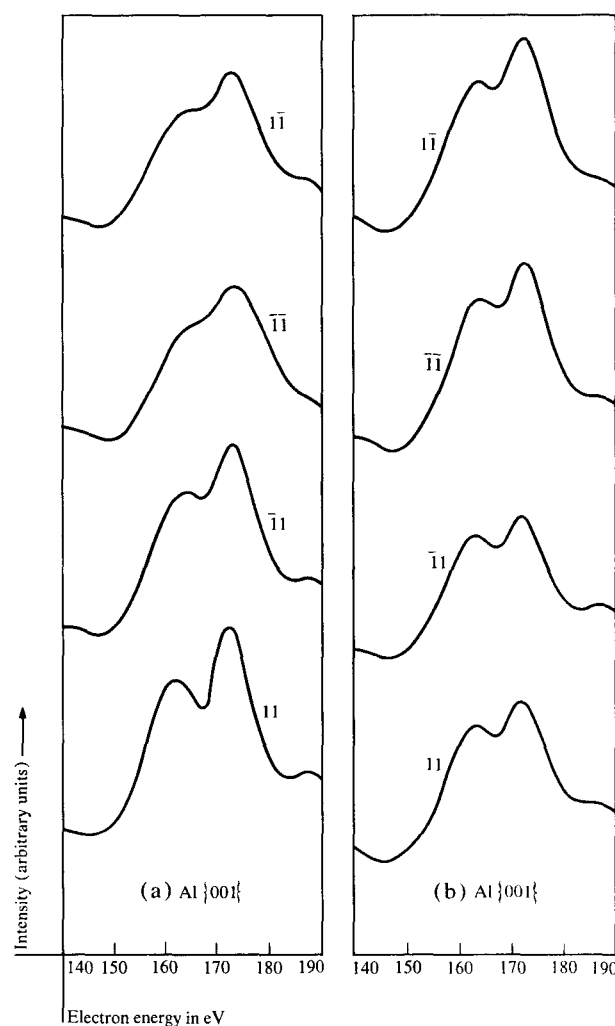


Figure 2 Al {100} surface: dependence of the intensities of the (equivalent) 11-type beams on orientation of the sample. The difference in orientation was about 1° between curves (a) and (b), the latter curve corresponding to normal incidence. These intensity curves were *not* normalized to constant incident current.

holder), which measures the angle θ . The procedure for this purpose was to record the intensity-versus-voltage curves of all equivalent nonspecular beams and to adjust both the tilt and the θ -rotation axis of the specimen holder⁵¹ until all such curves were identical. The equivalent nonspecular beams are, e.g., for the {100} surface, the 11, $\bar{1}1$, $\bar{1}\bar{1}$ and $1\bar{1}$ beams and the 20, 02, $\bar{2}0$ and 0 $\bar{2}$ beams; for the {110} surfaces, the 10 and $\bar{1}0$ beams, the 01 and $0\bar{1}$ beams, etc. (For the indexing used here, see Figs. 5, 6 and 7.) This procedure is quite laborious and time consuming. Fortunately, for all three surfaces of Al considered here a voltage range was always found in which the intensities of some nonspecular beams were

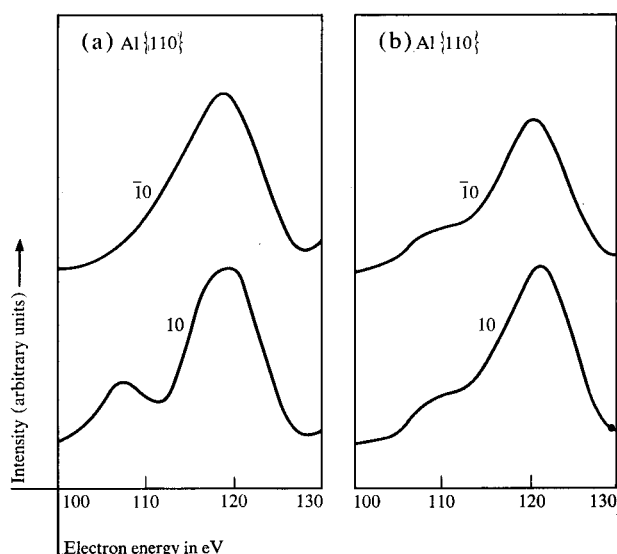


Figure 3 Al $\{110\}$ surface: same as in Fig. 2 but for the 10 and $\bar{1}0$ beams.

very sensitive to small changes in orientation of the sample. Figures 2, 3 and 4 show this sensitivity for the $\{100\}$, $\{110\}$ and $\{111\}$ surfaces of Al, respectively. For the $\{100\}$ surface, the spectra of the 11-type beams are very sensitive to the sample orientation in the range 150 V to 180 V (Fig. 2), while the 20-type spectra are little affected by small changes in orientation of the sample; for the $\{110\}$ surface, the 10 and $\bar{1}0$ spectra are sensitive in the range 100 V to 120 V (Fig. 3), the 11-type spectra in the range 70 V to 100 V; for the $\{111\}$ surface, the 10, $\bar{1}1$ and $0\bar{1}$ spectra are particularly sensitive to the sample orientation in the voltage range around 80 V (Fig. 4). In each case, the sample orientation required for equivalence of the sensitive spectra was taken as the one corresponding to normal incidence of the electron beam. Accordingly, the angle θ was measured with reference to this orientation as the one defining $\theta = 0$.

• Determination of the azimuthal angle

For each surface the reference axis that defines $\phi = 0$ must be chosen and identified. Figures 5, 6 and 7 show schematically the LEED patterns obtained from Al $\{100\}$, Al $\{110\}$ and Al $\{111\}$, respectively. In each case the reciprocal-lattice axes k_x and k_y are indicated; the azimuthal angle ϕ is defined as the angle between the projection of the incident wave vector on the plane of the figure and the k_x axis. Assuming that the θ -rotation axis is vertical, i.e., that the projection of the incident wave vector is horizontal, we have indicated in each figure the appearance of the corresponding pattern for selected values of the angle ϕ .

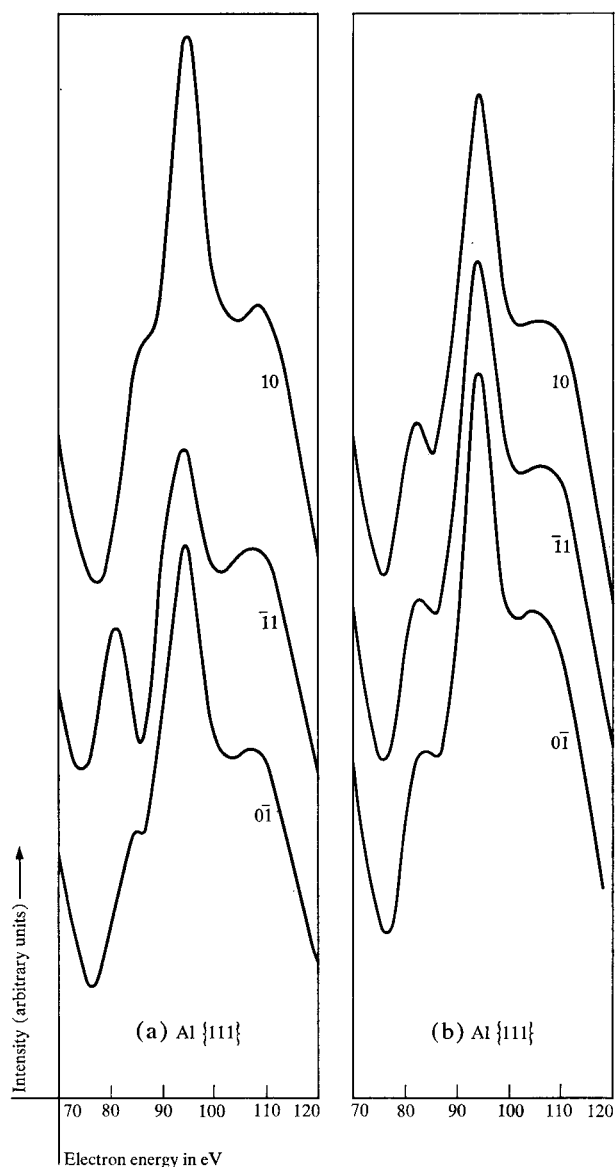
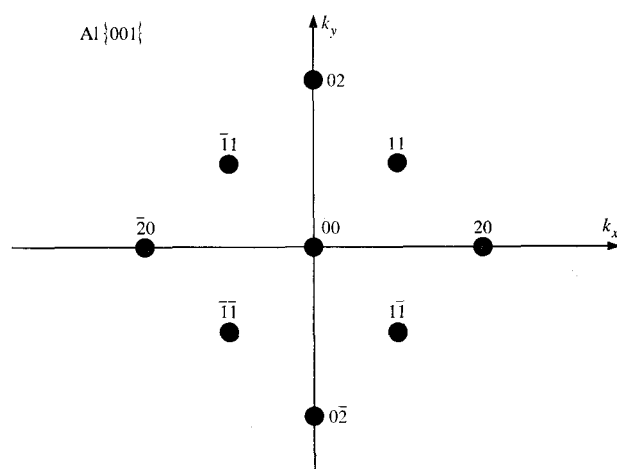
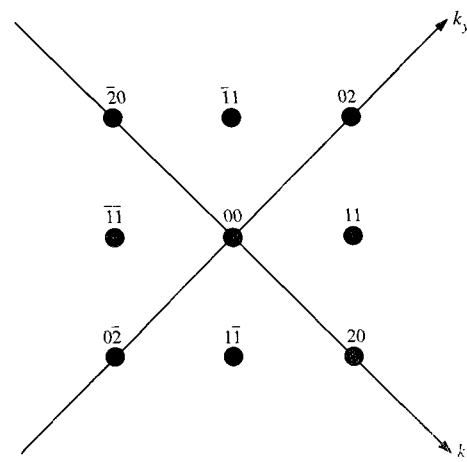


