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CONF-8006174--1

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RELIABILITY OF CONTEMPORARY DATA-ACQUISITION TECHNIQUES FOR LEED ANALYSIS

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October 1980

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RELIABILITY OF CONTEMPORARY DATA-ACQUISITION TECHNIQUES FOR LEED ANALYSIS*

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ABSTRACT

It is becoming clear that one of the principal limitations in LEED structure analysis is the quality of the experimental I-V profiles. This limitation is discussed, and data acquisition procedures described, which for simple systems, seem to enhance the quality of agreement between the results of theoretical model calculations and experimental LEED spectra. By employing such procedures to obtain data from Cu(100), excellent agreement between computed and measured profiles has been achieved.

INTRODUCTION

As may be seen from an inspection of the content of the papers in these Proceedings (also see, e.g., refs. 1-4), considerable improvement has occurred in dynamical LEED analysis during the last decade. Although this improvement has enable the surface crystallography for a considerable number of clean surfaces and surfaces with ordered layers of adsorbates to be determined, some debate still continues concerning the reliability of LEED analysis for the determination of surface structure. Therefore, it appears timely to address questions concerned with the improvement of the accuracy and sensitivity of LEED analysis. This paper, as well as several of the other papers in these Proceedings, addresses such questions.

*Research sponsored by the Division of Materials Sciences, U. S. Department of Energy under contract W-7405-eng-26 with Union Carbide Corporation.

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Some features of the analysis which limit the accuracy obtainable by LEED are details of the basic theoretical model employed to perform dynamical calculations and the applicability of specific values of the nonstructural parameters used in the calculations. However, since some of these are discussed in a companion paper,[5] the limitations of the theoretical analysis are not the focus of this paper. Rather, here we address the equally serious limitation of obtaining accurate experimental LEED intensity versus voltage (I-V) spectra. Of course, data collected in a manner designed to minimize errors should always be employed in a LEED analysis. However, it also should be realized that finite errors are introduced into any LEED data, which, especially with existing LEED scattering chambers, can ultimately degrade the structural accuracy obtained in an analysis. One approach[6,7] which has been used in an attempt to reduce the influence of experimental error has been the collection (at several scattering geometries) of as large a number of I-V profiles as time and patience allow. Then this large number of individual profiles is used in the subsequent analysis. However, we recently have been concerned with an assessment of an alternative approach for the collection of LEED data to be used in the analysis. The present status of the assessment is that our approach appears both to reduce the experimental error present in the data and to improve the accuracy and sensitivity of the analysis.

Our approach has been to measure the I-V profiles of all symmetrically equivalent beams for several nondegenerate beams rather than collect data for a large number of independent beams. Then profiles for beams related by symmetry are averaged to produce a single profile for each nondegenerate beam. It is these averaged I-V profiles that are used in the analysis. In specific analyses where we have employed such averaged profiles, results have been obtained which are sensitive to changes in atomic positions of the order of 0.02 Å. Very low Zanazzi and Jona[8] total reliability factors also have been obtained in these investigations; e.g., in our analysis of Cu(100) a total R-factor of 0.044 was obtained.

EXPERIMENTAL PROCEDURES

As gen rally recognized, there are several aspects of the apparatus and sample surface which degrade the quality of experimental LEED spectra. Among these are residual electromagnetic fields, electron beam coherence, alignment reproducibility and accuracy, and surface topography. Naturally, experimental procedures must be developed to minimize possible deliterious effects and to help specify the limitations of the apparatus. Several procedures are used by the Surface Physics Group at Oak Ridge National Laboratory to insure high quality I-V profile data.

For example, the residual magnetic fields are reduced by using three orthogonal sets of Helmholtz coils adjusted to minimize field strength as measured with a magnetometer. By proper choice of coil currents, the fields in the electron scattering region are below 15 mGauss, and below 5 mGauss at the sample position. Effects of the electrostatic fields are reduced by keeping the sample, first grid of the electrostatic analyzer and the chamber at ground potential. Therefore, the electrons are scattered in a nearly field free region.

