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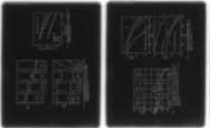
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RESEARCH AND DEVELOPMENT TECHNICAL REPORT

DELET-TR-79-20

**NEW SAMARIUM-2 COBALT-17- BASED COMPOUNDS FOR
PERMANENT MAGNET APPLICATIONS**

F. Rothwarf
H. A. Leupold
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NEW $\text{Sm}_2\text{Co}_{17}$ -BASED COMPOUNDS FOR PERMANENT MAGNET APPLICATIONS

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ABSTRACT

The magnetic properties of several $\text{Sm}_2\text{Co}_{17}$ - based alloy systems potentially useful for microwave/millimeter wave applications are reviewed. Two types of materials are discussed. One contains heavy rare earth substituents for some samarium in an attempt to achieve intrinsically temperature stabilized magnets. The other contains Mn or Cr substituents for some cobalt in the compound $\text{Sm}_2\text{Cu}_{1.6}\text{Zr}_{0.16}\text{Fe}_{3.3}\text{Co}_{12}$ in an effort to enhance the coercivity and to attain a more linear demagnetization curve for this material, which already has an energy product of 30 MGOe.

INTRODUCTION

Recently Rothwarf, Leupold and Jasper¹ noted that the new magnetic circuit designs being considered for microwave and millimeter wave tubes require permanent magnets with high energy products, i.e., $(\text{BH})_{\text{max}} > 30$ MGOe, low reversible temperature coefficients of magnetization, α , and linear demagnetization curves. Some of our early attempts to attain compounds with such properties were recently reported.^{2,3} In the present paper we review our preliminary results on two types of compounds. The first type employs heavy rare earth substituents for some samarium in an attempt to achieve intrinsically temperature compensated 2-17 magnets. This is an approach that was first successfully applied by Benz, Laforce and Martin⁴ to SmCo_5 . The systems being studied are $\text{Sm}_{2-x}\text{R}_x\text{Co}_{17-y}\text{Mn}_y$, where $\text{R} = \text{Gd}$, Er and Dy . We report here on just the $\text{Sm}_{2-x}\text{Gd}_x\text{Co}_{17-y}\text{Mn}_y$ compounds. The other work will be reported elsewhere.⁵ The Mn is being used since we had previously found⁶ that its presence in the quaternary system $\text{Sm}_2\text{Mn}(\text{Co},\text{Fe})_{16}$ significantly enhanced the anisotropy fields H_A over those measured for the corresponding compounds in the ternary system $\text{Sm}_2(\text{Co},\text{Fe})_{17}$. Thus, we decided to investigate the magnetic properties of these systems with the hope that the heavy rare earth substituents would yield low α 's in compounds having their anisotropy fields enhanced by the Mn substituent.

The second type of compound is one developed by Ojima and co-workers⁷⁻⁹ during their recent study of the effects of Nb, V, Ta and Zr substituents in the $\text{Sm}_2(\text{Co},\text{Cu},\text{Fe})_{17}$ quaternary alloys. They determined that the optimal compound was $\text{Sm}_2\text{Cu}_{1.6}\text{Zr}_{0.16}\text{Fe}_{3.3}\text{Co}_{12}$. (We shall abbreviate its formula as FCo_{12}). It had an energy product of 30 MGOe for a particular heat treatment. In our work we have been investigating the effects of Mn/Cr substituents and a different heat treatment on the saturation magnetization, $4\pi M_s$ and anisotropy field H_A . The rationale for using Mn or Cr substituents is based upon our previous study⁶ and the work of Nagel¹⁰ who found that adding these elements to the $\text{Sm}_2\text{Co}_{17}$ and $\text{Sm}_2(\text{Co},\text{Fe})_{17}$ compounds significantly enhances their coercivities and energy products. In the present paper we review our preliminary results on the compounds $\text{FCo}_{12-x}\text{Mn}_x$ and $\text{FCo}_{12-x}\text{Cr}_x$.

