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20. Abstract (cont'd)

The values of the saturation magnetization for the quaternary compounds showed much better temperature stability, increasing by about 5 percent in going from 300 K to 4.2 K as compared to 15 percent for the ternary system. A comparison of the present results with previous anisotropy measurements obtained by extrapolation from applied fields up to 20 kOe showed the latter to be too low by as much as 35 percent.

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CONTENTS

	Page
INTRODUCTION	1
EXPERIMENTAL	2
RESULTS	7
DISCUSSION AND CONCLUSIONS	9
ACKNOWLEDGMENTS	12
REFERENCES	14

TABLE

1. Physical Parameters of $Sm_2(Co_{1-x-y}Fe_xMn_y)_{17}$ 11

FIGURES

1.	Applied room temperature anisotropy fields (H_A^0) of $Sm_2(Co_{1-x-y}Fe_xMn_y)_{17}$ as a function of Co atoms replaced.	3
2.	Typical hard direction magnetization curves used to obtain $\mathrm{H}^{0}_{A}.$	5
3.	The anisotropy (H_A) of the system $Sm_2(Co_{1-x-y}Fe_xMn_y)_{17}$ as a function of the fraction of Co atoms (x+y) displaced.	8
4.	Saturation magnetization $(4\pi M_s)$ of $Sm_2Co_{1-x-y}Fe_xMn_y)_{17}$ for y=0 and y=0.059 at room and liquid helium temperatures as a function of fraction of cobalt atoms replaced (x+y).	10
5.	Comparison of room temperature anisotropy constants $K_1 \approx (H_A M_S)/2$ as a function of cobalt atoms replaced as obtained from extrapolated H_A values with those derived from directly measured H_A^S .	14

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MAGNETIC ANISOTROPY IN THE SYSTEMS Sm₂(Co_{1-x}Fe_x)₁₇ AND Sm₂Mn(Co_{1-x}Fe_x)₁₆

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ABSTRACT

The anisotropy fields (H_A) and saturation magnetizations (4π M_S) of the systems Sm₂(Co_{1-x}Fe_x)₁₇ and Sm₂Mn(Co_{1-x}Fe_x)₁₆ were measured at 4.2 K and 300 K in applied fields up to 100 kOe. The presence of the Mn in the latter system raised the anisotropy fields by as much as 55% over those measured for the corresponding compounds in the ternary system. The augmentation of anisotropy was accompanied by a slight decline in 4π M_S of approximately 5% for the materials of highest H_A. The values of 4π M_S for the quaternary compounds showed much better temperature stability, increasing by about 5% in going from 300 K to 4.2 K as compared to 15% for the ternary system. A comparison of the present results with previous anisotropy measurements obtained by extrapolation from applied fields up to 20 kOe showed the latter to be too low by as much as 35%.

INTRODUCTION

During the past decade the use of $SmCo_5$ has been having a significant impact on expanding the range and nature of the applications of permanent magnets. The high coercivities and energy products of $SmCo_5$ -based alloys have led to several novel designs for magnetic devices. These alloys had their initial impact on the design of microwave traveling wave tubes and lately are beginning to be used in rotating electrical machinery, bearings, magnetic suspensions, actuators, speed sensors, tunable biasing magnets, gyroscopes and accelerometers.¹ Typical commercially available $SmCo_5$ magnets have a remanence, Br, of 9.3 kG, a coercivity, H_c, of 9.0 kOe and an energy product, (BH)max, of 21 MGOe.² However, for some time workers in the field have speculated that the Sm_2Co_17 -based compounds have the possibility of yielding still higher energy products, well above 30 MGOe.³ Ray and Strnat³⁻⁵ have pointed to the $Sm_2(Co_{1-x}Fe_x)_{17}$ system as being potentially suitable for high energy, high coercive force permanent magnet applications in that it has

