

Synthesis and AC magnetic susceptibility measurements of $\text{Fe}_{(2-x)}\text{Al}_x\text{O}_3$ compounds

M.E. Villafuerte-Castrejón^{a,*}, E. Castillo-Pereyra^a, J. Tartaj^b, L. Fuentes^c,
D. Bueno-Baqués^c, G. González^a, J.A. Matutes-Aquino^c

^a*Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Ciudad Universitaria, A. P. 70-360, México, D.F. 04510, Mexico*

^b*Departamento de Electrocerámica, Instituto de Cerámica y Vidrio, CSIC, Camino de Valdelatas s/n 28049 Cantoblanco, Madrid España, Mexico*

^c*Centro de Investigaciones en Materiales Avanzados. Miguel de Cervantes 120, Chihuahua, Mexico*

Abstract

FeAlO_3 have orthorhombic unit cell and $\text{Pna}2_1$ space group. It is ferrimagnetic, magnetoelectric, and piezoelectric with strong magnetic anisotropy. Conventional ceramic and oxalic precursors routes require high temperatures and second phases appear in the final product. We prepared a nonstoichiometric serie by precipitation of an aqueous solution containing the precursor salts at lower temperature. X-ray diffraction, densities and AC susceptibility were measured. © 2004 Elsevier B.V. All rights reserved.

PACS: 75.50.gg

Keywords: Aluminum iron oxide; Solid solution; Ferrimagnetism; Magnetoelectricity; Piezoelectricity; Coprecipitation

1. Introduction

The FeAlO_3 crystallizes in an orthorhombic cell with parameters: $a = 8.5661 \text{ \AA}$, $b = 9.2491 \text{ \AA}$, $c = 4.9892 \text{ \AA}$, $V = 394 \text{ \AA}^3$, $Z = 8$, $D_x = 4.411 \text{ g/cm}^3$ and space group $\text{Pna}2_1$. This structure has hexagonal and cubic closed packing of oxygen ions. There are four cations sites: Fe1, Fe2, Al1 and Al2. The Al1 coordination is tetrahedral and quasi-octahedral for the rest of cations. The fractions f_i of iron ions over the four cation sites are $f_1 = 0.78$, $f_2 = 0.76$, $f_3 = 0.10$ and $f_4 = 0.34$. This compound undergoes a ferrimagnetic phase transition at about 280 K with strong 180° cation–anion–cation super exchange antiferromagnetic interactions. The FeAlO_3 compound is piezoelectric magnetoelectric, ferrimagnetic and at low temperature it shows an extremely high magnetic anisotropy [1].

The standard routes of synthesis of these compounds are complicated due the very narrow range of stability in the binary equilibrium diagram $\text{Fe}_2\text{O}_3\text{–Al}_2\text{O}_3$. Several authors claim for the evidence of “intermediate structures”, these assertions are supported by X-ray diffraction peaks nonattributable to Fe_2O_3 , Al_2O_3 or FeAlO_3 [2], or the presence of either alumina or hematite contaminating the orthorhombic phase FeAlO_3 when it is obtained by solid state reaction [3–5].

By other hand, other nonconventional routes have been proposed: oxalic precursor methods were reported in Refs. [1,6]

In this study, ten different nonstoichiometric compounds were obtained and characterized by X-ray powder diffraction, density and AC susceptibility measurements.

2. Experimental

The compounds serie were prepared by coprecipitation [7], starting from stoichiometric solutions of

*Corresponding author. Tel.: +52-55-56-22-46-46; fax: +52-55-56-16-13-71.

E-mail address: mevc@servidor.unam.mx (M.E. Villafuerte-Castrejón).

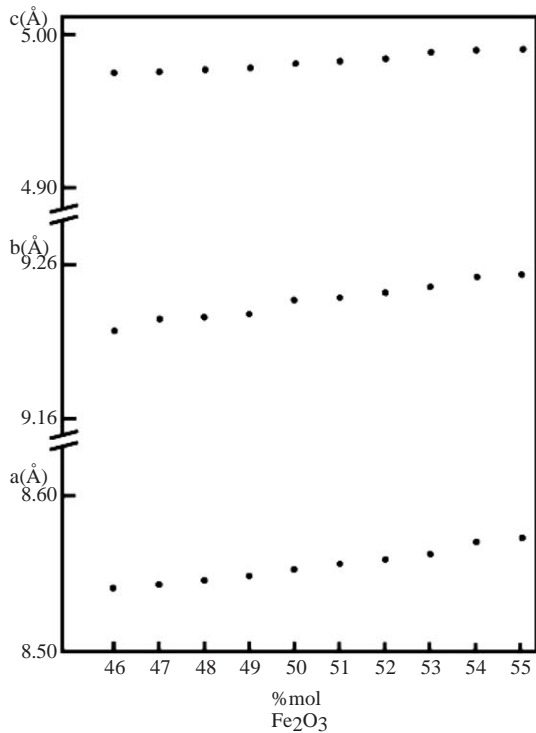


Fig. 1. Variation of lattice parameters of $\text{Fe}_{(2-x)}\text{Al}_x\text{O}_3$ compounds.

