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Electron-energy-loss studies of dislocations in diamond

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Electron-energy-loss spectra recorded from very small volumes of diamond containing individual dislocations show extra intensity within the band gap just below the 1*s*–to–conduction-band threshold energy, when compared to spectra recorded from neighboring defect-free regions. This is interpreted as direct evidence for the presence of vacant defect states associated with the dislocation structure. The contribution of the π^* states from the surface layers to this region of the spectra is completely removed by calculating the difference between the spectra recorded on and off the defect.

I. INTRODUCTION

Spatially resolved electron-energy-loss spectroscopy (EELS) in the scanning transmission electron microscope (STEM) is a proven technique for examining the electronic, chemical, and structural detail in very small areas of material.[1] Here, we use it to investigate the electronic structure associated with individual dislocations in diamond.

Interest in the electronic structure associated with dislocations in group-IV semiconductors dates back over 30 years to the pioneering work of Shockley[2] and Read.[3] Since that time there have been numerous attempts[4–8] to determine by experiment and theory both the lowest-energy atomic configurations and any shallow- or deep-level defect states which may be present in the band gap. However, the problem of identifying particular measured signals with particular defects has remained and theories have been unable to develop to a stage where one can confidently draw any firm conclusions. To date, most calculations for diamond do not agree exactly over the energy dispersion and spatial extent of these states. Yet there is a general trend among them: Structures with dangling bonds have either a wide half filled band or one filled and one empty band within the gap whereas those with reconstructed bonds are virtually clear of states in the energy gap.

In the diamond structure, perfect screw and 60° dislocations have a Burgers vector of the type $\frac{1}{2}\langle 1,1,0\rangle$ and glide on the $\{1,1,1\}$ slip planes, where 60° denotes the angle between the Burgers vector and the dislocation line marking the region between slipped and unslipped material. Perfect dislocations are those having a Burgers vector equal to a lattice vector. Recently, Pirouz *et al.*[9] have shown using weak beam microscopy that these perfect dislocations in type-II (i.e., low nitrogen content) diamond are in fact dissociated into two closely spaced Shockley partial dislocations which bound a stacking fault and have a separation of a few nanometers. The Burgers vector of a partial dislocation does not equal a complete lattice vector. Dissociation of the 60° perfect dislocation leads to one 30° and one 90° partial and the screw splits into two 30° partials. Other dissociated dislocation configurations (i.e., Lomer-Cottrell edge dislocations, extended nodes, and faulted dipoles) were also observed typical of those found in materials having fcc structures such as silicon and germanium. Despite such TEM studies the precise atomic arrangement and degree of reconstruction at any dislocation in diamond remains unknown.

Kiflawi and Lang[10] were first to demonstrate that the dislocations in diamond were electrically active by correlating the cathodoluminescence image with the x-ray topographic image in a bulk diamond with low dislocation density. In this and subsequent work Lang and coworkers[10–12] were able to determine that the polarization of the **E** vector is strongest parallel to the dislocation line. Pennycook *et al.*[13] used the STEM to acquire the cathodoluminescence signal simultaneously with the transmitted-electron image from individual dislocations in a thin sample with high dislocation density. They reported that the luminescence was independent of dislocation type but that not all dislocations were luminescent. In their study the separate potentials were note resolved. Yamamoto *et al.*[14] examined the spectrum, intensity, and polarization of the cathodoluminescence from individual dislocations in a suitably modified TEM. They reported that the light, of energy 2.85 eV and bandwidth 0.42 eV, emitted from the dislocation was polarized parallel to the line of the dislocation core and independent of the Burgers vector. Nonluminescent dislocations were also found and curved dislocations were less luminescent than straight ones.