Figure 4 Al $\{111\}$ surface: same as in Fig. 2 but for the 10, $\bar{1}1$ and $0\bar{1}$ beams.

Experimentally the condition $\phi = 0$ was established by adjusting the azimuthal orientation of the sample (by means of the sample holder mentioned previously) in such a way that upon varying θ all diffraction spots lying on the k_x axis passed through the same reference point on the fluorescent screen. The same procedure was followed to establish special values of ϕ , i.e., $\phi = 45^\circ$ for Al $\{100\}$ in Fig. 5(b); $\phi = 90^\circ$ for Al $\{110\}$ in Fig. 6(b); and $\phi = 30^\circ$ and $\phi = 60^\circ$ for Al $\{111\}$ in Figs. 7(b) and 7(c). Intermediate values of ϕ could be obtained in all cases by calibrating the feedthrough controlling the ϕ rotation in the sample holder.⁵¹



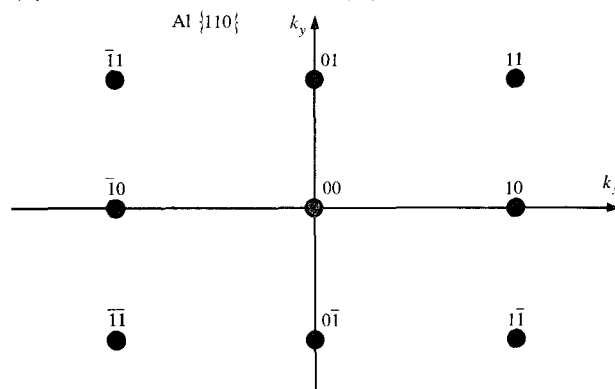
(a) $\phi = 0$



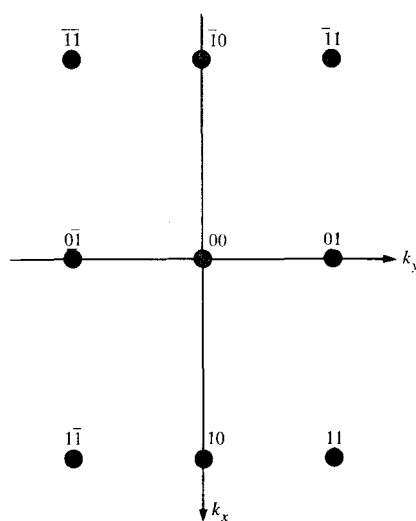
(b) $\phi = 45^\circ$

Figure 5 Schematic LEED patterns of Al {100} surfaces: (a) defines the condition $\phi = 0$; (b), $\phi = 45^\circ$.

Figure 6 Schematic LEED patterns of Al {110} surfaces: (a) defines the condition $\phi = 0$; (b), $\phi = 90^\circ$.



(a) $\phi = 0$



(b) $\phi = 90^\circ$

• *Use of a bias voltage between sample and first grid*

Normally both the sample and the first grid of the electron-optics assembly¹⁹ are kept at ground potential to provide a field-free space for the electrons. Under these conditions the nonspecular beams, say for normal incidence, are not visible on the screen at very low voltages, because they emerge from the sample almost parallel to the surface plane. To bring them onto the screen, so that the corresponding diffraction spots can be measured with the spot photometer, one generally applies a bias voltage of 100 V to 200 V between the sample and the first grid.¹⁹ If such a bias voltage is used it is advisable to determine anew the sample orientation that corresponds to normal incidence, as this is sometimes slightly different from that pertaining to normal incidence for zero bias. It is also advisable to check that the nonuniformity of the biasing field does not shift the positions of maximum intensity in any given spectrum with respect to the zero-bias condition. This is done by measuring the same spectrum twice, once with and once without the bias. If the positions of maximum intensity are found to be the same in the overlapping energy range (which was the case in the present work), it may be assumed that these maxima are correctly located in the low energy range that can be measured only with a nonzero bias.

• *Collection of intensity data*

Figures 8 through 26 depict some of the intensity data collected with the procedures described above. The two-digit indices labeling the curves correspond to the indexing of the diffraction spots defined in Figs. 5, 6 and 7. Each curve has been normalized to a constant incident current of $0.2 \mu\text{A}$. This was done by digitizing the recorded spectra and then performing the normalization operations on the

