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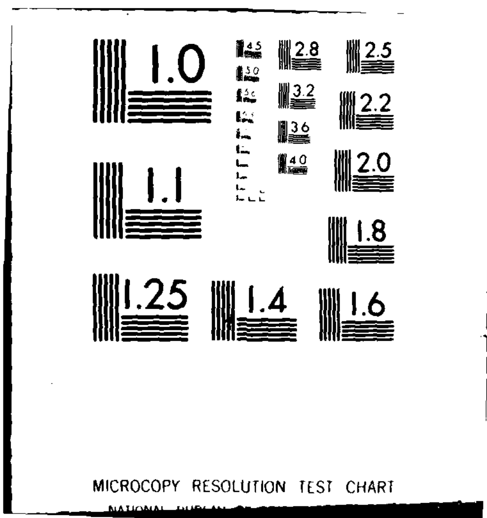
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TECHNICAL REPORT #4

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Ga and As in Cleaved and Sputtered GaAs(110)

by Means of Auger Line Shapes*

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ABSTRACT

Site-specific densities of states for the Ga and As sites in GaAs(110) are derived from the $M_1M_{45}V$ Auger lines and compared for the cleaved and sputtered surfaces. X-ray photoelectron spectroscopy, scanning electron microscopy, and LEED results for these surfaces are also presented. The results are interpreted in terms of a structure for the sputtered surface that consists of a two-phase mixture of Ga and disordered GaAs.

Introduction

The nature and extent of interfacial chemical reactions are major factors affecting the electronic properties of semiconductor-metal or semiconductor-semiconductor interfaces. Studies have recently been made to investigate aspects of this problem in a variety of systems.[1-5] A number of techniques have been used in these studies, including some that give structural information [low-energy electron diffraction (LEED), transmission electron microscopy (TEM), reflection high-energy electron diffraction (RHEED), and x-ray diffraction] and others that give electronic, chemical, or elemental information [x-ray and ultraviolet photoelectron spectroscopy, (XPS, UPS) and Auger electron spectroscopy (AES)]. There has, however, been little effort in correlating structural and chemical properties. It is well known that the extent and rate of chemical phase formation at a surface or interface may depend strongly on the surface or interface structure and the extent and nature of structural defects. It is even possible that new, chemically distinct phases may form because of certain kinds of damage that change the surface stoichiometry. Changes in reaction rates for ordered and disordered surfaces have been observed.[6] However, because of the difficulty in identifying different types of surface defects, it has not been possible to relate the chemical activity of a surface to specific types of structural damage.

The defect structure at semiconductor surfaces has been much less extensively investigated[7-10] than their other properties. In fact, except for an early paper,[11] only recently has the analysis of surface defects attracted significant attention.[12] LEED has been the major tool in these studies, with most applications

directed toward steps.[13-16] Recently also other types of surface extended defects, such as finite island sizes, domains, strain, and antiphase boundaries, have been quantitatively identified.[17,18] As a result of these studies, it now appears possible to measure the major elements of the defect structure at compound semiconductor surfaces and in epitaxial films.[19]

A first step to understanding defect-related chemical phase formation in epitaxial (as well as nonepitaxial) overlayers is a correlation of local chemical properties with structural defects at a clean surface. We present here initial attemps to do so by comparing Auger line shapes for cleaved and sputtered GaAs(110) surfaces and the corresponding XPS-determined valence band density of states with LEED and scanning electron microscopy (SEM) measurements. The most widely used technique to identify chemical states at a surface has been photoelectron spectroscopy (PES). Shifts in core-level XPS spectra and features in both valence band UPS and XPS spectra give this information. AES provides in addition the possibility of observing chemical properties that are site-specific.[20] Core-level chemical shifts can in principle also provide some site-specific information. Because core level shifts result from charge transfer, a charge redistribution within the valence band without significant charge transfer to neighboring atoms, e.g. because of the rearrangement of atoms at a surface, may lead to unmeasurably small chemical shifts. We are aware of only one study of core level chemical shifts associated with surface structural rearrangement.[21] While photoemission valence band spectra show observable changes with surface structural rearrangement, for multicomponent materials they do not give site-specific information, because the photoelectrons are

the result of excitations from extended states that include contributions from widely separated sites.[22] On the other hand, because the probability of existing an Auger electron falls off as $1/R^6$ away from the core hole, Auger transitions involving the valence band view the charge density in the valence band preferentially around each ion core.[20] Thus it is possible to obtain the site-specific density of states around different ion cores in the surface of multicomponent materials. We have domonstrated this for several materials.[23,24] In addition, the rearrangement of charge associated with a rearrangement of atoms in the surface of a compound or any multicomponent system may lead to large changes in the Auger line shapes from the individual ion cores even when there is insufficient charge transfer to observe a chemical shift. We report such a result here.

In addition, because Auger spectroscopy is sensitive to only the outer layers of a solid, the electronic structure of the surface layer is predominantly measured. Thus different binding states for a species on the surface should be mirrored in the Auger line shapes.

GaAs(110) surfaces have been extensively studied.[9,25-27] Some work has been done on sputtered GaAs(110).[9,27] The band structures of both crystalline and amorphous GaAs have been determined by comparison of photoelectron energy distribution spectra with calculations.[28] In the next section we briefly describe the various experimental techniques that we have used. In Sec. III the results of the Auger line shape measurements are presented. In Section IV the changes in the site-specific densities of states between the sputtered and cleaved surfaces are discussed and an attempt is made to correlate these changes with the observed structural changes.