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Address inquiries to: H. Leupold, US Army Electronics Technology & Devices Laboratory, US Army Electronics Research & Development Command, ATTN: DELET-ES, Fort Monmouth, New Jersey 07703 high saturation magnetizations and Curie temperatures as well as large uniaxial magnetocrystalline anisotropies. However, while Sm₂Co₁₇ has a higher room temperature saturation magnetization $4\pi M_s$ ($\sim 13 \text{ kG}$)^{4,6} than does SmCo₅ (11.0 kG), its low anisotropy field H_A (70-110 kOe)^{4,6,7} as compared to SmCo₅ (with quotes as high as 350 kOe)⁶ has discouraged its intensive development as a permanent magnet material, since a high H_A is a necessary (but not always sufficient) condition for attaining a high coercivity. For the three studies ⁶⁻⁸ that have already been done on the Sm₂(Co_{1-x}Fe_x)₁₇ system, it should be noted that the anisotropy fields do not agree in their absolute values^{6,7} and their composition dependence.⁶⁻⁸ Some of the previous H_A data for the system are presented in Fig. 1. To resolve these discrepancies it was decided to remeasure H_A for Sm₂(Co_{1-x}Fe_x)₁₇ in fields up to 100 kOe thus eliminating reliance on the excessively long extrapolations heretofore used to obtain H_A.

During the course of a previous study of the magnetic anisotropy in the Th(Co_{1-x}Fe_x)₅ and Y(Co_{1-x}Fe_x)₅ systems,⁹ F. Rothwarf et al had occasion to measure H_A for the compound Sm₂Co₁₆Mn which had previously been studied by Schaller, Craig and Wallace (SCW).¹⁰ H_A was found to be 150 kOe. Since this was significantly higher than the values generally quoted for Sm₂Co₁₇, it was decided to initiate a magnetic anisotropy study of the quaternary system Sm₂(Co_{1-x-y}Mn_yFe_x)₁₇ where (x+y) < 1. The previous work of SCW¹⁰ on Sm₂Co_{17-x}Mn_x (1 < x < 3) indicated that the best technical results were achieved with Sm₂Co₁₆Mn. Thus, the initial quaternary system Chosen for consideration was the one with y=0.587, corresponding to the system Sm₂Mn(Co_{1-x}Fe_x)₁₆. In this paper we present our encouraging results on the system.

EXPERIMENTAL

The various quaternary alloys were prepared by arc melting and rapid quenching in a copper hearth under argon or by induction melting in a boron nitride crucible and also rapidly quenching under argon. All starting metals were at least 99.9% pure. An attempt was made to homog**enize each** compound by annealing it in a tantalum container sealed into an evacuated guartz tube. The annealing temperatures ranged from 950-1000 C and were maintained from two to four weeks. All samples were **checked** to determine if they were single phase by x-ray diffractometry. **Only results** for the single phase materials are reported here. For the annealing procedure used, the single phase materials were those with the lower Fe concentrations up to Sm2MnCo10Fe6. Reference to the phase diagrams for the Sm-Coll and Sm-Fel2 systems, makes it clear that a much higher annealing temperature, above 1200 C, followed by a rapid quench would be necessary to obtain single phase samples of the iron-rich compounds. Cylindrical quaternary specimens were produced by suspending powders of the various compositions in an epoxy matrix and aligning them in a magnetic field while the epoxy hardened. Powder with grain sizes equal to 37 µm and smaller was prepared by grinding under argon in a glove box in a boron carbide mortar. The powder was thoroughly mixed with Shell B28 epoxy and Shell B40 hardener in a one-to-one proportion and poured into two identical cylinders. While the resin hardened in about one hour at a temperature of \sim 100 C produced by heat gun, a field of 28 kOe was applied to orient the particles in one cylinder parallel



Fig. 1. Applied room temperature anisotropy fields (H_A^O) of $Sm_2(Co_{1-x-y}Fe_xMn_y)_{17}$ as a function of Co atoms replaced. y=0 for all curves.

to and in the other perpendicular to the cylinder axis. The anisotropy fields were determined from magnetization curves of the magnetically aligned cylinders.¹³ The specimens of the ternary alloys were also in the form of powders suspended in epoxy cylinders and were obtained through the kindness of K. Strnat in whose laboratory they were prepared.

