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HIGH VOLTAGE ELECTRON MICROSCOPY OF DEFECTS IN LITHIUM FERRITE SPINEL

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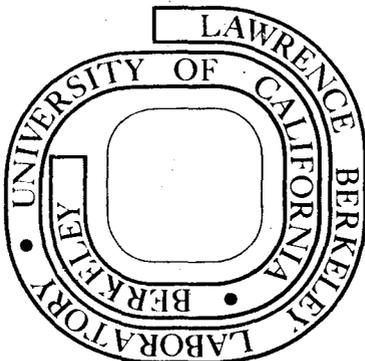
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## HIGH VOLTAGE ELECTRON MICROSCOPY OF DEFECTS IN LITHIUM FERRITE SPINEL

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Ferrimagnetic oxide spinels are an important class of modern technological ceramic materials. These have long been recognised as being predominantly ionic in nature. Hornstra's<sup>(1)</sup> crystallographic study of planar and line defects in spinel structure shows that dislocations of Burgers vector  $1/2\langle 110 \rangle$  can dissociate into two partials  $1/2\langle 110 \rangle = 1/4\langle 110 \rangle + 1/4\langle 110 \rangle$  bounding a cation stacking fault on  $\{111\}$  glide planes. These partials, in turn, can dissociate into quarter partials of Burgers vector  $1/12\langle 112 \rangle$  on  $\{111\}$  planes, reducing strain energy and at the same time preserving electro-neutrality through a synchroshear mechanism. The current paper describes experimental studies on the nature of the glide dislocations and their dissociation in lithium ferrite (inverse spinel) using high voltage transmission electron microscopy. High order bright field<sup>(2)</sup> electron micrographs taken in the HVEM together with the computed image profiles implementing multibeam dynamical theory are used to determine the fault plane and estimate the stacking fault energy.

Glissile dislocations are introduced by plastically deforming single crystal specimens at high temperature (1200°C). Thin foils for examination in the microscope are prepared from these specimens by mechanical thinning followed by ion bombardment. The foils are examined in Hitachi HU-650 electron microscope operating at 650kV. Figures 1a and 1b show dislocation networks in a particularly thick ( $>5\xi_g$ ) region of the specimen under different diffracting conditions. The Burgers vectors of the total and partial dislocations are determined from diffraction contrast experiments. The results show that almost all dislocations are dissociated according to the reaction  $1/2\langle 110 \rangle = 1/4\langle 110 \rangle + 1/4\langle 110 \rangle$ . Determination of the fault plane is less direct due to absence of stacking fault fringes ( $\xi_g \approx 10^3\text{\AA}$ ) and is done from geometrical considerations. From the micrographs, it is seen that the images are parallel to the intersection of the  $\{011\}$  planes with the specific foil plane (110). This result has been confirmed by examination of dislocations in a large number of foils in different orientations. No dislocations on  $\{111\}$  planes have been found in  $\text{LiFe}_5\text{O}_8$  in the entire course of this investigation contrary to Hornstra's analysis and some reported observations in  $\text{MgAl}_2\text{O}_4$ .<sup>(3)</sup>

Figure 2a shows dissociated dislocations imaged with  $s_{4g} = 0$ . Figures 2b and 2c are the microdensitometer trace at A and the corresponding computed image profile respectively. Dislocation A is a mixed dislocation of total Burgers vector  $1/2[011]$ . The dissociation can be represented as  $1/2[011] = 1/4[011] + 1/4[011]$  on the (011) plane connected by a cation stacking fault. The average value of the nonconstricted part of the extended dislocations is measured to be  $(150 \pm 20\text{\AA})$  from microdensitometer traces. This corresponds to a stacking fault about  $220\text{\AA}$  wide in the cation sublattice on the (011) plane. The stacking fault energy can then be calculated from the theory of dislocations. In the anisotropic case, the value determined is  $75 \text{ ergs/cm}^2$ . It may be remarked that although the value of  $\gamma$  reported here seems relatively low for an ionic compound, this agrees well with the calculated value of  $\gamma$  for  $\text{LiFe}_5\text{O}_8$ .<sup>(4)</sup>

