A Rietveld and infrared study of synthetic amphiboles along the potassium richterite–tremolite join

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ABSTRACT

Amphiboles were synthesized at 750°C, 1 kbar (H2O) for compositions at 20% intervals along the join potassium richterite–tremolite. Structural variations, site occupancies, and modal analyses of the experimental products (amphibole + minor diopside, quartz, and enstatite) were characterized by Rietveld structure refinement, with final R_Bragg indices in the range 4–6%, and by infrared spectroscopy in the principal OH-stretching region. Amphibole compositions were determined by (1) site-scattering refinement for the A and M4 sites that are occupied by (K, □) (□ = vacancy) and (Na,Ca), respectively; and (2) mass-balance calculations involving the modal analysis and the nominal experimental product composition. These measurements agree within 1% absolute and show close agreement with electron-microprobe compositions for the two samples that we could analyze. Deviations from nominal amphibole composition are up to 19% absolute. The resulting relations between cell dimension and composition are linear. The major change in cell dimensions is a decrease of 0.25 Å in a with increasing tremolite component.

The infrared spectra show two principal peaks at 3735 and 3675 cm⁻¹, corresponding to the local arrangements MgMgMg-OH-K (the Kr band) and MgMgMg-OH-□ (the Tr band), respectively. The relative variation in peak intensity as a function of amphibole composition shows that the molar absorptivities of the two bands are significantly different. The ratio of the molar absorptivities for the two bands is 2.2.

INTRODUCTION

A long-standing problem of amphibole synthesis is the adequate characterization of the product amphiboles. Critical examination of the products of amphibole synthesis (Hawthorne 1983a) showed that compositions frequently depart from their nominal values. Furthermore, spectroscopic examination showed that unexpected ordering patterns could be encountered (e.g., Raudsepp et al. 1987a), raising the question of whether some synthetic amphiboles are good analogues of their natural counterparts. These problems received an increasing amount of attention recently. Raudsepp et al. (1987a, 1987b, 1991), Robert et al. (1993), Della Ventura et al. (1993a, 1993b, 1995, 1997a), and Jenkins and Hawthorne (1995) have examined synthetic amphiboles by Rietveld structure refinement, a very powerful technique for the characterization of composition and ordering in amphiboles that have significant differences in X-ray scattering power of the components involved in the disorder. Della Ventura (1992), Della Ventura and Robert (1990), Della Ventura et al. (1991, 1993a, 1993b, 1995, 1997a), and Robert et al. (1989) showed how infrared spectroscopy in the principal OH-stretching region can give critical information on site occupancies when combined with careful and systematic synthesis of amphiboles. Maresch and Czank (1983a, 1983b) pioneered the use of high-resolution transmission electron microscopy (HRTEM) in the study of synthetic amphiboles, and the use of HRTEM characterization of synthetic amphiboles is now common (Maresch and Czank 1988; Maresch et al. 1994; Ahn et al. 1991; Pawley et al. 1993; Smelik et al. 1994). These methods, together with the more extensive use of scanning electron microscopy (SEM) and electron-microprobe analysis, have led to a resurgence of work on amphibole synthesis and phase relations.

Pawley et al. (1993) synthesized amphiboles along the join richterite–tremolite, ideally NaCaNaMg5Si8O22(OH)2, □Ca,Mg,Si5O18(OH)2, and showed the resulting amphiboles to be essentially defect-free (except for compositions close to tremolite) by HRTEM and close to ideal composition by electron-microprobe analysis and hydro-

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