Magnetization curves (0-15 k0e) were obtained in the alignment direction and normal to it by means of a PAR vibrating sample magnetometer in the temperature interval from 4.2 to 300 K. $4\pi M_S$ values were obtained from these data. The magnetization curves for the hard direction (0-100 k0e) were measured by means of a magnetometer employing an **0.S. Walker** Co. integrating fluxmeter and an American Magnetics superconducting magnet at 4.2 K. 14 The magnetization curves out to 100 kOe were obtained from these data. The magnetization curves for the easy axis show saturation effects at low fields, whereas those for the hard axis were quite linear above 60 kOe. Extrapolation of the easy/hard axis curves to the point of intersection gives an estimate of the anisotropy field H_A. It should be noted that of the ternary compounds only Sm2Co17 required an extrapolation over an interval of more than 10% of the H_A value, and all compounds with x > 0.1 were saturated at less than 100 kG and required no extrapolation. For the quaternaries more extrapolations were required, but only for SmpMnCo16 did the extrapolation interval exceed 0.25 H_A. Because of imperfect alignment of the suspended particles in the epoxy based samples, the linear approach to saturation of the magnetization curve was relatively shallow for some samples, thus contributing to extrapolation uncertainties as high as \pm 10%. For this reason as many as 20 determinations of H_A were made for samples in which long extrapolations were necessary, and an arith**metic** mean of the results was taken to be the anisotropy field. For samples in which saturation was possible, measured values of HA were generally reproducible to within one or two kG. Figure 2 illustrates both a saturated (A) and an extrapolated (B) curve.

Because of the large uncertainties encountered by previous investigators who relied on very long extrapolations (~ 0.8 H_A) to estimate H_A, demagnetization corrections were generally not attempted for specimens of cylindrical form. In the case of the present work however, demagnetization corrections are for the most part larger than the experimental uncertainties. These corrections are very easily made when H_A is determined from cylindrical samples with their easy axes aligned along a diameter.¹⁴ The external field H_A is then applied along the cylinder axis to magnetize the sample in a hard direction. The applied field at which saturation occurs is then the sum of the anisotropy field and the demagnetization field at the center of the cylinder because the center is where the total field is smallest and hence the last part of the sample to saturate. Thus to determine H_A one need only find the field in a particle at the center of an axially saturated cylinder in an applied field H_A. H_A is then given by

$$H_A = H_A^O - H_D^C + H_D^E - H_D^P$$

(1)

where H_D^C , H_D^E , H_D^P are demagnetization fields arising from magnetic poles

respectively located on the ends of the cylinder, on the epoxy side of the boundary between a particle and the epoxy and on the particle side of the epoxy-particle boundary.

 H_D is equivalent to the field produced midway along the axis of two oppositely poled circular laminae corresponding to the cylinder bases with surface pole densities \pm SM_S.

A straightforward integration over these laminae yields

$$H_{D}^{C} = 4\pi SM_{S} \left(1 - \frac{1}{(1+r^{2})^{\frac{1}{2}}} \right)$$
(2)

where S is the ratio of powder to sample volume, r is the sample diameter to length ratio and M_S is the saturation magnetization. If one assumes an average spherical shape for the powder particles, integrations over the surface of the particle and over that of the spherical cavity in which it is lodged, respectively yield:

$$H_D^P = \frac{4\pi M_s}{3}$$
(3)

$$H_{\rm D}^{\rm E} = \frac{4\pi \rm SM_{\rm S}}{3} \tag{4}$$

Substituting (2), (3), and (4) in (1) yields

$$H_{A} = H_{A}^{0} - 4\pi SM_{s} \left(\frac{2}{3} + \frac{1}{3S} - \frac{1}{(1+r^{2})^{\frac{1}{2}}} \right)$$
(5)

For dilute samples such as those used in the present measurements (S \approx 0.02), the second term in the parentheses of Eq. (5) is dominant. Its form is a consequence of our assumption that the particles are on the average spherical in shape. Scanning electron micrograph (SEM) scans of powders of materials of this type reveal a variety of irregular shapes with their three dimensions approximately the same and with the **directions** of magnetic alignment in individual particles apparently random with regard to the directions of the longest or shortest dimensions of these particles. For regular objects for which demagnetization corrections can be calculated and for which the three dimensions are approximately equal, demagnetization fields are found to be within about 10% that of a sphere. Thus the assumption of sphericity for the individual particles is probably not a bad one. Further, the total demagnetization corrections to the anisotropy fields are only about 5% or less so the error incurred by our assumption is not likely to be much greater than (0.10)(0.05) = 0.005 or 0.5% of H_A.

When one considers a solid transversely aligned cylinder, i.e., when S=1, (5) reduces to

$$H_{A} = H_{A}^{0} - 4\pi M_{s} \left(1 - \frac{1}{(1+r^{2})^{\frac{1}{2}}} \right)$$
(6)



A shows the room temperature results for $Sm_2(Co_{0,9}Fe_{0,1})_{17}$ which was saturated. The dotted line indicates an extrapolation to $4\Pi M_s$ from the neighborhood of H^0 = 20 kOe with the resulting underestimate of H^0_A .



