Title

ELECTRON MICROSCOPY OF EARLY STAGES OF PRECIPITATION IN LITHIUM FERRITE (LiFe5O8) SPINEL

Author

Mishra, R.K.

Publication Date

1975-03-01

Published in the 33rd Annual Proceedings
Electron Microscopy Society America,
Las Vegas, NV, 1975. G. W. Bailey (ed).,
pp. 62 - 63

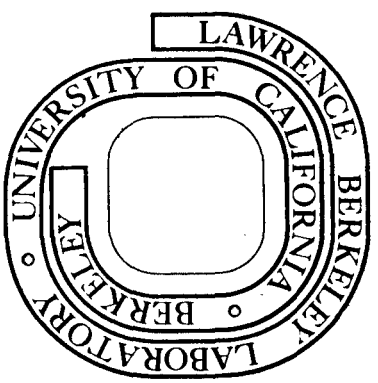
LBL-3770

ELECTRON MICROSCOPY OF EARLY STAGES OF PRECIPITATION IN LITHIUM FERRITE ($LiFe_5O_8$) SPINEL

R. K. Mishra

March 1975

Prepared for the U. S. Energy Research and
Development Administration under Contract W-7405-ENG-48



LBL-3770

DISCLAIMER

Reprinted from
"33rd Ann. Proc. Electron Microscopy Soc. Amer."
Las Vegas, Nevada, 1975. G. W. Bailey (ed.).

ELECTRON MICROSCOPY OF EARLY STAGES OF PRECIPITATION IN LITHIUM FERRITE ($LiFe_5O_8$) SPINEL

R. K. Mishra

Department of Materials Science and Engineering, College of Engineering, University of California, Berkeley, California 94720

The advent of high voltage microscopes with penetrating power higher than that of conventional microscopes and development of ion-milling technique for preparing ceramic specimens have opened up the field of microstructural characterisation of ceramic materials. The present paper describes the electron microscopy of the early stages of precipitation in ferrimagnetic lithium ferrite ($LiFe_5O_8$) spinel. These materials are of interest for applications in electronics industry. Thin slices (30μm in thickness) from a slightly lithium deficient lithium ferrite single crystal are aged at 1200°C in air for a sequence of times. Specimens for examination in HU 650 HVEM operating at 650kV are prepared from the center of these slices by mechanical polishing followed by ion-thinning.

Fig. 1(a) shows the microstructure obtained after the shortest aging time (~10 minutes at 1200°C) and Fig. 1(b) shows its symmetrical SAD pattern in (110) orientation. The redial diffuse intensity distribution around the diffraction spots in the plane of the pattern is apparent. The area imaged in Fig. 1(a) comes from a region approximately one extinction distance in thickness (~1000Å) and particles with an average diameter of 200Å are seen to be dispersed in the bulk of the specimen. Detailed contrast analysis shows that these particles exhibit strain contrast characteristic of isotropic symmetry (1). It is very likely that the radial diffuse intensity distribution around the diffraction spots in Fig. 1(b) is a result of the isotropic strains. The bright field micrograph in Fig. 2(a) is obtained from a specimen aged for about 25 minutes in air at 1200°C. This picture is taken from a particularly thick region of the foil (about 5 extinction distances thick) where absorption can be utilised to detect any strains around the particles. As can be seen in the figure, there is no noticeable strain contrast around the large particles (~2000Å in size). These particles are octahedra with faces parallel to {111} planes. Fig. 2(b) shows the (110) projection of an octahedron. The measured values of the angles and ratio of the diagonals in the micrograph correspond to that in Fig. 2(b). Thickness fringes at the inclined faces of the octahedron are visible in Fig. 2(a). Interestingly enough, it has been noticed that the particles are in best contrast in bright field when imaged in a systematic diffracting condition with $s = 0$ rather than $s \neq 0$. Electron metallography of subsequent stages in the precipitation reaction has shown that the particles in Fig. 2(a) are lithium ferrate ($LiFeO_2$ - a paramagnetic phase) although the available phase diagram (2) does not suggest any such phase transformation at the temperature and composition examined.

De Jonghe and Thomas (3) have reported the formation of cuboid shaped precipitates in the early stages of precipitation in cobalt ferrite spinel. Also Bansal and Heuer (4) have suggested that the pre-precipitates in non-stoichiometric magnesium aluminate spinel are nearly spherical in shape. These results along with the current findings indicate that formation of nearly spherical nuclei (small cuboids or octahedra) is favoured in a spinel structure which is to be expected if the strain energy is small. The possibility of obtaining higher coercive force in ferrimagnetic ceramic magnets by a dispersion of nonmagnetic particles as in the present system is currently being investigated.

