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MASTER

MAGNETIC PROPERTIES OF SOME INTRA-RARE EARTH ALLOYS*

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Abstract - Paramagnetic susceptibilities of samarium structure alloys in the systems La-Gd, Nd-Y, Nd-Tm, and Ce-Y were measured from 4.2 - 300°K to determine what effect the formation of this structure had on the magnetic properties of the alloys. Susceptibility maxima were observed in the Nd-Y and Nd-Tm alloys, at 34.5 and 28.5°K, respectively. Slope changes were observed in the curve of $1/\chi$ versus T for the La-Gd alloy at 155 and 130°K, and the susceptibility of this alloy remained temperature-independent from 130-7°K. A rather poorly-defined region of constant susceptibility was also observed for the Ce-Y alloy, from 26-37°K. Above these transition temperatures, the alloys obeyed the Curie-Weiss law, the susceptibilities of the La-Gd and Ce-Y samples indicating the possibility of electron transfer occurring in these cases.

INTRODUCTION

It has long been known that the rare-earth series of elements is built up through the addition of electrons to the 4f subshell. Since this

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is an "inner" shell, shielded from external influences by s and p electrons, the magnetic properties arising from the unpaired 4f electrons have been of interest to a number of workers. The first thorough study was carried out by Klemm and Bommer,⁽¹⁾ and a great many investigations have followed, especially at low temperatures. Recently, the temperature range of such investigations has been extended to 1500°K by Aarås and Colvin.⁽²⁾ The experimental results have shown that, with the exceptions of Sm, Eu, and Yb, the rare earths can be depicted as consisting of non-interacting tripositive ions, their paramagnetism arising from the electrons in the 4f subshell, which afford values of the paramagnetic susceptibility in accordance with the theories of Hund and Van Vleck. The magnetic data also indicate that the elements Eu (above 100°K) and Yb are divalent in the metallic state, i. e., these metals have assumed the more stable $4f^7$ and $4f^{14}$ electronic configurations. In addition, for the elements Sm and Eu, and to a lesser extent in Pr and Nd, it has been found that higher J levels can become populated and contribute to the paramagnetic susceptibility. Paramagnetic-antiferromagnetic transitions have been observed at very low temperatures for Ce, La, and Sm by Lock⁽³⁾ and in Eu by Bozorth and Van Vleck,⁽⁴⁾ while Gd undergoes a paramagnetic-ferromagnetic transition at 291°K.⁽⁵⁾ The rare earths Tb through Tm exhibit paramagnetic-antiferromagnetic-ferromagnetic transitions on cooling, as observed by a number of workers.⁽⁶⁻⁹⁾

The fact that ferromagnetism is exhibited by some of the rare earths and not others, even though unpaired 4f electrons are present in the metals Ce through Tm, is probably related to the crystal structure differences which exist among the members of the series, as suggested by Gschneidner

and Matthias.⁽¹⁰⁾ The so-called "heavy" rare earths, Sc, Y, and Gd through Lu are hexagonal metals, with c/a ratios of approximately 1.57, while the "light" members, La through Nd, exhibit h. c. p. structures with a double c -axis, leading to c/a ratios of about 3.1. Cerium (above 263°K) and Yb are f. c. c., while Eu is b. c. c., and Sm is rhombohedral. Thus, since the normal hexagonal metals become ferromagnetic, while those with a double c -axis do not, a relationship appears to exist between crystal structures and magnetic properties. It becomes of interest then, first to examine the alloying behavior of a light rare earth with a heavy, and secondly, to investigate the magnetic properties of such alloys. Valletta et al.⁽¹¹⁾ have shown, in the systems Ce-Y, Pr-Y, Nd-Y, La-Y, La-Gd, and Nd-Tm, the existence of a phase, usually occurring at compositions close to 50 atomic per cent, which has the rather remarkable characteristic of displaying the crystal structure of Sm, with approximately the same lattice parameters. It thus appears that the unusual crystal structure exhibited by Sm is actually a sort of transition structure between the light and heavy rare earths, since the atomic number of Sm places it at about the midpoint of the rare-earth series.

In this work, the magnetic properties of four of these samarium structure alloys (hereafter referred to as δ -phase alloys) have been investigated, in order to determine what effect, if any, the formation of this structure has on such properties. It will be noted that, in the La-Gd alloy system, the light rare earth is diamagnetic and the heavy, paramagnetic; in the Nd-Y system, the reverse is true, while in the Nd-Tm alloy, both constituents are paramagnetic. Thus all three different types of those δ phases which afford a measurable paramagnetic

susceptibility have been investigated. The Ce-Y alloy was examined because of the low temperature allotropy of cerium; Gschneidner and Smoluchowski⁽¹²⁾ have found that the low temperature form of this element has a valence of 3.62 at 116°K, indicating that a transition of the type $Ce^{+3} \rightarrow Ce^{+4}$ occurs on cooling. Thus, if any valence change took place in the cerium contained in the Ce-Y alloy, it would be apparent from the susceptibility data.

