Electron-momentum spectroscopy of the core state of solid carbon

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Electron-momentum spectroscopy [binary encounter $(e, 2e)$] experimental results are presented for the core state of an amorphous carbon allotrope. The $(e, 2e)$ cross section has two identifiable regions. One is a narrow-energy-width core-band peak that does not disperse with momentum. At higher binding energies there is an energy-diffuse multiple-scattering continuum, which is a consequence of $(e, 2e)$ collisions with core electrons that are accompanied by inelastic scattering of one or more of the incoming or outgoing electrons. Comparisons of experimental momentum distributions with the Hartree-Fock atomic carbon $1s$ orbital are presented for both regions.

I. INTRODUCTION

$(e, 2e)$ reactions are electron impact ionization collisions in which the kinematics are completely determined. Specifically, the kinematical parameters that must be defined or measured are as follows: incident electron kinetic energy E_0 and momentum vector $\mathbf{p}_0$, scattered electron kinetic energy E_A and momentum vector $\mathbf{p}_A$, and ejected electron kinetic energy E_B and momentum vector $\mathbf{p}_B$. One arbitrarily defines the "scattered" electron to be the faster of the two outgoing electrons and the "ejected" electron to be the slower one.

At high incoming and outgoing electron energies and large momentum transfer $\mathbf{K} = \mathbf{p}_0 - \mathbf{p}_A$, the $(e, 2e)$ cross section is dominated by binary electron-electron collisions, and is well described by the plane wave impulse approximation.[1-3] This $(e, 2e)$ reaction kinematical regime is conducive to electron-momentum spectroscopy, since the cross section is directly proportional to the square magnitude of the momentum space overlap between the many body initial target and final ion states. If the initial target state has negligible electron correlations, the $(e, 2e)$ cross section simplifies to be directly proportional to the square magnitude of the momentum space orbital of the target electron. In this circumstance, the proportionality factor, or spectroscopic strength, is essentially the probability that the final ion state is a particular independent particle hole in the initial target state.

The binary $(e, 2e)$ cross section for condensed matter targets is proportional to the *spectral momentum density*.[1,4] Specifically, for crystals, an occupied band n with band structure $\varepsilon_n(\mathbf{q})$, is characterized in $(e, 2e)$ experiments by the observed momentum $\mathbf{q} = \mathbf{p}_A + \mathbf{p}_B - \mathbf{p}_0$ in association with the observed binding energy $\varepsilon_n(\mathbf{q}) = E_0 - E_A - E_B$. The spectral momentum density for general condensed matter, including disordered phases, is discussed by Caprari.[4]

To our knowledge, the first $(e, 2e)$ experimental observation of the carbon core state ($1s$ atomic orbital) was the coplanar symmetric experiment on Formvar

$[CH_2(OCH_3)_2]$ reported by Amaldi *et al.*[5] By scanning the incident electron energy over an 800 eV range, and keeping each outgoing electron energy fixed at 7.3 keV, three broad peaks corresponding, respectively, to the totality of valence molecular orbitals, the carbon $1s$ orbital, and the oxygen $1s$ orbital, could be distinguished within the $(e, 2e)$ cross section. The cross section was afflicted by severe peak overlap, due to the inadequate binding energy resolution of 135 eV full width half maximum (FWHM). Nevertheless, the differences in the $(e, 2e)$ cross section at two momentum values revealed with clarity the much greater momentum space extent of the carbon and oxygen $1s$ orbitals compared to the valence molecular orbitals. The $q \approx 0$ energy spectrum indicated that the maximum $(e, 2e)$ cross section of the valence molecular orbitals was over 20 times that of the carbon $1s$ orbital, with the oxygen $1s$ cross section being too small to register in that spectrum.

The $(e, 2e)$ spectrometer of Amaldi *et al.* was subsequently utilized by Camilloni *et al.*[6] to conduct detailed coplanar symmetric $(e, 2e)$ experiments on evaporated carbon films. An energy resolution of 90 eV for each outgoing electron allowed the valence bands to be distinguished from the core state, but was insufficient to resolve energy band dispersion with momentum, or the presence of plasmon energy loss peaks accompanying the pure $(e, 2e)$ scattering peak. Maintaining invariant electron energies corresponding to the carbon $1s$ binding energy, and symmetrically varying the outgoing electron polar angles, yielded $(e, 2e)$ cross section measurements of the carbon $1s$ core state at several distinct momentum values between -3.4 and 4.0 a.u.

