Characterization of mineral surfaces using FIB and TEM: A case study of naturally weathered alkali feldspars

MARTIN R. LEE,^{1,*} DAVID J. BROWN,^{1,†} CAROLINE L. SMITH,² MARK E. HODSON,³ MAUREEN MACKENZIE,⁴ AND ROLAND HELLMANN⁵

¹Department of Geographical and Earth Sciences, University of Glasgow, Gregory Building, Lilybank Gardens, Glasgow, G12 8QQ, U.K. ²Department of Mineralogy, Natural History Museum, Cromwell Road, London, SW7 5BD, U.K.

³Department of Soil Science, School of Human and Environmental Sciences, The University of Reading, Whiteknights, Reading, RG6 6DW, U.K. ⁴Department of Physics and Astronomy, University of Glasgow, Glasgow, G12 8QQ, U.K.

partment of Physics and Astronomy, University of Glasgow, Glasgow, G12 8QQ, U.K.

⁵Environmental Geochemistry Group, LGIT, CNRS UMR 5559-OSUG-Université J. Fourier, France

ABSTRACT

Using a focused ion beam (FIB) instrument, electron-transparent samples (termed foils) have been cut from the naturally weathered surfaces of perthitic alkali feldspars recovered from soils overlying the Shap granite, northwest England. Characterization of these foils by transmission electron microscopy (TEM) has enabled determination of the crystallinity and chemical composition of near-surface regions of the feldspar and an assessment of the influence of intragranular microtextures on the microtopography of grain surfaces and development of etch pits. Damage accompanying implantation of the 30 kV Ga⁺ ions used for imaging and deposition of protective platinum prior to ion milling creates amorphous layers beneath outer grain surfaces, but can be overcome by coating grains with >85 nm of gold before FIB work. The sidewalls of the foil and feldspar surrounding original voids are also partially amorphized during later stages of ion milling.

No evidence was found for the presence of amorphous or crystalline weathering products or amorphous "leached layers" immediately beneath outer grain surfaces. The absence of a leached layer indicates that chemical weathering of feldspar in the Shap soils is stoichiometric, or if non-stoichiometric, either the layer is too thin to resolve by the TEM techniques used (i.e., ≤ 2.5 nm) or an insufficient proportion of ions have been leached from near-surface regions so that feldspar crystallinity is maintained. No evidence was found for any difference in the mechanisms of weathering where a microbial filament rests on the feldspar surface. Sub-micrometer-sized steps on the grain surface have formed where subgrains and exsolution lamellae have influenced the propagation of fractures during physical weathering, whereas finer scale corrugations form due to compositional or strain-related differences in dissolution rates of albite platelets and enclosing tweed orthoclase. With progressive weathering, etch pits that initiated at the grain surface extend into grain interiors as etch tubes by exploiting preexisting networks of nanopores that formed during the igneous history of the grain. The combination of FIB and TEM techniques is an especially powerful way of exploring mechanisms of weathering within the "internal zone" beneath outer grain surfaces, but results must be interpreted with caution owing to the ease with which artifacts can be created by the high-energy ion and electron beams used in the preparation and characterization of the foils.

Keywords: FIB, TEM, alkali feldspar, weathering