EELS of dislocations in diamond has been carried out previously by Pennycook.[15] The energy-loss spectrum is proportional to the probability of various inelastic scattering events as the fast electron traverses the material. At energy losses comparable to the binding energy of the core electrons, which is about 290 eV for 1*s* electrons in diamond, the bound core electrons can be excited to vacant energy levels lying above the Fermi level. For intrinsic bulk material these levels are in the conduction band and consequently the spectrum in this range has an absorption edge resembling the shape of the conduction-band density of states. For 1*s* electrons this is conventionally termed the *K* edge. At a defect, electronic states may be present within the band gap. Depending upon the position of the Fermi level, excitations to these states can produce extra absorptions preceding the bulk *K* edge (in Ref. 15, however, no effect was observed due to a poor signal strength and crystal surface absorptions). Here we report EEL spectra with improved statistics and energy resolution which show that in addition to the surface contribution there is a broad band of absorption within the band gap originating from the dislocated region. We find that the energy-loss spectrum is extremely sensitive to small variations in the number of localized defect states within the fine electron probe of the STEM. In these experiments the lateral resolution appears to be limited principally by the probe size. Batson[16] has reported similar effects in the energy loss at the aluminum-silicon interface.

II. EXPERIMENT

The diamond examined in this study was of the semiconducting type-II*b* variety. The stone had been prepared for electron microscopy by traditional mechanical polishing followed by an argon-ion milling process, a technique which leads to regions of suitably thin diamond covered by an amorphous surface layer. The foil was oriented in the microscope a few degrees off the [1,1,0] pole. The characteristics of this stone have been described previously.[13,15,17]

The analysis was performed on a V.G. Microscope's HB501 STEM fitted with a high-energy resolution Wien filter electron spectrometer. The spectrometer has been described in detail by Batson.[18] Spectra with an energy resolution of 0.35 eV may be routinely recorded with a 100-keV beam and a focused probe diameter of about 0.7 nm. The absolute energy calibration is known to be better than 30 meV. The collection and convergence semiangles of the beam at the specimen were both ~8 mrad. The current at the specimen is estimated to be of the order of a few nanoamperes. The total acquisition time for each spectrum was about 1 min. The vacuum in the microscope was better than 1×10^{-9} Torr and there was no sign of specimen contamination.

The effect of dislocations on the EELS is examined using "the background subtraction technique." It involves recording two spectra; one with the beam located on the region of interest and a second with the beam passing very close by it ($\lesssim$ 5 nm). The difference of the two spectra reveals any extra states on a very low background. The specimen is viewed immediately prior to and after each spectrum acquisition. If there is any apparent specimen drift during the analysis the data are not recorded and have no significance in the following discussion. This technique has been used previously to detect very small amounts of nitrogen in nanometer structures in diamond.[10] Straight segments of six separate dislocations of unknown Burgers vector were examined in this study.[20] Central segments are chosen for those dislocations threading through the foil from top to bottom. With the imaging conditions employed we were unable to resolve the separation of the dislocations into partials.

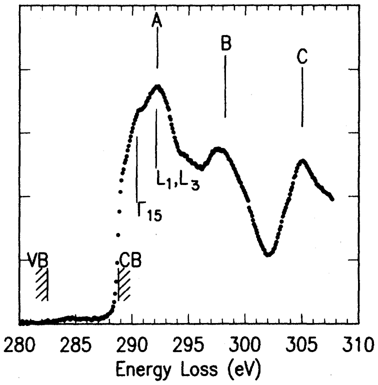

FIG. 1. Near-edge structure of type-II*b* diamond. The positions highlighted correspond to the energy separation of the 1*s* core level and levels close to the Fermi level. VB corresponds to the surface valence-band maximum determined by photoelectron spectroscopy (Ref. 28). CB to the bulk conduction-band minimum, and the lines marked *A*, *B*, and *C* correspond to gross features in the calculated (Ref. 24) *p* projected density of states where *A* is aligned to first peak in the EEL data at 292.3 eV. The points labeled Γ_{15} and L_1,L_3 denote the approximate energy positions of high symmetry, critical points in the Brillouin zone determined by theory (Ref. 23).