More recent core state $(e, 2e)$ experiments have been conducted on C_2H_4, C_6H_6, and N_2 molecules by Bharathi *et al.*[7] These noncoplanar symmetric $(e, 2e)$ experiments satisfy the binary encounter reaction conditions that are required for electron-momentum spectroscopy. The core molecular orbitals of these polyatomic molecules consist primarily of various superpositions of the $1s$ atomic orbitals of each atom, although the hydrogen atoms have only a very slight effect on organic molecule core orbitals.

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Individual molecular orbitals in momentum space exhibit strong anisotropic interference effects. However, since the experiments were conducted on randomly oriented molecules, and since the different core molecular orbitals are degenerate to within the experimental energy resolution, the experimental cross sections did not reveal any remnants of the interference effects. Bharathi *et al.* obtained good agreement between their measurements and calculated self-consistent field orbital momentum distributions.

Avaldi *et al.*[8] have undertaken $(e, 2e)$ experiments on the carbon core state in C_2H_2 molecules using coplanar asymmetric kinematics, with rather low momentum transfer. In this case, the binary encounter approximation is not valid, and the $(e, 2e)$ cross section as a function of ejected electron polar angle exhibits a pronounced "recoil peak" directed approximately in the opposite direction to the momentum transfer, in addition to a similarly intense "binary peak" directed approximately along the momentum transfer. This reaction regime is unsuitable for electron-momentum spectroscopy, because the cross section is dominated by poorly understood collision dynamics that obscure the information about the target electronic structure. The significance of these experiments of Avaldi *et al.* resides in the perspectives they provide on the electron impact ionization mechanism for core states.

II. EXPERIMENTAL ASPECTS

The present experiment is conducted on a thin film of the amorphous carbon allotrope known as *evaporated carbon* (e-C), purchased from the Arizona Carbon Foil (ACF Metals) Company. Although the atomic and electronic structure of e-C is still equivocal, the consensus of opinion is that it is a graphitic amorphous semimetal, consisting of regions of imperfectly trigonally coordinated atoms embedded in a less voluminous skeleton of imperfectly tetrahedrally coordinated atoms. Thin films of e-C (thickness $\approx$ 8 nm) are fabricated by applying an electric discharge between two graphite electrodes to evaporate carbon atoms from the electrodes, and rapidly condensing the carbon vapor on a cold substrate, such as glass. The e-C film is "floated off" in deionized water and transferred to a sample holder with 2-mm holes. For the present experiment, the film is annealed by heating to >600 °C in ultrahigh vacuum. The purpose of the annealing procedure is to expel physisorbed contaminants from the film.

The current electron-momentum spectroscopy experiment is conducted with an asymmetric, noncoplanar $(e, 2e)$ spectrometer. This spectrometer is distinguished by the simultaneous detection of outgoing electrons within both a finite energy range and a finite azimuthal angle range, by using two-dimensional position sensitive detectors. Simultaneous detection of $(e, 2e)$ reactions in a four-dimensional kinematical continuum (both energy and azimuthal angle of both scattered and ejected electrons) is particularly beneficial for core state experiments, since the diffuse (in momentum space) core orbital has a much smaller differential cross section than the compact (in momentum space) valence orbitals.

A description of the experimental apparatus has been published by Storer *et al.*[9] and a numerical algorithm that is used in the calibration of the two-dimensional detectors has been presented by Caprari.[10] The reaction kinematics observed in this experiment correspond to electron energies in the vicinity of the values $E_0 = 20\,290$ eV, $E_A = 18\,798$ eV, and $E_B = 1192$ eV; and outgoing electron polar angles with respect to the incoming electron direction of $\theta_A = 14°$ and $\theta_B = -75.6°$. Outgoing electron azimuthal angle ranges are discussed by Storer *et al.*[9]

The carbon core state energy is a substantial 260 eV below the valence band lower edge in amorphous carbon. Nevertheless, the observed core state $(e, 2e)$ cross section exhibits a significant (>10%) background contribution from valence band $(e, 2e)$ collisions that are accompanied by multiple elastic and inelastic scattering processes.

