Mottled contrast in TEM images of mica crystals

DAVID CHRISTOPHER NOE* AND DAVID R. VEBLEN

Department of Earth and Planetary Sciences, The Johns Hopkins University, Baltimore, Maryland 21218, U.S.A.

ABSTRACT

Mottling in [υ0υ] micrographs is a common feature of phyllosilicates observed with transmission electron microscopes and has been attributed to various mechanisms. The similar appearance of the mottling among various samples suggests that it arises from a common mechanism. Diffraction-contrast experiments demonstrate that it is caused by variations in basal plane spacings. A second type of mottling was identified that has not been previously described. This mottling is most apparent in dark-field images obtained with the electron beam oriented approximately normal to the basal plane, but the mechanism responsible for this mottling could not be determined. Both types of mottling occur as primary features but can be affected or even created by beam-induced changes. The induced nature of some mottling suggests that caution should be used when interpreting mottled features.

INTRODUCTION

Mottling in TEM images of phyllosilicates is common and has been described by numerous previous investigators. A relationship between mottling and defects such as microcleavages and phase boundaries has been noted and generally attributed to strain contrast (Livi et al. 1997; Merriman et al. 1995; Jiang and Peacor 1993; Livi and Veblen 1987). Mottling has also been attributed to beam damage (Shau and Peacor 1992; Ahn et al. 1986), exsolution effects (Ferrow et al. 1990), Guinier-Preston zones (Page 1980), and “destabilized states” (De Parseval et al. 1994).

This study considers two different types of mottling. The first type is visible in micrographs that have an end-on view of the (001) basal planes and is here referred to as type 1 mottling. The second (type 2 mottling) has not previously been described and is best seen in dark-field (DF) images viewed normal to the basal planes. A combination of high-resolution TEM (HRTEM), selected-area electron diffraction (SAED), analytical electron microscopy (AEM), and electron microprobe analysis (EMP) has been used. Our goal is to determine if mottling is a primary feature or if it results from sample preparation or beam damage for this important group of minerals.

EXPERIMENTAL TECHNIQUES

Phyllosilicate crystals were selected for analysis from the Williams research collection at the Johns Hopkins University Department of Earth and Planetary Sciences, the U.S. National Museum of Natural History, and private collections. Data on sample localities and sources are in Table 1.

Crushed-grain mounts were prepared by grinding small cleavage flakes in an agate mortar and suspending the resultant powder in ethanol or water before deposition onto a holey carbon grid. The grinding process was of concern due to the possibility of deformation-produced features. A comparison of powder mounts and cleavage flakes from identical localities indicated that the features of concern were present in both types of samples. The risk of introducing mottling from grinding therefore appears to be small.

Cleaved samples were prepared by gluing small cleavage flakes to copper washers (hole grids) and splitting them with adhesive tape or a razorblade until the samples were thinner than approximately 30 µm. A few of these samples were then ion milled; other samples were further split until small holes were produced. The regions around these holes were extremely thin and did not display the characteristic amorphization produced by ion milling.

A few specimens were prepared by embedding pieces in epoxy and thin sectioning. This method was used for the preparation of samples to be imaged parallel to the cleavage planes. These specimens were then thinned by argon-ion milling.

TEM and AEM

All samples were examined in the Philips 420ST TEM in the Johns Hopkins University Department of Earth and Planetary Sciences. The instrument was operated at 120 keV and analytical data were collected using an energy dispersive spectroscopy (EDS) system with an Oxford Analytical detector and Princeton Gamma-Tech System IV spectrum analyzer. The data were treated as described by Livi and Veblen (1987).

Electron microprobe analyses

EMP analyses (Table 2) were acquired with the JEOL 8600 Superprobe located in the Johns Hopkins University Department of Earth and Planetary Sciences. Quantitative analyses were obtained with a circular, approximately 5 mm, 15 keV, 20 nA beam. Standards used were albite for Na; enstatite for Mg; anorthite for Al, Si and Ca; orthoclase for K; anatase for Ti; rhodonite for Mn; fayalite for Fe; and fluorphlogopite for F. Data reduction was performed with the CITZAF program (Armstrong 1989).

*Current address: 11860 Wilshire Dr., N. Huntingdon, PA 15642, U.S.A. E-mail: Noed@hwr.com