RADIATION DAMAGE IN THE TEM

R.F. Egerton, Physics Department, University of Alberta, Edmonton, Canada T6G 2J1
Email: regerton@ualberta.ca

Radiation damage to a specimen provides the *ultimate* limit to the information that can be obtained by electron-beam observation, and it becomes the *practical* limit as the instrumentation and operating procedures are perfected. Nearly all specimens suffer some form of radiation damage. Even simple metals can show knock-on displacement damage: the creation of crystal defects due to the energy transferred to atomic nuclei during high-angle *elastic* scattering. Needing an energy transfer greater than 10 eV [1], displacement occurs only for higher incident energies; see Table 1. A directly related effect is the electron-induced sputtering of surface atoms, which requires energies of less than 10 eV and (for 200keV primary electrons) occurs for nearly all elements of atomic number below 50; see Table 2. Although the cross section for such damage is relatively low, thinning rates of many nm/s can be predicted for a focused field-emission probe (Table 2) and possibly ten times higher for an aberration-corrected probe. The removal of atoms from a specimen surface leads to thinning (mass loss) and eventually to the formation of a hole in the TEM specimen. In principle, sputtering can be avoided by coating one or both surfaces of the specimen with a thin layer of high-Z material (threshold energy > incident-electron energy) but a suitable coating material (which forms a structureless continuous film) may not exist.

In compounds, *radiolysis* (resulting from *inelastic* scattering of the incident electrons) greatly increases the damage rate. Hole drilling can occur in oxides [3] and for halides both defect creation and mass loss [4] have been studied. With organic and biological specimens, radiolysis effects are rapid and place a practical limit on the spatial resolution of TEM imaging, electron diffraction and spectroscopy. Various procedures have been used to reduce the damage rate, including surface coating and cooling the specimen below room temperature. It has been reported that the damage is less (for a given dose, expressed in C/cm^2) in the case of very small probes [5,6]. A plausible explanation [6] is that the damage is caused mainly by fast secondary electrons (FSE) that deposit their energy over a distance of several nm and damage the surrounding region of specimen rather than the probed area; see Fig. 1. Monte Carlo calculations of FSE trajectories suggest that this effect reduces damage within the probe by only a modest factor. Nevertheless, it is likely that secondary electrons (including slow secondaries, energy < 50 eV) are the major source of damage in organic materials, in both the TEM and the x-ray transmission microscope.

Elemental carbon represents an interesting situation, intermediate between organic and inorganic materials. Whereas damage in diamond, graphite and carbon nanotubes probably arises only from knock-on displacement [8], damage to solid C_{60} (Fig. 2) appears to involve radiolysis [9].

References

[1] Hobbs, L. in *Quantitative Electron Microscopy* (SUSSP, 1984), Chapter 11.
[2] R.F. Egerton, P. Li and M. Malac, Micron **35** (2004) 399.
[3] C.J. Humphreys et al., Scanning Microsc. Suppl. **4** (1990) 185.
[4] A. Muray et al., J. Vac. Sci. Technol. B **3** (1985) 367.
[5] K. Varlot et al., Ultramicroscopy **68** (1997) 123.
[6] K. Siangchaew and M. Libera, Phil. Mag. A **80** (2000) 1001.
[7] R.F. Egerton and M. Malac, Microsc. Microanal. **10** (Suppl. 2, 2004) 1382.
[8] V. Crespi et al., Phys. Rev. B **54** (1996) 5927.
[9] R.F. Egerton and M. Takeuchi, Appl. Phys. Lett. **75** (1999) 1884.

material	E_d(eV)	E_0(min) (keV)	$T_{0.5}$(s)
diamond	80	330	2.5
graphite	30	140	0.53
aluminum	17	180	0.16
silicon	16	175	0.13
iron	17	330	0.12
copper	20	420	0.14
gold	34	1340	

Table 1: Energy E_d required for atomic displacement in various materials, together with the threshold incident-electron energy E_0(min) and the calculated average time between atomic displacements for a electron current density of 5 x 10^4 A/cm^2 (focused 200keV field-emission probe *without* aberration correction).

element	E_0(min) (keV)	S(nm/s) for spherical SP	S(nm/s) planar SP
Be	13	136	22
C	38	83	20
Mg	16	1005	179
Al	40	382	95
Si	56	270	76
Ti	96	248	86
V	110	194	71
Cr	88	354	118
Mn	67	661	197
Fe	98	329	115
Co	107	288	104
Ni	108	305	110
Cu	92	486	164
Zn	38	2280	555
Ge	114	353	130
Zr	213	0	0
Mo	239	0	0
Pd	161	189	82
Ag	127	557	213
Sn	147	368	152

Table 2: Threshold incident energy E_0(min) for electron-beam sputtering of elemental solids. The last two columns give the sputtering rate S for a focused field-emission probe (5 x 10^4 A/cm^2) of 200keV electrons, calculated for a clean surface using Mott cross sections with two approximations for the symmetry of the surface potential (SP).

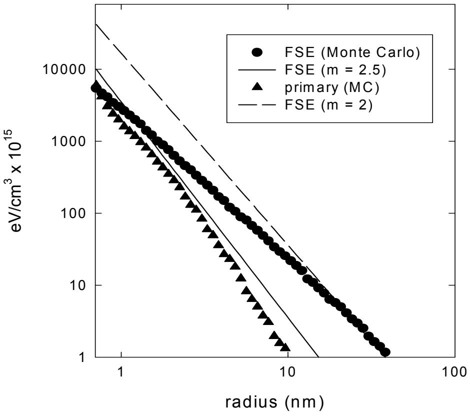

Figure 1. Energy deposition per unit volume (proportional to damage rate) in PMMA, as a function of distance from the centre of a small-diameter probe of 200keV electrons. Circles and triangles show the results of our Monte Carlo calculations; lines represent the predictions of an analytical formula [7], assuming the energy-loss spectrum to be proportional to E^{-m} for E > 50 eV.

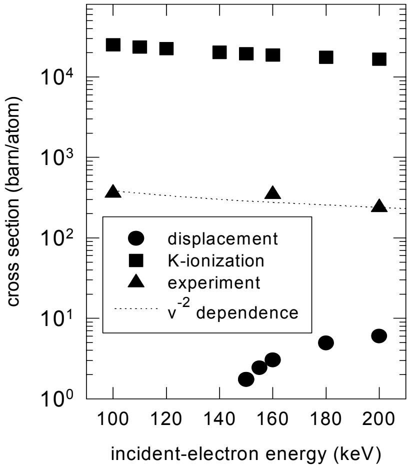

Figure 2. Cross sections (per atom) for carbon-atom displacement and for K-shell ionization, as a function of primary-electron energy, compared with the damage cross section measured for films of solid C_{60} [9].