III. RESULTS

Figure 1 represents a typical *K*-shell absorption spectrum of diamond acquired over the energy range from 280 to 310 eV. A smooth background, of the form E^{-r}, fitted in a 10-eV energy region preceding the edge structure, has been subtracted from the data. The background fit in this narrow energy range is apparently adequate for this 30-eV window above the carbon edge. Naturally a quantitative analysis requiring an accurate fit of over 50

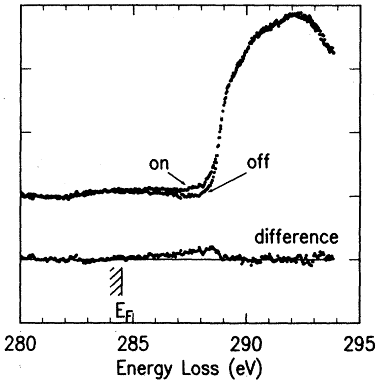

FIG. 2. Spectra recorded on and off a dislocation are displayed overlaying each other. The spectra are normalized in the region from 290 to 294 eV. The difference represents excitations to vacant states in the close proximity (<1 nm) of the dislocation. The position of the Fermi level (E_F) at the dislocation, calculated by Jones and King (Ref. 6), is marked for reference.

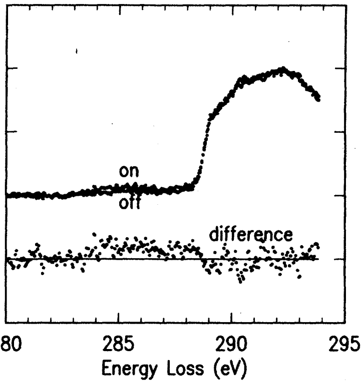

FIG. 3. The difference between two spectra recorded on and off a second dislocation to reveal the pre-edge structure due to defect states. The difference is scaled up by a factor of 3 for display purposes. As in Fig. 2, the spectra are normalized in the region of the bulk K loss. Compare with Fig. 2 to notice differences in the pre-edge structure from the different dislocations.

eV would demand a similar increase in the pre-edge fitting region. The structure of the edge in this window has a number of features due to bulk and surface losses: The midpoint of the rise for the bulk K-edge threshold energy is located at 288.8 eV in these data with peaks at 292.3, 289.0, and 305.0 eV which are associated with similar variations in the p-symmetry projected conduction-band density of states. Superimposed on these gross features there are a number of finer scale features. These may correspond to critical points in the Brillouin zone. The effect of surface scattering gives rise to the pre-edge intensity, with an onset located at 282.6 eV. The origin of these excitations is discussed in the following section.

The EELS data from four of the six dislocations showed a finite and reproducible increase in absorption in the band-gap region, which is only revealed using a "difference technique." Figures 2 and 3 illustrate this absorption from two different dislocations. The spectra from different dislocations do not match exactly, as illustrated. Generally, the difference between the dislocation and the diamond matrix spectra shows an onset at 283.6 eV and monotonically increasing up to the conduction-band minimum. Only a very slight normalization ($\lesssim 1\%$) of the core edge was found necessary to provide an accurate precise subtraction over the entire energy range of the recorded spectra. The reproducibility of each measurement can be seen in Fig. 4. In this case spectra were recorded on either side of a dislocation. The spectra are identical to within the noise limit of the data. In addition, spectra recorded over a period of over 10 min show no significant differences. In two of the six dislocations there was a decreased absorption in the pre-edge structure with the beam located on the visibly strained region of the dislocation. These effects are discussed in the following section.

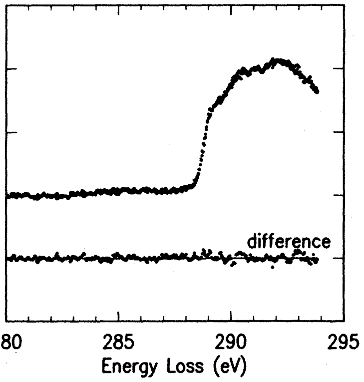

FIG. 4. Spectra recorded on either side of a dislocation. There is no detectable difference confirming the reproducibility of these measurements and the validity of the "difference technique" for eliminating surface effects.