EXPERIMENTAL

$\text{Sm}_{2-x}\text{Gd}_x\text{Co}_{17-y}\text{Mn}_y$ samples were made for $x = 0, 0.2, 0.4$ and 0.6 for each value where $y = 0, 1$ and 2 . They were prepared by induction melting of the elements in a water-cooled copper boat under a titanium-gettered argon atmosphere. These alloys were remelted several times to assure homogeneity. The samples were then annealed at 950 C in tantalum foil in quartz tubes under a helium atmosphere for two weeks. They were then rapidly quenched in ice water. X-ray diffraction patterns showed these compounds to be single phase except for the cases $x = 0.6$, where less than 5% of a second phase was indicated. The lattice constants obtained for the $\text{Sm}_2\text{Co}_{17-y}\text{Mn}_y$ compounds were in agreement with those reported by Perkins and Strassler.¹¹ The systematic variation of the lattice constants with x and y will be reported elsewhere.

The $\text{Sm}_2\text{Cu}_{1.6}\text{Zr}_{0.16}\text{Fe}_{3.3}\text{Co}_{12-x}\text{M}_x$ (where $M = \text{Mn}$ or Cr and $x = 0.5, 1$ and 2) samples were prepared by induction melting the appropriate constituents in a boron nitride crucible in an overpressure of 60 psi argon atmosphere using a model MP crystal growing furnace made by Arthur D. Little, Inc. The cast ingots were then heat treated as shown in Table I.

The magnetization measurements (0-15 kOe) were taken on loose powders with a Princeton Applied Research vibrating sample magnetometer over a temperature interval of 300 K to 425 K after an initial one-hour heat treatment at 250 C. The powder for a given sample was contained in a boron nitride (BN) holder having a loosely fitting BN plug. This holder was in turn sealed into a quartz ampule containing a helium atmosphere to prevent oxidation of the powder when magnetization measurements were made at elevated temperatures. The resultant α 's have an uncertainty of $\pm 15\%$.

Cubic samples were produced by mixing powders of the various compositions with an epoxy binder and aligning them parallel to a cube side in a magnetic field while the epoxy hardened. The anisotropy fields were determined by the use of an integrating flux magnetometer described elsewhere.^{6,12} These measurements were taken with fields supplied by a superconducting magnet, in the range 0-100 kOe. Measurements were taken at temperatures of 2.3 K and 273 K. Experimental reproducibility for the magnetization σ is about $\pm 1.5\%$. The reproducibility for H_A when the material is saturated ($H_A < 100$ kOe) is about $\pm 1\%$. The values for the extrapolated H_A 's are $\pm 2.5\%$ for $H_A = 125-150$ kOe, and $\pm 8.0\%$ for $H_A > 150$ kOe.

The cubic, epoxy-matrix samples were also used to determine magnetization σ measurements (0 - 15 kOe) as a function of temperature in the range from 4.2 K to 350 K using the PAR vibrating sample magnetometer. While these samples did not have the initial one hour heat treatment at 250 C given to the powder samples, their σ (T) values at 15 kOe corresponded quite well with those obtained for the powdered samples in the temperature interval $300\text{ K} < T < 375\text{ K}$. Thus, the α values listed in Table II and plotted in Fig. 2 are for the cubic, epoxy-matrix samples. One discrepancy did show up in the case of the $\text{Sm}_2\text{Co}_{17}$ cubic sample for which α_H was found to be -0.011 \%/K as compared to the value of -0.060 \%/K for the previously reported² heat-treated powder sample. We have not yet had time to resolve this discrepancy.

RESULTS AND DISCUSSION

A. The $\text{Sm}_{2-x}\text{Gd}_x\text{Co}_{17-y}\text{Mn}_y$ System

Some of our preliminary magnetization results are shown in Fig. 1 for the $x = 0$ and $x = 0.4$ samples. From these types of curves an average reversible temperature coefficient of magnetization was obtained, where α is defined as

$$\alpha = \frac{\Delta M}{M_1 \Delta T} = \frac{M(T_2) - M(T_1)}{M(T_1) (T_2 - T_1)} \times 100\% \quad (1)$$

We chose to list α 's for a high temperature region, α_H , where $300 \text{ K} < T < 425 \text{ K}$ and for a low temperature region, α_L , where $225 \text{ K} < T < 300 \text{ K}$. In each case T_1 was taken to be 300 K. ΔM was always chosen as the maximum magnetization change over the temperature interval ΔT .

