

Electron Radiation Damage of Pentacene Thin Films Measured in TEM

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We have investigated the mechanism of radiation damage (RD) in pentacene thin films by diffraction ring fading (loss of crystallinity). Pentacene has application in large area flexible organic electronics [1]. Pentacene films can be studied by TEM if artifacts from RD are avoided.

We have grown 70nm thick pentacene films onto carbon coated TEM grids by sublimation from a Knudsen cell. The samples were characterized in a JEOL 2010 at 200 kV. The intensity of each diffraction ring was integrated over all azimuthal angles. The integrated diffraction intensity (IDI) fitted to a Lorentzian was plotted as a function of electron dose. Fig. 1 shows the typical initial film (lower left) and a damaged film (upper right) and corresponding diffraction patterns while Fig. 2 shows dependence of IDI on radiation dose for a sample at room temperature (RT) and 90 K. The characteristic doses $D_{1/e}$ estimated from the region of Fig. 2 where IDI for a given reflection decreases (excluding the latent dose where little change of IDI takes place) are given in Table 1.

Measured $D_{1/e}$ were compared to calculated $D = e/\sigma$; here e is electron charge and the cross section σ was calculated for carbon 1s using the SIGMAK3 program [2]. The σ for valence electrons (including hydrogen 1s shell) was calculated from measured plasmon energy $E_p = 23.6$ eV and width of the plasmon resonance $\Delta E_p = 16.7$ eV using

$$\sigma_{tot}^{val} = \frac{1}{\pi a_0 m_0 v^2 n_a} \int_0^{E_0} (\ln[\theta_E^2 + 2\theta_E] - \ln[\theta_E^2]) \Im\left(-\frac{1}{\epsilon(E)}\right) dE$$

where the imaginary part of dielectric function $\epsilon(E)$ is

$$\Im\left(-\frac{1}{\epsilon(E)}\right) = \frac{E \Delta E_p E_p^2}{(E^2 + E_p^2)^2 + (E \Delta E_p)^2}$$

a_0 is Bohr radius, E_0 incident energy, m_0 electron mass, v electron velocity and n_a is number of atoms per unit volume. These calculated doses $D_{1/e}$ per atom were converted to $D_{1/e}$ per molecule by multiplying by the number of atoms (22 for C 1s, 36 for valence shells) [3]. IDI for higher-index rings in Fig. 2 show a monotonic decay with dose but lower- index rings either stay unchanged or become more intense before decaying. This might be explained by considering two competing mechanisms: the decrease of IDI due to damage of the crystal structure and a mechanism of IDI increase (such as change of crystallite orientation closer to Bragg condition). It should be noted that $D_{1/e}$ (Table 2) for the valence excitations is too low to explain the measured $D_{1/e}$ (Table 1) unless there is an efficient healing mechanism repairing about up to 45% of bond at RT and about 99 % of bond at 90 K. $D_{1/e}$ at 90K corresponds well to $D_{1/e}$ for a single carbon-1s excitation per molecule, suggesting (but not proving) that the damage mechanism is related to carbon 1s excitation [4].

References

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- [3] Egerton RF, Li P, Malac M, *Micron*, 35 (2004), p. 399.
- [4] Reimer L, *Laboratory Investigation*, 14 (6), (1965) page 344.

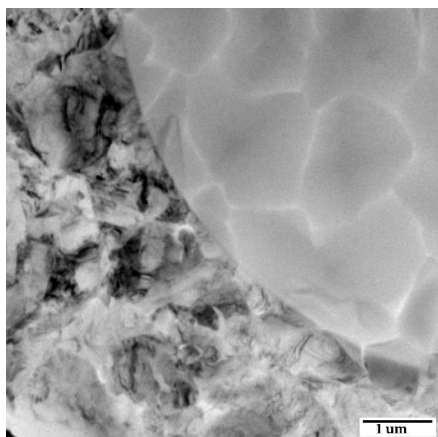


Figure 1 a) Pentacene film. Damaged area in upper right, pristine film in lower left.