II. Experimental

All measurements were made on (110) faces of undoped GaAs. Crystals were cleaved in situ using the knife edge and anvil technique, at pressures in the high 10^{-8}Pa (10^{-10}Torr) range. No surface contaminants were seen after cleavage within the detectablility limits of AES. The crystals were subsequently sputtered with 2KeV Xe ions for approximately 10 minutes.

The Auger line shape data were taken with a PEI single-pass cylindrical mirror analyzer with coaxial electron gun. Modulation voltage was 1eV peak-to-peak. The Ga and As $M_1M_{45}V$ transitions, in the kinetic energy range 115-160 eV, were recorded in the dN/dE mode. To obtain a sufficiently good signal-to-noise ratio, the data were signal averaged for approximately one hour. They were then transferred via paper tape to a computer for data reduction.

The XPS measurements were made in a separate chamber using an unmonochromatized Al source and a double-pass CMA with retarding grids operated at a resolution of 1.5eV. Sampels were cleaved in situ and then sputtered. Both valence band and 3d core-level spectra were recorded in the N(E) mode. Standard pulse counting techniques were used with signal averaging up to 2 hours.

LEED patterns could be obtained in the chamber used for the Auger line shape measurements. Because the LEED optics in this chamber are rather crude, LEED was used only to ascertain that the diffraction spots were "sharp", i.e., that a cleave was reasonably good, or that no spots existed after sputtering. Quantitative LEED measurements were made in a separate chamber(29) that uses a vidicon camera to scan the angular profile of a diffraction spot. Subsequent on-line computer analysis allows interpretation of the angular width of reflections in terms of steps and other extended defects. Results for cleaved and sputtered GaAs(110) have been presented elsewhere.(14)

Scanning electron microscopy (SEM) measurements were made on sputtered and annealed GaAs(110) surfaces, produced in the above experiments, on an instrument equipped with a field emission source. Data on the surface morphology were obtained also as a function of partial reannealing of the surface.

Both AES and XPS measurements suffer from several distortions. For AES, these include the finite resolution of the analyzer, the inelastic scattering as the Auger electrons propagate through the lattice, and the finite lifetime of the core levels involved in the "down" transition. XPS spectra obviously do not involve other levels, but there is the additional contribution of the finite linewidth of the x-ray source as well as any satellite emission lines if the source is not monochromatized. In both cases there will be an incoherent background due to cascade processes from transitions occurring at higher energies. These will be negligible for photoelectron spectra of the valence band, however, since there are no higher-lying transitions.

Auger and XPS spectra were corrected for these distortions using a procedure pioneered by Mularie and Peria[30] and developed by Madden and Houston.[31] We have applied this method to deconvolution of Auger spectra[23,24] as well as XPS core-level[32] and valence-band spectra,[35] and have considered the reliability of the technique in some detail.[24,32] Briefly, in this procedure, analyzer and loss broadening are modeled by an electron backscatter spectrum taken using the same experimental parameters as for the data and with a primary energy equal to that of the transition of interest. This spectrum is then deconvoluted from data using the van Cittert algorithm.[34] To correct for core-level broadening of the Auger lines, we have deconvoluted additionally a Lorentzian with full width at half-maximum (FWHM) equal to the sum of the FWHM's of the core levels involved in the transition.[32] This gives the site-specific valence band density of states. For the XPS spectra the

achromaticity of the Al source, which causes both a simple broadening of the features in the data because of the finite width of main $K\alpha_{12}$ x-ray line and a repetition of the major features of the data with 10% of the intensity at approximately 10eV higher in kinetic energy because of the $K\alpha_{34}$ emission, was taken into account in a similar manner. A source function that represents the emission from our x-ray source(32) was generated and convoluted with the backscatter spectrum. This function was then deconvoluted from the XPS data to give the corrected valence band DOS.(33)

The Ga and As Auger lines for a given surface were measured on the same scan. Because they are close enough in energy, a single backscatter spectrum was recorded and used in the deconvolution process for both lines. We have previously checked that diffraction effects did not distort the loss spectrum over the energy range of these two lines.(23,24) This procedure preserves the relative intensities of the lines for a given surface. To compare the intensities of the lines for the sputtered and cleaved surfaces, a normalization is required. The Ga and As lines may individually change in unknown ways when the surface is sputtered. However, if it is assumed that the total charge at the surface is constant and that the matrix elements do not change, the area under the sum of the Ga and As lines should be constant for the two surfaces. This normalization was used to obtain the relative intensities of the individual lines.

III. Results

Figure 1a shows a scanning electron micrograph of the surface of a cleaved single crystal of GaAs after it had been sputtered. Apart from the macroscopic fracture regions that occur infrequently on the cleavage

face, there are additional, smaller defects scattered over the flat face that we believe to be Ga bubbles. These do not occur on a face that has only been heated (at least below 550°C, the highest annealing temperature we have used) and also not on the freshly cleaved face. They thus appear to be a result of sputter damage. In addition to the large bubbles, which are on the order of 1μm in size, there is a larger concentration of smaller bubbles. The estimated surface coverage is 20%. Interestingly, if a sputtered surface is annealed, some of these Ga bubbles preferentially align along crystallographic directions that may be step edges. Figure 1b shows an example. The alignment direction is [100], determined from x-ray and LEED analysis. The bubbles themselves become oblong, with their major axis along this direction.