In Fig. 1A the $4\pi M_s(T)$ curves for $x = 0$ and $y = 0, 1, 2$ are presented, while in Fig. 1B the curves for $x = 0.4$ and $y = 0, 1, 2$ are shown. It is clear that the substitution of $\text{Gd}_{0.4}$ dramatically reduces the slopes of corresponding curves for $y = 0, 1, 2$, thus demonstrating the validity of using the heavy rare earth substituents to attain a lower α in the 2-17 based compounds.

The α 's for all the materials investigated are shown in Table II together with room temperature (300 K) saturation magnetization values σ_s and the anisotropy fields at 273 K for the various compounds. Approximate $4\pi M_s$ values are also tabulated. These were obtained by assuming an average density of 8.6 g/cm^3 for all of the compounds, since our preliminary x-ray diffraction results indicate that the densities of all the compounds are the same to within a few percent. The α_L and α_H values are plotted in Fig. 2 as a function of Gd concentration for the various fixed values of Mn substituent. Making use of Lemaire's magnetization data for $\text{Gd}_2\text{Co}_{17}$ ¹³ and our data for $\text{Sm}_2\text{Co}_{17}$, we have plotted the linear variation of α with Gd concentration expected from the additivity approximation¹ for the $y = 0$ case. Within experimental error this approximation seems to have some validity. However, the shapes of the $y \neq 0$ curves which are similar to the $y = 0$ case lead one to believe that the functional dependence of α with Gd concentration may be more complex. From a practical standpoint it seems that the compound $\text{Sm}_{1.6}\text{Gd}_{0.4}\text{Co}_{16}\text{Mn}$ would furnish the best intrinsically compensated magnet material for the temperature range of military interest, $225 \text{ K} < T < 425 \text{ K}$.

The final choice of an optimal alloy to achieve a zero temperature coefficient (ZTC) material is influenced by the variation of magnetization and anisotropy, as well as ultimately the coercivity and maximum energy product, with Mn and Gd concentration. In Fig. 3 we plot the variation of $4\pi M_s$, and H_A with Mn concentration for the different Gd concentrations for temperatures of 2.3 and 273 K. From Fig. 3A it is clear that there is a monotonic decrease in $4\pi M_s$ as the Gd substituent is increased. This behavior is what one expects from the antiferromagnetic coupling of Gd to the other moments in the system. For a given x , however, a shallow maximum occurs in the 273 K or 300 K curves for the substitution of one Mn atom for

cobalt. A similar monotonic decrease is apparent from Fig. 3B for the variation of H_A with Gd concentration and with the exception of $x = 0.6$ a maximum also occurs for the case of one Mn atom substituted for cobalt. The microscopic rationale for these trends are presently not well understood. However, these considerations show that the achievement of a ZTC material with $x = 0.4$ and $y = 1$, will most probably be attained at the cost of a lower remanence, energy product and coercivity than would be possible with the substitution of less Gd.

B. The $\text{Sm}_2\text{Cu}_{1.6}\text{Zr}_{0.16}\text{Fe}_{3.3}\text{Co}_{12-x}\text{M}_x$ Compounds

The effect of a heat treatment other than the one employed by TDK was studied for the TDK optimal compound FCo_{12} . The temperature dependence of magnetization for three heat treatments is shown in Fig. 4. As shown in Table III, the heat treatment applied at the Electronics Technology and Devices Laboratory (ET&DL) improves the temperature coefficient by a factor of about two. Microprobe studies³ show that a significant variation in the size and the composition of the principal and the grain boundary phases occurs with the two different heat treatments. Preliminary results in our laboratory show that another modification of the TDK anneal yielded a still further improvement of α_H by a factor of two giving $\alpha_H = -0.011 \text{ \%}/\text{K}$. This modification involved changing the first three steps of the TDK anneal to the following: Step 1, 1150 C for 1.5 hour; Step 2, the same; and Step 3, 940 C for 2 hours. More attention to further optimization of the heat treatment procedures might be important, since significant changes in α_H have been noted. However, we caution that no actual magnets have been synthesized using these new heat treatments and their influence on coercivity and $(\text{BH})_{\text{max}}$ remain to be determined.

