

A Technique for Observing Exsolution Lamellae in Pyroxenes with the Scanning Electron Microscope

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Abstract

Exsolution in pyroxenes can be studied with the scanning electron microscope by applying an HF acid etch to polished thin sections or oriented grain mounts. The technique requires a minimum of sample preparation time, thereby permitting investigations of submicron exsolution features in pyroxenes on a routine basis.

It is well known that exsolution features occur in pyroxenes with dimensions far below the resolution of the optical microscope. These features have been observed through transmission electron microscopic studies of crushed grains (*cf* Clark, Ross, and Appleman, 1971) and ion-beam-thinned samples (*cf* Champness and Lorimer, 1971). Although use of the transmission electron microscope for this type of study has many advantages, searching for thin edges on crushed grains with the proper orientation is often a painstaking technique, and ion beam thinning is time consuming and somewhat tedious. For this reason, a more expedient means of studying fine scale exsolution in pyroxenes was sought through the use of the scanning electron microscope (SEM).

Because one is, for the most part, observing surface features with the SEM, one can visually distinguish between host and lamellae most readily by obtaining relief between the components of the exsolution set. To achieve this relief, a technique of etching with HF acid was used in a manner similar to that described by Greer (1970) in his study of submicron unmixing in the feldspars. Miller and Philpotts (1973) have used an HF vapor etch to study pyroxene exsolution, but their technique was restricted to optical microscopic observations of tarnishing effects the vapor had on the host and lamellae.

During the course of the investigation a technique was developed whereby submicron-size exsolution lamellae in pyroxenes could be routinely observed with the SEM. The best results were achieved by lapping either thin sections or oriented grain mounts to a high polish prior to etching. The polished samples were then etched for various lengths of time

by immersion in either concentrated HCl or HF acid for times ranging from five to forty-five seconds. Following the etch the samples were thoroughly washed, dried, and prepared for the SEM by vacuum evaporating a gold film of approximately 75 Å on the surface.

The samples were analyzed with an ETEC Auto-scan scanning electron microscope equipped with an ORTEC energy-dispersive X-ray analytical system. For the purpose of this investigation the exsolution features were located by standard techniques and the chemistry of the host and lamellae were subsequently semi-quantitatively analyzed with the energy dispersive system to insure that the features were indeed exsolution lamellae.

To illustrate the success of the technique, electron micrographs of three samples etched by immersion in concentrated HF are presented. Figures 1a and 1b are electron micrographs of exsolution in a hypersthene and an augite host, respectively, which occur in a harzbergite from the Stillwater complex, Montana. Both samples reveal two sets of exsolution with the finer lamellae in Figure 1a having an apparent width of 700 Å. Etch times for the two samples were ten seconds and forty-five seconds, respectively.

Figure 2 is an electron micrograph of an augite host containing three sets of exsolution lamellae. This specimen occurs in a pyroxenite from the Giant Mascot Mine near Hope, British Columbia. The etch time in concentrated HF acid is thirty seconds, and the irregular shaped particles are residue left from the etching process. This residue can be removed by ultrasonic cleaning in alcohol. In all the pyroxenes examined, HF acid etched the Ca-rich phases at a slightly greater rate than the Ca-poorer

