Magnetic Properties of $\text{PbFe}_{12}\text{O}_{19}$ Hexaferrites Powders Obtained by Metallorganic Decomposition

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Abstract. Magnetoplumbite ($\text{PbFe}_{12}\text{O}_{19}$) powders were obtained by the thermal decomposition, at 900 °C of nitrates dissolved in ethylene glycol. The hysteresis loops showed that the predominant magnetisation mechanism is coherent rotation and a remarkably high value of coercivity (5 kOe) was obtained. Cell parameters, Curie temperature and hysteretic properties are reported. A study of the magnetic after effect is also presented. The results are compared with those obtained for Sr-M sintered compacts with submicronic particles obtained from chemically coprecipitated powders. The dependence of the viscosity coefficient (S) and the viscosity parameter (S_v) on the applied magnetic field were determined at temperatures between 77 and 300 K.

1. INTRODUCTION

The preparation of Ba- and Sr-M hexagonal ferrites using the metallorganic decomposition method (MOD) has been reported by [1,2]. The main advantages of this route are a lower crystallisation temperature for the process and the possibility of preparing submicronic particles with a coercivity suitable for recording applications. Preliminary studies of the synthesis Pb-M hexaferrites using this route have been presented previously [3-5]. The present work intends to review the magnetic and crystallographic data for Pb-M ($\text{PbFe}_{12}\text{O}_{19}$) powders obtained by MOD. In addition the time dependence of the magnetisation was measured between 75 and 300 K in order to determine magnetic viscosity and viscosity parameter in these materials.

2. EXPERIMENTAL PROCEDURE

The MOD method is described by Surig et al. [1]; a solution of Pb^{2+} , Fe^{3+} nitrates in ethylene glycol is heated to induce a polymerisation and subsequently a pyrolysis to remove the organic material. The precursor obtained was heated in air to 920 °C for 2 h to form the hexaferrite.

X-ray powder spectra were recorded using $\text{CoK}\alpha$ radiation ($\lambda = 1.789 \text{ \AA}$) with a Philips PW 1050/25 diffractometer. The magnetic properties were measured using an Oxford model 3001 vibrating sample magnetometer ($H_{\text{max}} = 16 \text{ kOe}$), with low and high temperature facilities. Magnetic viscosity measurements were performed between 75 and 300 K on small disc samples, prepared by pressing and sintering the $\text{PbFe}_{12}\text{O}_{19}$ powders for one hour at 925 °C, and after this procedure a small reduction in the coercivity takes place. $M(t)$ was recorded for different steady reverse magnetising fields, around iH_c , for 1200 s. Before each measurement the sample was saturated at H_{max} .

3. RESULTS AND DISCUSSION

Table 1. Cell parameters and RT magnetic properties of the Pb-M samples studied and literature data (* M_s at $H_{\text{ext}} = 16 \text{ kOe}$).

	a ($\text{\AA}$)	c ($\text{\AA}$)	σ_s (emu/g)	iH_c (Oe)	H_a (Oe)	M_s^* at 0K (emu/g)	T_c (K)
this work	5.877	23.119	54.00	5000	-----	82.00	721
literature data for ceramics [6]	5.877	23.07	56.00	-----	13750	82.00	725

The powder X-ray diffraction pattern confirmed that the magnetoplumbite structure was the main phase present and no spurious or secondary phases were detected. In Table 1 the lattice parameters and intrinsic magnetic properties are reported and compared with the literature data. As observed the agreement between them is quite good. The high value of coercivity (5 kOe) is an indirect indication of the presence of submicronic particles.

In permanent magnets, the variation of the magnetisation with time due to thermally activated processes can be written as:

$M(t) = C + S \ln(t + t_0)$, where S the viscosity coefficient is related to the viscosity parameter, S_v , through the equation

$S_v = S(1 - D \chi_{rev}) / (\chi_T - \chi_{rev})$; D is the demagnetising factor of the sample; the total differential susceptibility, χ_T , was obtained by differentiating the demagnetising $M(H)$ curve; and the reversible susceptibility, χ_{rev} , was estimated from the slope of the minor hysteresis loop described when the field is decreased from the applied field (i.e. $\chi_T = \chi_{rev} + \chi_{irr}$).

Fig. 1 shows the dependence of S and χ_{irr} on the applied field, for different temperatures. The maxima in both, $S(H)$ and χ_{irr} move toward higher fields as the temperature increases. This behaviour is consistent with those observed in sintered hexaferrites by Nishio et al. [7]. Fig. 2 shows that, in all cases, S_v falls with increasing applied magnetic field. This is more evident in the measurements at 295 K and 225 K and, contradicts the results reported in [7,8], in which S_v remains constant over a well defined range of applied field. However, this dependence of S_v on the field has been also observed by us, at room temperature, in submicronic Sr-M hexaferrite powders, prepared by chemical coprecipitation.

Figure 1. Field dependence of the viscosity coefficient S (solid symbols) and irreversible susceptibility χ_{irr} (hollow symbols) at 75, 150, 225 and 295 K.

Figure 2. Field dependence of the viscosity parameter, S_v .

In summary, $\text{PbFe}_{12}\text{O}_{19}$ powders with high coercivity (5 kOe) have been successfully obtained using the MOD procedure. The maxima of $S(H)$ and $\chi_{irr}(H)$ move towards higher fields as the temperature increases and, a dependence of S_v on the field was found.

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