B illustrates an extrapolation of the He temperature magnetization curve of $Sm_2MnCo_{12}Fe_4$ to 4TTMs from the neighborhood of H^O - 100 kOe, the highest field obtainable in the present experiment.

Fig. 2. Typical hard direction magnetization curves used to obtain H^0_A .

Of course, for a very long cylinder $r \rightarrow 0$ and $H_A = H_A^0$.

RESULTS

Figure 1 compares the room temperatures anisotropy fields, H_A^0 , measured in our experiments with the extrapolated values previously obtained by Perkins et al on single crystal spheres and with those of Strnat et al on epoxy-powder cylinders similar to those used in the present work. Since the latter results were uncorrected for demagnetization fields, H_{A} is plotted rather than H_{A} to provide a closer correspondence between the compared fields. It is clear that the values of HX obtained by **extensive** direct extrapolation are much too low, although our overall curve shape is very similar to that obtained by Perkins et al.^b The values deduced by Strnat from the anisotropy constants obtained from H/M vs M^2 plots are higher than those of the present work for (x+y) < 0.2 but are consistent with the low temperature measurements on the present specimens (see Figs. 1 and 3). Another Sm₂Co₁₇ sample furnished by W.E. Wallace, previously measured at room temperature by Rothwarf, showed an HX of 114 kOe as compared to the 88 kOe measured on the Strnat sample.

The salient feature of Fig. 3 is the dramatic increase of anisotropy field for (x+y) < 0.4 resulting from the presence of a single atom of manganese per formula unit, 35-40% at 4.2 K and 45-55% at room temperature. The experimental curves for the anisotropy fields of the ter**nary** and quaternary compounds at 4.2 K are qualitatively similar to each other as well as to the room temperature quaternary curve. All three curves show the same initial steep decline in H_A with increasing replacement of cobalt with iron followed by a region of positive curvature bounded by inflection points at (x+y) = 0.18 and 0.30, and by slow linear decrease in HA thereafter. The quaternary samples with (x+y) of 0.5 and greater were not obtainable as single phase materials, and the results in these regions should not be taken too seriously. It is, however, interesting to note that HA's measured for these materials tend to fall about the same straight line obtained from the measurements on the ternary compounds. This region of linear decline in HA may result from a change from an easy axis to an easy plane regime at (x+y) = 0.5. Such a transformation is known to occur for the ternary system at room temperature, 3 but it is not certain whether such a change occurs at 4.2 K.¹⁵ It is curious that the room temperature curve for the ternary compounds differs qualitatively from that taken at 4.2 K, **although no such difference is manifested by the quaternaries. If,** however, one uses previous measurements of Rothwarf on the Wallace sample of Sm_2Co_{17} as the true value of H_A , this anomalous aspect of the ternary curve disappears and the room temperature curve becomes more like that at 4.2 K. Apparently the behavior of H_A for this compound is very de-

pendent on specimen preparation and/or stoichiometry. Figure 1 shows the values of HA which would have been obtained had our room temperature magnetization curves been extrapolated from H=20 kOe. (See Fig. 2a for an example.) These are compared with the values of other investigators who used such an extrapolation. The resulting excellent agreement, with the work of Perkins et al,⁶ clearly





illustrates the necessity of using applied fields as near to saturation as possible to avoid the substantial errors incurred by long extrapolations.

The saturation magnetization $(4\pi M_s)$ as a function of composition for room and liquid helium temperatures is shown in Fig. 4. The curves for the ternary compounds show the same general shape commonly obtained for 2-17 compounds except that the initial rise in magnetization with increasing iron content becomes much more abrupt in the vicinity of the easy axis-easy plane transformation at x=0.5. This abruptness occurs at both room temperature and at 4.2 K even though the easy plane phase may not exist at liquid helium temperature. The saturation magnetizations of the quaternary compounds are generally somewhat smaller than those of their ternary counterparts, but as can be seen in Fig. 4 there is little difference in the neighborhood of y=0.059, the region of highest anisotropy and therefore of greatest potential for permanent magnet application. The quaternary materials have the additional advantage of showing much less temperature variation of saturation magnetization $4\pi M_s$. For example, for x+y < 0.5, the spread between the ternary curves for 4.2 K and 300 K is approximately triple that of the quaternary curves. The composition dependence of $4\pi M_s$ is also less marked for the quaternaries. Table I compares the present values of $4\pi M_S$ and other parameters with those obtained in previous investigations. Agreement in $4\pi M_s$ is reasonable as for the most part individual measurements fall within ±5% of the mean for the three sets of results. This agreement in $4\pi M_s$ contrasts sharply with the lack of agreement for the HY values which we have already attributed to the long extrapolations used by previous investigators.