The experimental LEED data are obtained in a bakeable UHV chamber capable of 3 x 10^{-11} Torr (4 x 10^{-9} Pa) base pressure. When the electron gun is operating, the pressure remains below 1 x 10^{-10} Torr. The scattered electrons are analyzed with a Faraday cup having an Einzel lense operated as a retarding field analyzer with an energy resolution of 0.5%. The electron gun used for the LEED experiments has been adjusted to produce a well focused, well collimated primary beam. The beam always has a spatial width of less than 1 mm diameter, with the greatest dispersion at low energies (~20 eV). Additionally, the energy dispersion of the beam has been studied. At the current densities typically used in LEED experiments, the beam has a FWHM of less than 0.4 eV at energies above 50 eV, and is approximately 1.5 eV at 20 eV. The elastically scattered electron intensity has been observed to be several orders of magnitude more intense than the inelastic component. Furthermore, the beam has an approximately gaussian profile for the systems investigated to date. The Faraday cup aperture has been selected to collect most of the scattered beam. Several cup apertures were tested until it was determined that a 1 mm aperture, corresponding to an acceptance angle of $\sim 2.5°$, would collect $\approx 90\%$ of the elastically scattered electrons and produce little distortion of the I-V profile shape. In addition the value of the retarding potential typically was -3 eV with respect to the incident electron energy. This retarding potential allowed most of the electrons elastically scattered by the sample to pass into the collector without significantly affecting the profiles' I-V structure, as has been determined by testing retarding fields between -0.5 and -10 eV.

One feature of this apparatus, and those used by other investigators, is the possible misalignment between the axis of rotation of the Faraday cup and the electron beam axis. This misalignment in our system is of the order of 0.1 mm. Therefore, the cup appears to precess about the electron beam axis as the cup is rotated azimuthally. As a result of the misalignment, there are azimuthal positions of the cup for which the measured polar angle, θ, is too large with respect to the true scattering angle and others where it is too small. At the poorest alignment conditions the measured values of θ appears to be within 1/2° of the

true polar angle. Importantly, however, it should be realized that the error is not random. Depending on the sector in which the cup is, the measured polar angle can be high or low. Therefore, systematic errors are introduced into measurements of scattering angles. This becomes especially important when there are no corroborating measurements of angle, such as for determination of the specular beam scattering angle.

Sample imperfections can contribute significantly to the quality of the experimental I-V spectra. In our work, several procedures are used to try to maximize surface perfection and reproducibility. A crystal is cut to expose the desired face, as determined from Laue x-ray patterns. The crystal is mounted on a polishing jig and aligned to the desired orientation by either Laue patterns or by measurement with an x-ray diffractometer. It is then lapped with alumina grit of selected sizes down to 0.3 μ diameter. The crystal is then annealed in hydrogen for several days to temperatures of approximately two-thirds of the material's melting temperature to reduce bulk damage and contaminants such as sulfur. As a final preparation step, the crystal is electropolished to produce an optically smooth strain-free surface. The crystal is then mounted in the UHV chamber, cleaned using low energy ion sputtering (< 1000 eV) and annealed in vacuum to ~400°C to remove sputter damage. The sputter-cleaning and annealing is repeated until all foreign elements are removed, as monitored with AES, and the surface appears well ordered.

Although attempts are being made to quantify the influence of surface topography on electron scattering, only a qualitative assessment of the effects of surface imperfection has been used in the work reported here. No LEED data were collected until the surface was clean and a sharp, well focused LEED pattern with low background intensity was observed. Using the 1 mm aperture, the spatial full-width at half maximum (FWHM) of a beam is limited by the Faraday cup's spatial resolution. This appears to be a satisfactory instrument resolution for measurement of LEED I-V spectra.[9]

Once the sample and electron diffraction system are optimized, a critical test for a LEED experiment is to compare I-V profiles for symmetrically-equivalent beams. Due to the various experimental limitations, perfect agreement is difficult to achieve. An illustration of this important fact is shown in Fig. 1, which is from our recent work on "normal incidence" LEED for Cu(100).[10] This figure illustrates that measured I-V profiles for beams that are related by symmetry can be noticeably different. Since changes in sample position for rotations of less than 0.1° about an axis in the surface further degrades the agreement, it is believed that the Cu(100) surface is aligned to be approximately normal to the incident electron beam for the data shown. Our work

