

MAGNETIC INTERACTIONS BETWEEN Co₅ Sm AND IRON COBALT FINE PARTICLE COMPOSITES

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ABSTRACT

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Ву

Manfred Doser

A brief history of magnetism and magnetic theory is presented. Various compositions of Co_5Sm and Lodex were pressed and their magnetic characteristics measured. The resulting magnetic interactions between the crystal-anisotropy Co_5Sm and the shape-anisotropy Lodex mixtures are recorded for remanence, intrinsic coercive force, and energy product. The interactions are also displayed by intrinsic hysteresis curves which show distinct "kinks" in the curves for composites containing from 20 to 80% Lodex.

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A THESIS

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LIST OF SYMBOLS AND NOMENCLATURE

- <u>Anisotropy</u>: Relating to the directional dependence of magnetic properties.
- H_C or <u>Coercive</u> <u>Force</u> (oersteds): The magnetizing force required to bring the induction to zero in a magnetic material.
- H_{Ci} or <u>Intrinsic Coercive</u> Force (oersteds): The magnetizing force required to bring to zero intrinsic induction of a magnetic material.
- <u>Energy-Product</u> <u>Curve</u>: The curve obtained by plotting the product of the coordinates of the demagnetization curve $B_d H_d$ as abscissa against the induction B_d as ordinate.
- <u>BH max</u> or <u>Maximum</u>, <u>Energy Product</u> (gauss-oersted x 10⁶) (m.g.o.): The value corresponding to the maximum value of the energy-product curve.
- B_i or <u>Intrinsic</u> <u>Induction</u> or <u>Remanence</u>: The excess of the induction in a magnetic material over the induction in vacuum for a given value of the magnetization force. $B_i = B - {}_VH$ where ${}_V$ is the permability of the material relative to space.
- Br or <u>Induction/Remanence</u> (gauss): The magnetic induction corresponding to zero magnetizing force in a magnetic material.
- B_{is} or <u>Intrinsic</u> <u>Saturation</u> <u>Induction</u> (gauss): The maximum intrinsic induction possible in a material.

INTRODUCTION

HISTORICAL

The use of permanent magnetism dates from 200 - 600 B.C. when loadstone (Fe₃O₄) was first used by the Greeks; (1) and many improved magnetic materials have been made through the centuries using iron, iron chrome, hardened steels, and iron alloys containing copper, silver, molybdenum, manganese, and carbon. Development of the first true hard permanent magnetic materials came in 1931⁽²⁾ following Mishima's discovery of Fe-Ni-Al alloys. In the years 1934 - 1938 the alloys Alnico 2, a Al-Ni-Cu-Co-Fe alloy, and Alnico 5, a field treated version of Alnico 2, were announced. Alnico 8 and 9 were announced in 1956. The Alnico 8 was a high titanium content alloy, and Alnico 9 was a directional grained version of this alloy. Magnetically, the alloys have energy products ranging from 3 to 10 m.g.o. (million gauss oersteds). Hexagonal crystal ferrite magnets with barium and strontium were announced in 1952 and 1954. Although the ferrite materials have low energy products (4 m.g.o. maximum), their coercive properties are as high as 3500 oersteds i.e., about 1500

oersteds higher than the best Alnicos. Lodex, (R) (General Electric trademark for fine particle permanent magnets) developed in 1955, consisted of single-domain iron particles imbedded in a lead matrix. The iron particles later were replaced by FeCo particles to obtain higher energy products. The advantage of this material was not the relatively modest 3 m.g.o. energy product but its ability to be formed in finished shapes without the need for sintering or heat treat-The next major advance toward higher coercive materials ment. (3, 4, 5,)occurred in 1969 - 70 with the discovery of Co₅Sm having maximum energy product (BHmax) values of 19 m.g.o. Since then many modifications of Co_5R (R = rare earth mixtures) compounds have been made, but the highest energy product in any material announced to date⁽⁶⁾ is a $Co_5Sm_{0.5}Pr_{0.5}$ alloy produced by General Electric having a maximum energy of 23 m.g.o. and a coercive force (H_C) of 6800 oersteds. The intrinsic coercive (H_{ci}) values in the cobalt rare earths have been reported to be in the 25,000 to 30,000 oersted range.

MAGNETIC THEORY

The magnetic flux B, in a premanent magnet, is the algebraic sum of two components, one of which results from an external magnetizing force H, and is the magnetizing force acting on the magnet. The other component B; is intrinsic to the magnet itself by virtue of its ferromagnetic This latter value, an internal magnetic field of nature. the magnet, is explained in modern theory as resulting from an alignment of magnetic moments (dipoles) associated with electrons in the atomic lattice. Permanent magnetic moments arise in systems in which unpaired electrons are present. (7) The magnitude of the aligning force is related to the distance between these electrons in neighboring atoms. With increasing temperature, the number of electrons with parallel moments decreases and finally reaches zero at the Curie temperature.

