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## Structural And Infrared Properties Of Mn-si Spinel Ferrite

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### Abstract:

*Structural and infrared properties of spinel ferrite system have been studied by means of X-ray diffraction and IR absorption spectra. Samples of system were prepared by ceramic technique. X-ray diffraction confirms single-phase formation of the samples. The lattice parameter is found to decrease linearly, which is attributed to ionic size difference of cations involved.*

*The IR spectra showed two main absorption bands  $\nu_1$  and  $\nu_2$  in the range of 400-600  $\text{cm}^{-1}$  arising from tetrahedral (A-site) and octahedral (B-site) interstitial sites in the spinel lattice. The band lengths  $R_A$  and  $R_B$  were found to increase with composition (x).*

### KEY WORDS:

MnSi ferrite, structural, Infrared Spectroscopy.

### INDRODUCTION

Studies on the solid state properties of the oxides spinels have been the subject of investigation since many years.(1-3) In particular the solid solutions have attracted a lot of attention in recent years. The interest in the spinel systems and their solid solution systems arises mainly from the fact that they exhibit interesting variations in cation distribution, magnetic and electric properties. These properties depend upon chemical composition, method of preparation and sintering temperature.

The infrared spectroscopic technique depends on the fact that a chemical substance shows marked selective absorption in the infrared region. Various bands present in IR spectrum correspond to the characteristic function groups and bonds present in the chemical substance but very less information exists in literature on infrared spectral study of ferrite system.

In this work, we report a detailed study of the structural analysis and infrared spectra of  $\text{Mn}_{1-x}\text{Si}_x\text{Fe}_{2-2x}\text{O}_4$  spinel system with  $x = 0.0, 0.1, 0.2, 0.3, 0.4,$  and  $0.5$

### EXPERIMENTAL

Six samples of  $\text{Si}^{4+}$  substituted  $\text{Mn}_{1-x}\text{Si}_x\text{Fe}_{2-2x}\text{O}_4$  system were prepared by usual double sintering ceramic technique for  $x = 0.0, 0.1, 0.2, 0.3, 0.4,$  and  $0.5$ . The starting materials were MnO,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ; all 99% pure and supplied by E. Merck. These oxides were mixed in stoichiometric proportions and presintered at  $950^\circ\text{C}$  for 12 hours. In final sintering process, the samples were held at  $1200^\circ\text{C}$  for 12 hours and then the furnace was allowed to cool to room temperature at the rate of  $2^\circ\text{C}/\text{min}$ . The X-ray

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### Structural And Infrared Properties Of Mn-si Spinel Ferrite

diffraction patterns were recorded using  $\text{CuK}_\alpha$  radiation on Philips diffractometer operated at 25 KV and 20 mA. X-ray Wavelength used for recording X-ray diffractograms was 1.5418 Å. IR spectra of the same samples were recorded on Perkin-Elmer IR spectrometer (Model No. 783) in the wavelength range (200-800  $\text{cm}^{-1}$ ) in the KBr medium.

### RESULTS AND DISCUSSION:-

X-ray diffraction patterns for the system  $\text{Mn}_{1-x}\text{Si}_x\text{Fe}_{2-2x}\text{O}_4$  with  $x = 0.0, 0.1, 0.2, 0.3, 0.4,$  and  $0.5$  at 300K are shown in Figure 1. The diffraction lines were found to be sharp corresponding to single-phase cubic spinel structure. The d-spacings for the recorded peaks are calculated according to Bragg's law. The lattice parameters were calculated using the following relation

$$\sin^2 \theta_{hkl} = \frac{\lambda^2}{4a^2} (h^2 + k^2 + l^2)$$

Where h, k, and l are the indices of spinel planes, 'a' is the lattice parameter and  $\lambda$  is the wavelength used. These values are tabulated in the Table 1. It is clear that lattice parameter decreases with composition (x), thus obeying the Vegard's law (4). Usually in a solid solution of spinels within the miscibility range, a linear change in lattice constant with the concentration of the composition is observed (5). The decrease in the lattice parameter is attributed to the replacement of large  $\text{Mn}^{2+}$  ions (0.80 Å) by smaller  $\text{Fe}^{3+}$  (0.64 Å) and  $\text{Si}^{4+}$  (0.41 Å) ions in tetrahedral site.

