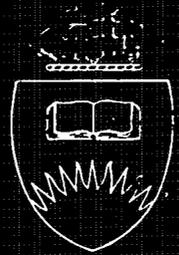


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**ELECTRONIC STRUCTURE OF MATERIALS CENTRE**

Electron momentum spectroscopy of the core state of solid carbon

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# Electron momentum spectroscopy of the core state of solid carbon

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## Abstract

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.

71.50.+t , 79.20.Kz , 73.90.+f , 34.80.Dp

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## I. INTRODUCTION

(e,2e) reactions are electron impact ionisation 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<sup>1-3</sup>. 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*<sup>1,4</sup>. Specifically, for crystals, an occupied band  $n$  with band structure  $\epsilon_n(\mathbf{q})$ , is characterised 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  $\epsilon_n(\mathbf{q}) = E_0 - E_A - E_B$ . The spectral momentum density for general condensed matter, including disordered phases, is discussed by Caprari<sup>4</sup>.

The first (e,2e) experimental observation of the carbon core state (1s atomic orbital) was the pioneering coplanar symmetric experiment on Formvar ( $\text{CH}_2(\text{OCH}_3)_2$ ) reported by Amaldi *et al*<sup>5</sup>. By scanning the incident electron energy over an 800eV range, and keeping each outgoing electron energy fixed at 7.3keV, 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 135eV 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 twenty 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 utilised by Camilloni *et al*<sup>6</sup> to conduct detailed coplanar symmetric (e,2e) experiments on evaporated carbon films. An energy resolution of 90eV 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  $-3.4\text{a.u.}$  and  $4.0\text{a.u.}$ .

More recent core state (e,2e) experiments, have been conducted on  $\text{C}_2\text{H}_4$ ,  $\text{C}_6\text{H}_6$  and  $\text{N}_2$  molecules by Bharathi *et al*<sup>7</sup>. These non-coplanar 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. 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*<sup>8</sup> have undertaken (e,2e) experiments on the carbon core state in C<sub>2</sub>H<sub>2</sub> 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 ionisation 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  $\approx$  8nm) are fabricated by applying an electric discharge between two graphite electrodes to evaporate carbon atoms from the electrodes, and rapidly condensing the carbon vapour on a cold substrate, such as glass. The *e-C* film is 'floated off' in deionised water and transferred to a sample holder with 2 mm holes. For the present experiment, the film is annealed by heating to  $>600^\circ\text{C}$  in ultra high 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 asym-

metric, non-coplanar (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*<sup>9</sup>, and a numerical algorithm that is used in the calibration of the two dimensional detectors has been presented by Caprari<sup>10</sup>. The reaction kinematics observed in this experiment correspond to electron energies in the vicinity of the values  $E_0=20290\text{eV}$ ,  $E_A=18798\text{eV}$  and  $E_B=1192\text{eV}$ ; and outgoing electron polar angles with respect to the incoming electron direction of  $\theta_A=14^\circ$  and  $\theta_B=-75.6^\circ$ . Outgoing electron azimuthal angle ranges are discussed by Storer *et al*<sup>9</sup>.

The carbon core state energy is a substantial 260eV 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 overwhelming excitation of volume and surface plasmons by the incoming and outgoing electrons. To account for a 260eV 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 ( $\sim 8\text{nm}$ ) film is quite low, its influence is enhanced by the numerical superiority of valence electrons (4 per C atom) over core electrons (2 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 charac-

terised by a critical scattering wavevector  $q_c = E_p/(2E)^{1/2}$ , beyond which the cross section is negligible, as discussed by Raether<sup>11</sup>. 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 momentum transfer of less than 0.1a.u. for plasmon creation by the ejected electron, which is the dominant multiple scattering mechanism (Storer<sup>12</sup>). 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 Section 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 can't be attributed to only plasmon scattering. The most likely mechanism for the momentum broadening is large angle elastic scattering, which is the amorphous solid analogue 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 9eV to 89eV 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 a 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 utilised in another, both experiments furnishing unnormalised 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 22eV 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*<sup>13</sup>. 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 (eg. 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 haven't 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 (<5eV). The 80eV 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 multiple 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 Figures 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  $\sigma(\epsilon, q)$ , which contains the total information content extracted from the (e,2e) experiment, is displayed in Figure 1.

