EELS characterization of highly irradiated PVC

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Highly irradiated PVC sample was analyzed by EELS in transmission using in a JEOL-2010 Transmission Electron Microscope equipped with a Gatan - 666 PEELS spectrometer. A primary energy of 200 Ke V was used and the average time for a single run was about 5 minutes. The results were compared with a clean HOPG surface (0001), amorphous carbon surface and a diamond thin film grown by Hot Filament Chemical Vapor Deposition (HFCVD) [1]. In order to obtain the local structure, the standard procedure developed for EXAFS was applied

Figure 1 shows the main features of the electron energy loss spectra near the K-edge in the transmission mode for (a) amorphous carbon, (b) HOPG,(c) diamond thin film (cross section), (d) natural diamond and (e) irradiated PVC. These spectra are shown without background subtraction. Amorphous carbon and HOPG have basically the same edge shapes which may be explained in terms of similarities in their bonding, particularly the σ^* and π^* transition are clearly absent as expected. Thus, through these spectra one may distinguish among different structures formed by carbon atoms. Similar results have been reported for graphite and natural diamond. The near edge structure is so different among these three forms of carbon that EELS spectra should suffice as a fingerprint for the identification of these structures. Highly irradiated PVC shows a smoothed shape indicating strong differences among the known carbon structures. At first glance the PEELS spectra of these materials show a resemblance to amorphous carbon even though at a microscopic level, the irradiated polymer shows a very well defined crystallographic structure, as shown in Figure 3, which has not been unambiguously identified [2].

Figure 2 shows the radial distribution function obtained by applying the Fourier transform to the EXEELFS spectra shown in Figure 1. Numerical values are shown in Table 1 where a comparison is made between both modes of observation for the set of samples under study. Also shown are results obtained by other groups.

We conclude that through a systematic study of different forms of carbon, we have obtained radial distribution functions allowing the possibility to distinguish different structures formed by carbon atoms in samples with a spatial resolution much less than 1µm (transmission mode). On the other hand we conclude that the crystalline structure of the highly irradiated PVC is not strictly a new form of carbon. However is necessary to perform additional experimentswith this sample.

1. G.A. Hirata, L. Cota, et. al., Diamond and Rel. Mat., (1993), 3,177

2. L. Cota et. al., Radiant. Phys. Chem. (1994),44,579

Table 1. Numerical values obtained applying the EXEELS analysis.

Structure	Our results (nm)	Other authors (nm)	References (nm)
Amorphous carbon	0.148±0.002	0.144±0.002	0.144
	0.248±0.002	0.254±0.003	0.256
Graphite (HOPG)	0.157±0.002	0.118±0.002	0.142
	0.244±0.002	0.227±0.002	0.246
Diamond thin film	0.156±0.002	0.161±0.003	0.154
	0.254±0.002	0.290±0.003	0.251
Highly irradiated PVC	0.150±0.002	no reference	no reference
	0.259±0.002		

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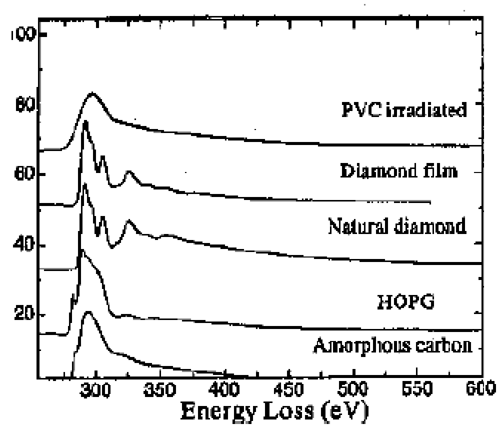

Figure1. Extended fine structure in the transmission mode

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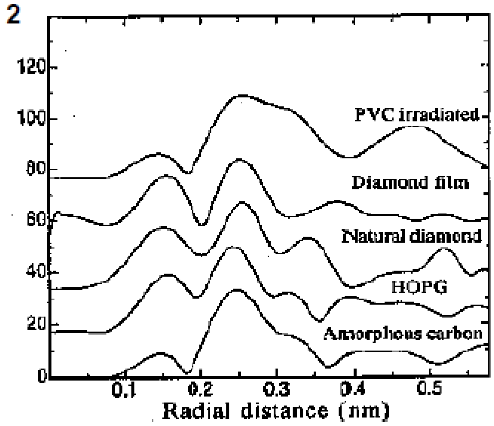

Figure 2. Radial Distribution Function corresponding to Fig. 1

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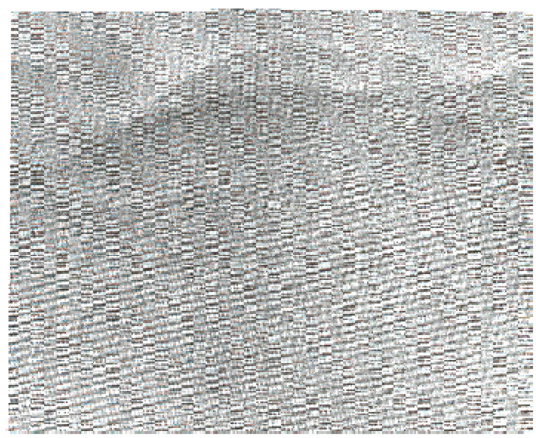

Figure 3. HRTEM micrographs of irradiated (10,000 Mrads) poly(vynilchloride)