The mechanism responsible for inelastic scattering is an overwhelmingly excitation of volume and surface plasmons by the incoming and outgoing electrons. To account for a 260-eV energy deficit, the electrons involved in a valence band $(e, 2e)$ collision must create a total of about ten plasmons. Although the probability of such a sequence of inelastic processes in such a thin ($\sim$8 nm) film is quite low, its influence is enhanced by the numerical superiority of valence electrons (four per C atom) over core electrons (two per C atom), and by the relatively broad momentum distribution and hence low momentum density of the core state. The mean free path of electrons for plasmon scattering increases with increasing kinetic energy, therefore, the low energy ejected electron is expected to create most of the plasmons, subject to all electrons traversing similar distances in the solid.

Plasmon creation is an extremely small angle scattering mechanism that can be characterized by a critical scattering wave vector $q_c = E_p/(2E)^{1/2}$, beyond which the cross section is negligible, as discussed by Raether.[11] This equation is expressed in atomic units, with E_p being the centroid of the plasmon loss energy peak, and E being the energy of the electron that excites the plasmon. This leads to a momentum transfer of less than 0.1 a.u. for a plasmon creation by the ejected electron, which is the dominant multiple scattering mechanism (Storer[12]). Consequently, plasmon creation does not appreciably alter the momentum dependence of the underlying $(e, 2e)$ cross section.

However, the experimental $(e, 2e)$ cross section within the energy interval between valence band and core state progressively broadens with increasing binding energy. In fact, as one progresses down in energy from the valence band to the core state, the width of the experimental $(e, 2e)$ cross section increases from that characteristic of the valence band, to more than twice the expected core state momentum width. As explained in Sec. III, one expects the core state momentum distribution to be accurately approximated by the square magnitude of the momentum representation of the carbon 1s atomic orbital. Such a momentum broadening cannot be attributed to only plasmon scattering. The most likely mechanism for

the momentum broadening is large angle elastic scattering, which is the amorphous solid analog of the Bragg diffraction that occurs in crystals.

In summary, valence band $(e, 2e)$ reactions in association with multiple plasmon and elastic scattering can contribute to the cross section in the core state energy interval. The intrinsic core state $(e, 2e)$ cross section is obtained by subtracting the valence band multiple scattering contribution from the measured $(e, 2e)$ cross section. This requires an estimation of the energy and momentum dependence of the valence band multiple scattering at, and below, the core state energy.

This estimate is derived by the following procedure. An $(e, 2e)$ experiment is performed on the same sample, with almost the same kinematics as the separate core state experiment, the exception being that the energy range of interest is from 9 to 89 eV above the core state. The $(e, 2e)$ cross section in this energy regime must be entirely due to valence band multiple scattering; therefore, it provides the basis for an empirical determination of the valence band multiple scattering cross section as a function of energy and momentum. The empirical formula that approximates this cross section is Gaussian in momentum, with width that increases linearly with binding energy, and magnitude that decreases exponentially with binding energy. This formula is extrapolated to the core state energy, and below. An unknown, but definite, scale factor is implied in this formula, since it is extracted from one experiment, and utilized in another, both experiments furnishing unnormalized cross sections. The scale factor is specifically chosen to make the corrected cross section in the core state $(e, 2e)$ experiment vanish for binding energies immediately below the core state spectrum (the observed binding energy range of the core state experiment actually extends 22 eV below the core state binding energy peaks).

An analogous phenomenon is apparent in the x-ray photoelectron spectroscopy (XPS) experiment on the carbon $1s$ core state in highly oriented pyrolytic graphite (HOPG) conducted by Sette et al.[13] The core state XPS spectrum measured by Sette et al. includes "a linear background" (their terminology), which they eliminate during their analysis of the spectrum, but without advancing a physical mechanism for this background. Based upon the reasoning enunciated above, one hypothesises that the background observed by Sette et al. is due to x-ray photons ejecting valence electrons deep within the solid, which subsequently undergo so many inelastic collisions (e.g., at least ten plasmon excitations) prior to emerging from the surface, that their emergent energy is equal to or less than that of core photoelectrons that have not undergone any inelastic collisions, because they have probably been ejected from close to the surface.