As a ferromagnetic material is cooled below its Curie temperature, electrons having their moments parallel group themselves into small volumes, called domains, which are regions spontaneously magnetized with an intensity dependent

upon temperature and material. A domain may contain several thousand atoms and have dimensions between $100A^{\circ}$ and $10,000 - 20,000A^{\circ}$.

These small domains tend to lie in particular crystallographic directions, but their magnetization directions are randomly oriented so that no magnetization is observed externally. Several domains may exist in a single crystal.

The laboratory measurement of the magnetic hardness of a ferromagnetic material is the intrinsic coercive force (H_{ci}) , which depends on the effectiveness of the various barriers to magnetization reversal.

When materials contain multidomains in a crystal, boundary or "bloch wall" movement is very evident and is shown in Figure 1.



Figure 1 - Representation of "Bloch Wall" Between Oppositely Directed Domains

The figure represents a schematic presentation of two domains of opposite polarity being separated by the "bloch wall." When an external field is applied, the "bloch wall" will tend to move toward one of the ends, and one of the domains will grow at the expense of the other. The "bloch wall will continue to move unless some pinning action, such as an impurity, stops its movement. This procedure is easily reversible, consequently materials having multidomains usually are those with low coercive forces. Frenkel and Dorfman⁽⁸⁾ early suggested that particle size might have a profound effect on coercive force. It was shown that the magnetostatic energy is a volume effect that varies with the third power of the particle radius, whereas the domain boundary energy is proportional to the square of the particle. radius. Below some limiting size particle there will be less energy associated with an external field than with a domain boundary; this is the critical diameter below which single domains are found.

The single-domain particle makes it possible to prepare materials containing no boundaries and having magnetizations which can be changed only by the simultaneous rotation of all the atomic magnetic moments in each particle. This procedure can be a more difficult process to accomplish than "bloch wall" movement. The obstacles to the rotation of the

magnetization vectors are related to the anisotropy energy.

By definition, "magnetic anisotropy" is the preference of magnetic moments to lie along certain specific directions. Anisotropy⁽⁹⁾ can be affected by strain, surface, shape and crystalline geometry. Most important are shape and crystal anisotropy which will be discussed in more detail since this study deals with materials having these properties. The coupling of the single domain with the anisotropy energy largely determines the H_{ci} value in a magnetic material.

"Shape anisotropy" arises from the interactions of dipole-dipole effects in various shaped particles. It can be $shown^{(2,9)}$ that for a spheroidal shaped particle having coherent magnetization reversals, where the moments rotate together and always remain parallel to each other,

$$H_{ci} = (N_b - N_a) M_s \tag{1}$$

where N_b and N_a are demagnetization factors along the minor and major axis respectively, and M_s is the saturation moment.

In an actual system, the moments will not necessarily rotate coherently because of the presence of other factors which cause lower-energy rotation mechanisms. The relatively weak dipole-dipole interactions, giving rise to shape anisotropy properties favor nonuniform magnetization because of long range effects. With no anisotropy present in a system, the dipole exchange would lead to a curling effect during

reversal. However, with anisotropy present and in cases where large particles make up the system, the curling action reduces to predominatly domain-wall motion. For extremely small particles, (single domain size), where domain wall movement is non existent, the particles may be in a state of uniform magnetization at zero field and reversal. In between these two extremes, the lowest energy has been found to be neither domain wall nor coherent reversal, rather a curling process. These mechanisms⁽¹⁰⁾ are shown in Figure 2.



Figure 2 - Schematic Representation of Magnetization Reversal Configurations. The Cross Sections Show Only the Transverse Flux Components.

The intrinsic coercive force for the curling mechanism can be expressed as,

$$H_{ci} = 2 \pi K A/b^2 M_s - N_a M_s$$
 (2)

where A is the exchange constant, b the semiminor axis, and K a shape factor constant varying from 1.08 for an infinitely long cyclinder to 1.38 for a sphere.

In practice irregularities in the shape of particles which deviate widely from ellipsoidal or rod shape give rise to more nearly a fanning mechanism and can be expressed by the relationship

$$H_{Ci} = \frac{1}{2} \mathcal{T} M_{S}$$
(3)

This is the best expression for materials such as Alnico where the precipitate particles are peanut-shape and have many cross ties. It has been shown that the behavior of specially grown elongated particles having diameters less than $1000A^{\circ}$ agree very well with the curling mechanism expression for H_{ci} .

