Analytical Electron Microscopy in Clays and Other Phyllosilicates: Loss of Elements from a 90-nm Stationary Beam of 300-keV Electrons

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Abstract: Diffusion of alkali and low-atomic-number elements during the microbeam analysis of some silicates by analytical electron microscopy (AEM) has been known for some time. Our repeated analyses at 300 kV of kaolinite, halloysite, smectite, biotite, muscovite and pyrophyllite, however, showed differential loss (relative to Si) of not only alkali elements (such as K, Na, Mg) and low-atomic-number elements (such as Al) but also higher-atomic-number elements (such as Fe, Ti). For AEM of these phyllosilicates, a Philips EM430/EDAX facility with a tungsten filament was used to provide a current of 0.3 nA in a stationary beam of nominal diameter 90 nm. The loss of Al in kaolin minerals during analysis is particularly severe. Kaolin crystals can be damaged by the electron irradiation over several seconds, making it the most sensitive clay to the electron beam; in general, relative phyllosilicate stabilities are kaolin < smectite < pyrophyllite < mica. A clear dependence of element loss on crystallographic orientation has been observed for layer silicates in our study; a greater element loss occurred when the plane of the specimen foil was perpendicular to the basal planes of the phyllosilicate crystals than when the foil was parallel to the basal planes. Lower beam current, larger beam diameter and thicker specimens all reduce the loss of elements. The initial stage of irradiation produces highest rates of element loss and the rate of loss can be fitted by an exponential decay law. The analyses at low temperature of phyllosilicates showed that element loss remains serious in our analytical conditions. Since the element loss appears to be instrument- and method-dependent, one should use closely related, well-characterized phyllosilicates as compositional standards to calibrate any AEM instrument that is to be used to analyze unknown phyllosilicates, and the standards and unknowns should be analyzed under identical conditions.

Key Words: Analytical Electron Microscopy • Clay Minerals • Diffusion • Element Loss • Low Temperature • Phyllosilicates • Sputtering • Stationary Beam

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