Sette et al. perceive this background to be linear, because of their narrow observed energy interval (<5 eV). The 80-eV energy interval observed in an adjunctive experiment to the one being presently reported (see two paragraphs back), is sufficiently broad to demonstrate that the background is actually not linear, but in fact decays gradually with increasing binding energy. One expects that the functional form of the valence band mul-

tiple scattering background is highly dependent upon the nature of the experiment [$(e, 2e)$ or XPS], and indeed the experiment kinematics.

All of the experimental $(e, 2e)$ cross sections presented in this article are "corrected" in the sense that the empirical valence band multiple scattering contribution has been subtracted from the intensity measured in the actual core state experiment. The cross sections displayed in Figs. 2 to 4 include the corresponding valence band multiple scattering cross section contribution as a broken line on the same graph. In each of these cases, the actual observed $(e, 2e)$ cross section is the sum of the broken line and the displayed experimental points.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The results of the electron-momentum spectroscopy experiment on the core state of annealed e-C are presented and discussed within this section. The fully differential cross section $\sigma(\varepsilon, q)$, which contains the total information content extracted from the $(e, 2e)$ experiment, is displayed in Fig. 1.

SOLID CARBON CORE STATE (e,2e) CROSS SECTION
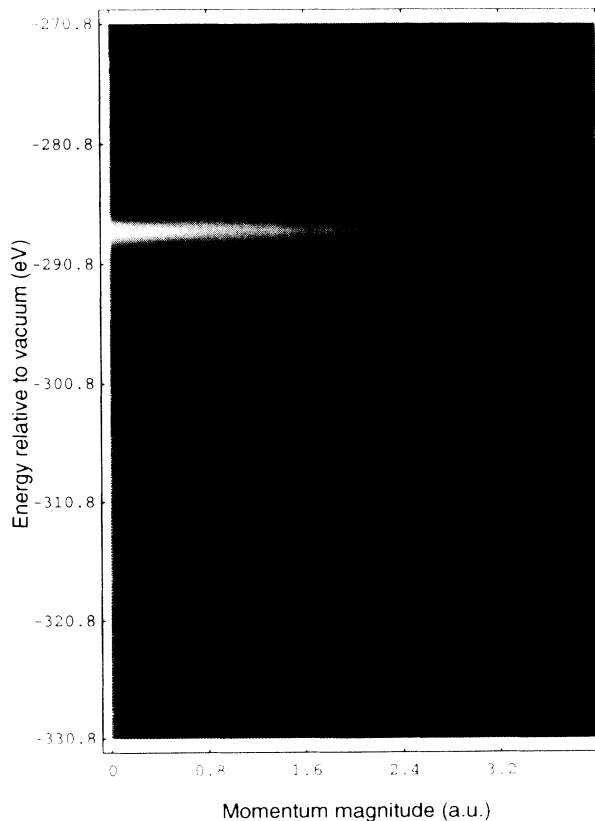

Momentum magnitude (a.u.)

FIG. 1. Fully differential $(e, 2e)$ cross section of the core state region of evaporated carbon. Magnitude is proportional to shading intensity, with black representing the minimum, and white the maximum. The spectrum has been corrected for valence band multiple scattering.

The core state binding energy spectrum is displayed in Fig. 2(a). This spectrum is derived from the fully differential $(e, 2e)$ cross section by an unweighted integration over the experimentally observable q domain ($q \in [0.0, 3.6]$a.u.) Consequently, it is not strictly a density of states spectrum, which requires a q^2 weighting factor. Introducing a q^2 weighting has the effect of enhancing the contribution from the high q cross section values, which are the most imprecise. Therefore, it would only serve to conceal features of the energy spectrum by increasing the statistical uncertainty.