Crystal anisotropy effects are intrinsic but are not fully understood. The electron orbits in these materials are coupled to the crystal lattice, causing the moments to align themselves along certain axes. For example, barium ferrites and new cobalt rare earth magnetic materials have hexagonal crystals in which the C axis is the preferred magnetic direction. The coercive force of these materials can be expressed as,

$$H_{ci} = 2K/M_{s}$$
(4)

where K is now the crystal anisotropy constant. The correlation between the calculated value and actual measurements agree only qualitatively and deviations are expected to result from compositional fluctuations, defects in crystal structure causing lowering of K locally, or positive or negative contributions from other anisotropies. As with the shape anisotropy materials, partical sizes equal to the single domain will produce the largest H_{ci} values and be the most resistive to demagnetization.

The reversals that take place in these domains can readily be seen by drawing complete hysteresis loops or partial demagnetization curves as shown in Figure 3.



A magnetic material just freshly prepared would have a net magnetic field of 0 until an external field is applied. When the field is applied, any domain walls would move first causing rotation of some of the moments. As more field is applied, rotation of moments in the domains takes place until they are aligned in the direction of the field. The field energy required to complete this alignment is called B_{is}, the saturation induction. Removal of the external field will cause the remanence to drop to B_r , which is called the residual induction of the magnet. B_r is directly dependent on whether the preferred shaped or crystal particle directions were lined up with the direction of external field magnetization. A measure of the degree of this alignment can be expressed in the ratio of B_r/B_{is} . (5)

A value of 1 would represent perfect alignment of the particles with the external field. This value is never obtained in practice because of the difficulty of getting all moments to rotate fully to their preferred directions. This is in part prevented by the inability to obtain perfect shaped particles or crystals.

When the external field is reversed, the demagnetization curve A_1 is obtained in the second quadrant showing how the moments are being reversed. When the remanence (B) equals zero, the H_{ci} value of the material is obtained. Further application of the external field will completely saturate the magnet in the reverse direction, as shown in the third quadrant of the figure. If the external field is reversed again, the hysteresis curve A_2 will be drawn and end at the original B_{is} point. To make the curve completely symetrical the external field must be large enough to fully saturate the magnet at both ends.

The curves for crystal anisotropy and shape anisotropy materials are very different in practice for hard magnetic systems. In shape-anisotropy materials, H_c and H_{ci} are nearly the same because of the inability to make perfectly shaped particles. In crystal anisotropy materials the H_{ci} is usually much greater than H_c , (Figure 4).



Figure 4 - Second-Quadrant Demagnetization Curves for Shape and Crystal Anisotropy Permanent Magnet Materials

The H_c curve is obtained by taking each B_i value on the intrinsic curve and subtracting the reverse applied H. The crystal anisotropy material "true" curve is usually a 45° line where H_c \approx B_r, and the energy product BH_{max} lies half-way between the two points. In shape-anisotropy materials, H_c is always much lower than B_r, and BH_{max} occurs at the knee of the curve.

To obtain the highest saturation magnetization for any particular material, a high volume fraction of magneticshaped precipitate or crystal particle must be held in a nonmagnetic matrix. Thus packing becomes a very important consideration whether the magnet is made by cast-precipitation techniques (as in Alnicos), order-disorder reactions (as in CoPt), sintering reactions (as in ferrites), or by synthetic methods where the magnetic particles are first made and then mixed with a non-magnetic matrix material (as in Lodex). Two extreme behaviors have been observed and are shown in Figure 5.

In all cases B_{is} increases linearly with increasing packing fraction p. The intrinsic coercive force remains constant for crystal anisotropy materials but decreases for shape anisotropy materials.

Neel⁽¹²⁾ derived an expression for an isotropic assembly of elongated particles whose moments are parallel and



Figure 5 - Theoretical Effect of Packing on an Aligned Sample of Fine Particals with Crystal and Shape Anisotropy (Courtesy of Parker and Luborski⁽²⁾)

magnetization is uniform,

$$H_{ci} = H_{ci}, p = 0 (1-p).$$
 (6)

Later it was shown that this expression would also hold for a random assembly of parallel infinite cylinders reversing coherently.

The graphs in Figure 5 are based on the assumption that all particles are perfectly aligned and the domains all are equally magnetized. This perfect alignment and magnetization does not happen under experimental conditions except at lower values of p.

The purposes of this study were to determine whether Co₅Sm would follow the theoretical crystal anisotropic curve and to determine the magnetic properties of mixtures of crystal and shape anisotropy materials.

EXPERIMENTAL APPROACH

Julien and Jones⁽¹³⁾ reported on the affects of $\propto -\gamma$ phases in Alnico and showed how the properties of the magnet decreased as the amount of γ phase increased. A pronounced "kink" was observed on the demagnetization curve when about 12% γ phase was precipitated. This paper stimulated a personal interest in determining if magnetic materials would react in a similar manner.