aluminum nitrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (Baker Analyzed) and iron nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (Aldrich, 99.99%)

Both solutions were mixed and agitated. A hydroxide ammonium solution (50%), was added drop by drop until rich precipitation. The coprecipitate was filtered, washed with water to remove residual ammonium nitrate salt until neutral pH and dried at 80°C in an oven. The dried powders were grounded in an agate mortar and heated in air using platinum crucible at 1350°C for a period from 1 to 2 h in a electrical furnace with temperature control $\pm 2^\circ\text{C}$. The resulting products were identified by X-ray diffraction. Powder patterns show a pure single phase in all obtained samples [7]. For the determination of lattice parameters a slow scan speed of $0.1^\circ \text{min}^{-1}$ was used, the data were calibrated using NaCl as an internal standard. The density measurements were performed using specific gravity bottles with CCl_4 as displacement liquid. AC susceptibility measurements were carried out.

3. Results

Ten different compositions were obtained by coprecipitation with formula $\text{Fe}_{2-x}\text{Al}_x\text{O}_3$ with $0.9 \leq x \leq 1.08$. The proposed replacement mechanism is $\text{Al}^{3+} \rightarrow \text{Fe}^{3+}$.

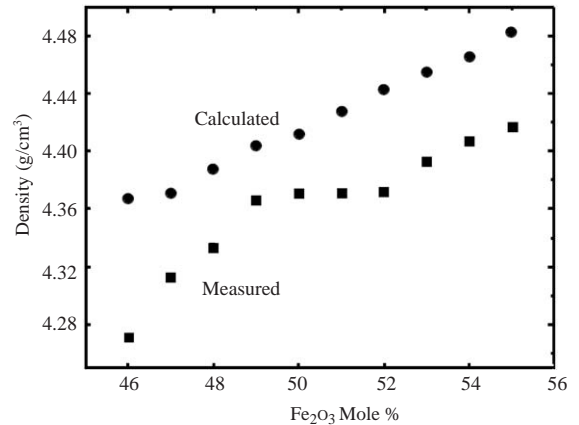


Fig. 2. Experimental and theoretical densities of $\text{Fe}_{(2-x)}\text{Al}_x\text{O}_3$ compounds.

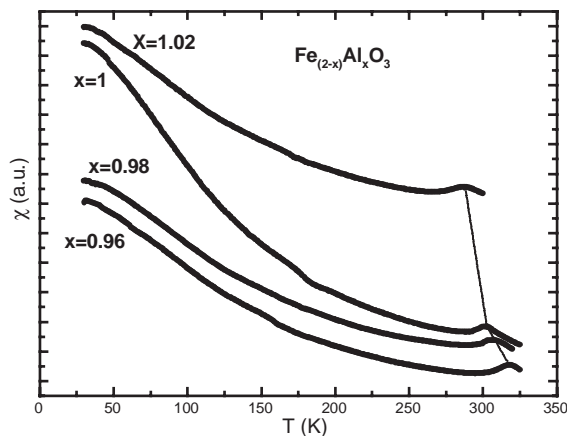


Fig. 3. AC susceptibility measurements show the change of the ferromagnetic transition temperature with composition.

From X-ray analysis one single phase is detected in each composition [7]. Fig. 1 shows the linear variation in lattice parameters a , b and c and the experimental and calculated densities are shown in Fig. 2.

Calculated density values were obtained from the proposed replacement mechanism, indicating its correctness. The samples are ferrimagnetic below the Curie temperature. Fig. 3 shows for the compositions $x = 1.02$, 1.00, 0.98 and 0.96 ferrimagnetic transition temperatures at 288, 301, 308 and 318 K, respectively, as obtained from AC susceptibility measurements.

References

- [1] F. Bouree, J.L. Baudour, E. Elbadraqui, C. Laurent, A. Rousset, Acta Crystallogr. B52 (1996) 217ff.

- [2] J. MacDonald, J.A. Gard, F.P. Glasser, *J. Inorg. Nucl. Chem.* 29 (1967) 661ff.
- [3] A. Muan, C.L. Gee, *J. Amer. Ceram. Soc.* 39 (1956) 207ff.
- [4] L.M. Atlas, W.K. Sumida, *J. Amer. Ceram. Soc.* 41 (1958) 150ff.
- [5] R.R. Dayal, J.A. Gard, F.P. Glasser, *Acta Crystallogr.* 18 (1965) 574ff.
- [6] X. Devaux, A. Rousset, J.M. Broto, H. Rakoto, S. Askenazy, *J. Mater. Sci. Lett.* 9 (1990) 371ff.
- [7] M.E. Villafuerte-Castrejón, E. Castillo-Pereyra, J. Tartaj, C. Moure, P. Durán, J.A. Matutes, L. Fuentes, *Key engineering materials, Proceedings Euro Ceramics VII, Vols. 206–213, Ed Trans Tech Publications, Suiza, 2002, pp. 1405–1408.*