The narrow, intense peak at lower ε (286–291 eV) is a consequence of solitary $(e, 2e)$ scattering reactions with core band electrons (hence it is denoted as the *core band peak*). The broad, weak hump that is discernable at higher ε is the *multiple-scattering continuum*. Since the valence band multiple-scattering contribution has been removed, the remaining intensity in the energy region of tighter binding than the core band peak is attributed to core state $(e, 2e)$ collisions plus multiple scattering. One notices that the $(e, 2e)$ cross section on the low binding energy side of the core band peak vanishes to within experimental error; this situation is enforced by appro-

priately choosing the scaling factor of the subtracted valence band multiple-scattering cross section, as stated in Sec. II. Both the core band peak and multiple-scattering continuum are readily discernible in the fully differential cross section of Fig. 1.

Plasmon creation is the only readily identifiable candidate for the inelastic multiple scattering. One may eliminate intraband transitions from contention, since the tightly bound carbon $1s$ energy bands are too narrow to account for the displacement and width of the continuum. Interband transitions are dismissed by the fact that the carbon $1s$ energy bands are too deep relative to the valence bands, for any such transitions to be observable. Intraband and interband transitions among valence bands are possible inelastic scattering mechanisms, however their cross section is relatively insignificant.

The binding energy spectrum is reproduced with higher resolution in Fig. 2(b) for an energy interval that encompasses the core band peak. Inspection of this spectrum indicates that the carbon $1s$ core band in e-C has a measured binding energy of 288.2 eV relative to the vacuum level, with a FWHM of 1.9 eV. XPS investigations of the carbon $1s$ core states of sputtered carbon (s-C), undertaken by Lascovich et al.[14] with an energy resolution of 0.2 eV, yielded a binding energy relative to the Fermi level of 285.0 eV with a FWHM of 1.50 eV for s-C that had not been subjected to argon ion etching, and a binding energy relative to the Fermi level of 284.4 eV with a FWHM of 1.55 eV for s-C that had been subjected to argon ion etching. Experimental evidence suggests that e-C and s-C have very similar atomic and electronic structures.

The apparent +3.2-eV binding energy offset of the present results compared with the former results of Lascovich et al. should be adjusted to account for the work function of e-C ($\sim$5 eV), because the present binding energies are with respect to the vacuum level, whereas the XPS binding energies are relative to the Fermi level. Thus, the actual binding energy offset of the present experiments compared with XPS experiments is approximately -2 eV. Furthermore, the precisely measured peak width of the former results of Lascovich et al. imply that the binding energy resolution of the present experiment is 1.2 eV FWHM.

Three factors possibly contribute to the actual binding energy discrepancy, each of which is individually at most a few eV. One is contact potentials between different metals in the pathway between the electrode that nominally defines the target potential, and the target film itself. Another is the Volta potential due to sample charging in ionization experiments (refer to Ley et al.[15] and McFeely et al.[16] for a discussion of this effect in the context of XPS). The remaining factor is inaccuracies in the spectrometer energy calibrations. In particular, calibration of the ejected (low energy) electron analyzer is conducted using electron beam energies much below those prevailing in actual $(e, 2e)$ experiments. Evidence is accumulating that the actual energy calibration in $(e, 2e)$ experiments for this analyzer, is marginally different from that measured during calibration experiments.

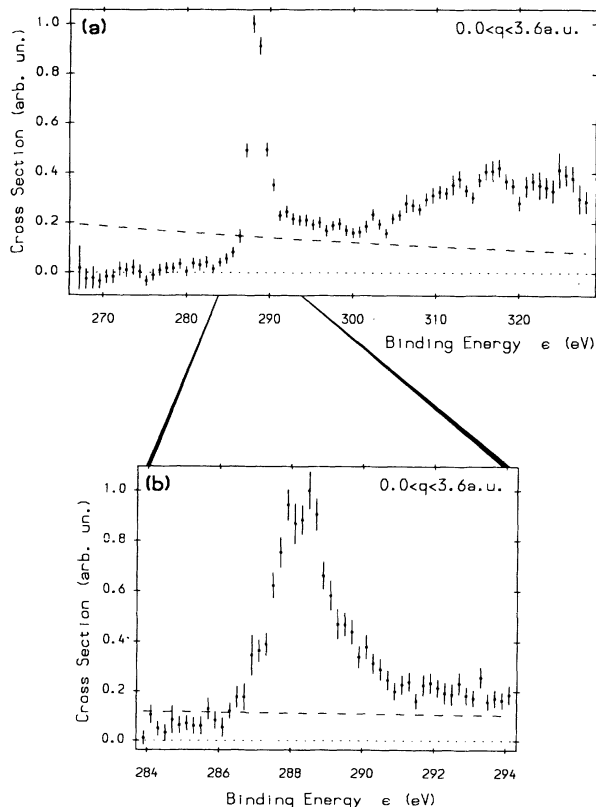The carbon $1s$ atomic orbital is concentrated in the