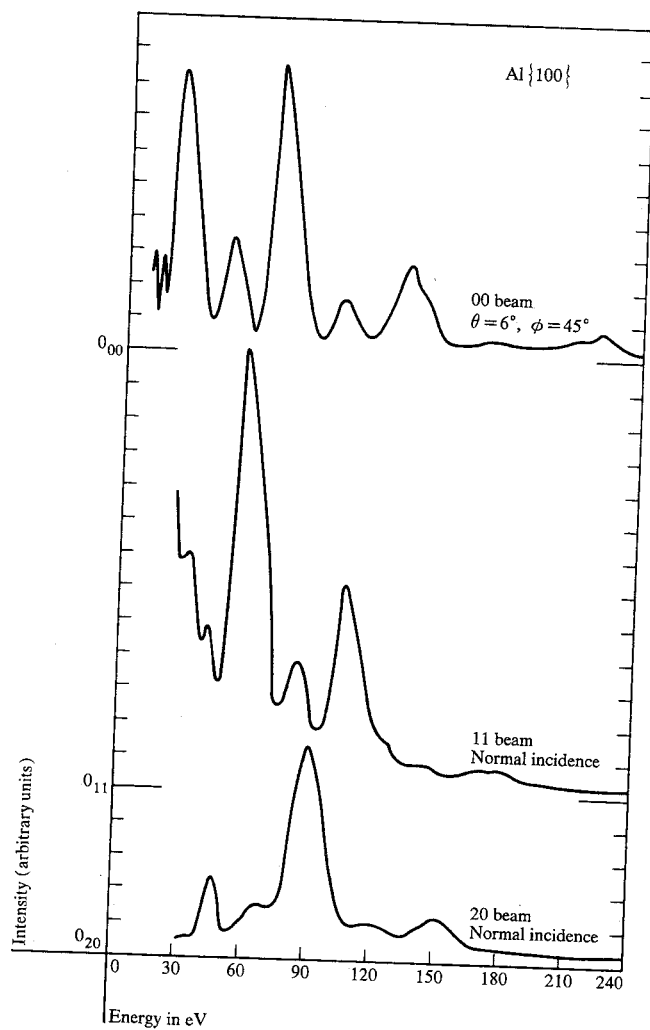


Figure 8

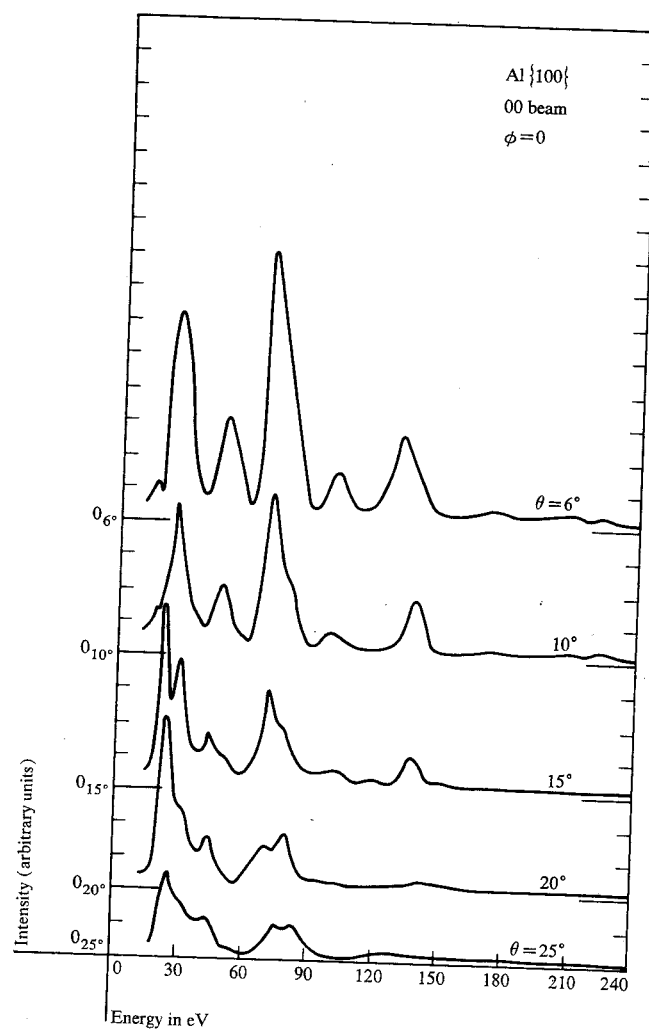


Figure 9

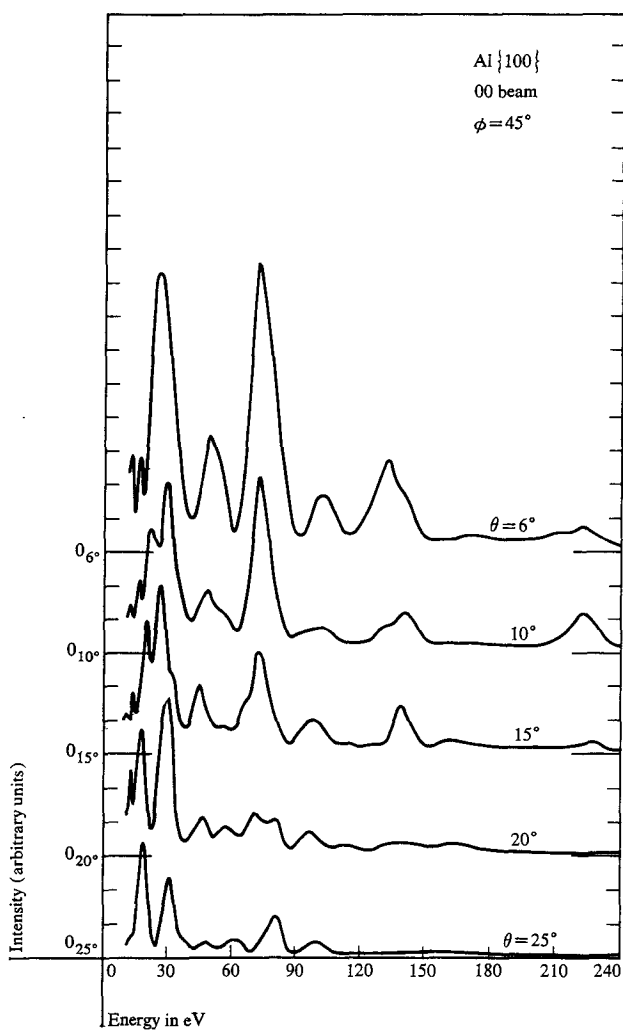


Figure 10

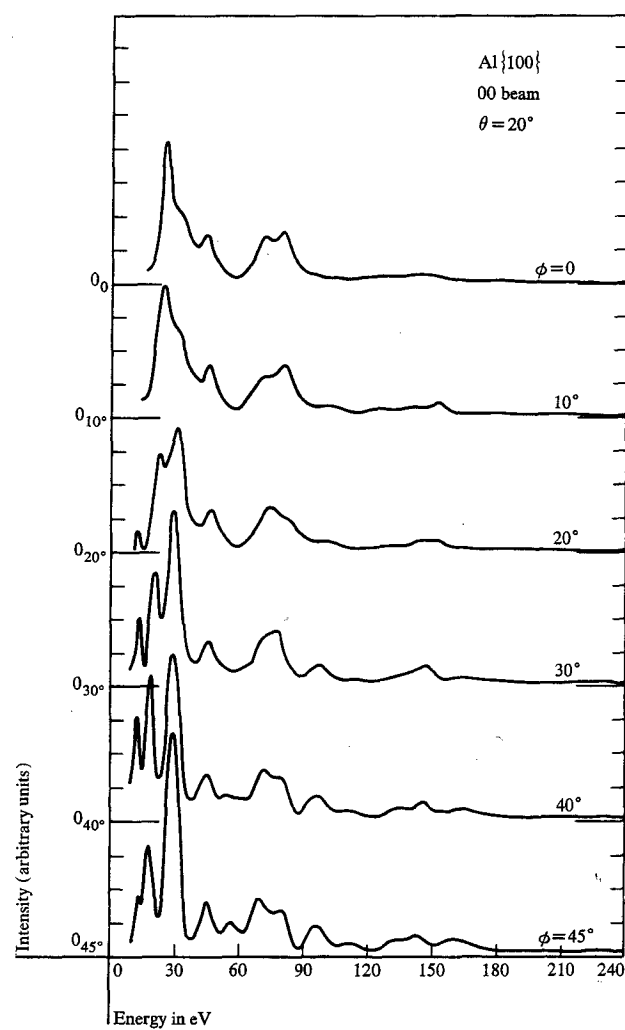


Figure 11

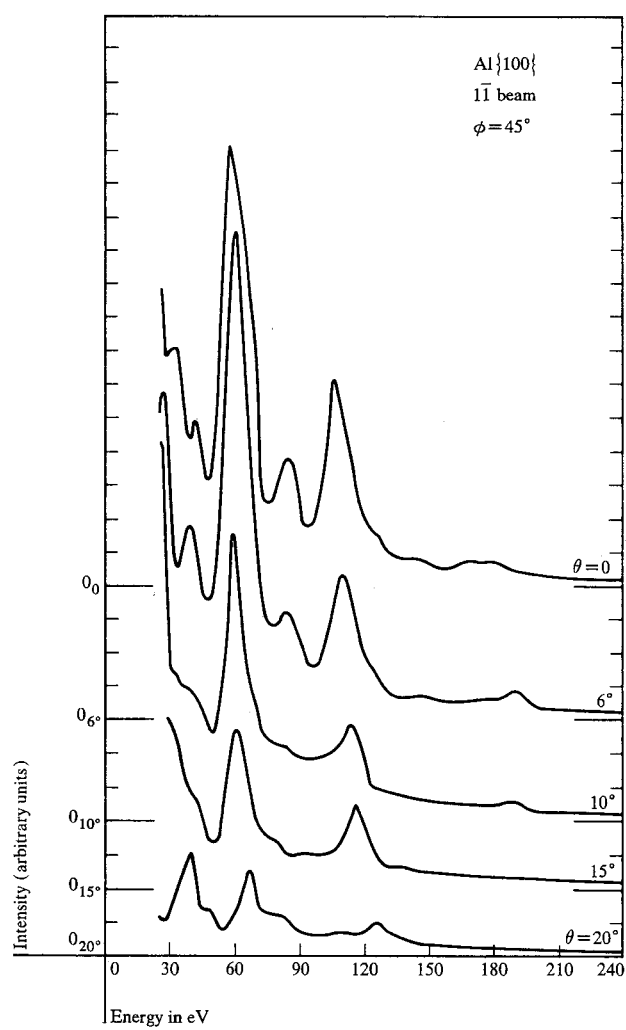


Figure 12

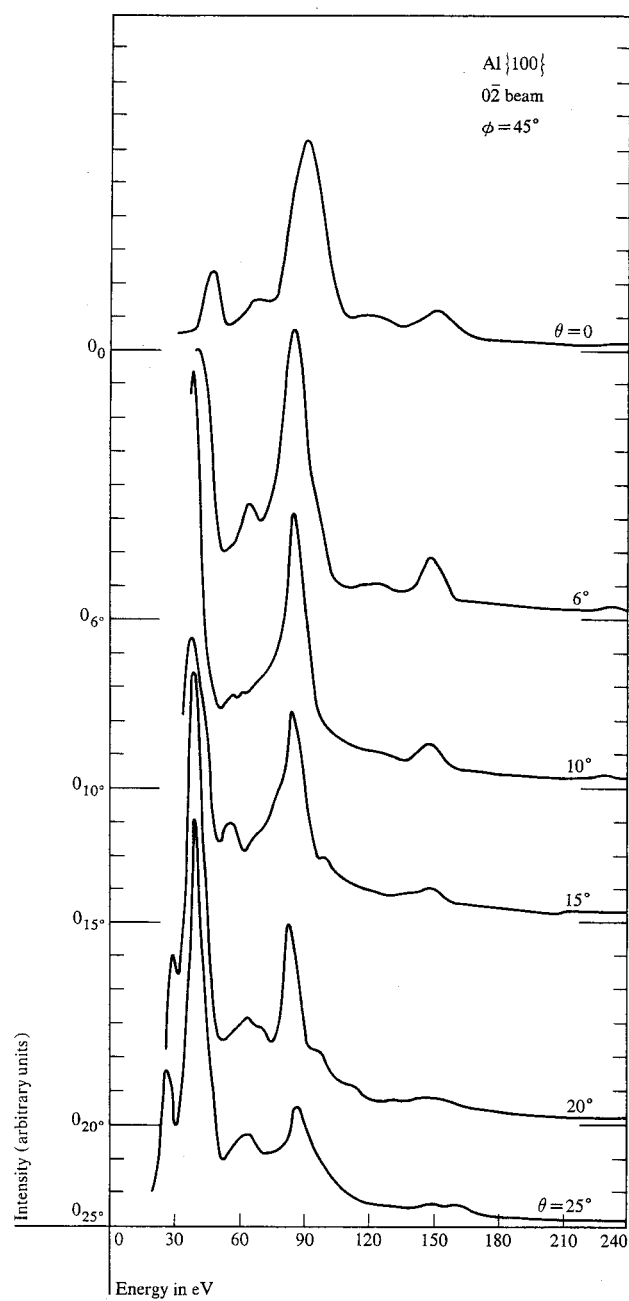


Figure 13

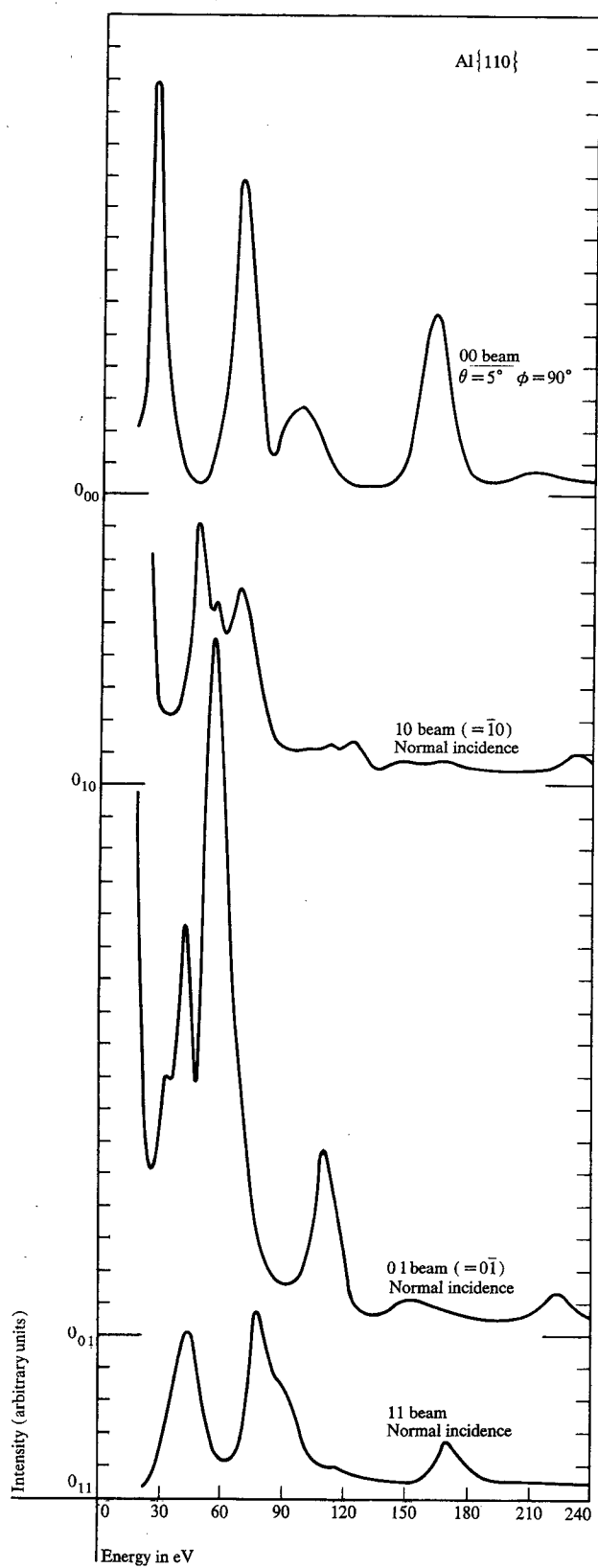


Figure 14

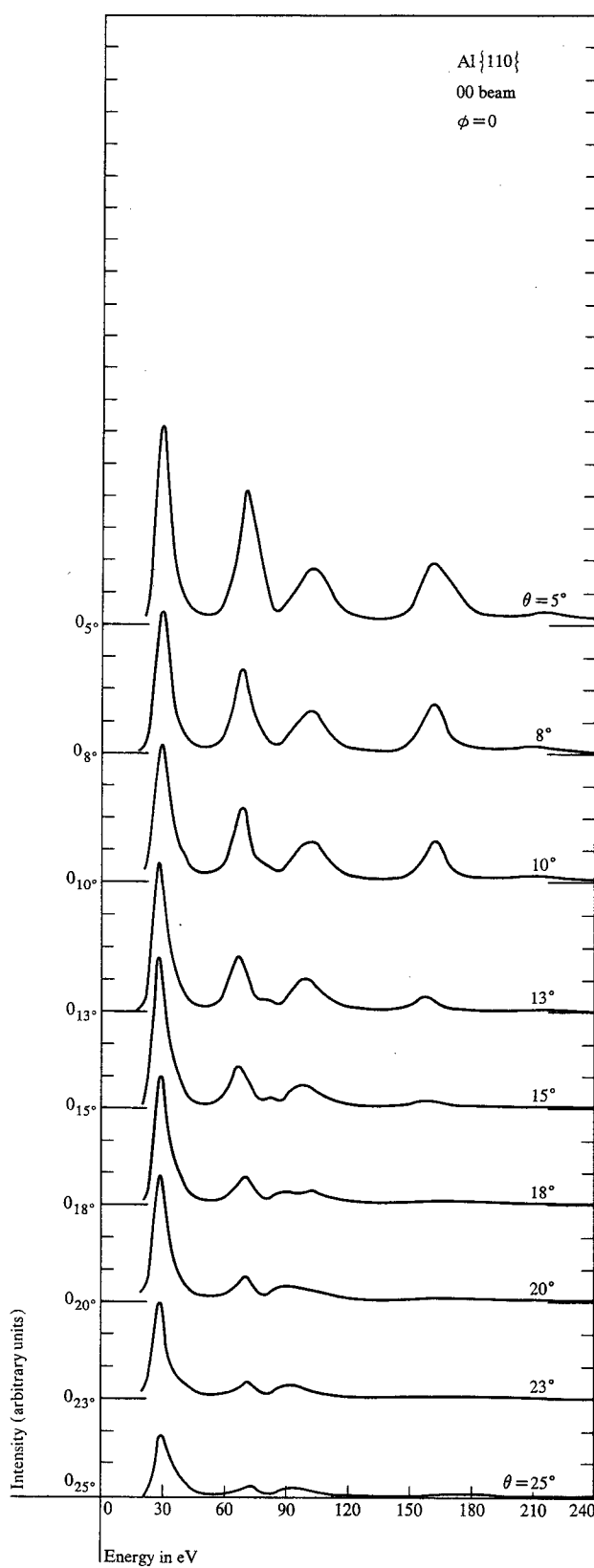


Figure 15

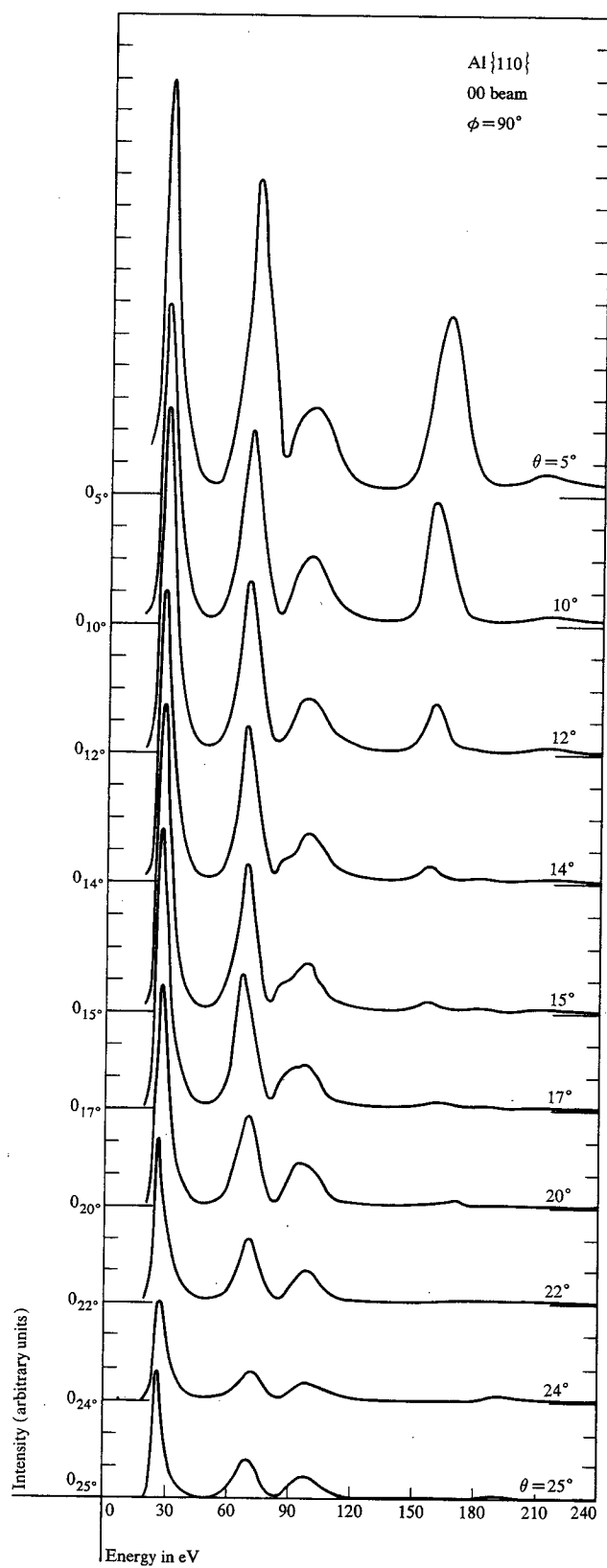


Figure 16

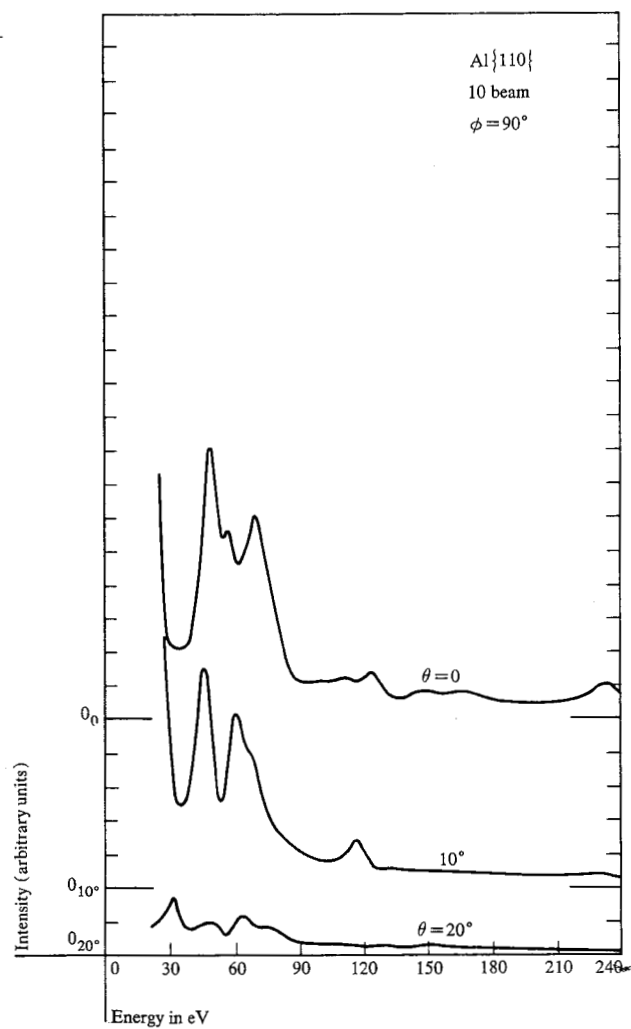


Figure 17

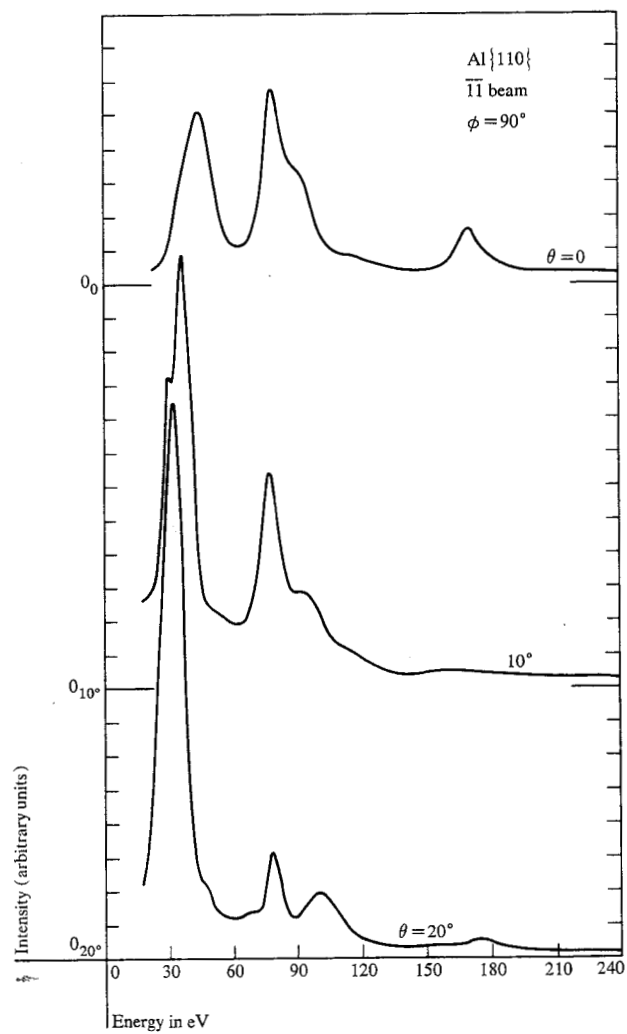


Figure 18

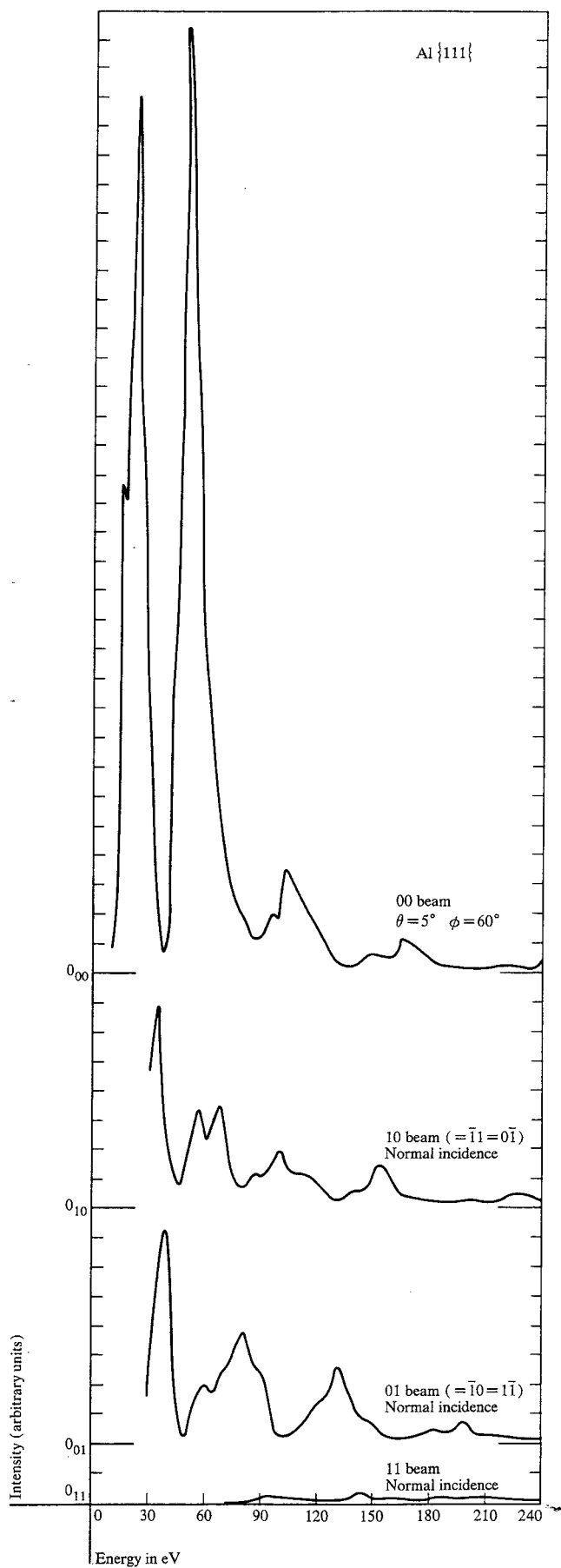


Figure 19

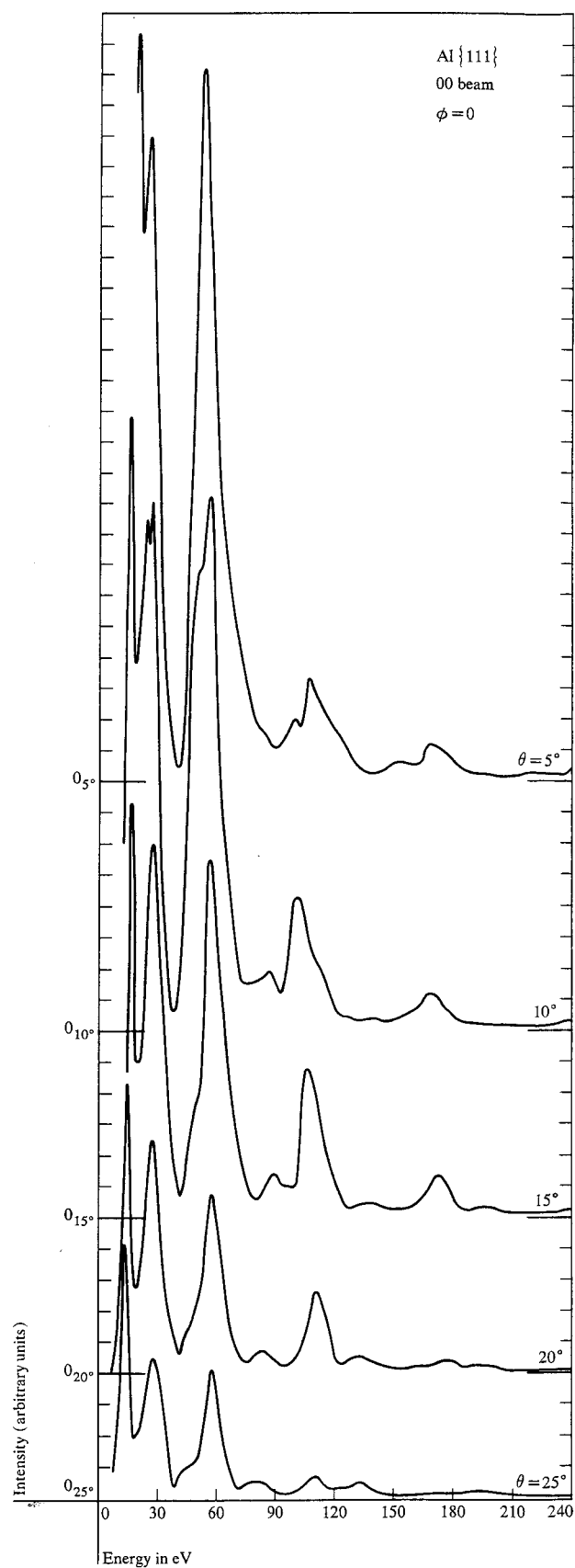


Figure 20

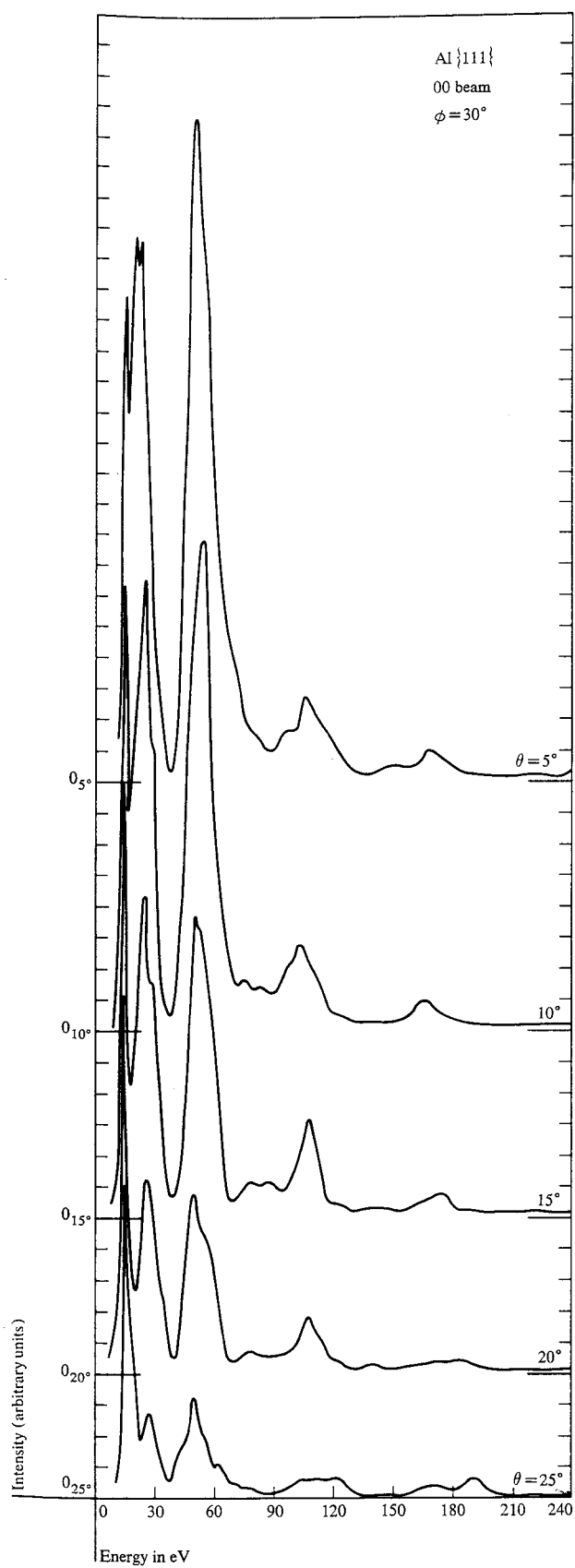


Figure 21

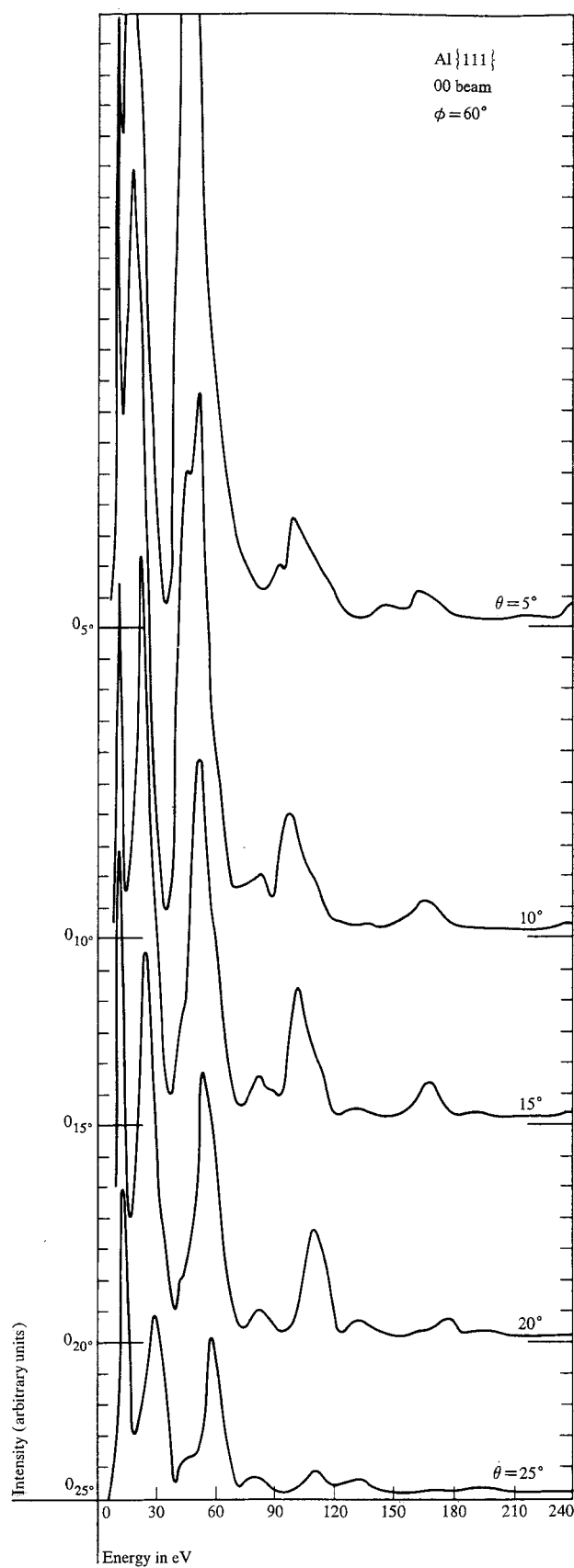