IV. DISCUSSION

A. Diamond core edge shape

The midpoint of the K-edge onset is located at 288.8 eV and corresponds to the lowest-energy transitions from the $1s$ core level to the p-like conduction-band states above the Fermi level. The edge shape reflects the local p-like projected density of states at the carbon atom sites which may be modified by any core-hole effects. The gross features of the near-edge structure beyond 0.3 eV above the onset is identical to previously reported absorption data[21,22] and there is a reasonable agreement with band-structure calculations.[23,24] However, we have been unable to detect the white line, described as an exciton, located at 0.2 eV below the conduction-band minimum which Morar *et al.*[25] observed by partial x-ray photoyield spectroscopy. The instrumental resolution in these EEL experiments should be capable of detecting such an exciton. Its absence may be due to either a momentum transfer effect (electrons transfer at least 1.6 times more momentum than photons), or a polarization effect (the direction of scattering dipole may be relevant if the exciton has a significant anisotropic dispersion). Alternatively, the peak observed by partial photoyield may be a surface excitation effect. An attempt to enhance the relative surface contribution by collecting spectra with the beam passing within 1 nm of the diamond surface did not prove conclusive due to the poor statistics (the scattering power falls off rapidly with increasing impact parameter). The white line is also absent in the photoyield data reported by Zhurakovskii and Zaulichnyi[26] and synchrotron x-ray absorption data[27] although in the latter work this was attributed to inadequate instrumental energy resolution. Whatever the final explanation, these results indicate that there are differences between absorption and partial yield which may not be ignored if fine detail in the near-edge structure is to be accurately interpreted.

B. Pre-edge structure

The structure preceding the bulk core edge from 282.6 to 288.8 eV represents excitations to empty states below the conduction-band minimum. These may be defect related, intrinsic surface states, or vacant π^* orbitals of unsaturated (or sp^2 hybridized) carbon. The pre-edge structure is broad and featureless and its integrated single scattering intensity is independent of the foil thickness, whereas the bulk K-loss intensity is linearly proportional to the foil thickness in thin areas. This is demonstrated in Fig. 5 where the spectra recorded at several thicknesses are compared. Each spectrum is multiplied by a normalizing factor of the form $\exp(t/\Lambda)$ to correct for plural scattering to higher energies, where Λ is the total inelastic mean free path of the fast electron in the material and t is the foil thickness. The ratio t/Λ is determined at each point by acquiring the low loss region of the spectrum from 0 to 100 eV. A reasonably accurate value of this dimensionless parameter is obtained by a Fourier deconvolution of the spectrum.[27] Figure 5 demonstrates that the pre-edge band is a surface effect. Both surfaces are believed to consist of a layer of ~5 nm of amorphized diamond, produced by the ion milling process. Such material is known to contain a significant amount of sp^2 hybridized carbon[22] and so the better part of the pre-edge intensity is due to this layer. Assuming that the cross section for scattering into an intrinsic π^*-like surface state is similar to that for the π^* band of amorphous carbon and that there are a few such surface states per unit cell of diamond then we estimate that intrinsic surface states, such as those described by Morar *et al.*,[28,29] contribute less than 10% to the pre-edge structure in this diamond specimen. This proportion will be different in samples prepared by alternative techniques which reduce the amount of amorphous carbon on the surface such as oxygen etching or cleaving. It is of interest to note that in most instances there is a fairly close resemblance between the pre-edge structure measured by EELS in this specimen and the photoyield data reported in Ref. 29.

C. Effect of dislocations

The presence of a dislocation is likely to affect the bulk K-shell loss locally and to produce extra states in the band-gap region. However, it is unlikely that the measured bulk core edge would be affected since atoms located on the dislocation core form only a small fraction of those probed by the beam. For example, there are about 10^4 carbon atoms in a pill box of diamond having a thickness of 100 nm and diameter equal to the probe size, i.e., ~8 Å. If this volume is threaded by a dislocation, only a few percent of these atoms would lie along the core. Convoluting the interaction impact parameter with the lateral probe size would reduce this number further. We expect the scattering cross sections for the 1s to σ^* transition to be similar for the tetrahedrally bonded atoms and the atoms located on defect sites. Thus the contribution of the dislocation core atoms to the measured intensity of the bulk σ^* edge should reflect their atomic fraction within the probed volume, which is of the order of a few percent. Variations on this scale would be difficult to observe.

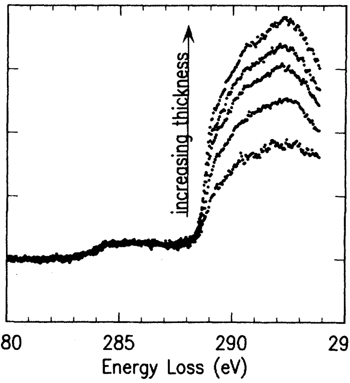

FIG. 5. Spectra recorded from a range of thicknesses. The pre-edge structure remains independent of foil thickness and is thus a surface effect. The region above 288.8 eV is proportional to thickness and is thus a bulk effect.

