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Effect of sintering temperature on the properties of Cu–Co ferrites prepared by oxalate precipitation method

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Abstract

Polycrystalline Cu–Co ferrite powder was synthesized following oxalate precipitation method. The samples of the compound $Cu_{0.5}Co_{0.5}Fe_2O_4$ were heated at different temperatures in the range of 773–1173 K and were characterized by X-ray diffraction and SEM techniques. The results of XRD show the formation of single-phase cubic spinel structure. The lattice parameter showed a minimum value for the sample heated at 1073 K. It has been observed that grain size increases with the increase in temperature and is maximum (3.2 μm) for the powder sintered at 1173 K.

Keywords: Ferrites; Chemical synthesis; X-ray diffraction; Scanning electron micrograph

1. Introduction

The chemistry of fine particle ferrite material is of interest in research due to its technological applications. The high-density ferrites having large grain size can be prepared at low temperature [1]. The microstructure and the surface properties of such fine powders have large implications in controlling the parameters required for any particular application which in turn depends on the method adopted for synthesis [2]. The conventional ceramic method for the preparation of ferrites has certain limitations such as long heating schedule and high temperature [3]. The necessity of such conditions results in the loss of certain elements leading to the formation of chemically inhomogeneous materials [4]. The oxalate precipitate method overcomes these limitations considerably and has become popular over the years. Other methods which include the co-precipitation method [5], sol–gel method [6], solvent evaporation method [7], hydrothermal method [8], citrate method [9] and combustion method [10] have also been developed by which ferrite particles ranging from 10 to 100 nm can be synthesized.

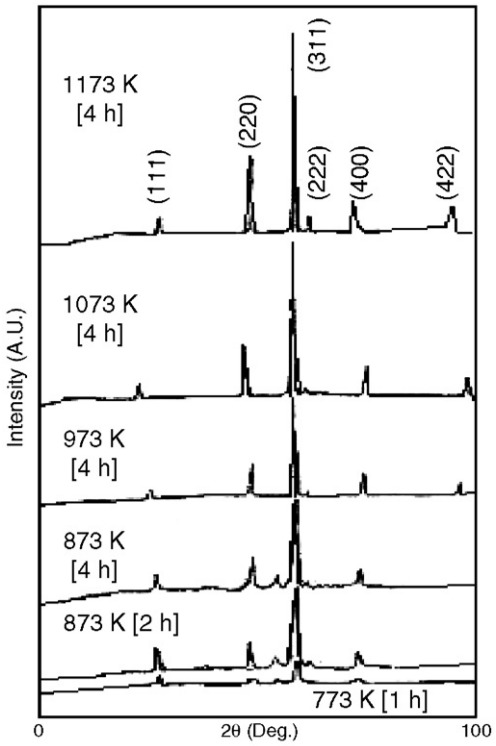

Fig. 1. XRD pattern of $Cu_{0.5}Co_{0.5}Fe_2O_4$ at different temperatures.

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Table 1
Physical parameters of $Cu_{0.5}Co_{0.5}Fe_2O_4$ at different temperatures

Temperature (K)	Sintering time (h)	Lattice parameter (Å) ±0.05	Radius of A-site ion (Å)	Radius of B-site ion (Å)	Average grain size (μm)
773	1	8.38	0.55	0.69	–
773	2	8.38	0.55	0.69	1.12
873	4	8.38	0.55	0.69	1.2
973	4	8.38	0.55	0.69	1.72
1073	4	8.37	0.54	0.69	2.3
1173	4	8.38	0.55	0.69	3.2

In the present investigation an attempt has been made to establish a new chemical route which is both affordable and versatile for the synthesis of fine mixed oxide powders. We report here the synthesis of ferrites by the oxalate method and their characterization using XRD and SEM techniques.