X-ray density for each composition was calculated using the relation

$$d_x = Zm / NV$$

Z - Number of molecules per unit cell (Z = 8)

M - Molecular weight

N - Avogadro's number ( $6.022 \times 10^{23}$ )

V - Volume of unit cell.

The variation of X-ray density with concentration is shown in Table 1.

In order to determine cation distribution, XRD intensity was calculated using the formula given by Burger (6)

$$I_{hkl} = |F_{hkl}|^2 \cdot p \cdot L_p$$

$I_{hkl}$  - Relative integral intensity.

$F_{hkl}$  - structure factor.

P - Multiplicity factor.

$L_p$  - Lorentz polarization factor.

$$L_p = 1 + \cos 2\theta / \sin 2\theta \cdot \cos \theta$$

According to Ohinishi and Teranshi (7) intensity ratios of the planes  $I_{220}/I_{400}$  and  $I_{422}/I_{400}$  are structural sensitive. The distributions of divalent, trivalent and tetravalent cations among tetrahedral and octahedral sites in  $\text{Mn}_{1-x}\text{Si}_x\text{Fe}_{2-2x}\text{O}_4$  ferrite samples are determined from these distribution intensity ratios and are given in Table 1.

Ferrites possess the structure of mineral spinel that crystalline in the cubic form with space group  $Fd3m-O_h$  (8). It is generally known that the spinel ferrites exhibit four IR active bands, designated  $\nu_1, \nu_2, \nu_3$  and  $\nu_4$ . The occurrence of these four bands has been rationalized on the basis of group theory calculations employing space group and point symmetries both in normal and inverse spinels. The first three bands are due to tetrahedral and octahedral complexes while the fourth one is due to some type of lattice vibrations.

IR spectra of present system are shown in the Figure 2. It can be seen from the figure that the IR spectra are found to exhibit two bands in the range 200-800  $\text{cm}^{-1}$ . The higher frequency band  $\nu_1$  is in the range 600-610  $\text{cm}^{-1}$  and low frequency band  $\nu_2$  is in the range 300-420  $\text{cm}^{-1}$ . These bands are common features of all the ferrites (9-10). Therefore the absorption band  $\nu_1$  is caused by tetrahedral site ions and absorption band  $\nu_2$  is caused by octahedral site ions. This difference in the band positions is observed because of difference in the  $\text{Fe}^{3+} - \text{O}^{2-}$  distance for tetrahedral and octahedral complexes. The center

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frequency of the bands  $\nu_1$  and  $\nu_2$  shows slight variation. The slight variation in  $\nu_1$  and  $\nu_2$  indicates that method of preparation, the grain size and sintering temperature can influence the band position. The band width  $\nu_1$  and  $\nu_2$  shift slightly towards higher frequency side indicating that there is decrease in site radius, (Table 2).

The force constant is a second derivative of potential energy with respect to the site radius, the other independent parameters being kept constant. The force constant for tetrahedral and octahedral sites  $k_t$  and  $k_o$  respectively are calculated from IR absorption data using the method suggested by Waldron (9). Bond lengths have been computed using formula suggested by Smith (11). The variations of  $k_t$ ,  $k_o$ ,  $R_A$  and  $R_B$  are listed in Table (2). The force constants are found to increase with site radii as shown in Table (2). This suggests strengthening of interatomic bonding. Normally increase in site radius leads to decrease in force constants. This can be attributed to the fact that under favorable conditions, oxygen can form stronger bonds with metal ions even at large inter nuclear separations (12-13).

### CONCLUSION

X-ray diffraction patterns for the system  $Mn_{1-x}Si_xFe_{2-2x}O_4$  with  $x = 0.0, 0.1, 0.2, 0.3, 0.4$ , and  $0.5$ . at 300K are found to be sharp corresponding to single-phase cubic spinel structure. The lattice parameter decreases with increase in  $Si^{4+}$  concentration. The infrared spectra of the system consist of two bands, which correspond to the intrinsic vibrations of tetrahedral and octahedral complexes. The difference in positions of the infrared band  $\nu_1$  and  $\nu_2$  is due to the difference in the angle between  $Fe^{3+}-O^{2-}$  for the octahedral and tetrahedral sites. The absences of bands  $\nu_3$  and  $\nu_4$  suggest the substitution of  $Si^{4+}$  does not cause significant vibrations that can be observed in the IR spectra.

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