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MINES BRANCH INVESTIGATION REPORT IR 69-58

THE PREPARATION AND EVALUATION OF ADDITIONAL ANISOTROPIC BARIUM FERRITE CERAMIC MAGNETS ON BEHALF OF NORTHERN PIGMENT COMPANY LIMITED, NEW TORONTO, ONTARIO

by

W. S. BOWMAN, G. E. ALEXANDER AND SUTARNO

MINERAL SCIENCES DIVISION

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SUMMARY OF RESULTS

Additional barium hexaferrite samples were prepared from several grades of iron oxide manufactured by Northern Pigment Company Limited, New Toronto, Ontario. Oriented disks have been fabricated and sintered. Various ceramic and magnetic properties of the specimens are presented.

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INTRODUCTION

The Northern Pigment Company Limited, New Toronto, Ontario, has been interested in the effect of the nature of the raw materials on the magnetic and ceramic properties of barium ferrite ceramic magnets for several years. Since the magnetic properties of such ceramic magnets can be greatly enhanced if the crystallites are oriented into a unidirectional array (1), the Mines Branch agreed that it would carry out this study since the necessary fabrication and testing equipment (2) are currently available in the Mineral Sciences and Mineral Processing Divisions. A similar previous study has already been completed and reported (3). It should be noted that the Northern Pigment Co. Ltd. has now acquired its own fabricating and testing equipment, and hence, further work of this nature can be conducted in their own laboratories.

In the present phase of the program, the Northern Pigment Co. Ltd. prepared nine aqueous slurries of finely ground barium ferrite from various grades of iron oxide using normal industrial technology. The contribution of the Mines Branch was the fabrication, sintering and measurement of ceramic and magnetic properties of oriented ferrite disks.

EXPERIMENTAL PROCEDURES

A. Preparation of Barium Ferrite Slurries (Northern Pigment Co. Ltd.)

Nine 800-gram mixtures of $BaCO_3$ and Fe_2O_3 were prepared by stirringthe raw materials in 1 litre of water in a Hobart Mixer for 30 minutes. After drying overnight at 400°F, the mixtures were passed through a 20-mesh screen and rolled in a glass mill for 15 minutes to ensure homogeneity. These mixtures were batch-calcined with a heating rate of 300 deg F per hour and natural cooling.

Kaolin (0.4 per cent by weight) was added to each sample after calcination. The samples were then wet-milled in a steel ball-mill in order to comminute the barium ferrite and homogenize the kaolin. The samples were shipped to the Mines Branch as aqueous slurries for fabrication and evaluation of test specimens. Details of the slurry preparation are given in Table 1.

Samples 1, 2 and 3 were prepared to examine the influence of CaO contamination on the magnetic and ceramic properties of barium ferrite. Samples 2, 4, 5, 6 and 7 were included to check the influence of different origin and morphology of iron oxides. From Samples 6, 8 and 9, it was hoped to see the influence of particle size of the iron oxide on the properties of the ferrite produced from it.

TABLE I

Details of Slurry Preparation

Sample Number	Composition	Iron Oxide Used	Calcination Time and Temperature	Ball-Milling Time
1	$BaO.5{\cdot}5Fe_2O_3$ +0.4 wt.% Kaolin	F-52 containing 0.04 wt.% CaO	2350°F/45 min	24 hr
2	"	F-52 containing 0.08 wt.% CaO	"	"
3	"	F-52 containing 0.11 wt.% CaO	"	"
4	"	3902 (synthetic)	"	"
5	"	F-56 (synthetic)	"	"
6	"	F-34 (natural)	2400°F/1 hr	32 hr
7	"	F-60 + 3902(50:50) (natural + synthetic)	2350°F/45 min	24 hr
8	"	Specularite(-100 mesh)	"	"
9	"	Specularite(-200 mesh)	"	"

B. Preparation and Evaluation of Oriented Disks (Mines Branch)

The slurries received from Northern Pigment Co. Ltd. were dried and re-slurried in ethanol by ball-milling them for 1 hour. Even though this procedure deviates from the normal commercial practice, experience in this laboratory has shown that alcohol slurries are much easier to handle in subsequent pressing operations and that the quality of the ferrite ceramic prepared from such slurries is in no way affected.

Four oriented 1.5-inch-diameter disks were prepared from each sample by pressing the slurries in a magnetic field of about 15,000 oersted at 5000 psi (2). The disks were dried and demagnetized at 950°F and their "green densities", D_g, were obtained from their weight and dimensional measurements. They were sintered for 30 minutes or for 1 hour in oxygen at a temperature of 2200°F with a heating and cooling rate of 180 deg F per hour.

