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INFLUENCE OF THE Fe TO Ba RATIO ON THE FORMATION OF HEXAFERRITE BY THE WET METHOD

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Hexagonal ferrite powders with *M* structure have been prepared by the decomposition of an amorphous organometallic precursor. The different ratio of Fe to Ba in the starting citrate-nitrate gels has produced various amounts of α-Fe_2O_3 and $BaFe_{12}O_{19}$ phases. The initial ratio of Fe to Ba ($n = 5.25$) allowed us to get single phase hexagonal ferrite powders with mean dimensions between 0.1 and 0.3 μm. For the study of the formation process of $BaFe_{12}O_{19}$ ferrites we have employed the Mössbauer spectroscopy and the X-ray diffraction analysis.

I. INTRODUCTION

Recently, several methods have been reported [1, 2] for the preparation of single domain particle samples of the hexagonal *M* type $BaFe_{12}O_{19}$. The highly homogeneous ferrite particles with the desired morphology have been produced by a citrate process [3]. The interest in these materials is because of their utilization mainly in the millimeter wave devices or high density magnetic recording applications [4, 5].

In this work we present the study of various initial ratios of Fe to Ba, in order to get single phase Ba hexaferrites. The amount of the α-Fe_2O_3 and $BaFe_{12}O_{19}$ phases was determined by X-ray diffraction and the Mössbauer spectroscopy. The particle sizes of the obtained crystallites were determined by a transmission electron micrograph.

II. EXPERIMENT AND DISCUSSION

In the investigation of the different initial ratio of $BaO \cdot nFe_2O_3$ compositions the values of $n = 6.0, 5.75, 5.5, 5.25, 5.0$ were used and the samples were labelled as BaM(a), BaM(b), BaM(c), BaM(d), BaM(e), respectively. The initial aqueous solution of $Fe(NO_3)_3 \cdot 9H_2O$ was precipitated with an concentrated ammonia

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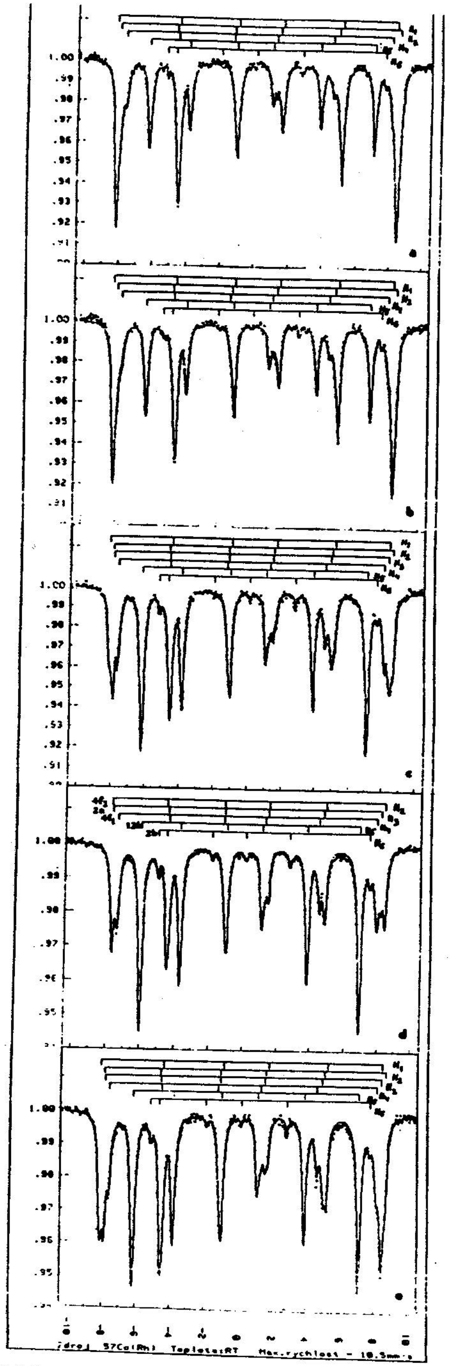

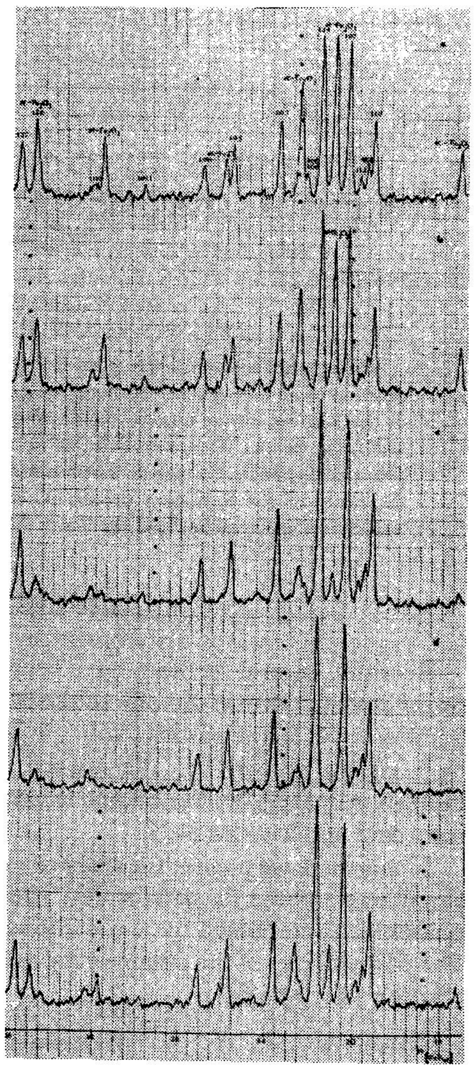

Fig. 2 X-ray diffraction patterns of the samples BaM(a, b, c, d, e)