Figure 2 shows the LEED results for a sputtered and a cleaved surface.(14) The angular width of the beams diffracted from the cleaved surface is almost instrument-limited, indicating large coherent regions >500Å in diameter. The angular width of the diffracted beams from the sputtered surface shows a marked oscillation with energy, an indication of steps.(13,14) In addition, it shows broadening beyond that due to steps, indicated by the rise in minimum achievable width relative to the cleaved surface as the energy of the incident electron beam increases. This is evidence for finite-size coherent domains or misalignment of domains on the surface.(17) A GaAs(110) LEED pattern is observable for this and all higher anneals.

The XPS valence band spectrum for cleaved GaAs(110) is shown in Figure 3 along with the Ga and As $M_1M_{45}V$ Auger transitions and their sum. The Auger results are slightly different than those reported earlier(23) because of a more accurate determination of the core level broadening(24)

(as well as the correction of an error in the computer program that caused a distortion in the deconvoluted spectrum). The position in energy of these transitions with respect to each other is determined by the respective core level binding energies; the shift with respect to the XPS spectrum is determined by aligning peak B with the respective XPS peak. This overall shift is within 2eV of that predicted from the core level binding energies and a CMA work function of 4.9eV. The intensity of the XPS spectrum is scaled so that it agrees with that of the sum of the Auger site-specific density of states at peak B.

Because, as already mentioned, the probability of exciting an Auger electron falls off a $1/R^6$ away from the core hole,[20] a Ga CCV transition should reflect valence band states local to the Ga ion while an As CCV transition should reflect those local to the As ion. The photoemission process, on the other hand, results in emission from extended states in R and thus is not site-specific. The Auger line shapes demonstrate that peaks B and C are localized around the Ga and As cores respectively, although there are some contributions to each of these peaks from the other constituent. Conversely there appear to be roughly equal contributions for each atomic species to peak A, although the low probability of emission makes such a conclusion tentative. In addition, because of the very short mean free path of these Auger electrons, the density of states local to the surface is measured. Thus these line shapes should reflect the charge configurations for the surface and near-surface atoms.

The deconvoluted XPS valence band spectra for cleaved and sputtered GaAs(110) are shown in Figure 4. The spectra are normalized by requiring that the sums of the peak heights of the Ga and As 3d lines are the same

for the two surfaces. Several differences between the XPS spectra of the cleaved and sputtered surfaces can be noted. For the sputtered surface, both peak A and peak C are reduced in amplitude. The decrease in intensity of peak A is not uniform across its width; those states at higher energy are reduced more than those at $\sim$-2eV. Unlike the other peaks, peak B increases in amplitude upon sputtering and becomes approximately twice as broad. The area under the curve remains approximately constant, an indication that our normalization for the Auger lines is reasonable.

A different perspective on these changes can be obtained by looking at the site-specific densities of states derived from the Auger line shapes. Figures 5 and 6 show a comparison of the densities of states for the cleaved and sputtered surfaces, respectively around the As site and the Ga site, derived from the $M_1M_{45}V$ transition. As mentioned earlier, the $M_1M_{45}V$ line shapes were normalized by requiring that the sum of the areas of the Ga and As lines be the same for both surface conditions.

There are comparatively few differences between the sputtered and cleaved As site-specific transition DOS's aside from an overal reduction in intensity, as shown by the difference curve in Figure 5. There is an indication, however, that the change in shape of peak A observed in the XPS data is also observed in the leading edge of the As line here.

Considerably greater changes are noted in the corresponding Ga site-specific transition DOS (Figure 6). The Ga contribution to peak C is significantly reduced, even though the overall intensity in the spectrum has increased. Conversely, the Ga-like character of peak A has increased. The Auger results also indicate an increase in both the intensity and the width of peak B.

IV. Discussion

The origin of the major peaks in the photoemission sprectrum of GaAs is well established by comparison of these measurements with calculations. Peak A consists of predominantly bonding p-like states, with the highest-energy states preferentially associated with the As sites. Peak B consists largely of Ga states, with a small admixture of As p-states. Peak C consists predominantly of As states, with some Ga s and p-states.[28]

The Auger line shapes for the cleaved surface mirror these assignments. The small amplitude of peak A in the Auger results is a matrix element effect. It causes the s-like states to dominate the valence band sprectrum determined from $M_1M_{45}V$ transitions in these elements.[20] Different degrees of hybridization of s-like and p-like states would also affect the intensity of this peak.

Most of the changes in the valence band DOS upon sputtering can be explained by preferential sputtering of As atoms, resulting in a Ga-rich surface. The area under the Auger lines indicates an As depletion of 20%, corresponding to an average near-surface composition that is 60% Ga and 40% As. An As depletion and Ga excess is borne out by a similar decrease in the As KLL Auger line, and an increase in the Ga KLL Auger line. The average Ga enrichment estimated from the XPS spectrum of the 3d core level is 15%.

The composition range over which GaAs can exist as a single phase is quite narrow, ranging from 49.935 to 50.015 atomic percent Ga.[35] Any Ga in excess of this composition range will form a second phase. This second phase is evident as the bubbles in Figure 1. We cannot separate Auger electrons emitted from a Ga ion in a GaAs environment from those emitted by a Ga ion in a Ga environment. Consequently the Ga line shape that we

have measured for the sputtered surface will be the weighted sum of the densities of states at the two types of Ga sites. This line shape is consistent with such a sum. The Ga density of states[36] has a sharp rise near the valence band edge, but is otherwise essentially featureless. In the line shape for the sputtered surface, a considerable increase in intensity is seen in peak A where the majority of Ga-Ga bonding states would be found. Conversely, the hybridization with the As s-like states in peak C due to Ga-As bonds is reduced. The relative intensities of this peak in the $M_1M_{45}V$ line shapes for the cleaved and sputtered surfaces are approximately in the same ratio as the amounts of Ga in the GaAs and Ga phases determined above. The overall broadening of the line shape in general and of peak B in particular for the sputtered surface are indicative of the disorder induced in the GaAs phase.