The same is not true for band-gap states, which occur on a significantly reduced background. Although the absolute absorption intensity for a 1s core state to a localized state drawn out of the conduction band into the band gap is difficult to calculate, a crude estimate of the intensity in the absorption spectrum involving a defect band as a fraction of the absorption to a perfect crystal band is given based on state counting arguments. The starting point for this estimate is the differential cross section in the first Born approximation,[30] which is typically expressed as a product of a joint density of states and an average transition matrix element. An important assumption is that the two terms are independent of each other. We shall briefly consider these two terms separately.

Typically, the matrix elements are slowly varying functions of energy that may impose certain selection rules determined by the symmetry of the system. For example, such symmetry might reveal itself in the following manner: In a dangling bond model for the dislocation core structure one envisages a linear array of sp^3-like orbitals lying along the length of the core. Each orbital points into the dislocation core and is capable of π bonding with neighboring half filled orbitals, leading to a defect band. In such a case, for scattering in the dipole limit, a scattering vector of the fast electron lying perpendicular to the dislocation line direction is necessary to yield a nonzero overlap integral for the excitation of a 1s core electron to the defect associated π-like state localized at the dislocation core. In the STEM, microscope apertures define a cone of momentum transfers collected by the spectrometer. Thus it may be possible to orient the dislocation such that its line lies either perpendicular or parallel to scattering vectors in the cone. A similar example of this technique has been illustrated with the planar hexagon rings in the graphite and BN systems.[31] Since the nature of the final state wave function depends upon the atomic structure and degree of dangling bond reconstruction along the dislocation core which is unknown at present, an absolute determination of the magnitude of the matrix elements is not reliable. We might expect, however, that the value of matrix elements for a defect state excitation is approximately equal to that for the bulk state provided it is symmetry allowed.

We turn now to the joint density of states, which approximates to the final state density of states in the present case because of the narrow width (~ 0.2 eV) of the core state. Although the precise energy dispersion of the localized states is open to question, the density of states of the localized state may show an enhancement relative to the bulk σ^* antibonding states if the bandwidth of the defect states is less than bulk. In other words, reducing the bandwidth increases the local density of states. Such an edge enhancement is well known in molecular systems, such as O_2 and N_2, that exhibit sharp "white line" spectra. We estimate its magnitude by assuming that the density of states scales inversely with bandwidth. Hence if each band approximates to a uniform distribution whose integrated area is equal to the total number of states contributing to the band then the relative magnitude of the localized state band to the bulk diamond crystal band is

$$\frac{D_{\mathrm{DB}}(E)}{D_{\mathrm{CB}}(E)}=\frac{\Delta_{\mathrm{CB}}}{\Delta_{\mathrm{DB}}}\frac{N_{\mathrm{DB}}}{N_{\mathrm{CB}}} \tag{1}$$