Figure 22

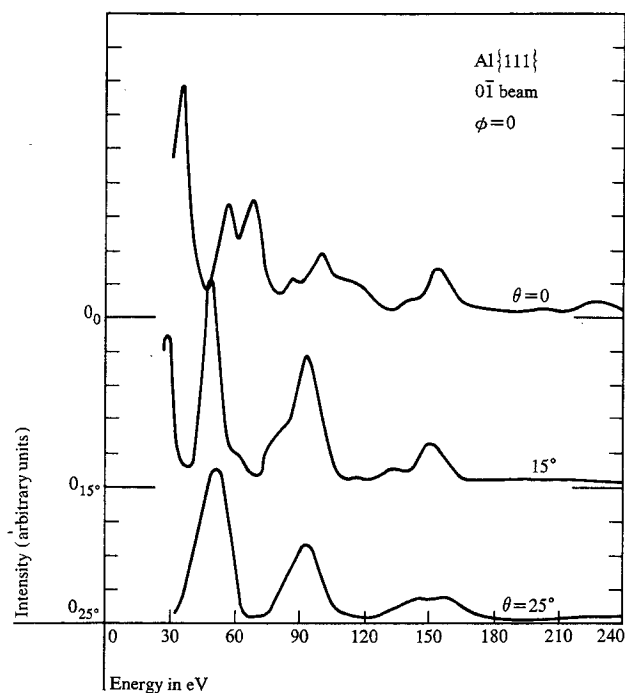


Figure 23

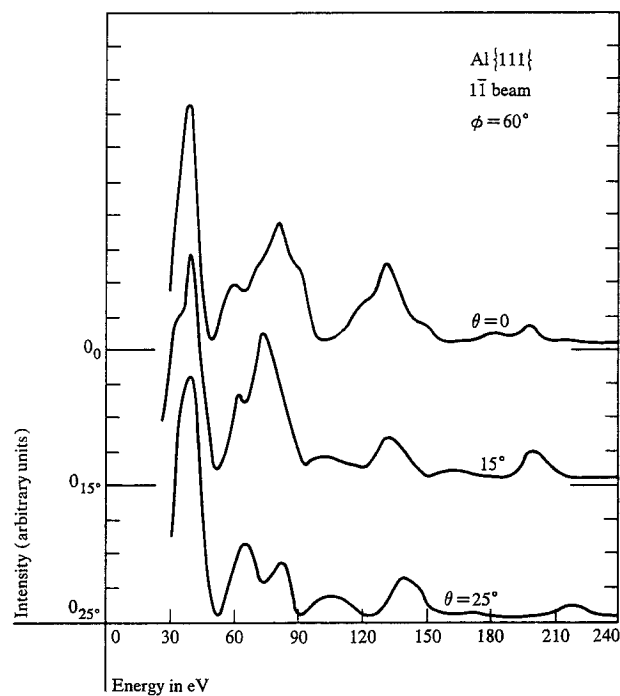


Figure 24

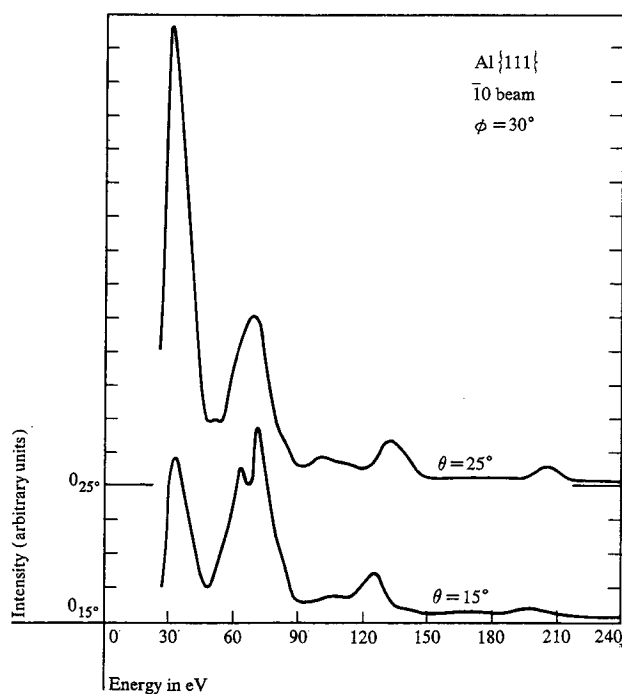


Figure 25

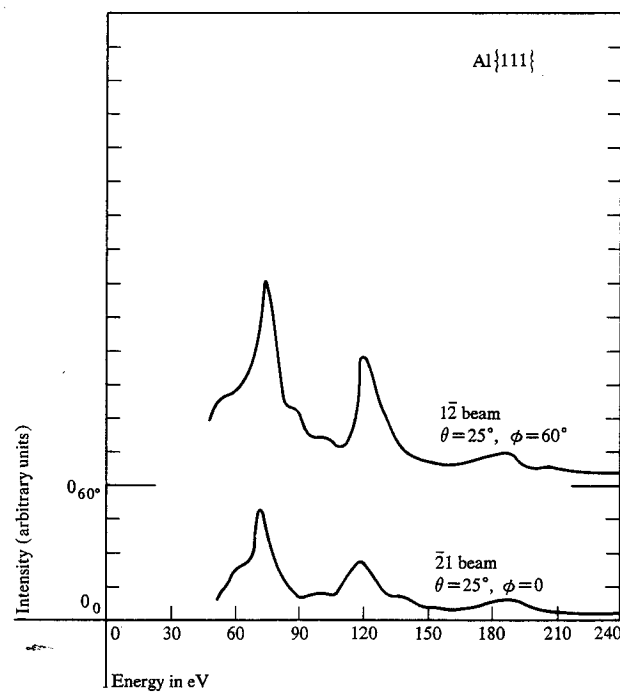


Figure 26

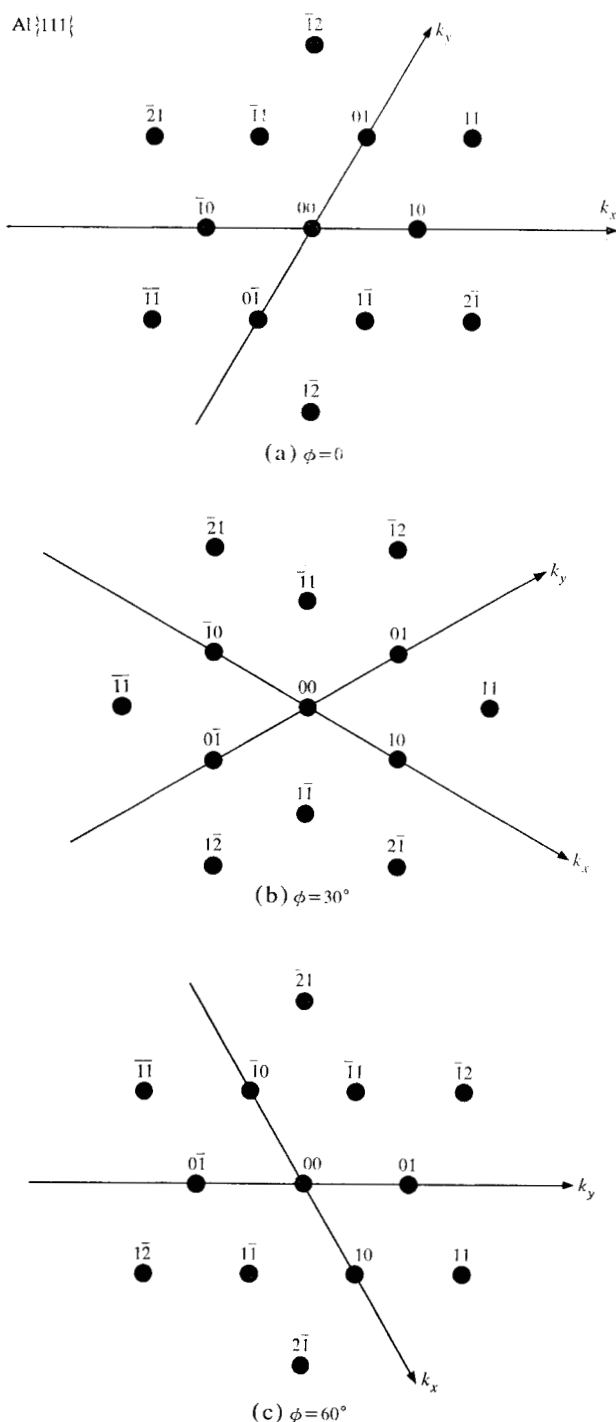


Figure 7 Schematic LEED patterns of Al {111} surfaces: (a) defines the condition $\phi = 0^\circ$; (b), $\phi = 30^\circ$; (c), $\phi = 60^\circ$.

basis of the appropriate normalizing curve (such as is shown in Fig. 1) on a computer. The normalized curves were then replotted by the computer in the form presented here.

For all three surfaces the 00 spectra were recorded