2. Experimental details

The high purity AR grade copper sulphate, zinc sulphate and ferrous sulphate were weighed carefully in a microbalance to

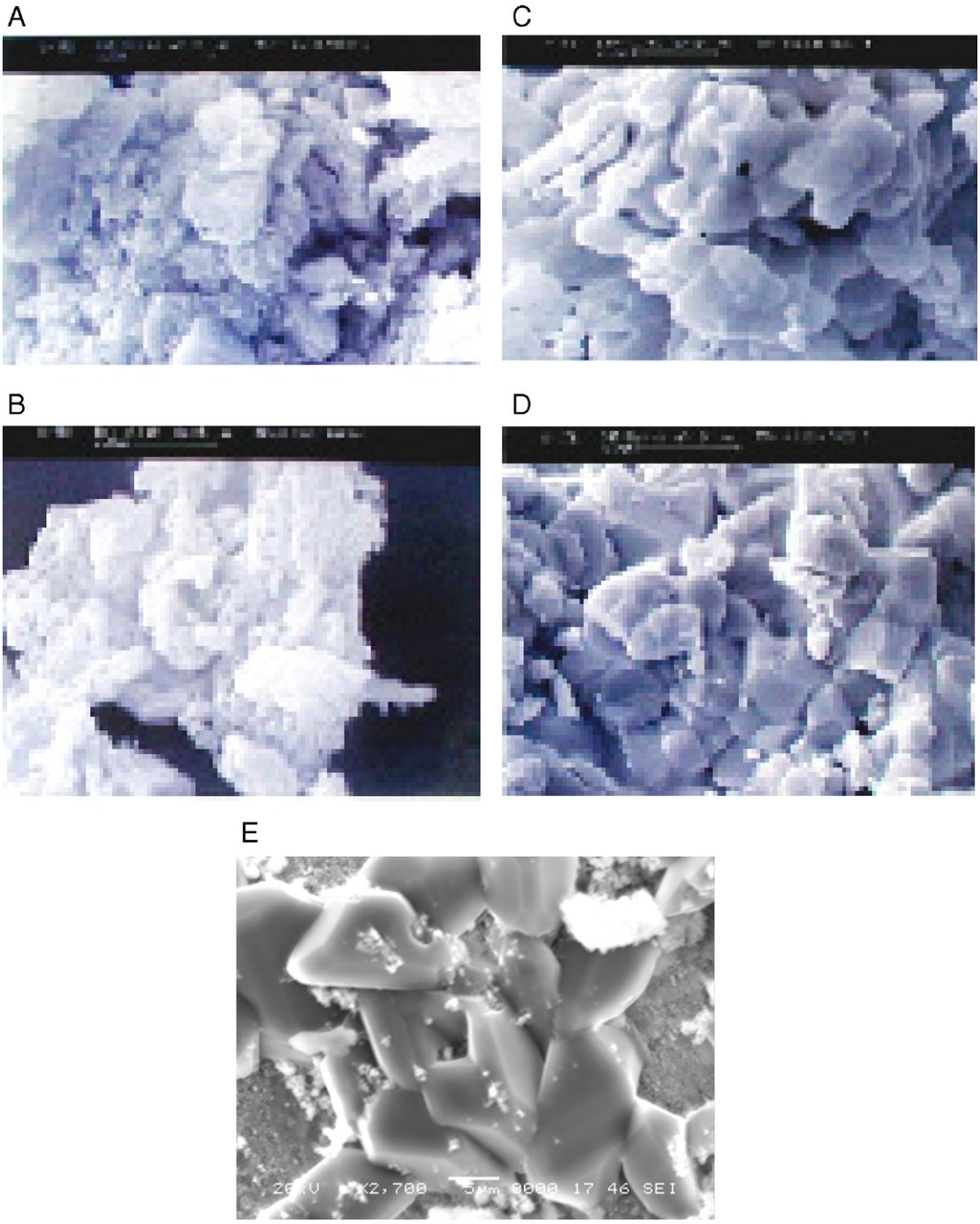

Fig. 2. Scanning electron micrographs of $Cu_{0.5}Co_{0.5}Fe_2O_4$ sintered at A) 773 K B) 873 K C) 973 K D) 1073 K E) 1173 K.