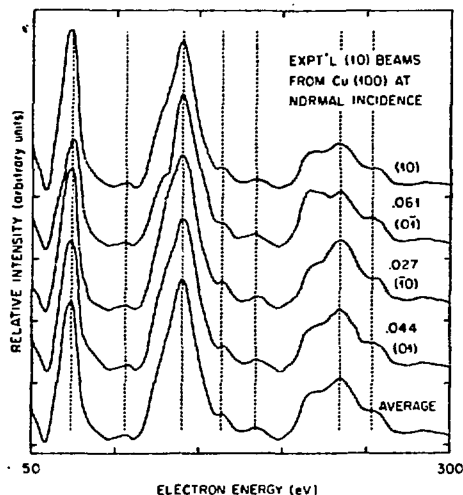

Fig. 1 Experimentally measured {10} profiles from Cu(100) and the average of the four. The single beam r-factors compare the other measured profiles with the (10) profile.

suggests that sufficient conditions for data acquisition are that the position of isolated peaks in a profile should agree within 1 eV in energy and ~10% in intensity, and that the overall shape of the curves should be nearly identical.

Another way to assess the quality of the data would be to compare profiles with single beam r-factors. The Zanazzi-Jona[8] r-factor has been used to compare the $(0\bar{1})$, $(\bar{1}0)$, and (01) profiles of Fig. 1 to the (10) profile. We have not attempted to define an r-factor criteria for the acceptability of the data for analysis, since we do not have a sufficiently large data base for a proper test, and the r-factors are not unique or relatable to an absolute measure at this time. Perhaps in the near future a more quantitative selection criterion will be available.[11] However, we have found that a simple, straightforward technique to obtain improved data for subsequent analysis is to determine the mean for the four "symmetry-equivalent" {10} profiles. Such a procedure produces the profile at the bottom of Fig. 1. Indeed, use of such averaged profiles leads to better agreement with calculated profiles.[5]

LIMITATIONS OF DATA

The data acquisition technique which we suggest is different from that advocated by others. Instead of collecting as many profiles as possible,[6,7] we suggest that data be collected for scattering geometries where symmetry-related beams exist; e.g., where the electron beam is incident normal to the surface.

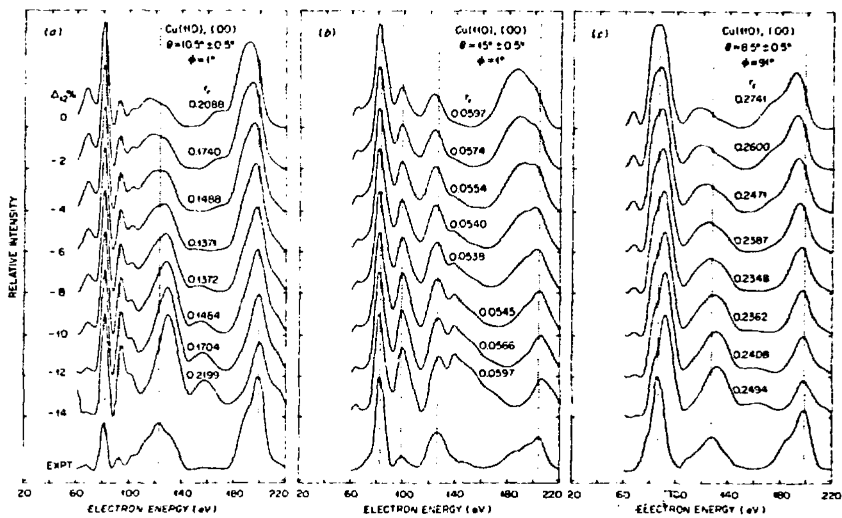

Fig. 2 Analysis of Cu(110) specular beam data at selected angles of incidence, (a) $\theta = 10.5°$, $\phi = 1°$, (b) $\theta = 15°$, $\phi = 1°$, and (c) $\theta = 8.5°$, $\phi = 91°$; $\phi = 0°$ is in the $(1\bar{1}0)$ azimuth.