The As line shape for the sputtered surface, on the other hand, will result only from As atoms in GaAs, because there should be no other chemical state of As present. We can not exclude the possibility, however, that some As-As bonds are present in the sputtered GaAs phase. Because the average energy of these bonding states is lower than that of the Ga-As bonding states[37] the relative increase in emission at $\sim$149eV in the As $M_1M_{45}V$ line shape may indicate a small amount of As-As bonding in the disordered GaAs phase. A small overlap at the low-energy end of the As line with the Ga transition will prevent seeing the bonding-antibonding split of peak C that is reported to be also present with As-As bonds.[37]

The disordering of the GaAs phase by sputtering is reflected in the increase in the width of peak C in the As line shape.

The XPS valence band DOS shows many of these same sputtering-induced changes, but not in the same detail. The observed changes have generally been explained only in terms of disordering. The changes are also consistent with two-phase coexistence arrived at from the Auger and SEM results. Thus the decrease of peaks A and C in the XPS valence band results can be ascribed to As depletion, because these peaks are mainly due to As states. On the other hand, the decrease of the Ga contribution at peak C is masked by the decrease of the As contribution, and the increase of the Ga contribution at peak A is masked by the decrease of the As contribution in that peak. Thus the XPS valence band DOS gives no evidence for two types of Ga environments, and it would have been difficult to arrive at this conclusion without the Auger line shapes.

The LEED results are also consistent with this description of the sputtered surface, although the evidence is circumstantial. Figure 2 showed the full width at half-maximum of the angular distribution of intensity in a LEED reflection. As mentioned earlier, oscillations in this width as a function of energy indicate the existence of steps[13,14] and a rise in the minimum width above the instrumental width indicates the existence of misorientation or finite-size effects.[17] The results for the cleaved surface are near the instrumental width and show no oscillations, indicating that this surface is free of steps and extended defects within the ability of the instrument to resolve them, which is about 500 Å in this case. The sputtered surface shows a large density of steps and also additional broadening. In all cases of sputter-annealed surfaces, the angular broadening remains larger than that for a cleaved surface.[14] Thus even though no differences are observable in I-V profiles,[27b] the angular profiles indicate surface imperfections in the annealed surface that are not present in the cleaved surface. We have not so far been able to say that

this broadening is due to the mixing of two phases or due to possible misorientation or finite-size effects in stoichiometric GaAs itself. This depends on the distribution of the second phase on the surface and more importantly on whether the second phase causes different regions of the GaAs to be incoherent with each other.

We have so far interpreted the results in terms of a two-phase mixture of Ga and GaAs on the surface. Another possibility that has been suggested is that a more or less continuous Ga layer forms on the surface. Although this possibility can not be absolutely eliminated, there is evidence against it. We are able to observe the GaAs LEED pattern except for the most severe damage, with ordered regions in the order of 50-150Å. If more than half a monolayer of Ga is deposited on GaAs, the LEED pattern disappears. Hence if the Ga is uniformly spread over the GaAs surface, there must be less than half a monolayer of it and it must be dispersed so that it does not affect the order of the GaAs underneath. This model would also severely limit the excess sputtering. The Auger line shapes are consistent with a Ga metal contribution, but this may, of course, also be true for a partial excess of Ga on the surface. This question could be answered only by depositing a fraction of a monolayer of Ga on the surface and observing the Auger line shape changes.

In summary, we have measured Auger core- core-valence line shapes for the individual atomic species in GaAs for two structural conditions of the surface, cleaved and sputtered. We have demonstrated 1) that the line shapes of transitions from the individual species are different and that these line shapes can be interpreted in terms of a site-specific density

of states; and 2) that the line shapes of transitions from the individual species change in different ways as the surface structure is changed. We believe these changes are consistent with a model of the sputtered surface as a two-phase mixture of Ga and GaAs, which is supported by SEM and LEED observations. This result cannot be derived solely from the valence-band photoemission spectra.

The surface structure modification by sputtering is admittedly severe. Less severechanges can be achieved by partially reannealing the surface, or by adsorption onto the surface. We have already made LEED(17) and SEM correlations of the structural changes with annealing. We are making Auger line shape measurements as a function of annealing to test the sensitivity of the Auger line shapes to the degree of structural disorder and the type of defect present. We also have measured Auger line shapes for O adsorption on GaAs and observed different changes in the Ga and As lines as a function of O exposure. These results will be reported elsewhere.(37)

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Figure Captions

Fig. 1. Scanning electron micrographs of a sputtered GaAs(110) surface. a) Unannealed surface, showing macroscopic fracture regions and smaller features that are Ga-rich; b) Partially annealed surface, showing preferential alignment of bubbles along line defects in the surface.

Fig. 2. LEED measurements of surface defects on cleaved and sputtered GaAs (110). The full width at half-maximum of the angular distribution of intensity in a reflection is plotted versus energy of the incident electrons. Dashed line: instrumental limit. Solid circles: cleaved surface, showing that it is free of extended defects to the extent that the instrument is able to resolve them. Squares: sputtered surface, showing large oscillations in width and a minimum width above the instrumental limits. These two effects are interpreted respectively as steps and finite-size domains or domain misorientation.

