American Mineralogist, Volume 89, pages 245–248, 2004

In situ observation of the thermal decomposition of weddelite by heating stage environmental scanning electron microscopy

J. THEO KLOPROGGE,[1,*] THOR E. BOSTRÖM,[2] AND MATT L. WEIER[1]

[1]Inorganic Materials Research Group, School of Physical and Chemical Sciences, Queensland University of Technology, GPO Box 2434, Brisbane, Q 4001, Australia

[2]Analytical Electron Microscopy Facility, Queensland University of Technology, GPO Box 2434, Q 4001, Australia

ABSTRACT

The morphological and chemical changes occurring during the thermal decomposition of weddelite, $CaC_2O_4 \cdot 2H_2O$, have been followed in real time in a heating stage attached to an Environmental Scanning Electron Microscope operating at a pressure of 2 Torr, with a heating rate of 10 °C/min and an equilibration time of approximately 10 min. The dehydration step around 120 °C and the loss of CO around 425 °C do not involve changes in morphology, but changes in the composition were observed. The final reaction of $CaCO_3$ to CaO while evolving CO_2 around 600 °C involved the formation of chains of very small oxide particles pseudomorphic to the original oxalate crystals. The change in chemical composition could only be observed after cooling the sample to 350 °C because of the effects of thermal radiation.