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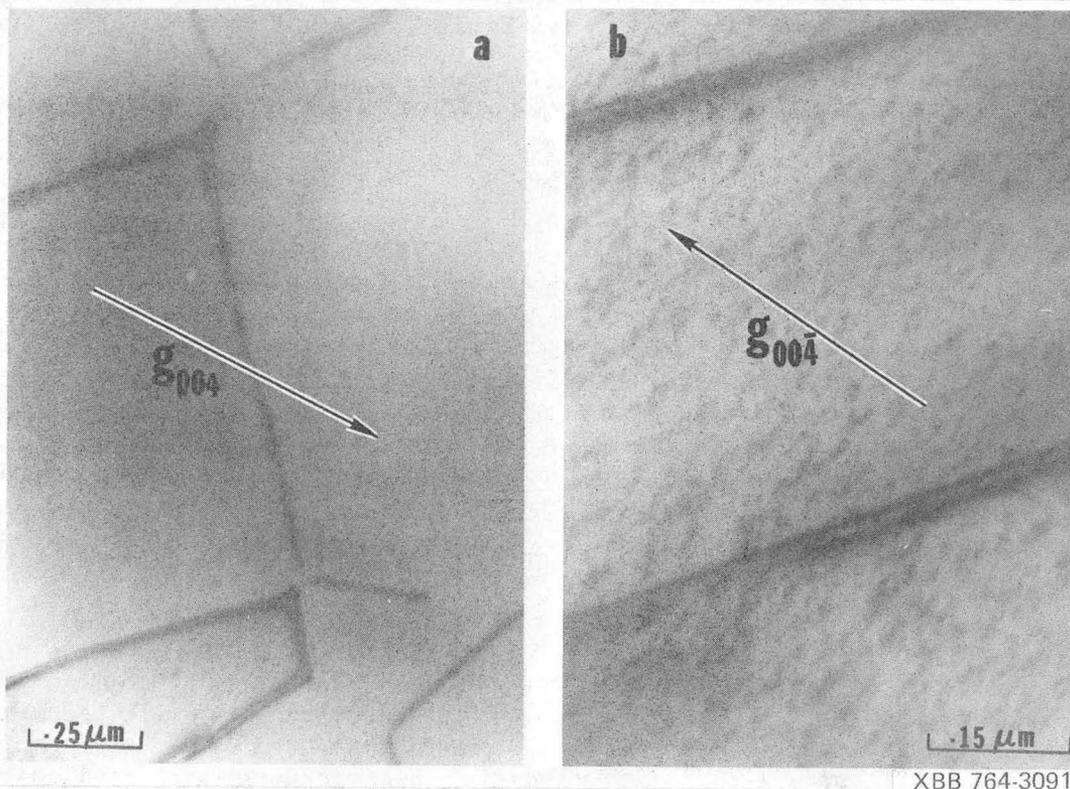


Fig. 1 Dislocation networks in plastically deformed  $\text{LiFe}_5\text{O}_8$ . Foil orientation near (110). The operating reflection is indicated (actually imaged in ng).

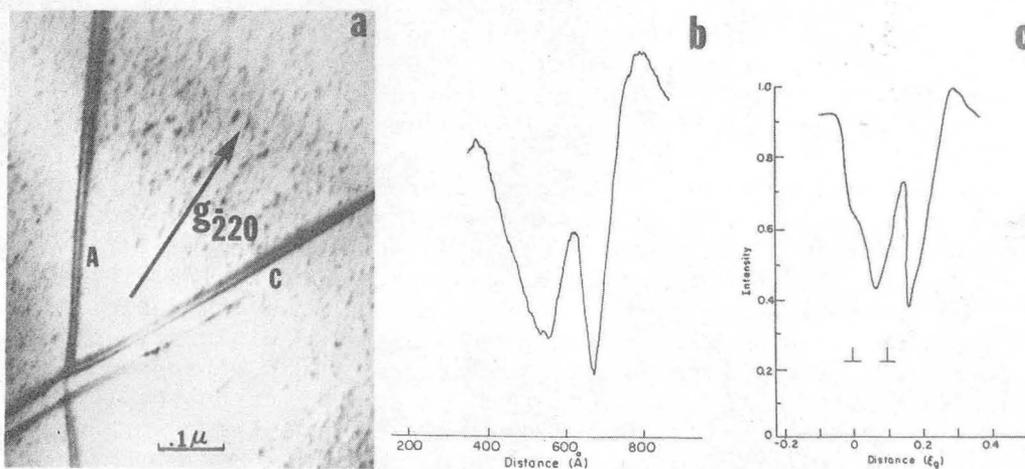


Fig. 2 a) Dissociated dislocations in  $\text{LiFe}_5\text{O}_8$  imaged near the (110) pole. b) Microdensitometer trace at A. c) Computer profile for  $1/2[011] = 1/4[011] + 1/4[011]$  (8 beam systematic  $\langle 220 \rangle$ ). Excellent agreement exists between theory and experiment.

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