A decision was made to test the effect of mixing a shapeanisotropy magnetic material such as Lodex with a crystalanisotropy magnetic material such as Co₅Sm. Since "kinks" occur in the demagnetization curves of Alnico, a similar reaction might be postulated for the Lodex and Co₅Sm system. A second purpose of the experiment was to determine what effect Lodex would have on remanence, intrinsic coercive force, and energy product of Co₅Sm.

[•]Lodex and Co₅Sm were chosen specifically because their magnetic properties are widely different and such large differences might result in more pronounced particle interactions and thus be more easily observable. Lodex, being a

magnetic material in a lead matrix, also allowed the two magnetic materials to be compacted without the use of . additional binder.

Lodex is an iron-cobalt magnetic material electroplated in a cell having a mercury cathode and an anode of iron and cobalt. The iron cobalt single-domain particles are formed in the mercury cathode. This mercury slurry later is heat-treated and matrix additions are made to obtain specific magnetic properties. It is preformed into an aggregate billet in which the single-domain ($50A^{\circ}$ diameter x $700^{\circ}A$ length) particles are aligned in one direction. The billet is then ground to a size which can be used for final pressing. The Lodex process is outlined in the block diagram of Figure 6.

The alloy Co₅Sm is produced by melting pure 99.9% cobalt and samarium in a vacuum induction furnace. The billets from this melt are crushed in a jaw crusher and further refined to a 12-micron size in a nitrogen gas-jet mill. Two compositions are made: one of 33% samarium content and the other a 60% samarium-rich phase used as a liquidphase additive. The two compositions are blended to produce an average composition of 37% samarium, and the blend is placed in a rubber tube and taken to a 60,000 oersted superconductor where the material is aligned and pressed



- US Figure 6 - Block Diagram of Lodex Manufacturing Process

sufficiently to hold the material together before final pressing.

The powder next is hydropressed at pressures of 200,000 psi to a density of 77 - 80% theoretical. After removal from the rubber tube the billets are sintered at 1120^OC in an argon atmosphere for 30 minutes to achieve a 90% density. The block diagram of the process is shown in Figure 7.

The experiment was started after obtaining the desired preformed Lodex and sintered Co₅Sm. The chemical and magnetic properties of these alloys are shown in Table 1. The Lodex billet was ground into powder using a Weber Boss pulverizer mill and screened to obtain the +200 -18 Tyler fraction.

The sintered Co₅Sm billet was ground in a jaw crusher and further reduced in a pulverizer to achieve a particle distribution as shown in Table 2.

By grinding the sintered Co_5Sm billet to this size powder an aggregate particle containing many crystals is obtained having the preferred magnetic axis aligned in the same direction. This in turn assures a maximum moment for field pressing. However, the main reason for making the large particles is to produce a stable powder resistant to oxidation; particles of Co_5Sm in the 10-micron range are very susceptible to rapid oxidation in air.



Figure 7 - Flow Diagram of Co₅Sm Manufacturing Process

Material			Co	mposit	ion Wt	. %		
	s _m	Co	Fe	s _b	Pb	s _n	н _д	Other
Lodex		14.1	21.4	5.09	55.1	.91	3.12	.28
Co ₅ Sm*	37	63						

Table 1 - Lodex and Co₅Sm Chemical and Magnetic Properties

	Magn	etic Prop	perties
	B _r	H _{Ci}	BHmax
Lodex	6500	900	2.2
Co ₅ Sm*	7890	17,600	14.8

- The magnetic properties of Co₅Sm are those obtained after sintering the aligned powder to a density of 90% and magnetizing the billet in a 60,000-oersted field.
- Table 2 Co₅Sm Powder Distribution After Grinding the Sintered Billet

Screen Size Microns	Weight gms	Wt. Percent
-61	11	2,08
+61 -104	70	12.8
+104 -147	122	22.3
+147 -208	176	32.2
+208 -295	126	23.1
+295 -417	33	6.05
+417	8	1.47

"As sintered" Co₅Sm properties are shown in Table 1 after being magnetized in a 60,000-oersted field.

After obtaining the desired magnetic powders, mixing experiments were carried out for various combinations of Co₅Sm and Lodex. The powders were accurately measured to the nearest 0.05 gm on a Mettler Pl200 balance. It was found necessary to add 0.8 wt% of MoS₂ as a lubricant to prevent laminations in the samples after pressing. Other lubricants probably could have been used, but this is a convenient one for Lodex die-pressing. The weighed powders were placed in 2-oz glass jars and rotated end for end at a speed of 12 rpm for 15 minutes to achieve blending.

A stainless steel nonmagnetic die was made that had a cavity $\frac{1}{4} \times 3/4 \times 1\frac{1}{2}$ inches with outside dimensions of 3 x 5 x $1\frac{1}{2}$ inches. A picture of the unassembled die is shown in Figure 8.