After measurement of the diametral shrinkage, S_d, the disks were lapped to perfect cylinders in order that accurate measurements of bulk sintered density, D_s, and magnetic properties could be made. The magnetic properties of the disks, including coercive force, ${}_bH_c$, intrinsic coercive force, ${}_iH_c$, remanent magnetization, B_r, and maximum energy product, $(BH)_{max}$, were obtained by measuring the hysteresis loops by the "pole-coil" method , as described by Steingroever (4).

RESULTS AND DISCUSSION

The ceramic and magnetic properties of the oriented disks are given in Tables II and III; duplicate disks were prepared and measured. A few disks were cracked during sintering and the values reported for these disks may be less accurate than those for uncracked disks.

In general, the disks sintered at 2200°F for 1 hour have higher values of D_s, S_d and B_r and lower values of ${}_iH_c$ and ${}_bH_c$ than the disks

sintered at 2200°F for 30 minutes. The disks sintered for the longer time have slightly higher $(BH)_{max}$ values due to the higher B_r values. Because $_iH_c >> 0{\cdot}5\ B_r$, the decrease in $_iH_c$ that is associated with the increase in B_r does not lower the $(BH)_{max}$.

The magnetic properties of disks made from Sample 7 are particularly poor, considering the high relative density (over 95% of the X-ray density).

It is difficult to make any comparison between the results obtained from different samples. It is also impossible to draw any general conclusions about the effects of a particular iron oxide or a particular amount of contamination, since it is known that the final properties of a ferrite ceramic are dependent on a host of chemical and technological variables (5). The results given in Tables II and III are valid for the particular sintering conditions employed and under the general conditions described in Table I.

ACKNOWLEDGEMENTS

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TABLE II

Ceramic and Magnetic Properties of Oriented Barium Hexaferrite Disks Sintered at 2200°F for 1 Hour

Sample No.	D_g (g/cm^3)	D_s (g/cm^3)	S_d (%)	B_r (gauss)	$_iH_c$ (oersted)	$_bH_c$ (oersted)	$[(BH)_{max} \times 10^{-6}]$ (gauss-oersted)
1	2.93	4.78	10.7	3410	3010	2980	2.8
	2.93	4.76	10.2	3480	2930	2900	2.8
2	2.97	4.80*	10.5	3650	2980	2950	3.0
	2.98	4.79	9.9	3680	2930	2920	3.2
3	3.02	4.82	10.5	3450	3000	2970	2.9
	3.05	4.75	10.1	3590	2920	2910	3.1
4	2.92	4.78	10.1	3720	2920	2900	3.3
	2.92	4.81	10.3	3730	2800	2780	3.3
5	2.97	4.79	10.9	3570	3140	3100	3.0
	2.93	4.79	10.9	3640	3020	2990	3.1
6	2.75	4.97	13.3	3720	2810	2790	3.3
	2.76	5.00	13.2	3750	2760	2740	3.3
7	2.62	5.07	16.2	3320	3000	2820	2.4
	2.63	5.09	16.2	3280	2920	2800	2.5
8	2.73	4.78	12.7	3520	2800	2750	2.9
	2.74	4.82	12.9	3560	2640	2580	3.0
9	2.71	4.91	13.3	3580	2770	2750	3.1
	2.71	4.91	13.5	3640	2700	2680	3.0

*Cracked disk.

TABLE III

Ceramic and Magnetic Properties of Oriented Barium Hexaferrite Disks Sintered at 2200°F for 30 Minutes

Sample No.	D_g (g/cm^3)	D_s (g/cm^3)	S_d (%)	B_r (gauss)	$_iH_c$ (oersted)	$_bH_c$ (oersted)	$[(BH)_{max} \times 10^{-6}]$ (gauss-oersted)
1	2.94	4.69	10.2	3390	3140	3070	2.7
	2.93	4.67	10.2	3380	3100	3050	2.7
2	2.95	4.64*	9.9	3370	3120	3050	2.7
	2.96	4.70	9.7	3640	3000	2970	3.2
3	3.01	4.72	10.1	3500	3100	3080	2.9
	3.00	4.71	10.1	3480	3000	2970	2.8
4	2.92	4.68	9.5	3590	3010	3000	3.1
	2.94	4.72	9.7	3720	2820	2780	3.3
5	2.93	4.66	10.5	3510	3230	3190	2.9
	2.93	4.72*	10.3	3550	3030	3000	3.0
6	2.76	4.93	12.9	3700	2920	2910	3.2
	2.79	4.95	13.1	3720	2840	2820	3.3
7	2.61	5.08	16.2	3200	3090	2850	2.4
	2.60	5.05	16.1	3190	3000	2760	2.4
8	2.70	4.75	12.6	3400	2890	2850	2.8
	2.71	4.77	12.8	3540	2770	2700	2.9
9	2.69	4.86	13.2	3550	2920	2850	3.0
	2.72	4.85*	13.3	3520	2780	2760	2.9

*Cracked disk.

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