FIG. 2. Core state binding energy spectrum as measured by an $(e, 2e)$ experiment on evaporated carbon. The measured fully differential cross section is integrated over the complete observable momentum interval. The same spectrum is displayed with binding energy quantization intervals of 0.8 eV in (a) and 0.2 eV in (b). Broken lines indicate the estimated valence band multiple-scattering contributions, which have been subtracted from the measurements.

immediate vicinity of the nucleus. Consequently, there is only a miniscule overlap between $1s$ orbitals on neighboring atoms in any condensed phase of carbon. The additional fact that there are no other atomic orbitals that are nearly degenerate with the $1s$ orbital, precludes the possibility of the $1s$ atomic orbital participating in a hybridization scheme. One, therefore, expects the condensed carbon $1s$ core state to be closely approximated by the carbon $1s$ atomic orbital. In particular, the energy band should exhibit negligible dispersion with momentum, and the $(e, 2e)$ cross section as a function of momentum, at the energy of the band, should be proportional to the square magnitude of the momentum representation of the carbon $1s$ atomic orbital.

The dispersion of the carbon $1s$ core band may be assessed from Fig. 3, which integrates the measured $(e, 2e)$ cross section as a function of energy over three momentum intervals. Only the core band peak is included in the energy domain. Inspection reveals the absence of any identifiable dispersion of the energy peak with momentum, as anticipated above. The absence of energy dispersion with momentum in the core band peak is also

readily apparent in the fully differential cross section of Fig. 1.

In principle, the core states of atoms in different bonding environments (trigonal sp^2, or tetrahedral sp^3) of amorphous carbon would be expected to have small relative chemical shifts in both their energy and momentum spectra. The observed $(e, 2e)$ spectrum is actually an incoherent sum of the $(e, 2e)$ spectra of the different types of carbon atom. These bonding induced perturbations in the $(e, 2e)$ cross section are too small to be convincingly resolved by the present experiment, due to its insufficient energy and momentum resolutions, and excessive statistical uncertainty. Indeed, any experiment capable of resolving the different components in the $(e, 2e)$ cross section would probably also resolve the small energy dispersion with momentum of the core band. This is manifestly not the case in the present experiment.

Previous XPS experiments indicate the presence of chemical broadening in the energy spectrum of the core state in amorphous carbon, although the core state peak is not obviously resolvable into subordinate peaks. The XPS experiment of Sette et al.,[13] conducted on purely sp^2 bonded HOPG, shows a natural linewidth of 0.210 eV FWHM, while the XPS experiment of Lascovich et al.,[14] conducted on sp^2/sp^3 bonded s-C, exhibits a much broader 1.50 eV FWHM natural linewidth.

The energy integrated differential cross section as a function of momentum is displayed in Fig. 4(a) for the energy range of the core band peak and in Fig. 4(b) for an energy range that partially covers the multiple-scattering continuum. Although the relative normalization of the two distributions is experimentally measured, it is somewhat arbitrary since it is dependent on the specific interval chosen for the binding energy integration of the multiple-scattering continuum. The decline of the $(e, 2e)$ cross section with increasing momentum in both energy regions is manifest in the fully differential cross section of Fig. 1.