where $D(E)$ is the number of states per atom per unit energy width contributing to the band, N is the number of states per atom in the band, Δ is the effective bandwidth of the distribution, and the subscripts DB and CB correspond to the defect and conduction band, respectively. In the linear combination of atomic orbitals (LCAO) scheme each tetravalently coordinated carbon atom in the diamond crystal provides four orbitals to the $\sigma(sp^3)$ bands whereas an atom at a dislocation core might provide three orbitals to the σ bands and one half filled orbital (i.e., either a dangling bond or a reconstructed π bond) to a defect band within the band gap. This is equivalent to one vacant state drawn down from the conduction band and one filled state pulled up from the valence band. The effective conduction-band width is estimated from absorption experiments to be about 24 eV and according to calculations[6,7] the defect-band width may be about 2 eV if the dislocation core were unreconstructed. Thus evaluating the ratio of Eq. (1) one might expect a three to one enhancement in the relative density of states and a similar threefold increase in the detection sensitivity if there were no other significant factors affecting the cross section. There are two factors that may be relevant. Firstly, according to the Bethe theory of inelastic scattering,[30] reducing the excitation energy enhances the cross section by a factor proportional to its inverse. In the present example, this corresponds to an enhancement of less than 2%. Secondly, the measured intensity of an edge is proportional to the summation of the differential cross sections over the possible initial and final states for all the atoms probed in the experiment. For nonlocalized excitations, such as valence-band–to-conduction-band transitions, this sum is not always straightforward to estimate. However, for core-level excitations, the initial state is localized: The spatial extent of the 1s shell in carbon is about a Bohr radius, which is approximately 0.003 nm for a binding energy of about 300 eV. In this case there is only a significant overlap between initial and final states of the bound electron in the spatial region defined by the tightly bound core state, thus the electron excitation is localized at the atom cores. The measured intensity is then proportional to the local density of states at each atom site summed over all the atoms probed in the experiment. For example, if the probed volume of diamond contains n_c atoms on bulk diamond sites and n_d atoms on defect sites, which are perhaps lying along a dislocation core, and $n_d \ll n_c$ then the intensity of the pre-edge structure due to the defect band relative to that of the σ bulk crystalline band would be

$$\frac{I_{\mathrm{DB}}}{I_{\mathrm{CB}}}=\frac{D_{\mathrm{DB}}}{D_{\mathrm{CB}}}\frac{n_d}{n_c} . \tag{2}$$

The dimension of the probed volume is determined by the product of the foil thickness with effective cross-sectional area of the beam-specimen interaction, the diameter of which is the beam size, ~ 8 Å, convoluted with the inelastic interaction parameter. The value of the interaction parameter is of the order of magnitude of the spatial extent of the electric field that is generated by the excitation in response to the applied field of the fast electron. An upper limit of the interaction parameter is given by the classical impact parameter for an inelastic scattering process (Colliex outlines a simple derivation of this parameter in his review[1]). A 290-eV loss by 100-keV electrons corresponds to an impact parameter of about 3.5 Å.

In favorable situations it has been predicted[1] that one can detect a few tens of atoms by EELS. This estimate is limited by signal counting statistics (which is a function of beam current density and counting time), accuracy of background removal, knowledge of the relevant cross section, and the collection efficiency and stability of the microscope system. Hence on a very low background, such as we have achieved in these present experiments, it would not be unreasonable to assume that the EEL pre-edge structure of a dislocation corresponds to about ten defect states over a line length of approximately 2 nm, that is, about one state per atom. This very small number suggests that the absolute intensity of the defect band is an extremely sensitive function of the experimental geometry. In fact the reduction in the intensity noted at two of the dislocations might be the result of a slight misplacement of the beam relative to the core. There is an uncertainty in its precise location because the width and position of the strain contrast image is a function of microscope imaging conditions. An alternative explanation for the variations in intensity at the dislocation is that there is some unseen modification of the surface layer produced by the specimen preparation technique. It would seem reasonable to assume that this effect would be limited to dislocations intersecting or passing very close, say within about 5 nm, to the foil surface. This is most unlikely in every case here because only short segments in the center of the dislocations were examined, leaving a small chance that the dislocation was of the $\langle 1,\bar{1},0\rangle$ family running very close and parallel to the surface. Further evidence against this is that spectra recorded at different locations around the dislocation show no detectable differences.

The effect of the long-range strain field around the dislocation requires some comment. Pennycook *et al.*[13] estimated that the strain alone, ignoring details of dislocation structure, might reduce the indirect band gap by as much as 0.6 eV from 5.5 to 4.9 eV. This would result from the reduction of the overlap of bonding *s* and *p* orbitals in the expanded lattice. In the energy-loss spectrum, this effect should be manifested as a shift in the *K*-edge threshold energy. We have been unable to observe any dependence of the threshold energy as a function of the probe location both on the strained regions around the dislocations and the strain-free energy. We note also that we have been unable to detect any shift in the range of diamond types (namely, types I*a*, I*b*, and II*a*).