◄ Fig. 1 Mössbauer spectra of the samples BaM(a, b, c, d, e)

solution. $Ba(OH)_2 . 8H_2O$, ethylene glycol was dissolved in a concentrated citric acid solution and hydroxide iron was added to the solution. The evaporation of the solution was at 70° C and then the rest was dried in a vacuum furnace at 120° C resulting in a precursor. The organic matter was ignited by heating at 450° C for 5h and consequently the BaM phase started to form at 600° C. The hematite was present at the BaM phase. Next a combined thermal treatment at 700° C for 2h and 950° C for 3h was carried out. After the thermal treatment a small amount of $BaFe_2O_4$ was formed. The samples were further treated in a 50% aqueous solution of HCl for 15 min and monoferrite did not occur as reported in [6].

Table I

Mössbauer parameters

Sample	H_1		H_2		H_3		H_4		H_5		H_6	
	MA/m	%	MA/m	%	MA/m	%	MA/m	%	MA/m	%	MA/m	%
BaM(a)	41.1	45.4	41.0	13.4			39,0	11.3	32.9	27.9	31.9	1.9
BaM(b)	41.1	44.4	41.0	12.8			39.2	11.3	32.9	29,6	31.9	1.9
BaM(c)	41.1	18.8	41.0	15.2	40.3	7.8	39.0	16.1	32.9	45.7	31.9	3.3
BaM(d)			40.9	16.2	40.2	8.2	38.8	18.3	32.8	50.7	31.7	6.7
BaM(e)	41.1	19,8	41.0	12.9	40.3	7.5	39.0	14.6	32.9	41.4	31.9	3.9

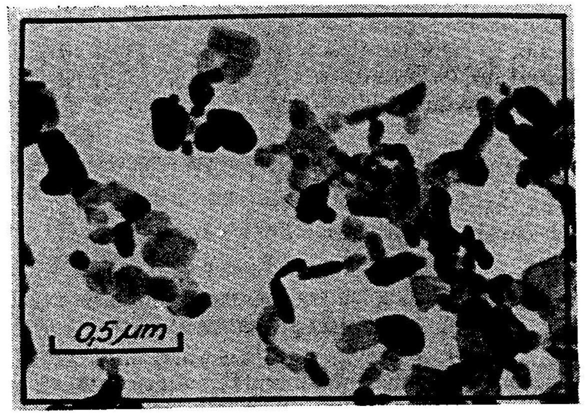

Fig. 3 Electron microscopy of the sample BaM(d)

The Mössbauer spectra were measured by a conventional constant acceleration Mössbauer spectrometer. The source was ^{57}Co in an Rh matrix.

X-ray diffraction analysis was applied using the X-ray diffractometer Phillips aquipped with a PW 1050 goniometer. Radiation of FeK_α was used and the measurement was performed within the Bragg angles from 15 to 36°.

A transmission electron microscopy model Tesla BS 242E was used.

The spectra and the resulting Mössbauer parameters of the samples BaM (a, b, c, d, e) are given in Fig. 1 and Table I, respectively. The obtained hyperfine field value H_1 in Figs. 1a, b, c, e, coresponds to the hematite (α-Fe_2O_3). For the sample BaM (d) with $n = 5.25$ the hematite has not risen. The obtained spectrum is the result of a superposition of five subspectras due to five iron sublattices ($4f_2$, $2a$, $4f_1$, $12k$, $2b$) of the M type of $BaFe_{12}O_{19}$. This conclusion was verified also by X-ray analysis. Diffraction patterns are shown in Figs. 2a, b, c, d, e. The patterns in Figs. 2a, b, c, e prove the presence of hematite. The pattern of the sample BaM(d) with $n = 5.25$ corresponds to the Ba hexaferrite. The electron micrograph of sample BaM(d) shows the particle diameter about 0.1—0.3 μm (Fig. 3), and proves that a good homogeneity is obtained.

III. CONCLUSION

Hexagonal Ba ferrite has been prepared by the decomposition of an organometallic precursor. The proper ratio for the composition $BaC.nFe_2O_3$ ($n = 5.25$) was found in order to get a single phase Ba hexaferrite with a mean particle diameter of 0.1—0.3 μm.

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ВЛИЯНИЕ ОТНОШЕНИЯ Fe К Ba НА ФОРМИРОВАНИЕ ГЕКСАФЕРРИТОВ МОКРЫМ СПОСОБОМ

Гексагональные ферритовые порошки с M структурой были приготовлены разложением аморфных органометаллических заготовок. Различное соотношение Fe и Ba в начальных цитрато-нитридных гелях приводит к различным количествам α-Fe_2O_3 и $BaFe_{12}O_{19}$ фаз. Первоначальное соотношение Fe и Ba ($n = 5{,}25$) нам позволило получить однофазных гексагональный ферритовый порошок со средним размером от 0,1 до 0,3 μм. Для изучения процесса формирования $BaFe_{12}O_{19}$ ферритов мы использовали мессбауэровскую спектроскопию и анализ дифракции X-лучей.