Charge contrast imaging of fine-scale microstructure and compositional variation in garnet using the environmental scanning electron microscope

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ABSTRACT

Gaseous secondary electron (GSE) imaging of eclogite garnets under the environmental scanning electron microscope (ESEM) at low chamber gas pressures reveals detailed image contrast patterns (charge-contrast images, CCI) that are not present in back-scattered or secondary electron images. Image intensity is a function of the amount of surface charge accumulation. Successful acquisition of CCI depends on frame size and beam scan rate at a given chamber gas pressure and beam current. Images are obtained in a few seconds, and are stable and reproducible. CCI patterns do not correlate with cracks or grain boundaries, but do correspond closely to variations in major-element composition, both in the form of concentric (growth) zoning, and branching, linear features interpreted as cracks that have been healed by new garnet growth. Causes of CCI are not yet well understood, but may be related to variations in lattice defect density and their influence on charge-trapping and dissipation. These in turn influence the rate of charge build-up at or very close to the specimen surface. One interesting possibility is that CCI images detect vacancies related to non-homovalent coupled substitutions involving, for example, REE and hydroxyl, so the method offers a way of imaging the distribution of these trace species in garnets. The CCI images are rich in microstructural detail and offer the potential for rapid, high-resolution, low-noise reconnaissance mapping of intragranular microstructure and compositional variation in both natural and synthetic garnets.

INTRODUCTION

Natural aluminosilicate garnets are well known to exhibit internal compositional and microstructural features that are potentially rich in information about the petrological and deformation history of their host rocks (Hirsch et al. 2003; Hwang et al. 2001; Matsuyk et al. 1998; Matthews et al. 1992; Prior et al. 2000; Spear and Daniel 1998; Terry and Heidelbach 2004; Treppmann and Stockhert 2002). Garnets are also important materials in magneto-ceramics and lasers. Thus, imaging of compositional variation and defect structures in synthetic garnet is important in the development of improved material quality and novel industrial applications (e.g., Zamoryanskaya and Pis’mennyi 2000). However, fine-scale variability of composition, defect density, and lattice orientation is usually invisible under the polarizing optical microscope, and may even be invisible to back-scattered electron (BSE) imaging on ordinary scanning electron microscopes (SEM). High-resolution X-ray compositional mapping on an SEM or electron microscope is time-consuming. It would be useful to have a rapid reconnaissance imaging method for investigation of fine-scale internal variability of garnet. Cathodoluminescence (CL) imaging fulfills this role for some silicates: normal CL detectors generally do not detect luminescence contrasts in common pyralspite garnets, although hot-cathode luminescence microscopy has recently been reported to exhibit internal variation in garnet, such as compositional zoning and healed microcracks (Schertl 2003).

Watt et al. (2000) have reported image contrast on uncoated, polished sections of natural silicates using a gaseous secondary electron detector (GSED) on an environmental scanning electron microscope (ESEM). These charge-contrast images (CCI) result from variable surface charge accumulation and dissipation, and closely resemble CL images for luminescent silicates such as quartz and zircon, suggesting a related causal mechanism. However, Watt et al. (2000) have shown that CCI is also possible for silicates that are not normally luminescent, such as cordierite and micas. They considered that this method has particular value in detecting the effects of otherwise cryptic, fluid-related alteration. Encouraged by these results, we have investigated the CCI characteristics of natural pyralspite garnets under the ESEM, and have found that it is possible to image fine-scale internal structure and compositional variation rapidly and conveniently, making it potentially very useful in reconnaissance studies of garnet microstructures. The purpose of this paper is to report our preliminary findings on the charge-contrast image characteristics of garnets, to make some initial deductions about their microstructural and crystal-chemical causes, and to highlight the potential usefulness of this technique for garnet studies in the mineralogical and materials sciences.

METHODOLOGY

The environmental scanning electron microscope (ESEM), or variable-pressure electron microscope, is capable of imaging specimens of insulating materials without the requirement for