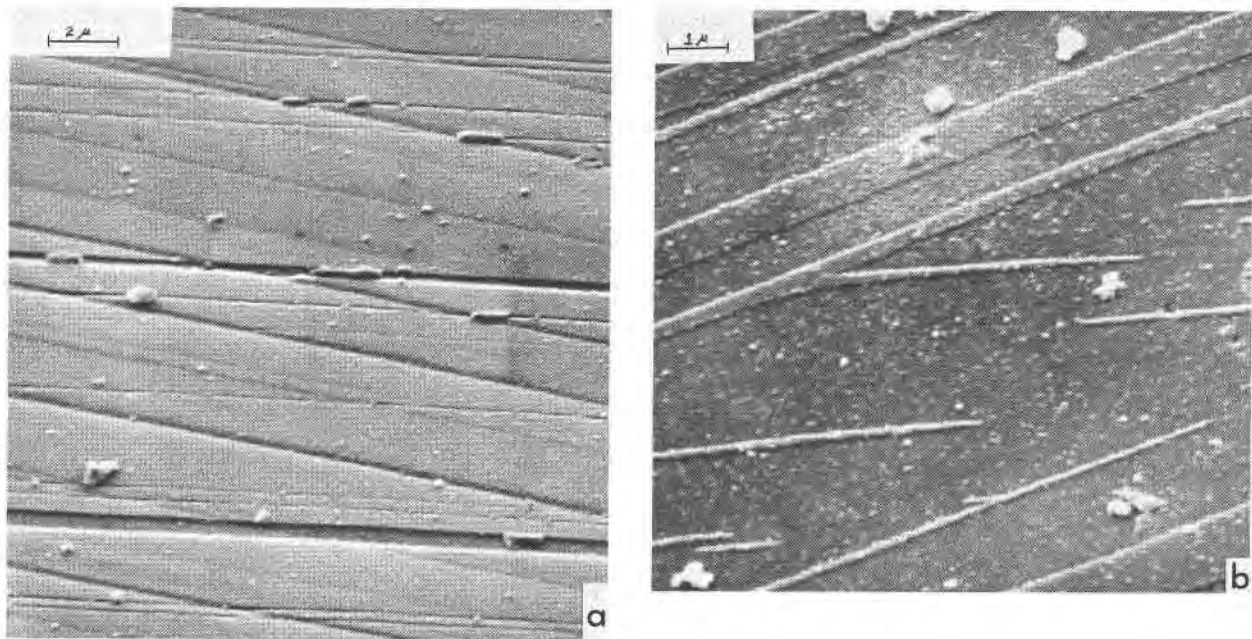


FIG. 1. SEM electron micrographs of exsolution lamellae in pyroxenes from the Stillwater complex, Montana. (a) A hypersthene host containing two sets of Ca-rich lamellae. (b) An augite host containing two sets of Ca-poor lamellae.

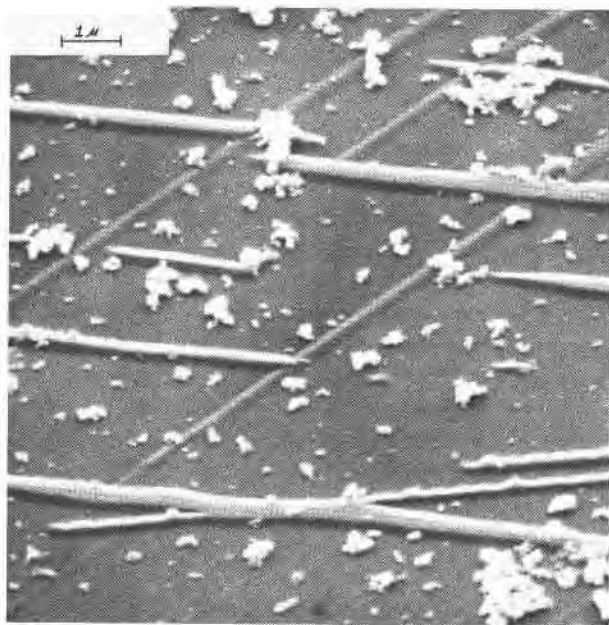


FIG. 2. SEM electron micrograph of an augite host containing three sets of exsolution lamellae. The lamellae oriented northeast-southwest possess a Mg/Ca ratio greater than that of the host and an Fe/Mg ratio approximately equal to that of the host. The two remaining sets of lamellae have compositions similar to each other with higher Mg/Ca and Fe/Mg ratios than the first set.

phases, thereby yielding the desired difference in relief.

The limits as to the size of lamellae which can be observed using this etch technique are not known. However, lamellae approximately 470 Å wide have been observed. It would appear that a similar technique could be applied to the amphibole group, thereby allowing investigations of submicron exsolution features in amphiboles and pyroxenes to be made on a routine basis.

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