The author gratefully acknowledges the continued guidance and encouragement of Prof. G. Thomas. This research is sponsored by the U.S. Energy Research and Development Administration through the Lawrence Berkeley Laboratory.

Reprinted from:
"33rd Ann. Proc. Electron Microscopy Soc. Amer."
Las Vegas, Nevada, 1975. G. W. Bailey (ed.).

References

1. L.M. Brown, Electron Microscopy in Materials Science, ed. U. Valdre, Academic Press (1970), 361.
2. D.W. Strickler and R. Roy, J. Am. Cer. Soc., 44,(1961), 225.
3. L.C. DeJonghe and G. Thomas, Mat. Sci. Eng., 8, (1971), 259.
4. G.K. Bansal and A.H. Heuer, Phil. Mag., 29,(1974), 709.

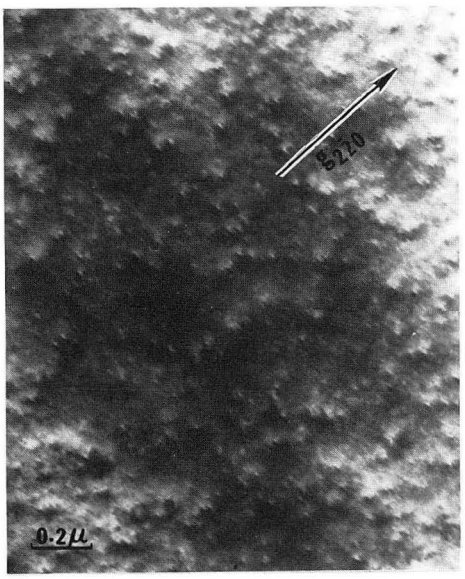


Fig. 1(a) bright field image showing strain contrast from small particles in lithium ferrite spinel (10 min. at 1200°C)

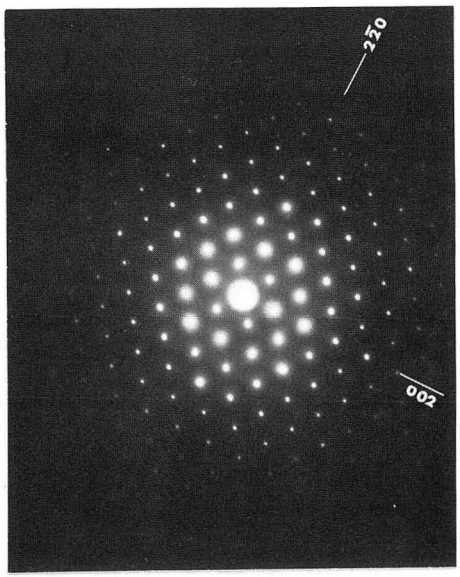


Fig. 1(b) SAD from the same area as (a) (110) foil orientation radial haloes exist around the diffraction spots.

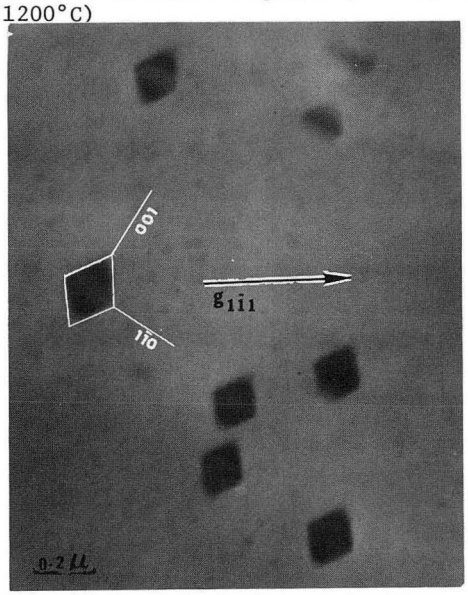


Fig. 2(a) octahedral shaped $LiFeO_2$ precipitates in athick region of the foil imaged near (110) pole.

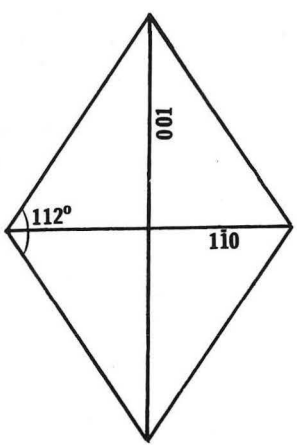


Fig. 2(b) sketch of (110) projection of a regular octahedron with {111} faces. Ratio of the diagonals in the figure is $\sqrt{2}$.

TECHNICAL INFORMATION DIVISION
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720