One advantage of the proposed data collection technique is that effects of errors can be reduced. Two examples are given below to illustrate the effects of errors. First, an analysis of LEED specular beams is used to demonstrate that loss of accuracy in sample alignment significantly affects the analysis. In the second example, different structural conclusions are found when the separate individual profiles for the {10} set are employed in an analysis.

In previous work the structure of the Cu(110) surface was determined by analyzing the six lowest order LEED beams for normal incidence scattering.[12] A good agreement between theory and experiment, as determined by both a visual test and r-factor analysis, was achieved using calculated profiles for an approximately 10% contraction of the first interlayer spacing. Further work, analyzing specular beam scattering from the same Cu(110) surface, was performed in an attempt to verify this result.[13] Although results of the specular beam analysis were consistent with the normal incidence study, serious problems in analyzing data collected for nonnormal incident scattering were noted.

The three independent scattering geometries are denoted in Fig. 2. The scattering angles were determined by positioning the sample such that the measured angles for the specular beam and several selected nonspecular beams satisfied the surface grating

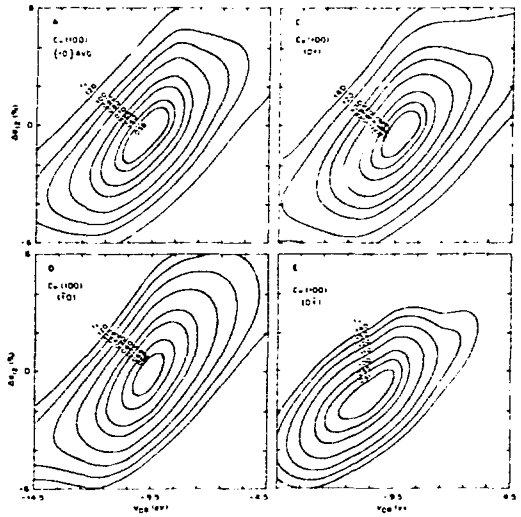

Fig. 3 Single beam r-factor analysis of the Cu(100) $\{10\}_{avg}$, (01) $(\bar{1}0)$ and $(0\bar{1})$ profiles.

equation. The (00) beam's profile was measured (bottom curve for each frame) and then compared with the results of model calculations. The calculated profiles of Fig. 2 are labeled with the values used for the first interlayer spacing contraction, Δ_{12}. All nonstructural parameter values were identical to those used in the previous "normal incidence" analysis. Each computed profile is compared with the experimental profile using the Zanazzi-Jona single beam r-factor. Results for the scattering condition θ = 8.5°, ϕ = 91° (ϕ = 0° is in (110) azimuth), Fig. 2c, illustrate the possible problem of the error due to misalignment of the Faraday cup with respect to the electron beam axis. The value θ = 8.5° was determined from the experimental measurements. As can be seen, the best agreement between calculated and experimental profiles is for an 8% interlayer contraction, although the r-factor minimum value of 0.235 is rather large. If it is recognized that the measured polar angle, θ, could be systematically different than the true scattering angle, it would be reasonable to compare the experimental profile with profiles calculated for other angles of incidence. In fact, by making the comparison using profiles calculated for θ = 9°,[13] the r-factor reduces to 0.15. It is important to note that the analysis of these specular beam data was much more sensitive to changes in polar angle than to interlayer spacing.

Although it is not directly related to equipment precision, Fig. 2 also serves to illustrate another possible problem with

the analysis of specular beam data. The measured profiles often are restricted by experimental limitations and are usually collected before the analysis is performed. However, as seen in Fig. 2b and 2c, the results obtained when analyzing some profiles can be relatively insensitive to changes in a structural parameter; e.g., an 8% change in the Δ_{12} values of Fig. 2b produces only a 0.006 change in the r-factor. Thus, insensitivity of the calculated results and apparatus imprecision can degrade a LEED analysis which employs nonnormal incidence data.