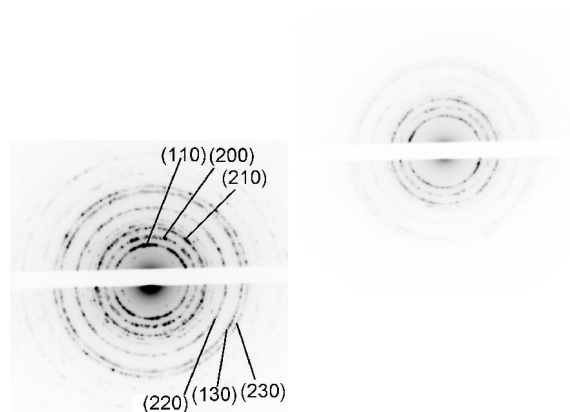


Figure 1b) Diffraction patterns from damaged (above) and pristine area (below).

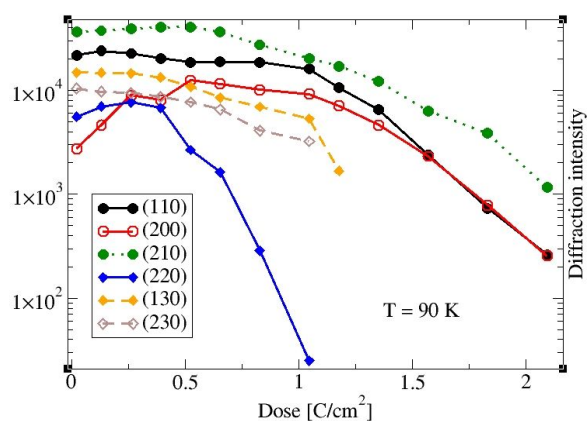


Figure 2a) Diffraction intensity as function of dose at 90K.

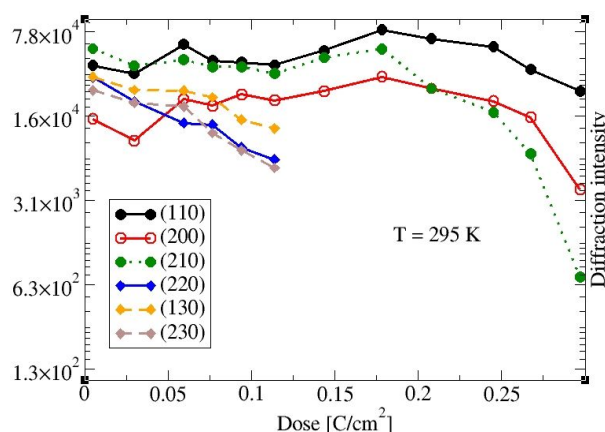


Figure 2b) Diffraction intensity as function of dose at 295 K.

TABLE 1 Measured damage doses.

Temp.	Dose rate j [A/cm ²]	Miller index	Latent dose D_L [C/cm ²]	Char. dose $D_{1/e}$ [C/cm ²]	Critical dose D_c [C/cm ²]
295K	0.6×10^{-3}	110	0.18	0.1	0.45
		200	0.18	0.06	0.45
		210	0.18	0.03	0.45
		220	< 0.03	0.07	0.14
		130	< 0.03	0.11	0.14
		230	< 0.03	0.07	0.14
90K	4.3×10^{-3}	110	1.1	0.5	2.4
		200	~1	0.4	2.4
		210	0.7	0.5	2.4
		220	NA	0.3	1.2
		130	< 0.3	0.6	1.2
		230	< 0.1	0.85	1.2

TABLE 2 Calculated damage doses.

C 1s $D_{1/e}$ 1/atom [C/cm ²]	valence elect. $D_{1/e}$ 1/atom [C/cm ²]	C1s $D_{1/e}$ 1/molec. [C/cm ²]	valence elect. $D_{1/e}$ 1/molec. [C/cm ²]
10	0.06	0.47	1.5×10^{-3}