Fig. 3. Comparison of valence-band density of states in GaAs determined from XPS and Auger line shapes. Solid curve: XPS; dotted curve: Ga site-specific DOS; dot-dashed curve: As site-specific DOS; dashed curve: sum of the latter two, representing the total valence band DOS.

Fig. 4. Comparison of XPS measurements of the valence band DOS for cleaved and sputtered GaAs(110).

Fig. 5. Comparison of As site-specific DOS for cleaved and sputtered GaAs (110) and difference curve.

Fig. 6. Comparison of Ga site-specific DOS for cleaved and sputtered GaAs (110) and difference curve.

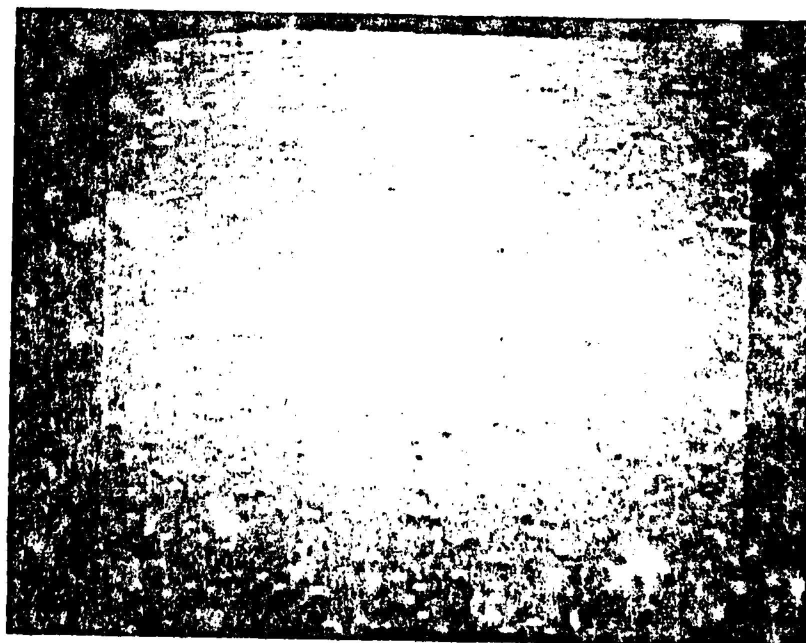

FIGURE 1.