Data for Cu(100) can be used to provide another illustration of some possible limitations of experimental data.[5] If an experiment was performed and data collected for only one of the beams in, say, the set of four {10} beams, the structural conclusion obtained in the analysis can vary with the choice of individual beam. For example, Fig. 3 contains plots of r-factors obtained by comparing calculated profiles with experimental profiles for the individual (01), ($\bar{1}$0), and (0$\bar{1}$) beams, and for the average of the four measured {10} beams. These are contour plots of the single-beam r-factor as a function of interlayer spacing, Δ_{12}, and the real component of the optical potential. The ($\bar{1}$0) beam has its minimum r value when $\Delta_{12} \simeq 0\%$, while the (0$\bar{1}$) beam has its minimum r value when $\Delta_{12} \simeq -1.5\%$. Similar scatter is found when the individual profiles for other {ij} sets are analyzed. The scatter also increases when nonnormal incidence data are included in the analysis. On the other hand, the use of the averaged data for the four non-specular beams, obtained for normal incidence, not only produces low single-beam r-factors, but the scatter among the structural conclusions derived for each of the four nondegenerate {ij} sets is less than 0.3%, which corresponds to less than 0.01 Å, for Cu(100). So if one employed only profiles for individual beams in the analysis, the Δ_{12} values obtained for the various nondegenerate beams could have greater scatter than when averaged data was employed. Although the scatter might appear rather small for Cu(100), ~1.5%, it corresponds to greater than 0.025 Å. However, the use in the analysis of experimental profiles obtained by averaging data significantly reduces the range over which the optimum Δ_{12} is determined for different parameters.

REASONS FOR IMPROVEMENT

It has been discussed how data averaging leads to an improved agreement between calculated and experimental profiles, as verified by r-factor reduction and small scatter in the structural conclusion of the analysis. Such results by themselves might be sufficient reasons to continue with data averaging in LEED analysis, but some further comments can be made to show that the averaging technique has a firmer basis.

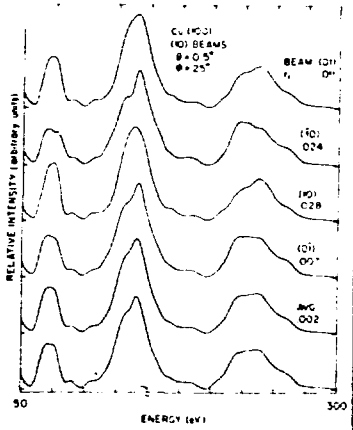

Fig. 4 Comparison between computed spectrum for the (10) beam from the Cu(100) surface at normal incidence with the computed profiles for the four {10} beams and their averages for a primary beam angle of incidence at $\theta = 0.5°$, $\phi = 25°$ ($\phi = 0°$ in the $(\bar{1}10)$ azimuth).

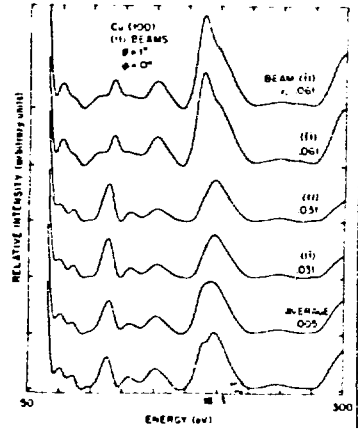

Fig. 5 Comparison between the (11) beam at normal incidence with the {11} beam and their average for beam incidence at $\theta = 1°$, $\phi = 0°$.

A simple reason why the data are improved follows from an elementary statistical analysis. Since one is determining a distribution by obtaining data for the individual symmetrically-equivalent beams, the variation of the mean profile from the true profile is reduced by $\sigma/\sqrt{N}$, where σ is the variance of the individual profiles from the population mean and N is the order of symmetry. Therefore, for the four-fold symmetry beams for Cu(100), the random error is reduced by one-half as compared with a single beam.