with no bias voltage between the sample and the first grid.¹⁷ The nonspecular beams, on the other hand, were measured with a bias for angles of incidence smaller than about 15° . For larger angles the nonuniform biasing field seemed to displace the intensity maxima with respect to their positions at zero field. Fortunately, a bias was not needed at larger values of θ because the geometry was such that some of the spots were visible on the screen immediately after emergence of the corresponding beam, and most of the other spots could have been maintained on the screen only with impractically high bias voltages (hence these latter spots were simply not measured).

Figures 8 through 13 present data for Al {100}; Figs. 14 through 18, Al {110}; and Figs. 19 through 26, Al {111}. In each group the first figure collects the spectra measured at (or near) normal incidence in the same plot; in particular, the nonspecular beams were measured at normal incidence. The 00 beam, which could not be measured at $\theta = 0$, was measured slightly off normal incidence (Fig. 8 for Al {100}, Fig. 14 for Al {110} and Fig. 19 for Al {111}). Two figures (three in the case of Al {111}) exhibit the θ dependence of the 00 beam for two values of ϕ . The ϕ dependence is exhibited explicitly only in one case (Fig. 11), while the remaining figures are concerned with the θ dependence of some nonspecular beams.

The precision is estimated to be about 1° in both θ and ϕ , and about 2 eV in the energy coordinate.

Discussion

A few *a posteriori* remarks may be useful in order to improve future data collection of this kind.

First, we think that intensity data collection by means of a Faraday box, rather than the fluorescent screen-photometer combination, is better on at least three counts: 1) It is more accurate; 2) it eliminates the need for a bias voltage, and any beam can be observed down to its emergence energy, thus avoiding the distortion that the bias voltage often introduces; and 3) the data can be more easily put on an absolute scale, an advantage that may be essential in comparing theoretical calculations with experimental data.¹⁷

Second, it may be more advantageous in some cases to present the data on a logarithmic, rather than a linear, scale. Although the logarithmic scale may be somewhat misleading if one is concerned only with the low energy range (say, below 100 eV to 150 eV), it is essential, at least for Al, if one is concerned with the energy range above 200 eV. Often there is considerable structure in the spectra in the higher energy range, which the normalization process and the linear plotting in the present work have hidden almost completely.

Third, it may be more convenient to carry out the normalization process electronically, and to record directly the ratio between the intensity being measured and

the intensity of the incident beam.⁵³ However, if the normalization process is to be done computationally *after* the experiment (as it was in the present work), it would be easier and faster to digitize the raw data *while* they are being collected and to manipulate them directly in the computer.

Acknowledgments

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