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Electron Irradiation Damage of MgO Nanocube

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The irradiation effect of ceramics has been intensively studied for many years due to the important applications of ceramics in irradiation environments. As one of the most important ceramic materials, MgO received special attention in the fields of high energy ionic radiation and surface science [1, 2]. In our work, the electron beam irradiation damage of MgO is studied *in-situ* with PEELS in a HF-3300 CFEG-TEM at an acceleration voltage of 300 KV. The MgO TEM sample was prepared by burning the Mg strip in the air and the smoke was collected on the holey carbon TEM grid.

Figure 1 shows TEM micrographs of MgO nanocubes suspended across a hole of carbon film, aggregated together by sharing the planes or edges. The dark speckles originate from the surface contamination during exposure in the air. The edge contrast reverses through focus as predicted by the projected charge density (PCD) approximation [3]. A single nanocube was selected with a [001] orientation to obtain uniform thickness along the electron beam direction and convergent-beam low-loss spectra collected to monitor irradiation effects. Mass loss was characterized in terms of relative thickness (t/λ), as shown in Figs. 2(a) and 2(b). The linear fits suggest mass loss by electron-beam sputtering and reveal a reduction about 28% when the MgO cube is on a carbon support than when MgO is in vacuum. However, the relative thickness increased and the fine structure in the MgO spectrum was gradually covered by the broad carbon plasmon peak, showing that carbon contamination can overwhelm mass loss due to irradiation.

Figure 3(a) shows a set of MgO valence-loss spectra collected on one nanocube, all normalized to the relative thickness (t/λ). The energy resolution measured from the full width at half maximum of zero-loss peak is 0.55 eV. A slight red-shift of the plasmon peak (around 22 eV) was observed in the damaged MgO nanocube. In addition, as shown in Figure 3(b), some weak peaks between 3.8 eV and 6 eV appeared with increasing electron dose, regardless of whether the MgO nanocube was supported by a carbon film or suspended in vacuum. Figure 3(c) obtained from the pure amorphous carbon (a-C) film shows π-plasmon peak around 5 eV appears apparently only when the thickness above 0.4 t/λ. The weak peaks in damaged MgO may have the contribution of carbon contamination; however, there are also contributions from oxygen vacancies (color centers) [4]: F^+ and F centers contribute to peak around 4.8 - 5.9 eV, while, M^+ and M centers appear around 3.9 - 4.7 eV. The peaks can not be attributed to Cerenkov radiation as they were observed only in damaged MgO and not observed in pristine MgO at both 300 kV and 100 kV.

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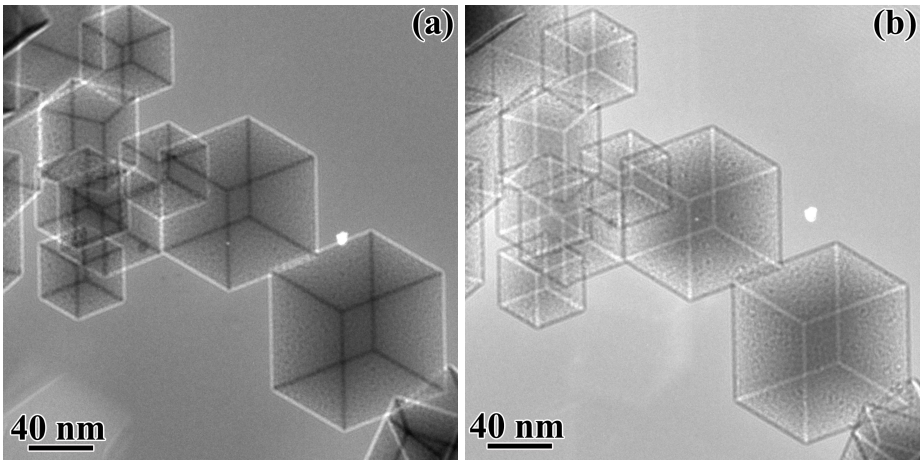

Fig. 1. (a) Under-focus and (b) over-focus TEM micrographs of MgO nanocubes suspending in the hole of the holey carbon grid, the white speckle in the image is a dust contamination in the CCD camera.

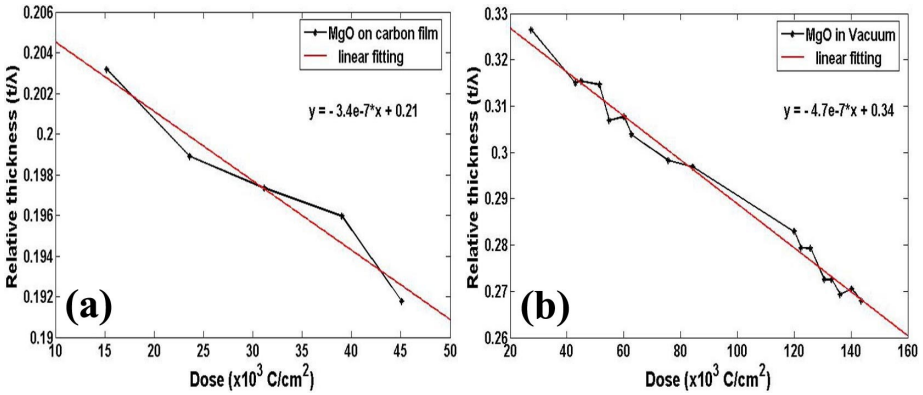

Fig. 2. The reduction of MgO nanocube relative thickness as a function of electron irradiation dose, measured for a MgO cube (a) on the carbon support and (b) in vacuum.

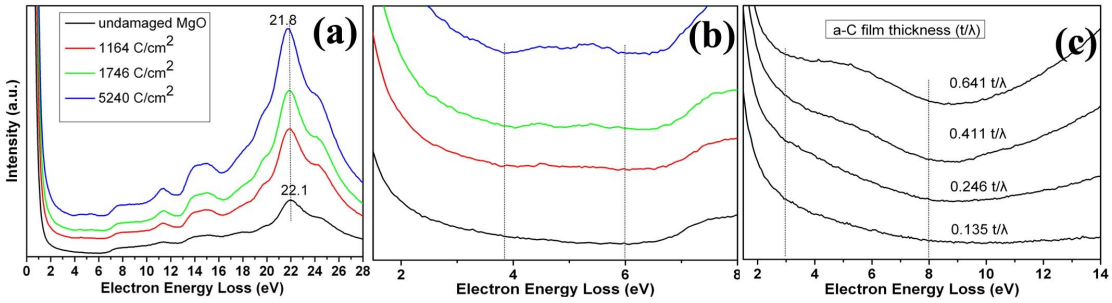

Fig. 3. (a) A set of low-loss spectra from an electron-irradiated MgO nanocube, (b) locally enlarged spectra, showing emerging fine structure, and (c) low-loss spectra of pure amorphous carbon film showing the π-plasmon peaks. All spectra are normalized to relative thickness (t/λ).

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