Superimposed upon the experimental $(e, 2e)$ cross section of Fig. 4 is the square of the theoretical Hartree-Fock wave function for the carbon $1s$ atomic orbital. The theoretical momentum distribution includes the effect of finite q resolution by convolving the direct Hartree-Fock momentum distribution with an effective q resolution function, but this has a negligible effect, since the distribution is very broad compared with the 0.15 a.u. resolution of the spectrometer, as reported by Storer et al.[9]

The experimental $(e, 2e)$ cross section displayed in Fig. 4 has been normalized by optimally scaling the core band momentum distribution in (a) to fit the absolute Hartree-Fock one-electron-momentum distribution. To maintain the correct measured relative cross section, the same scaling factor is applied to the experimental multiple-scattering momentum distribution in Fig. 4(b). Also shown in (b), is the same Hartree-Fock momentum distribution scaled by a factor of 0.54.

Inspection of Fig. 4(a) indicates subjectively that the agreement between the carbon $1s$ and e-C core band momentum distributions is quite satisfactory, especially considering the statistical uncertainty. There is no identifiable systematic deviation between the two momentum
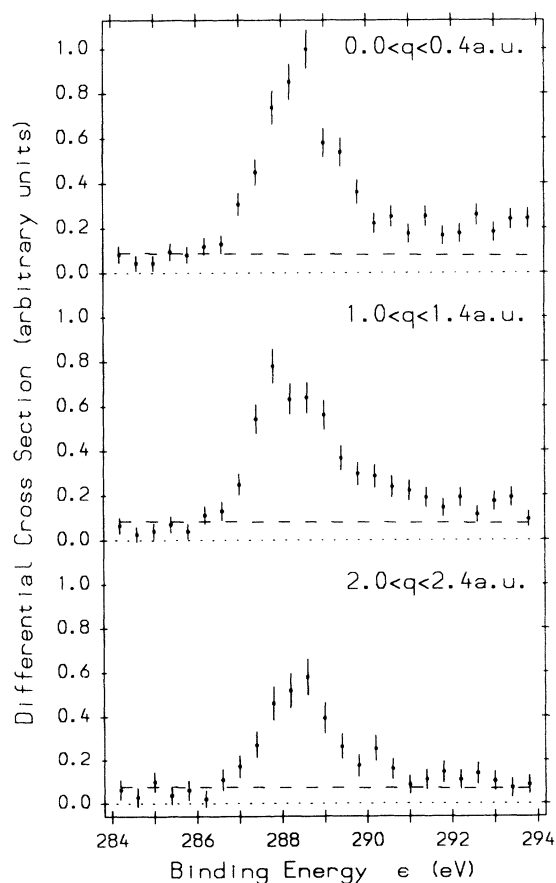

FIG. 3. Evaporated carbon core state $(e, 2e)$ cross section, integrated over the indicated momentum intervals. A common normalization factor is applied to all spectra. The binding energy quantization interval is 0.4 eV. Broken lines indicate the estimated valence band multiple-scattering contributions, which have been subtracted from the measurements.
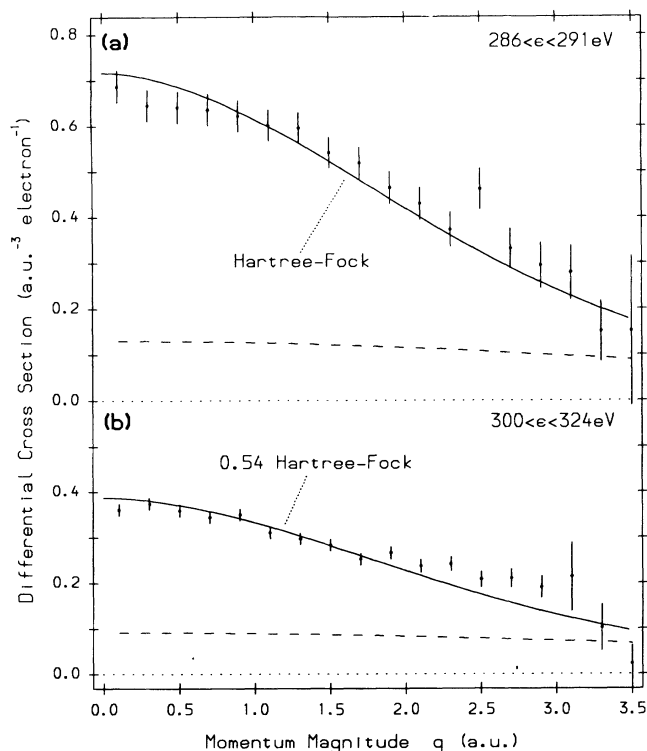
FIG. 4. Core state fully differential cross section of evaporated carbon integrated over the indicated binding energy intervals. The experimental relative cross sections have been scaled so that the core band cross section in (a) fits the absolute Hartree-Fock atomic carbon 1s one-electron-momentum space probability density. The momentum quantization interval is 0.2 a.u. Broken lines indicate the estimated valence band multiple-scattering contributions, which have been subtracted from the measurements.

