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The Interaction of Low-energy Electrons with Soft Materials in the Electron Microscope

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Experimental studies of soft materials in the electron microscope are constrained by the damage caused by inelastic scattering processes. Typically, most soft materials can tolerate incident electron doses on the order of about 10 - 10,000 e/nm^^2, depending on the material, before exhibiting significant changes to their structure and/or chemistry. There has recently been substantial interest in using lower-energy electrons for soft-matter characterization. However, while lower-energy incident electrons can reduce knock-on damage, ionization damage remains the limiting factor, and, like the scattering cross section for inelastic scattering, it increases with decreasing electron energy. We have used a combination of scanned-probe microscopy, scanning electron microscopy (SEM), and confocal immunofluorescence imaging to study the effects of focused electron beams with energies ranging from 2 - 30 keV and point doses ranging from 10 - 1000 fC on thin films of poly(ethylene glycol) [PEG] ~100 nm thick on silicon substrates. STEM EELS studies at 200 keV show that PEG evolves hydrogen when irradiated and suggests that it undergoes free-radical radiolytic polymerization. We have observed at 2-30 keV that electron irradiation ultimately crosslinks the PEG and grafts it to the underlying substrate to create surfaces patterned with PEG microgels of interest for biomaterials applications. Consistent with the experimental data, Monte Carlo simulation of electron energy deposition identifies three types of structure within each microgel: a highly crosslinked core near the point of electron incidence; a lightly crosslinked near-corona surrounding the core; and a far-corona localized at the PEG-Si substrate created by electrons backscattered from the silicon substrate. The nature and relative sizes of these three regions and, hence, the protein-interactive and cell-interactive character of the microgels depend strongly on the incident electron energy and electron dose as well as on the molecular weight and thickness of the precursor PEG thin film.