A major objective of our work involved the substitution of Mn or Cr in place of Co in hopes of enhancing H_A . These results at 273 K are presented in Table III, and are plotted in Fig. 5. The values of H_A at 273 K show a monotonic increase with Mn substitution from 88 kOe for $x = 0$ to 112 kOe for $x = 2$, an increase of 27%. The same increase is obtained for Cr but at the lower concentration of $x = 0.5$. Such results encourage speculation on the possibility of an increase in the intrinsic coercivity; H_C and in turn the possible extension of the linear reversible portion of the demagnetization characteristic when actual magnets are made from these materials.

The temperature coefficients for the Mn and Cr substituted materials are also listed in Table III where the results reflect primarily the TDK anneals. Fig. 6 gives the $4\pi M_s$ temperature dependence of these TDK annealed materials in the temperature interval $4.2 \text{ K} < T < 400 \text{ K}$. It is of interest to note that the alloy with two Cr atoms substituted had the fairly low α_H of $-0.022 \text{ \%}/\text{K}$. It is possible that our ET&DL anneals, when applied to these materials, may yield the lower α 's noted for the basic compound. It would also be of interest to see if the use of the heavy rare earth substituents in these compounds would also produce ZTC materials.

We have previously noted³ Livingston's observations¹⁴ that coercivity and $(\text{BH})_{\text{max}}$ of rare earth-cobalt magnets are very dependent upon their microstructure, since second-phases can lower coercivity by nucleating reverse domains or raise coercivity with domain wall pinning.¹⁵ The TDK compound FCo_{12} shows a fine cellular structure⁹ which may be responsible for the domain wall pinning that controls its coercivity. Our results show that

the microstructure of this material is very sensitive to heat treatment. For many dynamic applications the TDK material is unsatisfactory because of its nonlinear demagnetization characteristic. Thus, it is important to improve this material by enhancing its coercivity. Nagel¹⁰ has demonstrated that the addition of Mn or Cr enhances the anisotropy fields and coercivities of nucleation-controlled 2-17 magnets but has not correlated that result with microstructure studies. His work, in part, motivated our current attempt to enhance H_A in the TDK compound. However, it is not certain that the 27% enhancement of H_A that we have noted as a result of using Mn or Cr substituents in $FeCo_{12}$ will indeed lead to the higher coercivities desired in these domain wall pinning materials. We are currently planning to fabricate actual bulk permanent magnets from these new compounds to answer this question.

TABLE I

Heat treatments for the $Sm_2Cu_{1.6}Zr_{0.16}Fe_{3.3}Co_{12-x}M_x$ compounds.

<u>TDK Anneal</u>			<u>ET&DL Anneal</u>		
Step	Temp. (C)	Time (hours)	Step	Temp. (C)	Time (hours)
1.	1200	2	1.	Same	
2.	Quenched in ice water		2.	Same	
3.	850	2	3.	Same	
4.	700	1	4.	Same	
5.	600	1	5.	Same	
6.	500	2	6.	Same	
7.	400	10	7.	400	4
			8.	280	12

TABLE II

Saturation magnetizations, anisotropy fields and average temperature coefficients for the system $\text{Sm}_{2-x}\text{Gd}_x\text{Co}_{17-y}\text{Mn}_y$