distributions. Inspection of Fig. 4(b) indicates that the agreement between the scaled carbon 1s and e-C multiple scattering momentum distributions is good at low momentum (q<1.8 a.u.), but degrades at higher momentum where the theoretical momentum distribution de-

clines with increasing q more rapidly than does the experimental cross section.

The most likely reason for the discrepancy between the momentum distributions of the core band peak and multiple-scattering continuum is the gradual deterioration with increasing binding energy of the quality of the empirical estimate of the valence band multiple scattering. In particular, on the basis of the momentum profiles of Fig. 4, one can conclude that the estimated valence band multiple-scattering cross section declines too rapidly with increasing binding energy, especially at high momentum. In such a circumstance, the "partially corrected" $(e, 2e)$ cross section in the multiple-scattering continuum would indeed be expected to have a broader momentum distribution than that of purely core band multiple scattering.

A more carefully constructed estimate of valence band multiple-scattering contributions needs to be developed, preferably one based upon theoretical principles governing the plasmon and elastic scattering mechanisms that occur in amorphous condensed matter. Research on this topic is presently being conducted at Flinders.

IV. CONCLUSIONS

This article presents an electron-momentum spectroscopy measurement of the carbon core state for an amorphous carbon allotrope. As expected for localized states, the core band energy peak is narrow and nondispersive with momentum variation. Also as predicted for tightly bound nonoverlapping orbitals, the core band momentum distribution is accurately described by the atomic carbon 1s atomic orbital.

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SOLID CARBON CORE STATE
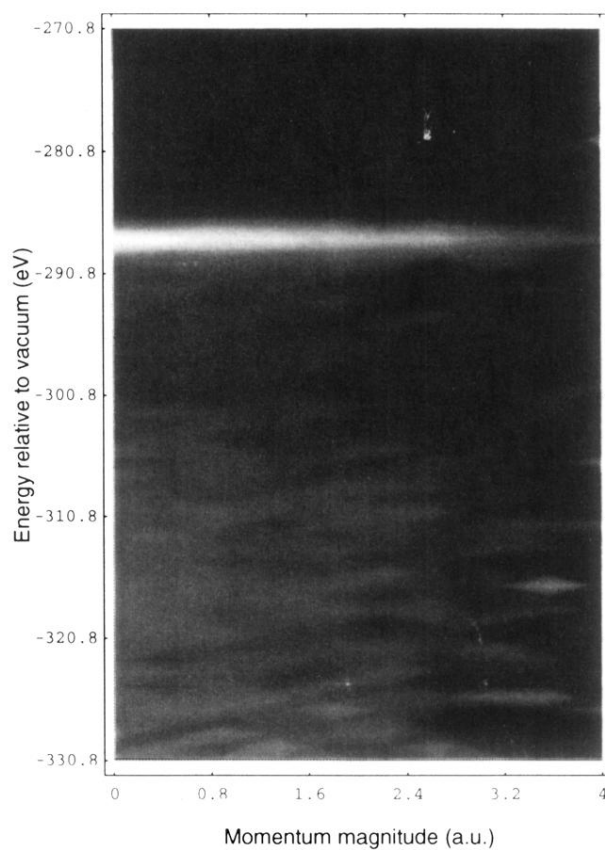## (e,2e) CROSS SECTION

FIG. 1. Fully differential $(e, 2e)$ cross section of the core state region of evaporated carbon. Magnitude is proportional to shading intensity, with black representing the minimum, and white the maximum. The spectrum has been corrected for valence band multiple scattering.