DISCUSSION AND CONCLUSIONS

Because the composition ranges of the initial decline in H_A (Δ (x+y) \gtrsim 0.18) and of the following region of positive curvature in H_A (Δ (x+y) \gtrsim 0.12) are respectively equal to the fraction of cobalt sites in the 9d and 6c sublattices, it is tempting to correlate the behavior in these regions with preferential substitution of Mn and/or Fe in these sites in a manner analogous to that in the RE(Co_{1-x}TM_x)₅ systems.9,16 Recent work by Perkins and Fischer,17 however, shows that while preferential substitution does occur in the Y₂(Co_{1-x}Fe_x)₁₇ system tested, such substitution appears only for (x+y) > 0.29 and then the preference shown is statistical rather than absolute. Further, only the 6c sites show this affinity for iron, whereas the simple interpretation conjectured would necessitate an even greater preference for 9d sites as well. It would therefore appear that the actual effect of iron substitution upon HA may be more complex than for the one-five compounds, and further investigations are required to unfold all of the details.

The substitution of a single atom of Co by an atom of Mn raises the anisotropy fields of the series $Sm_2(Co_{1-x}Fe_x)_{17}$ to values consistent with high coercive forces and energy products. If one applies the rule of thumb that the potentially obtainable coercivity, H_c , is the order of one tenth the anisotropy field then one would expect an H_c of approximately 13-16 kOe for a properly prepared magnet of SmMnCo₁₆. Together



Fig. 4. Saturation magnetization $(4TTM_s)$ of $Sm_2Co_{1-x-y}Fe_xMn_y)_{17}$ for y=0 and y=0.059 at room and liquid helium temperatures as a function of fraction of cobalt atoms replaced (x+y).

							-X-1X-	-y x y	17			
		-		4.1	×				300 K			
Compound	ŧ	Density	4mH	8n/n	0.4	RA A	4 mM 8	u/u	HA (20 k0e)	0.4	-	Ref
	1	gm/cm	2	1	kOe	koe	kG		k0e	koe	koe	1
5 Co 17	0.0	8.673	14.0	29.9	119	114	12.0	25.8	68.5	88.7	85.0	M
	1	1	13.7	1	1	1	12.8	27.4	69.3	1	65.0	6.19
	1	1	1	1	1	1	11.8	25.2	70.0	1	1	3,4,5
	1	1	1	29.1	1	1	1	1	:	:	;	20
•	:	1	1	1	1	1	;	1	1	112	1	6
Sm2(Co0.9Fe0.1)17	0.1	8.599	15.0	32.1	113	108	12.8	27.5	72.0	91.0	86.8	M
	1	1	15.2	1	1	1	13.6	29.0	71.5	:	67.0	6,19
	1	1	1	1	1	1	12.8	27.5	74.0	1	:	3.4.5
Sm ₂ (Co _{0,8} ^{Fe} 0,2)17	0.2	8.503	14.7	31.7	90.9	86.1	13.1	28.5	70.0	87.9	83.6	M
	1	:	15.1	1	1	1	14.0	30.1	69.6	1	65.0	6.19
	1	1	1	1	1	1	13.6	29.4	19.0	1	1	3.4.5
Sm2(Co0.7 ^{Fe} 0.3)17	0.3	8.381	15.8	34.4	88.9	83.8	13.9	30.3	59.0	80.8	76.3	M
	1	1	16.1	1	1	1	14.6	32.0	58.7	1	54.0	6119
	1	1	1	1	1	1	14.3	31.5	78.0	1	1	3.4.5
Sm2(Co0.6Fe0.4)17	0.4	8.286	15.4	34.2	85.6	80.5	14.0	30.8	40.0	73.7	69.1	Md
	1	1	16.4	1	1	1	15.2	33.5	39.1	1	34.0	6.19
	1	1	1	1	1	1	14.7	32.3	67.0	:	1	3.4.5
Sm2(Co0.5Fe0.5)17	0.5	8.258	16.2	35.9	81.8	76.5	14.6	32.1	1	63.6	58.8	Md
	:	1	1	:	1	1	15.7	34.6	1	1	1	9
	1	1	1	;	1	1	14.9	32.8	67.0	1	1	3.4.5
		-										