A more important reason for the data enhancement is related to the change of diffracted current with changes in the angle of incidence of the primary beam. If the reflection coefficient is a slowly varying function of the primary electron's angle of incidence, which seems to be a reasonable assumption, then energy removed from one diffraction beam due to a change of angle will be deposited into a symmetry related beam. This hypothesis can be tested both experimentally and by use of calculated results. An inspection of Figs. 4 through 6, provides a calculational test of this hypothesis. All but the bottom two curves of each of these

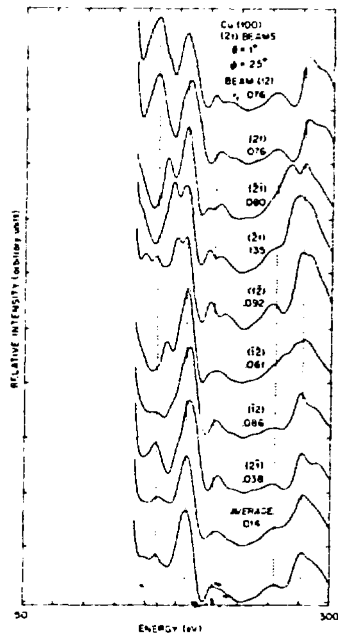

Fig. 6 Comparison between the (21) beam at $\theta = 0°$, with the eight $\{21\}$ beams and their average at $\theta = 1°$, $\phi = 25°$.

figures are calculated I-V profiles for the specified individual beams for the denoted angles of incidence. The profiles at the bottom of each figure was calculated for exact normal incidence. The single-beam reliability factor[8] has been used to furnish a quantitative comparison of all profiles with the profile at the bottom of each figure. In other words, the individual profiles and their average are tested to determine how well they agree with the profile for normal incidence, which is the experimentally desired alignment condition. Interestingly, misalignment of the surface by only 0.5°, a reasonable error using the current sample manipulator, produces visible changes in the individual profiles (see Fig. 4). The overall features are reproduced, but the relative intensities of closely spaced features are significantly altered. On the other hand, the average of the profiles for the $\{10\}$ set is almost identical with the normal incidence profile. One feature which should be noted is that the averaging process does not significantly broaden the spectral peaks, as verified by the very small r-factor comparing the average with the normal-incident profiles.

As seen by an inspection of Figs. 5 and 6, other calculated $\{ij\}$ sets also have similar changes in individual profiles caused by minor misalignment. Although the average of the $\{11\}$ set (see

Fig. 5) reproduces the normal incidence profile quite well, the 1° misalignment does cause some features not to be faithfully reproduced (e.g., the structure near 190 eV). The decreasing quality of agreement is still better illustrated by Fig. 6 for the {21} set. For a 1° misalignment the average profile is noticeably different from the normal incidence profile. These discrepancies serve to illustrate that caution must be applied when attempting to improve LEED data by averaging. First, there are limits beyond which the averaged profile no longer reasonably approximates the normal incidence profile. In addition, the higher order beams are significantly more sensitive to misalignment than the lower order beams. In fact, this may be one of the reasons that experimental profiles for the higher order beams usually have worse agreement with calculated profiles than the lower order beams in some LEED analyses. Although the use of the mean of the set of profiles has its limitations, it is preferable to employ the mean in an analysis over any one of the individual profiles. For example, if one employed in a normal incidence analysis the ($\bar{2}1$) profile of Fig 6, its large deviation (r-factor value of 0.135) from the normal incidence profile would certainly introduce errors into the analysis. However, the averaged profile has relatively small deviations from the normal incidence profile.

It is interesting to note that the differences between the calculated individual {10} profiles of Fig. 4 are similar to those between the measured profiles of Fig. 1. It is believed that one of the major sources of error in experimental profiles is surface imperfection. However, since surface imperfection could be viewed as a random distribution of domains which are misaligned with respect to other domains, differences between symmetry related profiles should be similar to differences introduced by misalignment. Thus, use of the averaged experimental profiles in an analysis might better represent a perfect surface.