Composition		σ_s	$4\pi M_s$	H_A	α_L	α_H
		300K	300K	273K	225-300K	300-425K
y	x	(emu/g)	(kG)	(kOe)	(%/K)	(%/K)
0,	0.0	117	12.7	85.3	-0.010	-0.011
	0.2	107	11.6	82.1	-0.010	-0.010
	0.4	96.2	10.5	78.7	0.000	+0.015
	0.6	97.3	10.6	66.5	0.000	+0.015
	2.0	67.0*	7.50*	--	+0.069*	+0.074*
1,	0.0	118	12.7	109	-0.021	-0.027
	0.2	110	11.9	98.5	-0.019	-0.017
	0.4	105	11.4	88.2	0.000	-0.006
	0.6	101	10.9	85.0	-0.002	-0.019
2,	0.0	114	12.3	90.0	-0.032	-0.038
	0.2	106	11.4	92.7	-0.022	-0.021
	0.4	91.0	9.82	86.7	-0.020	-0.019
	0.6	96.3	10.4	109	-0.029	-0.029

* R. Lemaire, Cobalt 33, 301 (1966)

TABLE III

Magnetic parameters of the compounds $\text{Sm}_2\text{Cu}_{1.6}\text{Zr}_{0.16}\text{Fe}_{3.3}\text{Co}_{12-x}\text{M}_x$
(where M = Mn, Cr)

Substituent	Type Anneal	σ_s	$4\pi M_s$		H_A	α_L	α_H
			300K	300K			
		emu/g	kG		%/K	%/K	
None	0	None	104	10.6	--	--	-0.036
		TDK	109	11.1	88	-0.013	-0.040
		ET&DL	108	11.0	--	--	-0.022
Mn	1.0	TDK	108	11.0	102	-0.034	-0.058
	2.0	TDK	104	10.2	112	-0.064	-0.087
Cr	0.5	TDK	103	10.5	112	-0.030	-0.035
	1.0	TDK	87.8	8.95	107	-0.021	-0.022

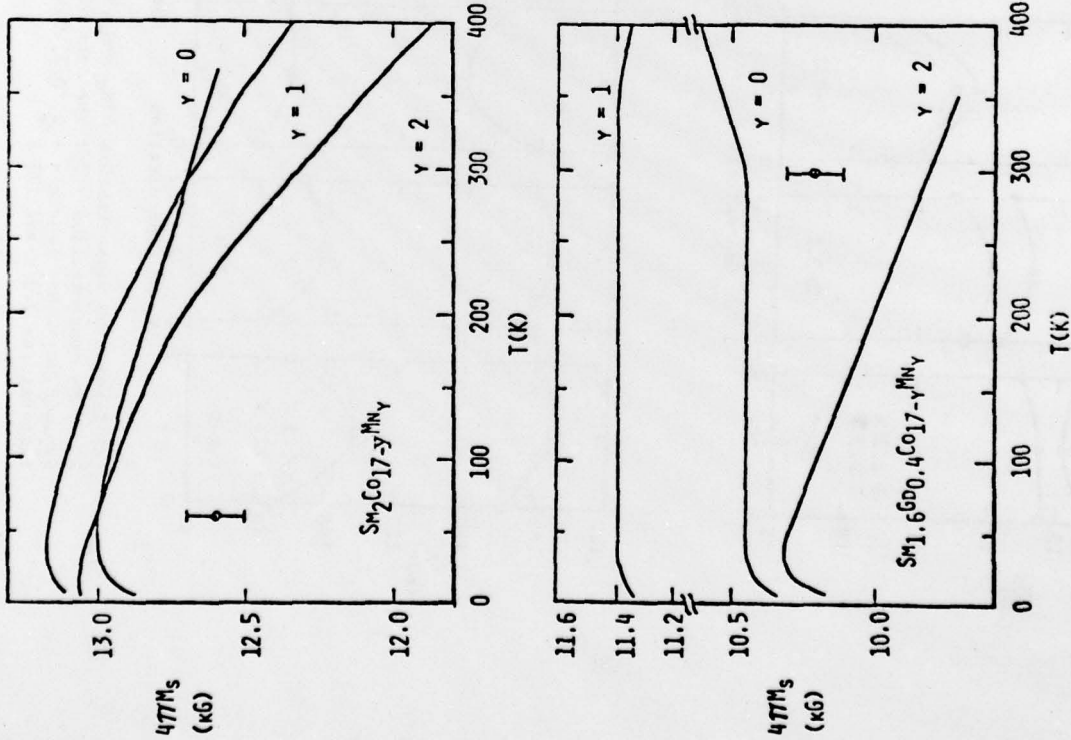


Fig. 1. Saturation magnetization $4\pi M_s$ vs temperature for the $Sm_2Co_{17-y}Mn_y$ and $Sm_{1.6}Gd_{0.4}Co_{17-y}Mn_y$ systems.