Then 6 grams of mixed powders was poured into the stainless steel die and placed in the magnetizing coil (Figure 9). This coil was equipped with tapered pole pieces that concentrate the magnetic field to 11,000 oersteds in a two inch diameter cylindrical region between the pole faces. The die was placed between the pole pieces so that the die cavity was in the center of the pole-face region. The magnetizer was switched on; and the powder was aligned in the 11,000 oersted



Figure 8 - Nonmagnetic Stainless Steel Die and Punches



Figure 9 - Electromagnet System for Alignment of Co₅Sm Powder

field. While the field was on, a pressure of 1300 psi was applied by the vertical ram to prevent the aligned particles from becoming random again when the field was removed.

The die then was removed from the magnetizer and pressed in a Pasadena hydraulic press (Figure 10) to a pressure of 210,000 psi.



Figure 10 - Hydraulic Press for Final Pressing Cycle

After removal from the die, the sample dimensions were measured and densities determined. In order to achieve reproducible magnetic measurements, the samples were all



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saturated at 60,000 oersteds in a General Electric Model 65E-175 amp superconducting magnet (Figure 11). The pressedmagnet size was small enough to allow the magnet to be inserted in the field and pulled out again without having to turn the field off. Fields of 25,000 oersteds which could be obtained from an electromagnet were not sufficient to saturate the composite materials.



Figure 11 - Superconducting Magnet

Final magnetic measurements were made on an O. S. Walker Model MH 5.0 Hysteresisgraph capable of measuring coercive forces as high as 25,000 oersteds (Figure 12) and of drawing curves on a X - Y console plotter. Accuracy of the electronic integrating flux meter is 1% or better, and the Hall-effect gaussmeter is $\frac{1}{2}$ % or better.



Figure 12 - 0. S. Walker Hysteresisgraph Curve Tracer

Figure 13 shows a block diagram of the test features of this equipment. The Hall probe measures the coercive force of the applied field. The B_i coil is placed around the sample. This coil is composed of two components; one measures the B or induction of the sample, and the other part measures the H of the applied field. Since⁽¹⁾

$$B_i = B + H \tag{7}$$

the resultant from these coils measures B_i directly.

The B_i coil consists of 490 turns of 3-mil wire having an overall resistance of 53.4 ohms. The effective area of





this coil is 913.4 $\rm cm^2$. For measurement of $\rm H_{ci}$ of these samples, a 290.3-ohm Hall probe capable of measuring up to 10,000 oersteds was used.

Output from these probes are fed into their respective amplifiers so the B_i and H_{ci} can be displayed on meters or directly plotted on the X-Y recorder. This equipment has a balancing circuit which allows stabilization of the electronic circuitry to prevent measurement drift.

To measure B_r the sample was placed between the pole pieces and the B_i coil placed over the magnet as shown in Figure 14.



Figure 14 - Closeup View of Hall Probe, B_i Coil and Pole Pieces

The gap between the pole faces was closed until the magnet touched both poles. The recorder was balanced to read full scale when the magnet was in this closed magnetic circuit.

Next the magnetic circuit was broken by opening the gap between the pole pieces. The flux measured at this point is the leakage flux from the magnet. Removal of the coil from the magnet causes a further change in flux measurement (Y direction) which represents the remainder of the induction or remanence of the magnet. The summation of these two values equals B_r (see Appendix A).

The B_i coil was placed over the magnet again, and the pole pieces were closed. Adjustments were made on the X - Y recorder, and the pen was set at the established B_r value.

By turning the H-drive control, a magnetic field was established counter to the field direction of the magnet. The B_i and H_{ci} values were recorded on the X - Y recorder. Only the second quadrant of the hysteresis loop was drawn. This equipment could not be used to draw a complete hysteresis curve since insufficient magnetic field was available to saturate these magnets in the reverse direction. Measurement errors were found to be less than 2% provided the samples were remagnetized each time at 60,000 oersteds in the superconductor.

EXPERIMENTAL RESULTS

Table 3 is a summary of the magnetic data obtained for various compositions of Lodex in Co_5Sm . Figure 15 is a graphical representation of how H_{ci} varied with various amounts of Lodex. Excellent data points were obtained on the Lodex-rich compositional side. The scatter obtained on the Co_5Sm -rich region was due to the poor structural strength of the compact. Corners of the magnets would break off easily with compositions containing less than 14% non-magnetic binder material. In order to reduce the scatter and obtain some repeatability, the samples were sprayed with lacquer after die-pressing.

