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***In situ* high-*P/T* behaviour of minerals by neutron scattering**S.A.T. REDFERN¹, H. STONE¹ M.T. DOVE¹ AND F. MEDUCIN²¹Department of Earth Sciences, University of Cambridge, Downing Street, Cambridge, CB2 3EQ, UK²Centre de Recherche sur la Matière Divisée, UMR 6619 - CNRS - Université d'Orléans, 1 bis, rue de la Férollerie, 45071 ORLEANS Cedex 2, France (satr@cam.ac.uk)

Apparatus for neutron diffraction at simultaneous high temperatures and pressures is reported. The basic design is based on the Paris-Edinburgh cell using opposed anvils, with internal heating. Temperature is measured using neutron radiography. The apparatus has been shown in both on-line and off-line tests to operate to a pressure of 7 GPa and temperature of 1700 °C. The apparatus has been used in a neutron diffraction study of the crystal structure of deuterated brucite, and results for 520 °C and 5.15 GPa have been obtained. The diffraction data that can be obtained from the apparatus are of comparable quality to previous high-pressure studies at ambient temperatures, and are clearly good enough for Rietveld refinement analysis to give structural data of reasonable quality.

Examples of the application of this apparatus will be discussed, and include the first structural data from BaTiO₃ perovskite at high P (3.2 GPa), demonstrating the usefulness of the apparatus for the study of displacive phase transitions, and measurements of the effect of pressure on the order-disorder behaviour of Mg and Al in spinel at high P and T. Neutron diffraction patterns of stoichiometric MgAl₂O₄ were collected on heating from room temperature to ~1600 K at pressures of ~2.6 GPa, and the cation distribution was determined directly from site occupancies obtained by Rietveld refinement. The high-pressure temperature-dependent behaviour demonstrates that disordering occurs to a much greater extent in MgAl₂O₄ at high pressure compared with the equivalent ambient-pressure behaviour, and that pressure favours disordering towards the inverse structure.

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Investigation of exsolution by energy filtered TEM

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Introduction

The new generation of energy filtered TEM's opens an avenue of applications. They offer the possibility to investigate the crystal structure in the range of Å and the chemical composition in the range of nm and by investigating the Electron Loss Near Edge Structure (ELNES) additionally the atomic environment and thus the valence state. Exsolution phenomena play an important role in the behaviour of many mineral systems and the relationship between nanostructure, chemical composition and magnetic properties is of great interest to understand the macroscopic properties.

Investigated mineral systems

In natural rocks remanant magnetisation occurs in various mineral systems, the most important of which are titanomagnetite (Fe₃O₄-Fe₂TiO₄) and titanohematite (Fe₂O₃-FeTiO₃) solid solutions. The composition modulation and size of fine precipitates resulting from exsolution generated during cooling control the magnetic properties. Exsolution in the titanomagnetite solid solution is not possible to study experimentally due to the low solvus temperature (<500°C) and the slow kinetics. An ideal analogue system is the magnetite (Fe₃O₄)-hercynite (FeAl₂O₄) system, which has a miscibility gap up to higher temperatures, forming a complete solid solution above 700 °C [1].

Results

The magnetite –hercynite solid solution has a spinel structure with a face centered cubic unit cell. Spinodal decomposition would be expected in this system in the early stages of unmixing and predicted to occur in the elastically soft <100> directions, leading to three sets of mutually perpendicular modulations. In spinodal decomposition this modulation is also correlated to a sinusoidal spatial variation of the chemical composition. By using element mapping we show for the first time the modulation of chemical composition, including the chemical gradients, and hence verify the theoretical expectation. The wavelength of the sinusoidal modulation and the amplitude offers the possibility of obtaining information about the kinetics of the unmixing mechanism.

The titanohematite exsolves by nucleation and growth to hematite and ilmenite rich lamellae and precipitates, where hematite contains Fe in a trivalent oxidation state and ilmenite Fe in a divalent oxidation state. This enables to distinguish between both phases by valence state mapping.