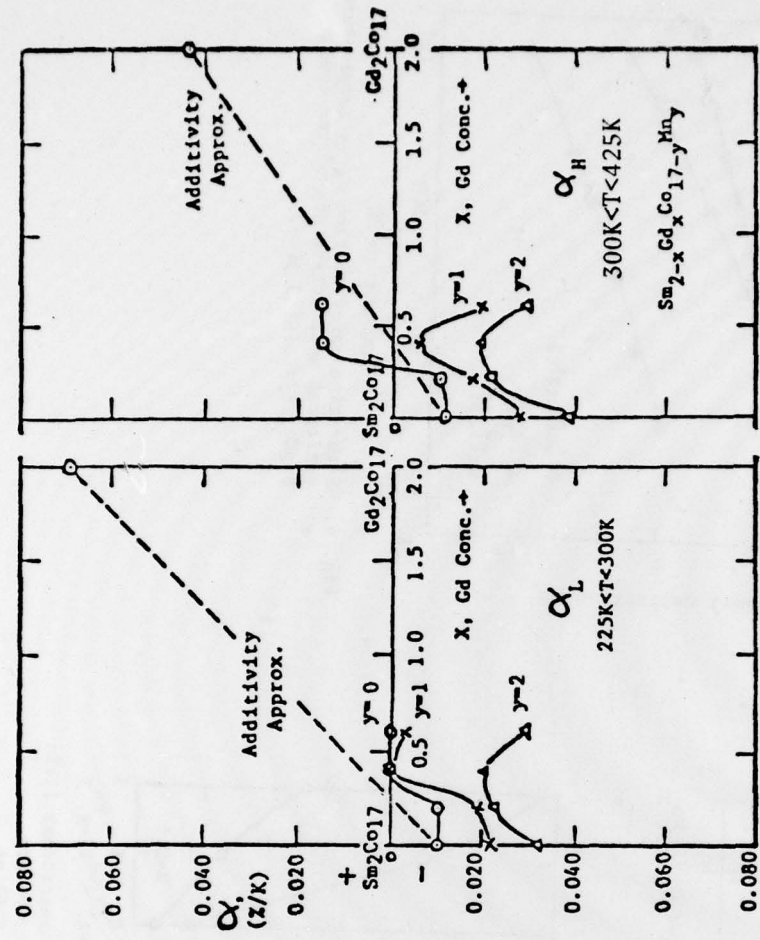


Fig. 2. Reversible temperature coefficients α_L and α_H vs Gd concentration x with Mn concentration y as a parameter ($y=0, 1, 2$).