Remanence (B_r) for Lodex-Co₅Sm compositions is plotted in Figure 16. The data shows remanence increasing as the Lodex content increases. From theoretical calculations (Appendix A) the B_r for the 100% Co₅Sm compact should be 6950 assuming perfect alignment and stress-free surfaces. Only 85% of this value was obtained in the experiment. It is relatively certain that some misalignment does exist since cogging of particles must take place during field

15R 5500 15R-1 5850 15R-2 5900		5700	۲ ۲	C TL	С ца	α C	
15R 5500 15R-1 5850 15R-2 5900	3000	5700	4.5	14.2	85.0	0.8	10.0
15R-1 5850 158-2 5900	3150	6100	4.65	14.2	85.0	0.8	10.0
15R-7 5900		6300		14.2	85.0	0.8	10.0
		6500		14.2	85.0	0.8	10.0
16 5750	3250	5800	5,3	10.0	89.2	0.8	7.28
16R 5800		2000		10.0	89.2	0.8	7.28
17 6000	3050	5700	5.0	20.0	79.2	0.8	13.7
17R 5500	2900	5900	4.4	20.0	79.2	0.8	13.7
18 5600	2400	4600	3.6	30.0	69.3	0.8	20.0
19 5500	1900	3300	3.0	40.0	59.2	0.8	26.6
20 6000	1700	2600	3 ° 0	50.0	49.2	0.8	33.0
20R 6000		2400		50.0	49.2	0.8	33.0
21 6000	1400	1850	2.6	60.0	39.2	0.8	39.5
21R 6050	1400	1900	3 ° 0	60.0	39.2	0.8	39.5
22 6050	1200	1400	2.4	70.0	29.2	0.8	46.0
23 Broke							
23R 6000	1000	1100	2.8	80.0	19.2	0.8	52.5
24 6000	870	950	2.0	0.06	9.2	0.8	58.8
12 6500	850	006	2.25	100.0	0	0	64.5

Samples aligned and pressed at 11,000 oersteds and 210 K psi and magnetized at 60,000 oersteds.

Table 3 - Co₅Sm - Lodex Summary Data









pressing which would prevent some particles from orienting completely in the proper direction. A further indication of misalignment is the scatter of points in the graph. What effect surface damage has on the ground Co₅Sm aggregates was not investigated in this experiment.

As predicted, "kinks" did appear in the demagnetization curves for certain mixtures of Co₅Sm and Lodex. Figure 17 shows typical intrinsic curves for 0, 30, 60 and 100% Lodex mixtures. These "kinks" were observed in mixtures containing from 20 to 80% Lodex.

Figure 18 shows how energy product varied with Lodex composition. The dashed line represents the region where the maximum "kinking" occurred in the curves. Since BH_{max} is the maximum area under the B-H curve, any "kink" in this curve would lower the BH value.

To assure that the "kinks" in the curves resulted entirely from the interaction of the FeCo particles in Lodex and not a dilution affect, mixtures of Co_5Sm and pure lead were measured. No "kinks" were observed in the demagnetization curves. Figures 19 and 20 demonstrate how B_r , H_{ci} and BH_{max} varied with lead composition.















DISCUSSION AND CONCLUSIONS

A previous study of two-phase ($\ll -r$) Alnico 8 alloy materials reveals the presence of a bend or "kink" in the demagnetization curve, after a pre-field treatment isothermal 900^OC temperature. The reasons for the occurrence of the "kinks" were unexplained. From this study, it was postulated that similar "kinks" might be found in the demagnetization curves of composites made from crystallographically-anisotropic and geometrically-anisotropic magnetic materials such as Co_5Sm and Lodex.

In the experimental results shown here, indeed such "kinks" are observed in the demagnetization curve of a combination of these two magnetic materials in the range of 20 -80 weight percent Lodex. This is found without the need for heat treatment, as was the case with the Alnico 8 alloys; however, in the Co_5Sm -Lodex composites the magnetic phases are already present and being held together with a lead matrix. A possible explanation of these "kinks" may involve an early flipover of domains of the lower H_{ci} Lodex material until all the domains are turned, while the remaining

demagnetization curve characteristics are solely those of the higher coercive Co_5Sm material. This would not explain why H_{ci} is still changing at either end of the "kinking" range.

The intrinsic curve on the Co₅Sm rich-composite varies linearly with Lodex additions; whereas, the curve for Lodex rich composite does not vary linearly. From theory⁽²⁾ if a crystal-anisotropy material such as Co₅Sm is diluted with a magnetically inert material, the H_{ci} should remain constant; the shape-anisotropy material such as Lodex, on the other hand should vary linearly under similar dilution. Some interaction mechanism must occur between the magnetic particle moments in addition to dilution material effects throughout the compositional range.

