The effect of heat treatment on the lattice parameter of nickel ferrite. By K. N. SUBRAMANYAM and L. R. KHARE, Physics Department, Al Fateh University, Box 656, Tripoli, Libya

(Received 24 April 1978; accepted 28 September 1978)

Abstract

Room-temperature X-ray precision measurements of the lattice parameters of spectroscopically pure polycrystalline nickel ferrite (NiFe₂O₄) samples, sintered in air in the range 1373–1773 K, show that the lattice parameter increases with increasing sintering temperatures; a result which is in conformity with the expected Fe³⁺ \rightarrow Fe²⁺ transformation.

Introduction

The general strategy in X-ray precision measurements of the lattice parameters of polycrystalline substances (Parrish & Wilson, 1959; Lipson & Steeple, 1968; Subramanyam, 1971*a,b*; Subramanyam & Khare, 1978; and others) has been carefully followed for the purpose of assessing the sensitivity of the lattice parameter of polycrystalline NiFe₂O₄ to the final temperatures at which the specimens have been sintered. Starting with spectroscopically pure oxides of Ni and Fe (supplied by Johnson Matthey Chemicals Ltd, UK), the final samples of NiFe₂O₄ were obtained by sintering the compressed mixture of oxides in air at closely controlled

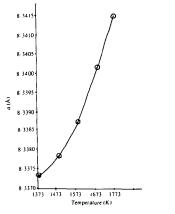


Fig. 1. Sintering temperature (K) vs lattice parameter a (Å) for NiFe₂O₄ samples sintered in air. 0567-7408/79/010269-01\$01.00

Acta Cryst. (1979). B35, 269

Table 1. Sintering temperature versus the lattice parameter for NiFe₂O₄ sintered in air

The reflections used throughout were 555 $(a_1 \text{ and } a_2)$, 662 (a_2) , 840 $(a_1 \text{ and } a_2)$ and 911 (a_1) .

Sintering temperature	Lattice parameter (a)
1373 K	$8.3373 \pm 2 \times 10^{-4} \text{ \AA}$
1473	8·3378 ± 3 × 10 ⁻⁴
1573	$8.3387 \pm 3 \times 10^{-4}$
1673	$8.3401 \pm 3 \times 10^{-4}$
1773	$8.3414 \pm 3 \times 10^{-4}$

temperatures (1373, 1473, 1573, 1673 and 1773 K) (see Fig. 1 and Table 1); subsequently, their X-ray photographs were taken with Co radiation ($Ka_1 = 1.7889, Ka_2 = 1.7928$ Å).

Discussion

The loss of oxygen (δ) that takes place during the sintering of NiFe₂O₄, which is given by Ni²⁺₁Fe³⁺₂O²⁻ \rightarrow Ni²⁺₁Fe³⁺₂Fe³⁺₂ $_{\delta}$ Fe³⁺_{2-\delta}, O²⁻_{4-\delta} + O²_{\delta}, shows that as the rate of evaporation of oxygen from the structure increases with increasing temperature, so does the ferrous-ion concentration. Indeed, the results presented here are in conformity with the fact that Fe²⁺ ions have a much larger radius than Fe³⁺ ions (Standley, 1962).

References

- LIPSON, H. & STEEPLE, H. (1968). Interpretation of X-ray Powder Diffraction Patterns, pp. 157–186. London: Macmillan.
- PARRISH, W. & WILSON, A. J. C. (1959). International Tables for X-ray Crystallography, Vol. II, pp. 216–234. Birmingham: Kynoch Press.
- STANDLEY, K. J. (1962). Oxide Magnetic Materials, p. 25. Oxford: Clarendon Press.
- SUBRAMANYAM, K. N. (1971a). Phys. Lett. A, 35(2), 125-126.

SUBRAMANYAM, K. N. (1971b). J. Phys. C, 4, 2266-2268.

SUBRAMANYAM, K. N. & KHARE, L. R. (1978). Acta Cryst. B34, 347–348.

© 1979 International Union of Crystallography

The crystal structure of tetracycline hexahydrate: erratum. By L. R. NASSIMBENI, Department of Physical Chemistry, University of Cape Town, South Africa

(Received 9 October 1978; accepted 16 October 1978)

Abstract

The paper by Caira, Nassimbeni & Russell [Acta Cryst. (1977), B33, 1171–1176] contains a typographical error. In Table 3 the z coordinate of C(12) should be 0.6088 instead of 0.4088.

All the relevant information is contained in the Abstract.