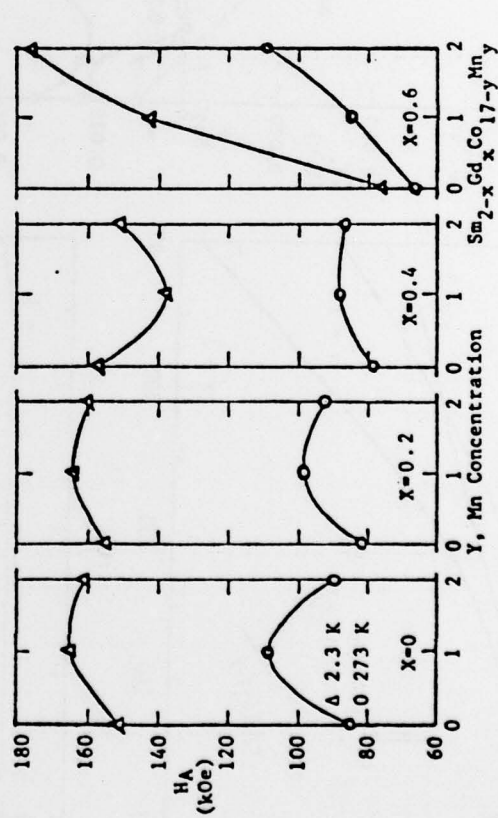
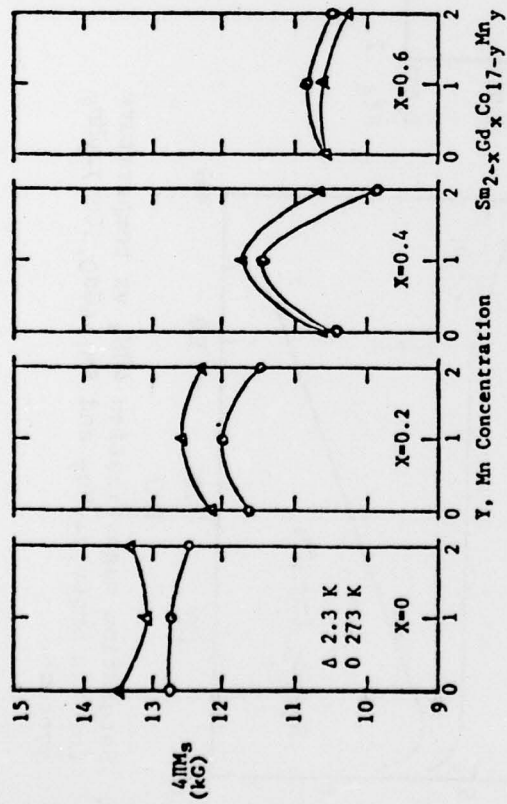


Fig. 3. (A) Saturation magnetization $4\pi M_s$ and (B) anisotropy field H_A vs Mn concentration y for the sets of compounds $Sm_{2-x}Gd_xCo_{17-y}Mn_y$ (where $x = 0, 0.2, 0.4, 0.6$) at temperatures 2.3 K and 273 K.

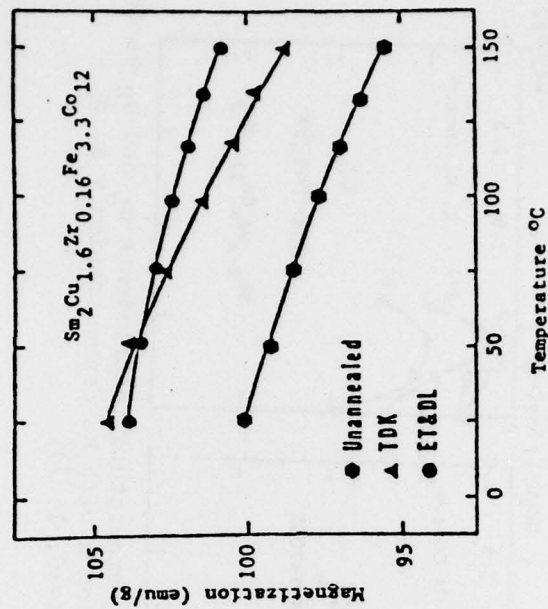


Fig. 4. Saturation magnetization vs temperature for different heat treatments of the compound $Sm_{2}Cu_{1.6}Zr_{0.16}Fe_{3.3}Co_{12}$.

