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Magnetic and Structural Characterisation of $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ ($1.0 \leq x \leq 6.0$) Hexaferrites Powders Obtained by Coprecipitation Method

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Abstract. The present work describes the preparation of chromium substituted magnetoplumbite ferrites from nitrate salts, and the effect of Cr on its structural and magnetic properties. Powders with nominal composition $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ ($1.0 \leq x \leq 6.0$) were obtained by thermal decomposition at 920 °C of coprecipitated hydroxide-carbonate mixed salts. A complete substitution of Fe^{3+} by Cr^{3+} was found and the influence of Cr on cell parameters, ordering temperatures and hysteretic properties are reported. The results are discussed and compared in connection with behaviour observed in isostructural Ba- and Sr-compounds.

1. INTRODUCTION

Most of the investigations carried out on M-type ferrites, $\text{MFe}_{12}\text{O}_{19}$ (M= Ba, Sr, Pb), have been dedicated to Ba and Sr compounds. For practical applications Pb-M has some drawbacks, such as a lower saturation magnetisation, anisotropy field and chemical stability. In addition, its preparation involves environmental pollution, consequently, the data in literature about Pb-M based compounds is very scarce [1,2] and, the effect on the magneto-structural properties of iron substitutions by other trivalent ions, and its preparation by unconventional methods remain unexplored.

The preparation of $\text{BaFe}_{12}\text{O}_{19}$ hexaferrite powders by calcination of coprecipitated hydroxide-carbonate salts was first reported by Haneda et al. [3]. Powders with submicronic particles having a good homogeneity in size and chemical composition are obtained using this procedure. The magnetisation processes of these particle systems is well described by the Stoner-Wohlfarth theory and, the coercivity of 6 kOe was the highest value reported for this material.

The experimental details of the preparation of $\text{PbFe}_{12}\text{O}_{19}$ hexaferrites, using the coprecipitation method and metallorganic decomposition (MOD) routes was reported by us in [4]. The present investigation reports the preparation of Pb-M powders by the chemical coprecipitation technique and describes the effects of substituting Fe^{3+} by Cr^{3+} on the magneto-structural properties. The powders with nominal composition $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ ($1.0 < x \leq 6.0$) were studied by means of X- ray diffraction, thermomagnetic analysis, and magnetic measurements.

2. EXPERIMENTAL PROCEDURE

The coprecipitated salts were obtained following the route proposed by Haneda et al. [3]. $\text{Pb}(\text{NO}_3)_2$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were used as starting materials. A cationic solution with the stoichiometric ratio $\text{Pb}/(\text{Fe,Cr}) = 1/12$ was poured drop by drop into a $\text{NaOH}/\text{Na}_2\text{CO}_3$ basic solution having a pH= 10.5. The precipitate thus obtained was repeatedly washed with deionized water till all the NO_3^- ions were eliminated. Afterwards, it was dried at 60 °C and finely powdered. The powders were heat treated in air at 920 °C for two hours in order to crystallise the hexaferrite phase.

X-ray powder spectra were recorded using $\text{CoK}\alpha$ radiation ($\lambda = 1.789 \text{ \AA}$) with a Philips PW 1050/25 modified diffractometer. Magnetic properties were measured using an Oxford model 3001 vibrating sample magnetometer ($H_{\text{max}} = 16 \text{ kOe}$) with low and high temperature facilities. Thermomagnetic analysis (TMA) was performed at 100 Oe with an extraction magnetometer. In all cases corrections due to the demagnetising fields, according the shape of the samples, were done.

3. RESULTS AND DISCUSSION

The X-ray diffraction patterns obtained for the $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ series ($1.0 \leq x \leq 6.0$) confirm, in all cases, the existence of M-type hexagonal structure and no spurious or secondary phases were detected. The a and c parameters both fell with the Cr substitution (5.88-5.85 Å and 23.04-22.71 Å, respectively), in good agreement with the values reported for $\text{BaFe}_{12-x}\text{Cr}_x\text{O}_{19}$ compounds ($1.0 \leq x \leq 6.0$), prepared by the standard ceramic method [5,6]. Moreover, the changes in Curie temperatures observed on the $\text{Pb}(\text{Fe,Cr})$ samples (see the Figure 1) are very similar to those of the $\text{Ba}(\text{Fe,Cr})$. Both types of behaviour clearly suggest that a complete substitution of Fe by Cr had occurred in the range taken into consideration.

Figure 1. Curie temperature for $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ coprecipitated powders.

Figure 2. Room temperature hysteretic properties of $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ coprecipitated powders

The dependence of the relevant properties of the hysteresis loops, measured at room temperature, is reported in the Figure 2. As can be seen the specific magnetisation, σ_s , decreases with Cr content down to 2 emu/g for $x=6.0$, in coincidence with the value reported for $\text{BaFe}_6\text{Cr}_6\text{O}_{19}$ ferrite [7-9]. Further substitution would eventually produce a paramagnetic compound. The coercivity is above 4.0 kOe, in agreement with results obtained for Ba and Sr-M compounds prepared by coprecipitation [3,8]; this may be an indirect indication of the presence of submicronic particles. The values of iHc remain practically constant for low levels of substitution ($1.0 \leq x \leq 2.0$), reaching a maximum for $x=3.0$ (8.5 kOe), and then decreasing to 5.8 kOe for $x=6.0$. The value of the coercivity and the orientation coefficient, σ_s/σ_{90} , in all cases around 0.5, indicate that the particle size is close to single domain.

In summary, $\text{PbFe}_{12-x}\text{Cr}_x\text{O}_{19}$ ($1.0 \leq x \leq 6.0$) hexaferrite powders were successfully obtained by coprecipitation as hydroxide-carbonate mixed salts. The main magneto-structural properties are very similar to those of $\text{BaFe}_{12-x}\text{Cr}_x\text{O}_{19}$ ($1.0 \leq x \leq 6.0$) ferrites. The substitution of Cr^{3+} by Fe^{3+} in the system causes the drastic reduction of σ_s (2 emu/g for $x=6.0$) and a maximum coercivity of 8.5 kOe for $x=3.0$.

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