Alternatively, the pre-edge structure may reflect the presence of extrinsic states due to impurity donor or acceptor species, such as boron and nitrogen or the decoration by mineral precipitates. Yamamoto *et al.*[14] preferred the donor-acceptor model as an explanation for the production of luminescence although there is no direct experimental evidence for impurity atoms along the dislocation. Examination of the EELS over an extended energy range has not revealed the presence of any other chemical species, although detection sensitivity may not be adequate to expose the total number of impurity atoms located along the short segment of dislocation which is probed. The presence of nanometer-sized inclusions is unlikely as these should have been observed in either the annular dark field image or the band-gap energy filtered image. Both techniques are able to reveal very small mineral particles in diamond.[32]

We do not expect to observe any effect due to the passivation of the defect states by hydrogen atoms because the electron beam is likely to desorb hydrogen rapidly from the probed volume.[33] Other radiation damage effects are unknown, but are not believed to be significant. There was no observable change in the bright field image of each dislocation following a spectrum acquisition.

Finally, we turn our attention to a more direct comparison of our measurements with the calculations of Jones and King[6] and Persson.[7] In the light of the foregoing discussion it would be naive at this stage to draw firm conclusions from such a comparison. Theory indicates that both the 60° and 30° unreconstructed partials contain broad bands in the energy gap of diamond which are removed from the gap upon reconstruction. If the Fermi-level position is between the filled and unfilled states within the gap then Ref. 6 predicts an onset of vacant states occurring at 3.5 eV below the conduction-band minimum. In these experiments, we measure the 1*s*-core-level–to–conduction-band minimum at 288.8 eV for the bulk material. The core binding energy level at the dislocation is likely to be reduced from its bulk value. Photoelectron data[28] show that the core-level binding energy at (111) diamond surfaces is reduced by 0.8 eV. If a similar shift occurs for atoms located along the core of a dislocation one might expect an onset for the defect band due to 1*s*–to–dislocation-state transitions at $288.8-0.8-3.5=284.5$ eV. We have ignored core-hole interactions in this estimate. A 0.2-eV bound exciton may be expected to alter this estimate slightly. It seems unreasonable to assume that all atoms located at the dislocation have the same core binding energy, but rather they would have a range of values, perhaps varying by as much as ±0.4 eV. Thus the observed pre-edge structure should be a self-convolution of the density of states function over this energy range which would have the apparent effect of filling the entire gap with vacant states as observed.

V. CONCLUSIONS

This study has examined the electron-energy-loss spectra in the narrow energy range centered on the *K*-edge threshold energy. The results are summarized as follows.

(a) The bulk absorption spectra is consistent with the detail of the conduction-band density of states.

(b) No core-hole exciton is observed in the present study.

(c) The major component to the pre-edge structure originates at the surface and is due to an amorphous layer or intrinsic surface states.

(d) In the neighborhood of a dislocation there is an additional component to the losses in the band-gap region. This is consistent with the concept of a band or deep-level states generated by the presence of dangling bonds or a π-bonded reconstructed structure along the dislocation core.

(e) The current study has failed to reveal additional chemical components or mineral particles thought to decorate dislocation structures.

The present studies suggest further work may be of interest. In particular, a careful correlation between the loss spectrum and the direction of the momentum transfer relative to the dislocation line direction and Burgers vector. This may provide detail on the symmetry and direction of the deep-level states that are pinning the Fermi level. Spectra acquired as a function of well-defined impact parameter should be informative as to physics of electron scattering near small scale structures.

The absence of the exciton is intriguing and has not been explained yet. Improved statistics of surface sensitive data to try to simulate the photoyield experiment may help to resolve this.

ACKNOWLEDGMENTS

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[1] See, for example, a review by C. Colliex, *Advances in Optical and Electron Microscopy*, edited by R. Barer and V. E. Cosslett (Academic, New York, 1984), Vol. 9.

[2] W. Shockley, Phys. Rev. **91**, 228 (1953).

[3] W. T. Read, Philos. Mag. **45**, 775 (1954).

[4] A general review is given by P. B. Hirsch, Mater. Sci. Technol. **1**, 666 (1985).

[5] J. R. Chelikowsky and J. C. H. Spence, Phys. Rev. B **30**, 694

(1984).

[6]R. Jones and T. King, Physica B+C **116B**, 72 (1983); J. Phys. (Paris) Colloq. **44**, C4-461 (1983).

