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Damage Generation in Ultra Nano-crystalline Diamond by Low-energy Electron Irradiation

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Gas-mediated electron beam induced etching (EBIE) is used to analyze damage generation in ultra nano-crystalline diamond (UNCD) irradiated by a low-energy electron beam. Specifically, we use H_2O-mediated EBIE to quantify the volatilization rate of UNCD, and show that it is rate-limited by an electron stimulated carbon restructuring process. The observed behavior contradicts existing EBIE models which predict a volatilization rate that is proportional to the precursor (H_2O) dissociation rate. The models are modified to reproduce the measured etch kinetics, and can now be used to characterize low-energy electron beam damage kinetics in UNCD. EBIE is a nano-scale, direct-write technique analogous to gas-assisted focused ion beam (FIB) milling. However, low-voltage EBIE is a chemical process that does not involve sputtering or knock-on damage. H_2O-mediated EBIE of carbon proceeds through electron induced dissociation of surface adsorbed H_2O molecules generating fragments (e.g., O and OH) that react with the substrate. Volatile species (e.g., CO and CO_2) produced in these reactions can desorb, thus giving rise to localized chemical dry etching under an electron beam. EBIE of diamond, carbon nanotubes and amorphous carbon has been demonstrated previously. However, the etch mechanisms have not been investigated in detail, and quantitative EBIE has previously not been used to compare volatilization rates and detect electron restructuring effects in different types of carbon. We show that as-grown UNCD and highly ordered pyrolytic graphite (HOPG) both exhibit negligible etching ascribed to a low volatilization rate of sp^2 and sp^3 rich carbon by reactive fragments produced by electron induced dissociation of H_2O adsorbates. Etching of UNCD accelerates significantly upon irradiation by low-energy (<~ 20 keV) electrons, and the rate scales inversely with electron beam energy and directly with energy density deposited into the solid. EBIE of UNCD is shown to proceed through an electron restructuring pathway that generates defect rich carbon that is susceptible to volatilization, a behavior that is not observed in HOPG, which etches slower than electron irradiated UNCD. Electron restructuring of a substrate has previously not been shown to rate limit EBIE. Most detailed prior work on restructuring of diamond and graphitic carbon by charged particles is limited to electrons with energies above ~ 100 keV, and energetic ions. Our results demonstrate the utility of EBIE for characterizing damage produced by low-energy electrons, where atom displacements caused by momentum transfer to carbon nuclei are negligible.