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Mechanisms of Radiation Damage and Electron-Beam Fabrication

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Electron beams can be used to modify material properties and to examine the changes produced. A good example is the work of Humphreys and co-workers, who used a CFEG-STEM to drill holes in thin alumina films and EELS to measure changes in thickness, formation of metallic aluminum and oxygen bubbles, etc. [1]. Hole drilling has been discussed in terms of a Coulomb explosion resulting from buildup of space charge [2] but this idea has been superseded by an exciton mechanism in the case of radiation-induced decomposition of alkali halides [3]. Clearly the mechanism of radiation-induced change depends on details of the bonding and energy-band structure.

Radiation damage in *organic* materials has been extensively investigated, yet there seem to be many open questions, such as how much of the damage is created by fast or slow secondary electrons and exactly how far these electrons travel. The greater radiation resistance of *aromatic* compounds is attributed to the high resonance energy of their ring structures but the role of K-shell excitation, leading to a possible threshold incident energy, remain controversial topics [4-7]. A reported huge increase in characteristic dose with decreasing electron-beam diameter [8,9] may be the result of delocalization of the damage (due to secondary electrons) combined with delocalization of the inelastic scattering giving rise to the 7eV peak that was used to monitor the damage [10].

Radiation damage mechanisms can be broadly divided into displacement or knock-on processes, caused by nuclear (elastic) scattering of the electrons, and radiolysis or ionization mechanisms that result from inelastic scattering by atomic electrons. Of the two, radiolysis is associated with lower characteristic dose D_e and higher cross section ($\sigma = e/D_e$) and dominates in nonconducting materials, which are therefore relatively radiation-sensitive; see Table 1.

While radiation damage may be the nanotechnologist's friend, it is certainly the electron microscopist's enemy, so various methods of minimizing the damage have been devised. They include: cooling or coating the specimen, substitution of hydrogen by heavier elements, dispersion and confinement (e.g. in nanotubes) and in the future perhaps fast irradiation, if femtosecond techniques (as used successfully for x-ray diffraction of virus and proteins) can be developed.

Once instrumental problems (drift, instability) are overcome, radiation damage provides the ultimate limit to the spatial resolution of electron-microscope imaging and elemental analysis. Because of its high signal-collection efficiency, EELS offers the possibility of detecting single atoms and even analyzing their electronic states (via ELNES), whereas x-ray emission spectroscopy seems unlikely to achieve the single-atom (rather than atomic-column) detection because the cross section for signal collection is lower than a typical cross section for damage; see Table 1.

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TABLE 1. Mechanisms of radiation damage and typical values of characteristic dose D_e, cross section σ per atom (in barn = $cm^2 \cdot 10^{-24}$) and displacement energy E_d. Also given are the EELS and x-ray cross sections for selected elements, showing that detection and ELNES analysis of single atoms is possible by EELS in most inorganic and conducting materials, whereas radiation damage precludes single-atom detection by EDXS, even with a high-efficiency detector (solid angle taken here to be 0.9 sterad).

mechanism	specimen	$D_e(C/cm^2)$	σ (barn)	E_d(eV)
radiolysis	organic	0.002 - 1	10^5 - 10^8	
radiolysis	inorganic	0.2 - 10^6	0.1 - 10^6	
bulk displacement	conducting	10^3 - 10^4	10 - 100	10 - 50
bulk diffusion	conducting		10^2 - 10^4	0.5 – 1.5
surface sputtering	conducting		10^2 - 10^3	1 - 10
surface diffusion	conducting		> 10^3	< 1
K-shell EELS	Z = 4 to 9		10^2 – 10^5	
K-shell ELNES	Z = 4 to 9		10 – 10^4	
K-shell x-ray peak	C,Na,Ti,Ni		2 – 10	

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