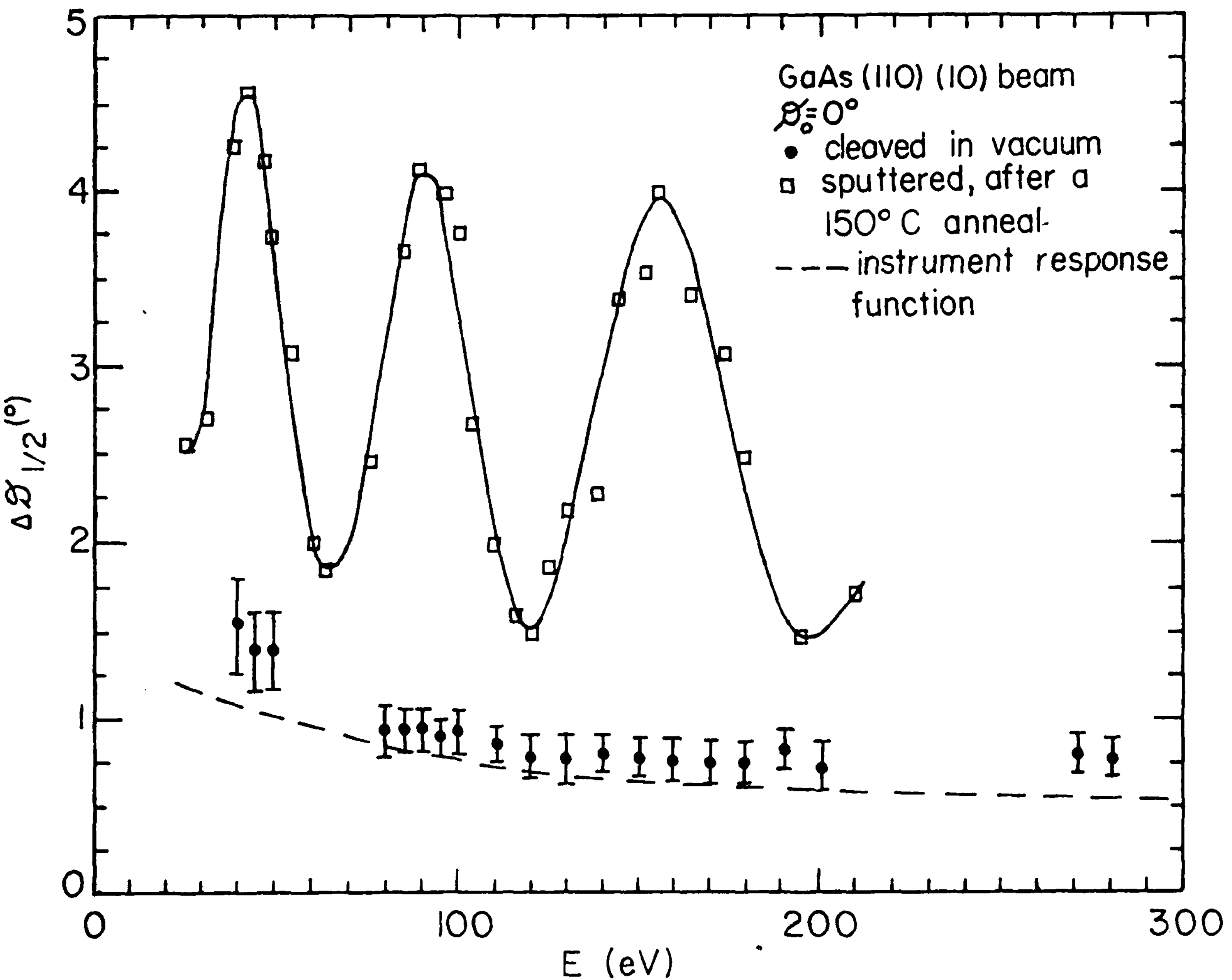

FIGURE 2.

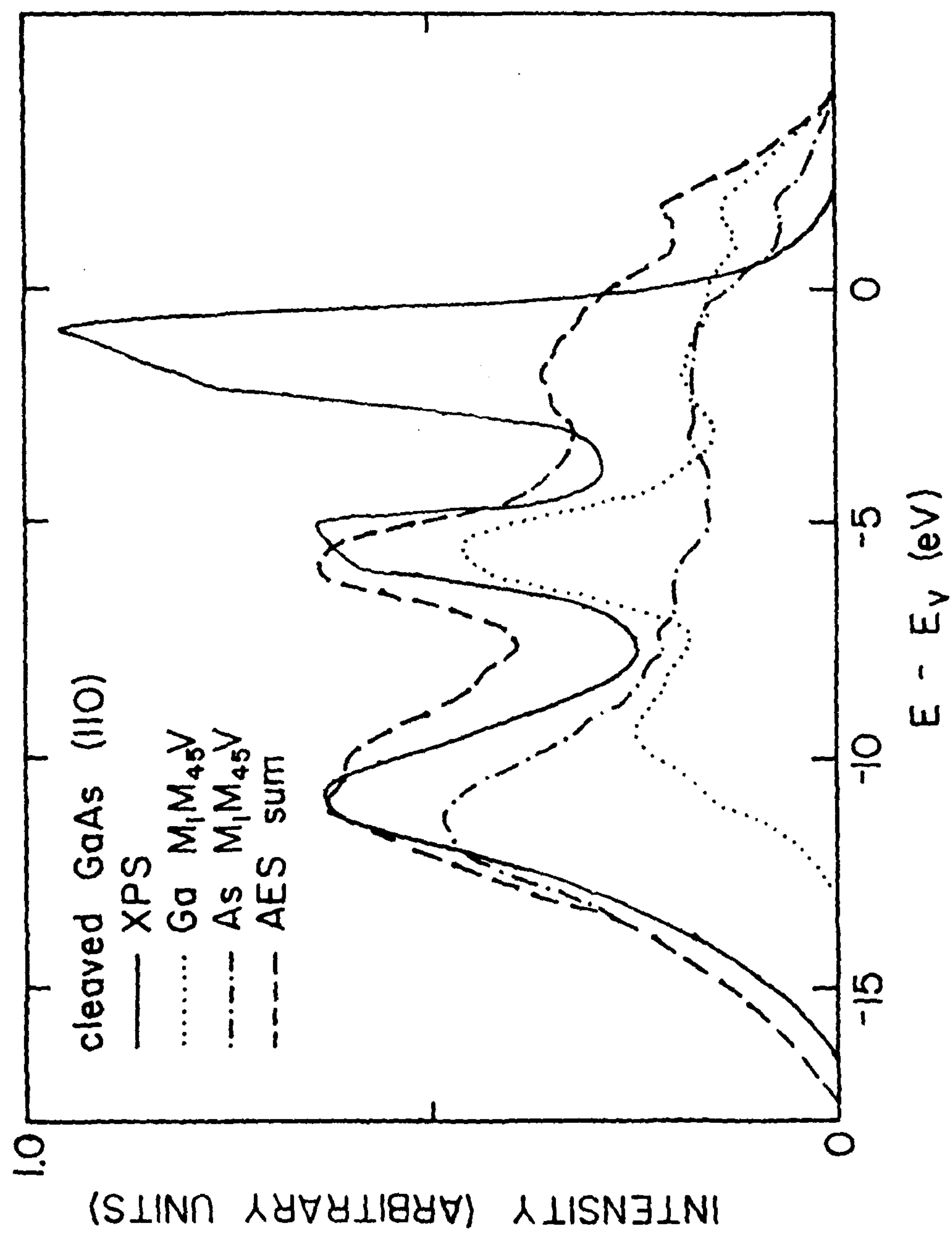

FIGURE 3.