The core state binding energy spectrum is displayed in Figure 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  $\epsilon$  (286–291eV) 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  $\epsilon$  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 appropriately choosing the scaling factor of the subtracted valence band multiple scattering cross section, as stated in Section II. Both the core band peak and multiple scattering continuum are readily discernible in the fully differential cross section of Figure 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 Figure 2(b) for an energy interval that encompasses the core band peak. Inspection of this spectrum indicates that the carbon 1s core band in  $\epsilon$ -C has a measured binding energy of 288.2eV relative to the vacuum level, with a FWHM of 1.9eV. XPS investigations of the carbon 1s core states of sputtered carbon ( $s$ -C), undertaken by Lascovich *et al*<sup>14</sup> with an energy resolution of 0.2eV, yielded a binding energy relative to the Fermi level of 285.0eV with a FWHM of 1.50eV for  $s$ -C that had not been subjected to argon ion etching, and a binding energy relative to the Fermi level of 284.4eV with a FWHM of 1.55eV for  $s$ -C that had been subjected to argon ion etching. Experimental evidence suggests that  $\epsilon$ -C and  $s$ -C have very similar atomic and electronic structures.

The apparent +3.2eV 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  $\epsilon$ -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 Lascovich *et al*'s former results imply that the binding energy resolution of the present experiment is 1.2eV 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 ionisation experiments (refer to Ley *et al*<sup>15</sup> and McFeely *et al*<sup>16</sup> 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 analyser 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 analyser, is marginally different from that measured during calibration experiments.

The carbon 1s atomic orbital is concentrated in the immediate vicinity of the nucleus. Consequently, there is only miniscule overlap between 1s orbitals on neighbouring 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 hybridisation 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 Figure 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 Figure 1.

In principle, the core states of atoms in different bonding environments (trigonal sp<sup>2</sup>, or tetrahedral sp<sup>3</sup>) 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*<sup>13</sup>, conducted on purely  $sp^2$  bonded HOPG, shows a natural linewidth of 0.210eV FWHM, while the XPS experiment of Lascovich *et al*<sup>14</sup>, conducted on  $sp^2/sp^3$  bonded *s*-C, exhibits a much broader 1.50eV FWHM natural linewidth.

The energy integrated differential cross section as a function of momentum is displayed in Figure 4(a) for the energy range of the core band peak, and in Figure 4(b) for an energy range that partially covers the multiple scattering continuum. Although the relative normalisation of the two distributions is as 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 Figure 1.

Superimposed upon the experimental  $(e,2e)$  cross section of Figure 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 0.15a.u. resolution of the spectrometer, as reported by Storer *et al*<sup>9</sup>.

The experimental  $(e,2e)$  cross section displayed in Figure 4 has been normalised 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 sec-

tion, the same scaling factor is applied to the experimental multiple scattering momentum distribution in Figure 4(b). Also shown in (b), is the same Hartree-Fock momentum distribution scaled by a factor of 0.54.

Inspection of Figure 4(a) indicates subjectively that the agreement between the carbon 1s and  $\epsilon$ -C core band momentum distributions is quite satisfactory, especially considering the statistical uncertainty. There is no identifiable systematic deviation between the two momentum distributions. Inspection of Figure 4(b) indicates that the agreement between the scaled carbon 1s and  $\epsilon$ -C multiple scattering momentum distributions is good at low momentum ( $q < 1.8 \text{ \AA}^{-1}$ ), but degrades at higher momentum where the theoretical momentum distribution declines 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 Figure 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 localised 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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## FIGURES

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.

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 quantisation intervals of 0.8eV in (a), and 0.2eV in (b). Broken lines indicate the estimated valence band multiple scattering contributions, which have been subtracted from the measurements.

FIG. 3. Evaporated carbon core state (e,2e) cross section, integrated over the indicated momentum intervals. A common normalisation factor is applied to all spectra. The binding energy quantisation interval is 0.4eV. 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 quantisation interval is 0.2a.u.. Broken lines indicate the estimated valence band multiple scattering contributions, which have been subtracted from the measurements.

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