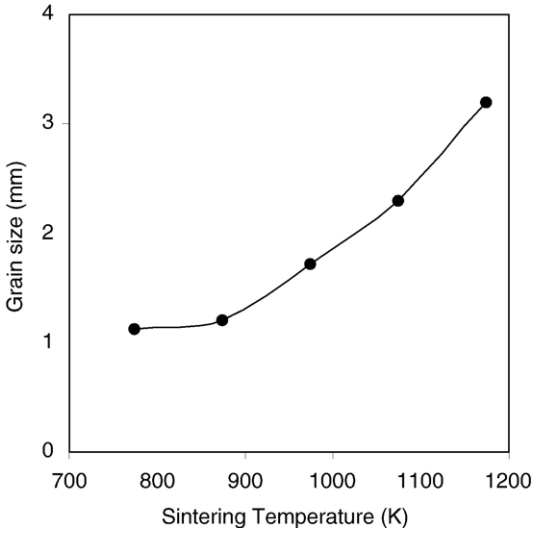

Fig. 3. Effect of sintering temperature on $Cu_{0.5}Co_{0.5}Fe_2O_4$.

have the proper stoichiometric proportion required in the final ferrite powder. A mixed solution of the above sulphates was prepared in double distilled water. The corresponding oxalate was made by adding oxalic acid maintaining pH at 4.7. The chemical reactions proceed as follows:

$$CuSO_4 + 2H_2O + C_2O_4^{-2} \rightarrow CuC_2O_4{\cdot}2H_2O + SO_4^{-2}$$

$$CoSO_4 + 2H_2O + C_2O_4^{-2} \rightarrow CoC_2O_4{\cdot}2H_2O + SO_4^{-2}$$

$$FeSO_4 + 2H_2O + C_2O_4^{-2} \rightarrow FeC_2O_4{\cdot}2H_2O + SO_4^{-2}$$

The precipitate was washed thoroughly with distilled water to remove the sulphate and filtered through the Whatmann filter paper no. 41. The absence of sulphate ions in the filtrate was confirmed with the barium chloride test. The precipitate was dried to obtain the powder, which was presintered at 773 K for 1 h in air. The presintered powder was milled in agate mortar with acetone as a base. The powder was then divided in to six parts and sintered at different temperatures.

X-ray powder diffractograms were recorded on Philips PW-3710 diffractometer by continuous scanning in the range 0 to 100° using a Chromium target ($\lambda = 2.2897$) with V_2O_5 filter. Lattice constants were calculated using the formula, $a = d(h^2+k^2+l^2)^{1/2}$ where the notations have their usual meaning . Scanning electron micrographs were recorded on JEOL, T 330 machine using 10 kV as the accelerating voltage and a magnification of 10,000×.

The radii of cations at A-site and B-site are shown in Table 1, which were calculated using the expressions,

$$\gamma_A = (u-1/4)a(3)^{1/2} - R_0$$

$$\gamma_B = (5/8-u)a - R_0$$

where γ_A and γ_B are the radii of the cations at tetrahedral and octahedral sites respectively, R_0 is the radius of oxygen ion and u the parameter.

3. Results and discussion

X-ray diffraction patterns of the representative $Cu_{0.5}Co_{0.5}Fe_2O_4$ sample at different temperatures are presented in Fig. 1. It is observed that the well-defined peaks with (311) reflection appear to be more intense. All the planes are allowed planes with no ambiguity. Fig. 1 also shows clearly that the sharpness increases with the increase in temperature. It also reveals that the peaks of the samples sintered at 1173 K are the most intense. The dependence of ferrite formation on sintering temperature has been noted for Ni–Zn–Cu ferrite sintered at different temperatures from 423 to 1223 K in the step of 100 K [11,12]. The lattice parameter shows minimum values at 1073 K. The ionic radii on A-site are less than that on B-site (Table 1).

Studies of scanning electron micrographs indicate that grain size increases with increasing temperature and sintering time (Fig. 2). In the present samples the grain size increases from 1.2 μm for the sample sintered at 873 K for 4 h to about 3.2 μm for the sample sintered at 1173 K. The variation in grain size calculated from scanning electron micrographs are plotted in Fig. 3. It is observed from Fig. 3 that the grain size increases with the increase in temperature. The slight variation in linearity of grain size is due to constant heating schedule. It is also observed that the grains are more or less spherical in shape, having smaller grain size distribution. These samples are also compared with those samples prepared by the ceramic method [13] which shows large grain size (7.8 μm) for $Ni_{0.4}Zn_{0.6}Fe_2O_4$ while our average grain size is 1.90 μm. The maximum grain size is observed at higher sintering temperature, 1173 K for 4 h.

4. Conclusion

It has been shown that fine grain Cu–Co ferrite powder can be prepared at low temperature using low sintering time by the oxalate precipitation method.

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