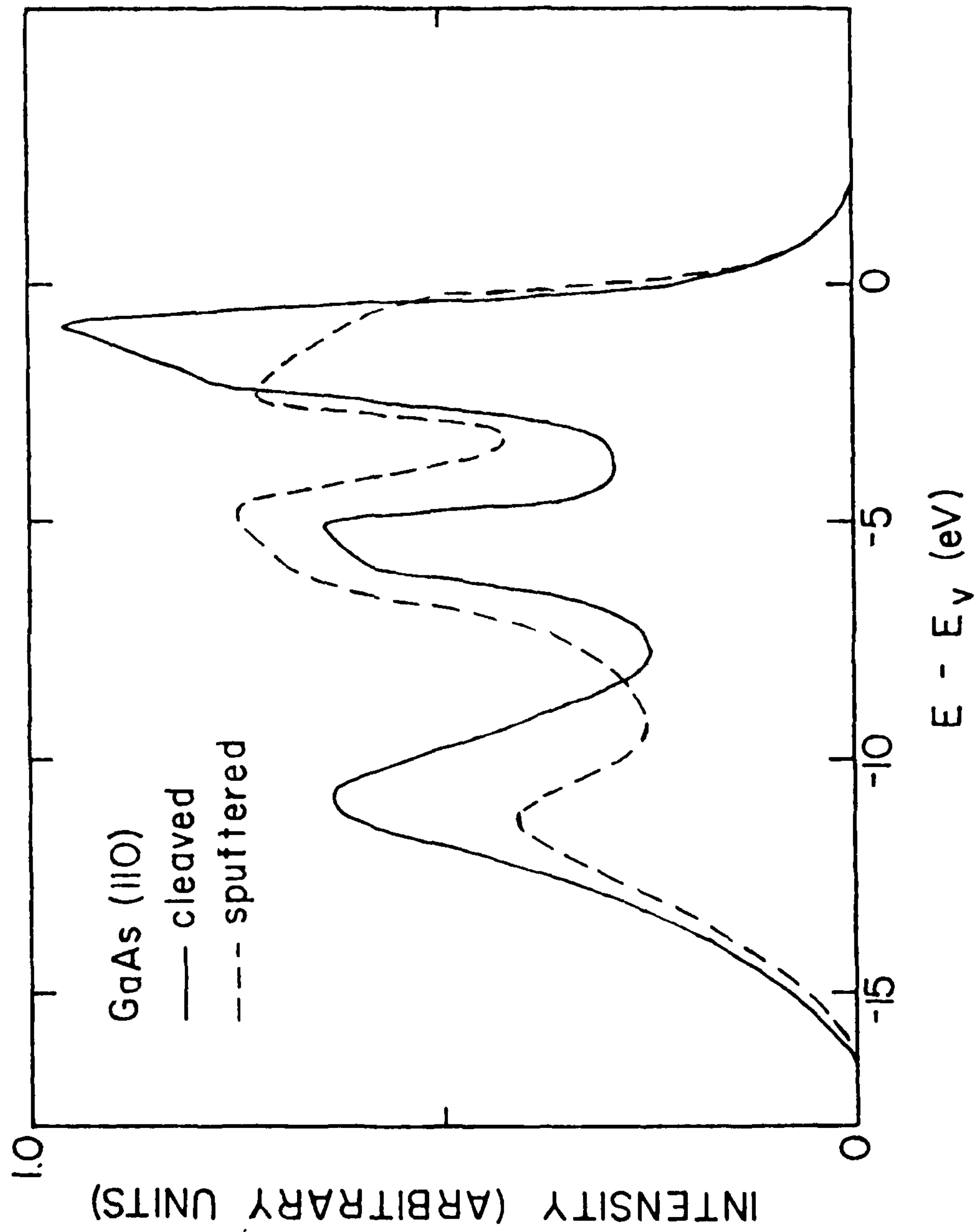

FIGURE 4.