Another indication of the presence of some interaction may be found in the slight increase of remanence (B_r) in the Co_5Sm -Lodex mixtures as the Lodex content is increased, in spite of the fact that the nonmagnetic content of the system changed from 22 to 64.5 wt. percent. In this set of experiments, the 100% Lodex material did have a higher B_r than the 100% Co_5Sm magnets, yet one would expect the B_r of either material to decrease with dilution by the nonmagnetic material.⁽²⁾

To further substantiate this premise, various compositions of Co_5Sm -lead magnets were made. The value of B_r for these magnets decreased with increasing amounts of lead; whereas, the value of H_{ci} remains nearly constant. These results, Figure 19, are in reasonable agreement with Néel's⁽²⁾ prediction for crystal-anisotropy materials. The observed slight deviations from the theoretical curves may result from imperfect alignment of particles or from crystal imperfections.

Based on a comparison of the results for Co_5Sm -lead with those of Co_5Sm -Lodex, it appears that some interaction between the magnetic particles must be occurring. This effect should be examined further to determine the exact mechanism, but such a study is beyond the scope of the present thesis.

Although the B_r remains almost constant and H_{ci} increases as the Co_5Sm is enriched in the composite systems, no striking increase in the maximum energy product occurs until 80 wt.% Co_5Sm is reached. Since BH_{max} is obtained by plotting the product of the coordinates of the demagnetization curve, the values are directly affected by the "kink" in the demagnetization curve. Thus very little increase in energy product will result from increase in H_c as long as "kinks" occur in this curve.

The physical properties of the pressed part exhibited

poor mechanical integrity until the lead binder content reached 14 wt. percent. Normal handling of the specimens resulted in edge breakage; consequently, there was considerable scatter in data from the Co5Sm-rich composites.

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In conclusion, the data clearly shows that Co₅Sm and Lodex cannot be mixed without generating "kinks" in the demagnetization curve. Whether composites of two or more shaped-anisotropic or two or more crystal-anisotropic magnetic materials held together with a nonmagnetic material would exhibit similar behavior is not known and would be a fruitful subject for further study.

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APPENDIX

APPENDIX A

Theory and Calculations For Magnetic Measurements

Br Measurement

In measuring a magnet open circuit, one cannot measure the B_r directly unless the length of bar is infinite. The magnet will operate somewhere down the curve depending upon the length-to-area ratio of the sample.



Figure 21 - Curve Depicting Open-Circuit Magnet Conditions Applying Parker and Studders analogy⁽¹⁾ for unit properties and geometry relationships for magnetic materials, gives,

$$L_{m}H_{m} - L_{q}H_{q} = 0, \qquad (A1)$$

where L_m = magnet length, H_m = magnetization potential of magnet per unit length, L_q = air-gap length, H_q = air-gap

unit potential. If the air gap and magnet neutral section are equated, and if it is assumed for the moment that all magnetic lines reach the air gap:

$$A_{m}B_{m} = A_{g}B_{g} \tag{A2}$$

where $A_m = magnet area$, $B_m = magnet unit density$, $A_g = gap$ area, $B_g = gap$ flux density. By combining equations 1 and 2,

$$L_{m} = L_{g}H_{g}/H_{m}, A_{m} = A_{g}B_{g}/B_{m},$$

$$V_{m} = L_{g}H_{g}^{2} A_{g}/B_{m}H_{m}$$
(A3)

where V_m = magnet volume and B_q = H_g

From Ohms law and using magnetic circuit analogy Flux = Magnetomotive Force divided by the Reluctance, it can be shown⁽¹⁾ that the "load line" as shown in Figure 21 is equal to,

$$B_{m}/H_{m} = L_{m}A_{q}/A_{m}L_{q} = L_{m}(1)/A_{m}(R)$$
(A4)

where R = reluctance.

Thus for a specific length and area of magnet one would measure the point H_m and B_m when placing a B coil over the magnet while having the magnet open circuited. By placing the magnet between two pole pieces with a magnet return path, the magnet load line will recoil up the demag curve to the B_r value provided the magnet L/D ratio does not put the magnet below the knee of the curve. If the load line is below the knee of the curve, the magnet would recoil at a lower value than Br.

One can calculate B_r from the open-circuit conditions once you know the B_m and H_m points. This is often hard to do since the exact load line is not known until the reluctance value is obtained.

By using the integrating flux meters in the O. S. Walker equipment, a relationship is developed

$$\int dB/dt = K \int E dt = B$$
 (A5)

where t = time, B = flux density, E = voltage measured by integrator amplifiers, K = constant for the particular sample (C.F.).

This relationship is shown in two parts in Figure 22.



Figure 22 - Curve Showing Integrating Flux Regions

By opening the pole pieces and getting the open-circuit conditions for the magnet, the O. S. Walker equipment will measure the first $\int E_1 dt$. When the B_i coil is removed, the

second integral E2dt is measured. By adding the quantities

$$K\left[\int E_1 dt + \int E_2 dt\right] = B_r$$

the remanence of the sample is obtained.