TABLE I. Physical Parameters of Sm2(Co1____Pe_Mn_)

				TA	BLE I	(cont'd)						
				4.2	X				300 K			
Compound	4¥x	Density*	4mM	8h/n	° ⁴	HA A	4TM	n/u	H ⁰ (20 k0e)	0_4	HA A	Ref
		gm/cm	k		k0e	k0e	kç		k0e	k0e	k0e	Ì
Sm2 (Co0.4 Pen.6) 17	0.6	8.136	18.3	40.8	76.8	70.8	17.0	37.9	1	:	1	PW
	1	1	1	1	1	1	15.6	34.6	1	1	1	3,4,5
Sm2(Co0.3Fe0.7)17	0.7	8.050	18.3	41.1	72.2	66.2	16.8	37.6	1	1	:	Md
	1	1	1	1	1	1	16.3	36.6	1	1	:	3,4,5
Sm ₂ (Co _{0.2} Fe _{0.8})17	0.8	7.992	17.9	40.1	68.7	62.8	17.0	38.3	1	1	:	μų
	1		1	,	1	1	15.3	34.5		1	1	3,4,5
Sm2(Co0.1Fe0.9)17	0.9	7.943	1	1	62.6	57.4	16.4	36.9	1	1	:	Md
	1	1	1	1	1	1	14.3	32.2	1	1	1	3.4.5
Sm ₂ Fe ₁₇	1.0	7.899	16.8	37.9	1	1	15.7	35.4	1	1	1	Md
	1	1	1	1	1	1	13.5	30.4	1	1	1	3,4,5
Sm ₂ MnCo ₁₆	0.059	8.628	12.7	27.3	160	156	12.1	25.9	82.0	139	135	Md
	1	1	14.0	30.0	1	1	1	1	1	1	1	18
	;	1	1	. 1	1	1	12.0	1	80.0	:	1	21
	1	1.	1	1	1	1	I.	1	•	150	146	6
Sm ₂ MnCo ₁₄ Fe ₂	0.176	8.521	12.8	27.7	140	136	12.2	26.3	1	123	119	Md
Sm ₂ MnCo ₁₂ Fe ₄	0.294	8.381	13.3	29.0	125	120	12.4	27.0	1	113	109	Md
Sm ₂ MnCo ₁₀ Fe ₆	0.412	8.275	13.6	29.9	87.0	82.0	12.6	27.8	1	80.	75.8	Md
Sm ₂ MnCo ₈ Fe ₈	0.529	8.224	14.2	31.3	76.8	72.0	13.7	30.2	1	:	:	Μd
* Densities are tho PW - Present Work	se of K.	Strnat via	private	commun	lcation	;						

with a remanence of 11-12 kG such a coercivity would result in an energy product of more than 30 MGOe as compared to the 24 MGOe currently attainable for SmCo5.

The saturation magnetization of Sm_2MnCo_{16} shows significantly less temperature dependence than either $SmCo_5$ or Sm_2Co_{17} with a fractional increase of M_S on going from 300 K to 4.2 K of only 5% as compared to the respective values for the latter compounds of 11% and 16%. This result is somewhat surprising in view of the results of Perkins and Strässler¹⁸ who found a higher Curie temperature for Sm_2Co_{17} (1200 K) than for Sm_2MnCo_{16} (1100 K). The foregoing advantages over the binary compounds coupled with increased corrosion resistance and lower cost resulting from a smaller Sm content make the $Sm_2(Mn,Co,Fe)_{17}$ system a very promising one for permanent magnet applications.

The use of extrapolated anisotropy field values in theoretical calculations^{18,19} of magnetic parameters can lead to significant deviations from those obtained by saturation of the material in the hard direction. As an example, Fig. 5 compares the values of the anisotropy constant K1 obtained by derivation from extrapolated values of HA¹⁹ with those calculated from the HA's of the present work. Differences as great as 30% are manifest and, of course, such underestimates would propagate through subsequent calculations such as the crystal field parameter $< r^2 > A_2^0$.¹⁸,20

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Fig. 5. Comparison of room temperature anisotropy constants $\rm K_1 \gtrsim (\rm H_AM_S)/2$ as a function of cobalt atoms replaced as obtained from extrapolated HA values with those derived from directly measured HA. The curves are for the ternary (y=0) compounds.

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