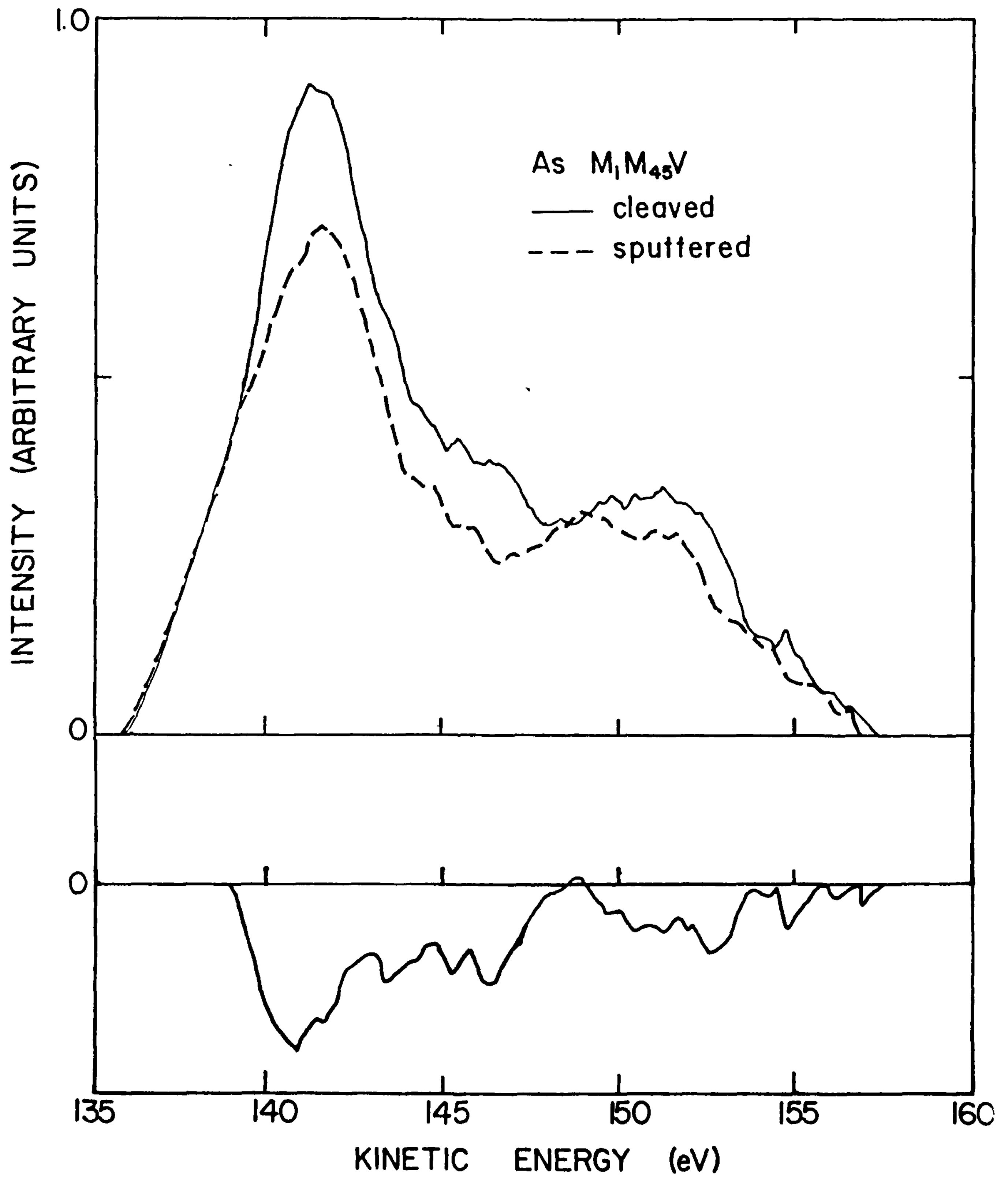

FIGURE 5.

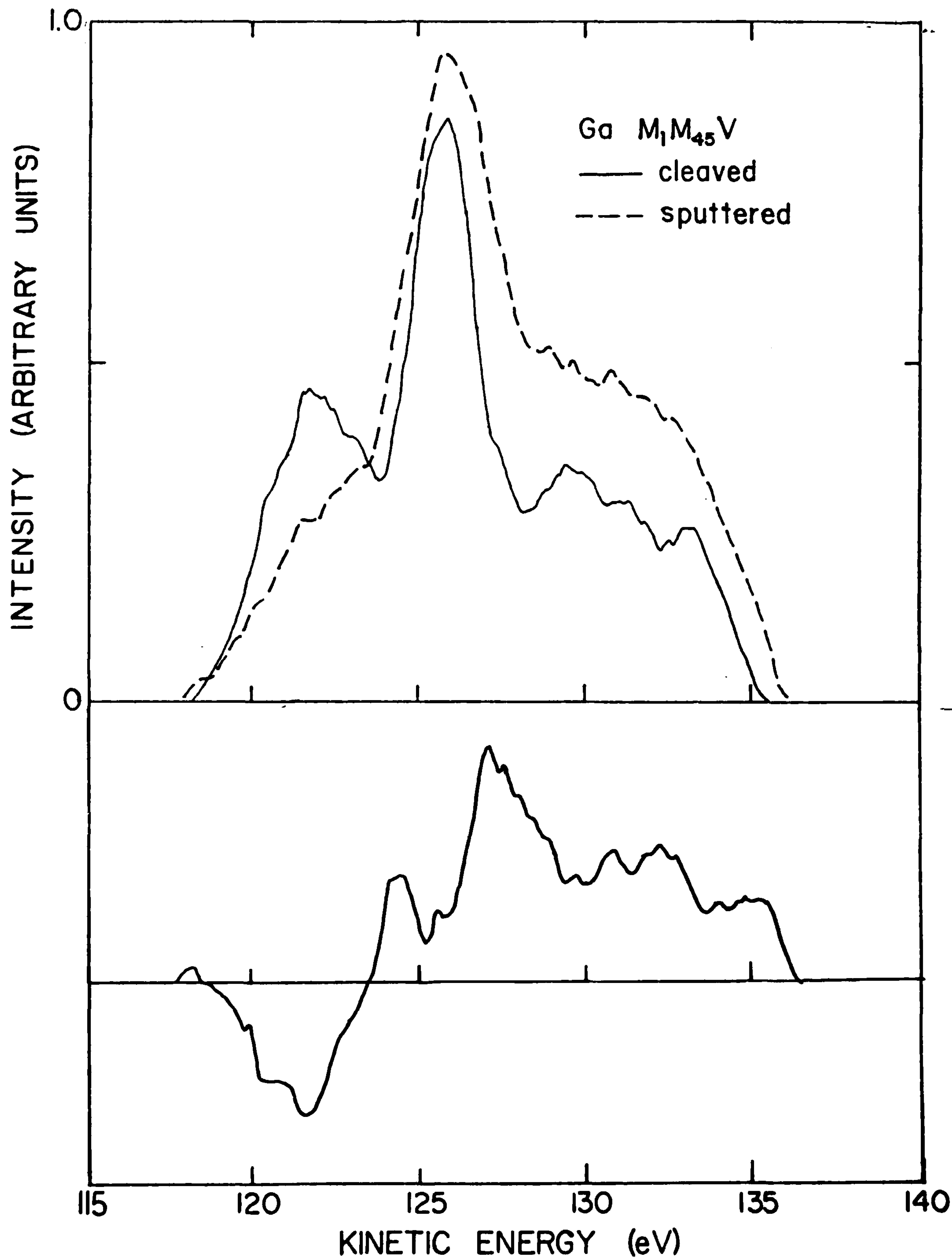

FIGURE 6.

-8