

## Ordering state in orthopyroxene as determined by precession electron diffraction

DAMIEN JACOB,<sup>1,\*</sup> LUKAS PALATINUS,<sup>2</sup> PRISCILLE CUVILLIER,<sup>1</sup> HUGUES LEROUX,<sup>1</sup>  
CHIARA DOMENEGHETTI,<sup>3</sup> AND FERNANDO CÁMARA<sup>4</sup>

<sup>1</sup>Unité Matériaux et Transformations, Université Lille1, CNRS UMR 8207, 59655 Villeneuve d'Ascq, France

<sup>2</sup>Institute of Physics of the Academy of Sciences of the Czech Republic, 182 21 Prague, Czech Republic

<sup>3</sup>Dipartimento di Scienze della Terra e dell'Ambiente, Università di Pavia, 27100-Pavia, Italy

<sup>4</sup>Dipartimento di Scienze della Terra, Università di Torino, 10125-Torino, Italy

### ABSTRACT

Fe<sup>2+</sup> and Mg distribution on octahedral M1 and M2 sites of the orthopyroxene structure is an indicator of the cooling rate and closure temperature of the mineral. It is generally obtained by single-crystal X-ray diffraction, which is limited in spatial resolution. In this work, we determine the cationic distribution at a submicrometer scale in a transmission electron microscope using precession electron diffraction. Two orthopyroxene samples coming from the same metamorphic rock are studied, a naturally ordered one and a disordered one. The latter was obtained from the ordered sample by annealing at high temperature and rapid quenching. Both samples have been first studied in X-ray diffraction and then in precession electron diffraction. Intensities recorded in zone-axis precession electron diffraction experiments have been quantitatively analyzed and compared to simulations, taking into account dynamical interactions between diffracted beams. Our structure refinement results are in good agreement with those obtained by single-crystal X-ray diffraction. They enable to distinguish between the ordered sample and the disordered one in terms of the observed molar fractions of Fe at M1 and M2 sites. We discuss the sensitivity of the method as a function of experimental parameters. The larger dispersion of the results obtained on the ordered specimen is attributed to structural heterogeneities inherent to the sample.

**Keywords:** Ordering, orthopyroxene, precession electron diffraction, site occupancy, structure refinement, transmission electron microscopy