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HOT STAGE HVEM DYNAMIC STUDIES OF ORDER–DISORDER TRANSITIONS IN LITHIUM FERRITE

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Lithium ferrite (LiFe$_2$O$_4$) is a ferrimagnetic compound with some interesting technological properties(1). In the disordered state the compound has the spinel structure with a random mixture of $\text{Li}^+$ and $\text{Fe}^{3+}$ on the octahedral sites (spacegroup Fd3m). Below 750°C the lithium ions and iron ions on the octahedral sites order and the spacegroup symmetry is lowered to $P4_332$ ($P4_332$). The resulting domain structure has been described in detail by Van der Biest and Thomas(2).

The kinetics of the ordering reaction have been studied "in situ" in the hot stage of a HVEM. Annealing in the normal atmosphere of the microscope leads to a reduction of the compound in a way similar to that found for CoFe$_2$O$_4$(3). In order to control the stoichiometry of the compound during the ordering reaction, it is necessary to maintain an oxygen atmosphere around the specimen, hence the use of an "environmental" cell is necessary. In this study the environmental cell designed by Swann(4) for the Imperial College HVEM was used. An oxygen pressure of 40 Torr was found to be high enough to prevent rapid reduction of the compound and low enough to have sufficient penetration and contrast in the images. Radiation damage definitely played a role during the experiments. Prolonged focussing of the beam (>10 min) on one spot caused voids to nucleate and grow until holes were formed. It was also observed that the amount of damage was proportional to the time the specimen had spent above the ordering temperature. This suggests that the rate of damage is an order of magnitude larger in the disordered state for approximately the same temperature. The specimen was heated up above Tc to about 770°C and then cooled as fast as the stage permits to temperatures slightly below the ordering temperature (735°C - 745°C). The changes in microstructure were followed in dark field with a superlattice spot. Images were recorded with a low light level TV system on videotape with a speed of 25 frames/sec. Fig. 1 shows a disordering sequence. It can be seen that disordering starts at the anti-phase boundaries, which are replaced by a layer of disordered material. The domains subsequently shrink and disappear, if heating is slow or the contrast may fade uniformly before the domains can disappear by shrinking. Fig. 2 shows an ordering sequence. Individual domains can be seen nucleating and growing, until they impinge and form antiphase domain boundaries.

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Fig. 1. Disordering sequence.
Fig. 2. Ordering sequence.
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