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Electron-irradiation-induced phase segregation in crystalline and amorphous apatite: A TEM study

A. MELDRUM, L.M. WANG,* AND R.C. EWING*

Department of Earth and Planetary Sciences, University of New Mexico, Albuquerque, New Mexico 87131-1116, U.S.A.

ABSTRACT

Single crystals of natural F-rich apatite and 800 keV Kr^{2+} ion-beam–amorphized apatite were irradiated by an electron beam in a transmission electron microscope over a range of beam energies and beam currents. Irradiation of crystalline apatite using a high current density (16 A/cm^2) caused the precipitation of cubic CaO from the crystalline apatite matrix. Using a lower beam current (1.6 A/cm^2), the formation of nanometer-sized voids was observed, but CaO did not crystallize even after prolonged irradiation. Amorphous apatite crystallized to a coarse-grained polycrystalline assemblage of apatite crystallites at 85–200 keV. Increasing the beam current through the sample caused the formation of fine-grained cubic CaO, and the crystallization of apatite was not observed, even at high doses. In each case, many beam-induced bubbles formed and were typically larger at the edge of the beam. Thermal annealing at 450 °C resulted in epitaxial crystallization from the thick portions of the TEM foil and resulted in a single crystal with a high defect density. Electron-beam irradiations at 300 °C confirmed that the difference in microstructural evolution as a function of current density is driven by dose-rate effects. In fact, temperature and dose rate are competing effects in the precipitation of CaO.