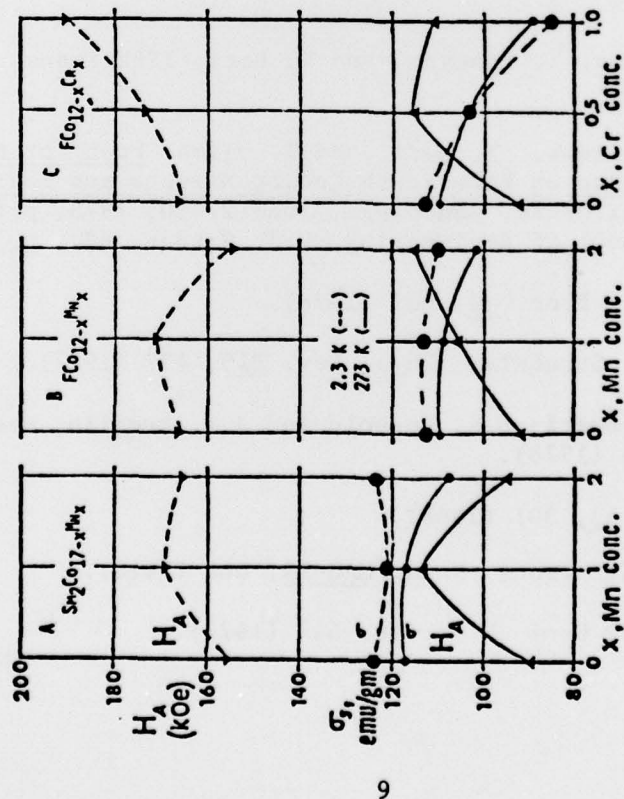


Fig. 5. Anisotropy field and saturation magnetization σ_s vs concentration Mn or Cr in the systems (A) $\text{Sm}_2\text{Co}_{17-x}\text{Mn}_x$, (B) $\text{FCo}_{12-x}\text{Mn}_x$ and (C) $\text{FCo}_{12-x}\text{Cr}_x$ at temperatures of 2.3 K (---) and 273 K (—).

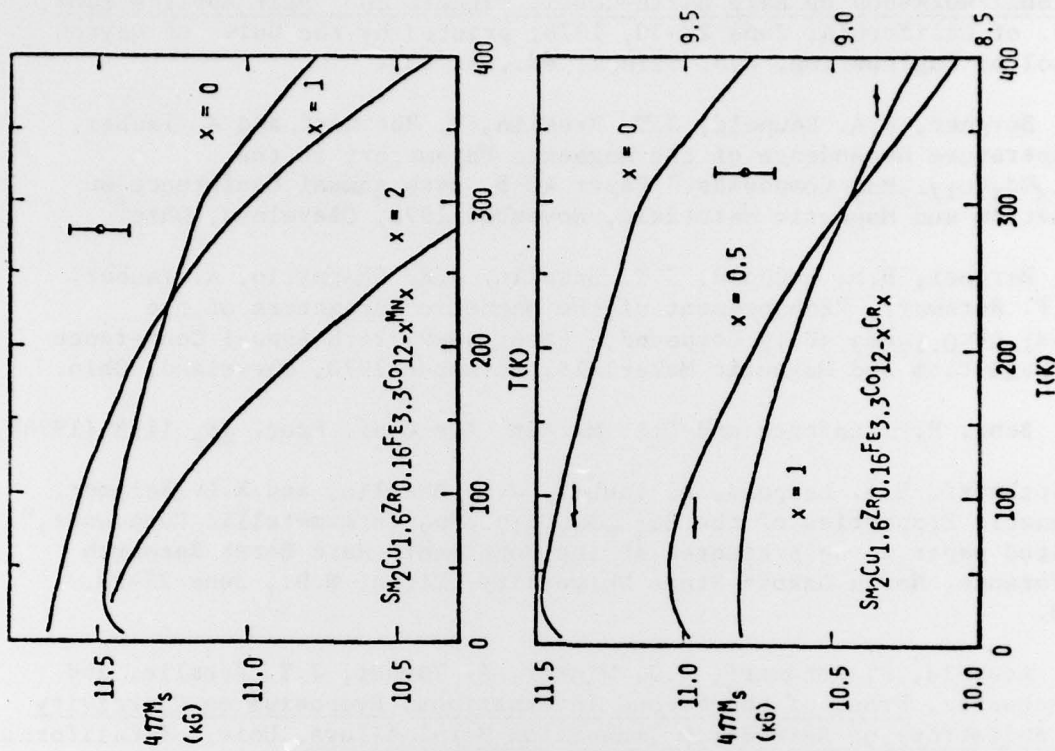


Fig. 6. Saturation magnetization, $4TM_s$ vs temperature for the $\text{FCo}_{12-x}\text{Mn}_x$ and $\text{FCo}_{12-x}\text{Cr}_x$ systems.

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