[7]A. Persson, J. Phys. (Paris) Colloq. **44**. C4-453 (1983); Ph.D. thesis, University of Umea, Sweden, 1983.

[8]A. A. Bonapasta, Solid State Commun. **65**, 245 (1988).

[9]P. Pirouz, D. J. H. Cockayne, N. Sumida, P. B. Hirsch, and A. R. Lang, Proc. R. Soc. London, Ser. A **386**, 241 (1983).

[10]I. Kiflawi and A. R. Lang, Proc. R. Soc. London, Ser. A **386**, 219 (1974); Philos. Mag. **30**, 219 (1974); **33**, 679 (1976).

[11]P. L. Hanley, I. Kiflawi, and A. R. Lang, Philos. Trans. R. Soc. London, Ser. A **248**, 329 (1977).

[12]N. Sumida and A. R. Lang, in *Microscopy of Semiconducting Materials,* Inst. Phys. Conf. Ser. No. 60, edited by A. G. Cullis and D. C. Joy (IOP, London, 1981), p. 319.

[13]S. J. Pennycook, L. M. Brown, and A. J. Craven, Philos. Mag. A **41**, 589 (1980).

[14]N. Yamamoto, J. C. H. Spence, and D. Fathy, Philos. Mag. B **49**, 609 (1984).

[15]S. L. Pennycook, Ultramicroscopy **7**, 99 (1981).

[16]P. E. Batson (unpublished); in *Proceedings of the 47th E.M.S.A., 1988,* edited by G. W. Bailey (San Francisco Press, San Francisco, 1989), p. 376.

[17]S. D. Berger and L. M. Brown, in *Proceedings of the Electron Microscopy and Analysis Group 1983,* Inst. Phys. Conf. Ser. No. 68, edited by P. Doig (IOP, London, 1984), p. 115.

[18]P. E. Batson, Rev. Sci. Instrum. **57**, 43 (1986); **59**, 1132 (1988).

[19]J. Bruley and L. M. Brown, Philos. Mag. A **59**, 247 (1989).

[20]A previous Burgers vector analysis of several dislocations in this stone by Pennycook *et al.* (see Ref. 13) showed them to be chiefly of the glide $\frac{1}{2}(1,1,0)$ variety with a small fraction of screw-type dislocations.

[21]G. Comelli, J. Stohr, W. Jark, and B. Pate, Phys. Rev. B **37**, 4383 (1988).

[22]S. D. Berger, D. R. Mckenzie, and P. J. Martin, Philos. Mag. B **52**, 285 (1988).

[23]G. S. Painter, D. E. Eillis, and A. R. Lubinskey, Phys. Rev. B **4**, 3610 (1971).

[24]X. Weng, P. Rez, and H. Ma, Phys. Rev. B **40**, 4175 (1989).

[25]J. F. Morar, F. J. Himpsel, G. Hollinger, G. Hughes, and J. L. Jordon, Phys. Rev. Lett. **54**, 1960 (1985).

[26]E. A. Zhurakovskii and Ya. V. Zaulichnyi, Fiz. Tverd. Tela (Leningrad) **27**, 3452 (1985) [Sov. Phys.—Solid State **27**, 2081 (1985)].

[27]D. Misell and A. F. Jones, J. Phys. A **2**, 540 (1969).

[28]J. F. Morar, F. J. Himpsel, G. Hollinger, G. Hughes, and J. L. Jordon, Phys. Rev. B **33**, 1340 (1986).

[29]J. F. Morar, F. J. Himpsel, G. Hollinger, G. Hughes, and J. L. Jordon, Phys. Rev. B **33**, 1346 (1986).

[30]M. Inokuti, Rev. Mod. Phys. **43**, 297 (1971).

[31]R. D. Leapman and J. Silcox, Phys. Rev. Lett. **42**, 1361 (1979).

[32]J. Bruley, L. M. Brown, and S. D. Berger, in *Proceedings of the Electron Microscopy and Analysis Group 1985,* edited by G. Tatlock, Inst. Phys. Conf. Ser. No. 78 (IOP, London, 1985), p. 561.

[33]S. V. Pepper, J. Vac. Sci. Technol. **20**, 643 (1982).