RESULTS OF USING DATA AVERAGING

A thorough structural analysis of the Cu(100) surface has been performed using the averaged experimental profiles.[5] In this analysis an excellent agreement has been obtained between the averaged experimental and calculated profiles (see Fig. 7). In addition, the multi-beam r-factor corresponding to Fig. 7 is 0.044, which is the smallest value reported to date for any LEED analysis. It should be mentioned that one alternative method for the analysis would be to collect data for the same number of beams as required to obtain averaged profiles, but analyze each separately and then statistically analyze the totality of results. Since the same beams would be included in the analysis it is believed that the same structural conclusion would be obtained by both methods. However, instead of a large number of

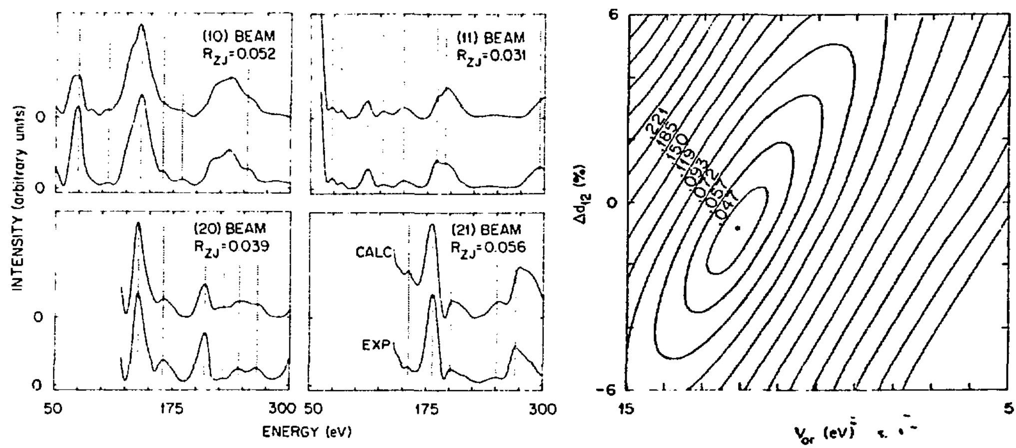

Fig. 7 Summary of the LEED analysis of the Cu(100) surface using the four averaged, experimental LEED beams. The topograph compares the four beam r-factor, as defined by Zanazzi and Jona, as a function of interlayer spacing, Δ_{12}, and real part of the optical potential, V_{or}.

individual profiles to analyze, with use of averaged profiles only four needed to be analyzed. This reduced the time and effort required by the analysis. Important results obtained by using the averaged profiles are the self-consistency between analyses for profiles of the different indexed beams and the insensitivity of the total data base to variation of nonstructural parameters. For example, using a given set of nonstructural parameters, the structural conclusions for the four averaged profiles all agreed to within less than a 0.01 Å change in the first interlayer spacing. At the same time, when comparing the structural conclusions obtained for different sets of nonstructural parameters, the distribution in spacings was contained within a range of ± 0.02 Å of the preferred value.[5]

Additional analyses using data averaging are now being performed.[14,15] The r-factors for each case are reduced by the use of averaged experimental profiles. A six-beam r-factor of 0.095 has been obtained for Ag(110), which is to be compared to the value of 0.17 obtained using unaveraged profiles.[14] The laser irradiation stabilized Si(111)-(1x1) surfaces is also being analyzed.[15] Although the analysis is quite preliminary, this metastable "(1x1)" surface appears to have a large contraction in the first interlayer spacing and an expansion in the second

interlayer spacing. At present, a Zanazzi-Jona r-factor of 0.115 has been achieved, which in part is due to the use of the averaged experimental profiles.

CONCLUSIONS

One current limitation to LEED structural analysis is the accuracy of the experimental data. Of course, refined data collection techniques are the preferred way to obtain improved experimental profiles, and new LEED chambers with high precision goniometers are beginning to be designed and placed in operation. However, an averaging technique can be applied, which seems to improve the quality of the data beyond that obtained using present-day instrumentation. By measuring all symmetrically equivalent LEED profiles and determining an average profile, the effects of errors on the data appear to be considerable reduced. Such averaged experimental profiles have been used in a thorough analysis of the Cu(100) surface. This determination of the value for the first interlayer spacing for Cu(100) appears to be sensitive to within ± 0.02 Å. More complex surfaces such as the Ag(110) surface and the Si(111)-(1x1) surface are being analyzed using averaged experimental profiles, and similar structural sensitivity seems to exist in these analyses.

ACKNOWLEDGMENTS

The authors would like to thank Drs. L. H. Jenkins and D. M. Zehner for helpful discussions.

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