Coil factor (K or C.F.) Calculation for the O.S. Walker
C.F. =
$$10^4$$
 x number of turns in B_i coil x specimen area x 10^{-3}
 10^2

C. F. for this experiment = $49 \times \text{specimen}$ area in (cm)

Theoretical Value for Br for Co5Sm Powder

From theoretical relationships (Figure 5) B_r is directly related to the density of the compact for shape anisotropy

$$\frac{B_{r} \text{ sintered}}{B_{r} \text{ at compact density}} = \frac{\text{density sintered}}{\text{density compact}}$$

 B_r sintered for Co₅Sm material used = 7890 and density = 7.37 gm/cc

$$\frac{7890}{B_{rn}} = \frac{7.37}{6.5}$$
 where B_{rn} = Induction for powder compact
 $B_{rn} = \frac{(7890) (6.5)}{7.37} = 6950$ if aligned perfectly

The measured B_r for the 100% Co_5Sm compact which was die pressed at 210 K psig = 5900 gauss

 $\frac{5900}{6950}$ = .85 = Fraction of sintered B_r obtained

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$\mathbf{B}_{\mathbf{r}}$		H _C	H _c i	BHmax.	Width Inch.	Thickness Inch.	Length Inch.	Area cm ²	Coil Factor	Strength	Density g/cc
		Brok	e								
5900	_	4050	7350	7.2	.290	.256	.761	.479	23.46	Poor	6.50
5600	~	3000	5700	4.5	.280	.257	.759	.463	22.68	Poor	6.72
550	0	3150	6100	4.65	.283	.260	.759	.474	23.25	Poor	6.57
585	0		6300		.277	.256	.758	.457	22.39	Poor	6.87
590	0		6500		.278	.255	.757	.457	22.39	Poor	6.83
575	0	3250	5800	5 . 3	.281	.257	.759	.466	22.83	Poor	6.68
580	0		7000		.284	.255	.758	.467	22.88	Poor	6.67
600	0	3050	5700	5.0	.271	.256	.758	.447	21.90	Fair	6.86
550	0	2900	5900	4.4	.275	.256	.758	.454	22.25	Fair	6.86
560	õ	2400	4600	3.6	.265	.257	.757	.439	21.53	Fair	7.11
55(0	1900	3300	3.6	.255	.255	.757	.418	20.54	Fair	7.47
600	õ	1700	2600	3.0	.244	.256	.756	.403	19.74	Good	7.76
600	õ		2400								
600	0	1400	1850	2.6	.236	.254	.758	.387	19.96	Good	8.05
605	0	1400	1900	3.0	.236	.254	.758	.387	18.96	Good	8.05
605	0	1200	1400	2.4	.255	.227	.757	.373	18.27	Good	8.37
		Brok	e								
600	0	1000	1100	2.8	.218	.254	.759	.356	17.50	V. Good	8.75
600	0	870	950	2.0	.213	.254	.754	.349	17.10	V. Good	8.98
650	0	850	006	2.25	.243	.255	.754	.399	19.55	V. Good	9.15

Table 4 - Magnetic and Physical Data of Co₅Sm - Lodex Composites

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- Magnetic and Physical Data of Co₅Sm - Lead Composites Table 5

Sample #	Wt% Pb	gm Pb	gm Mo S ₂	gm T Co ₅ Sm	hickness Inch.	width Inch.	Length Inch.	Area cm ²	Coil Factor	Density g/cc
25	14	.84	.05	5.11	.256	.256	.761	.437	21.44	7.11
26	20	1.2	.05	4.75	.255	.256	.758	.421	20.63	7.41
27	30	1.8	.05	4.15	.255	.242	.757	.398	19.50	7.84
28	40	2.4	.05	3.55	.254	.230	.757	.377	19.46	8.28
28R	40	2.4	.05	3.55	.254	.229	.758	.375	18.38	8.32
29	50	3.0	.05	2.95	.253	.218	.755	.356	17.43	8.78
29R	50	3.0	.05	2.95	.255	.215	.755	.354	17.33	8.84
30	60	3.6	.05	2.35	.253	.208	.754	.339	16.61	9.24
30R	60	3.6	• 05	2.35	.253	.209	.756	.341	16.70	9.17
Sample #	$\mathbf{B}_{\mathbf{\Gamma}}$	Hc	Hci	BHmax						
25	5550	4100	7600	6.7						
26	5150	3950	7950	5.8						
27	4600	3700	8100	4.75						
28	Broken									
28R	4500	3600	8000	4.7						
29	4000	3300	8150	3.6						
29R	3900	3200	8150	3.4						
30	3400	2900	8200	2.7						
30R	3450	2950	8200	2.8						

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Figure 23 - Co-Sm Phase Diagram

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