EXPERIMENTAL

Two different methods for the measurement of the paramagnetic susceptibility were employed, the Gouy technique from 77-300°K, and a mutual inductance bridge technique from 4.2-ca. 90°K. The apparatus used in the former method is shown in Fig. 1, the sample being suspended from a Mettler B-5 single-pan analytical balance, while the mutual inductance bridge used was constructed and described by Jennings⁽¹³⁾ and Gerstein.⁽¹⁴⁾ The electromagnet and current controller employed in the Gouy measurements were constructed in this laboratory, and the 20 amperes and 200 volts required by the magnet at maximum field were supplied by a direct current generator. The fields employed in these measurements were of the order of 3000 oersteds, and were measured by an NMR technique. Three copper-constantan thermocouples were used to determine the temperature of the Gouy sample, and the heater was wound non-inductively with copper-beryllium wire. A room temperature measurement was obtained by weighing the sample in and out of the field, and then the Dewar was filled with liquid nitrogen. Another weighing was carried out at 77°K, after which the vacuum chamber was pumped down

Fig. 1. Dewar assembly for the Gouy apparatus.

to about 1×10^{-5} mm Hg in order to insulate the sample from the liquid nitrogen; by means of the heater, the sample could then be warmed to 300°K.

Over the temperature range 4.2-90°K, the susceptibilities were determined by measuring the mutual inductance of a coaxial coil system when the sample was positioned at the center of the coils, and again when the sample was removed from the coils. By suitable calibration with a paramagnetic salt, the difference in mutual inductance could be related directly to the susceptibility of the sample. In these measurements, the field experienced by the sample was approximately two oersteds. The spherical sample was contained in a bakelite sample holder which was surrounded by a heater shell of heavy paper, on which was wound, non-inductively, copper-beryllium wire. The temperature was determined with a gold-cobalt versus copper thermocouple, placed in contact with the sample. The heater shell containing the sample was placed at the bottom of a long quartz tube, which could be evacuated and filled with helium exchange gas. The tube was joined to a double dewar assembly via an "O"-ring slip seal which enabled the tube to be moved vertically inside another tube on which the coils were wound, thus moving the sample in and out of the coils. With exchange gas in the system, the sample was allowed to equilibrate at 4.2°K, after which the exchange gas was pumped out, and electrical heating employed to warm the sample.

The alloys were prepared by arc-melting the constituent metals together in an argon atmosphere. The samples were remelted several times to ensure homogeneity, and finally melted down into a mold about 5 in. long by 3/8 in. wide, resulting in rod of elliptical cross section.

The arc-melted products were then cold-swaged until cylindrical rods were obtained, at which point annealing was usually required before further reduction in cross section could be effected. After annealing in vacuo for about 8 hr at 550°C, swaging was continued until rods of the desired size were obtained. For the alloys of high moment, i. e., La-Gd and Nd-Tm, the rods used were 0.25 cm in diameter, and approximately 15 cm in length; for those of lower moment, a diameter of 0.5 cm was employed, over the same length, in order to obtain minimum weight changes of 0.1 g. in the Gouy measurements. Because of the high vapor pressure of thulium, there was danger that some of this metal would be lost during the arc-melting procedure, so it was necessary to prepare the Nd-Tm alloy by casting the constituent metals together in a tantalum crucible which was sealed under a partial pressure of helium gas. The casting was carried out in a high-vacuum system, using r-f induction heating, the alloy being held molten, at ca. 1400°C, for approximately 1 hr, then allowed to cool slowly through the melting point to prevent the formation of voids. After radiographic examination showed the sample to be free from holes, the tantalum was turned off on a lathe, and the swaging operation outlined above was carried out on the alloy. All of the alloys were annealed after swaging for approximately 2 hr at 550°C to remove the effects of cold working. The short annealing time was employed in order to avoid producing appreciable grain growth in the samples. Swaging tends to break up the large crystals formed during melting, and together with the recrystallization during annealing, reduced the risk of preferred orientation effects showing up in the magnetic measurements.

Spherical samples were used for the low temperature measurements, since at such temperatures demagnetization effects may be important, and the correction factor for samples of other than spherical geometry is difficult to determine. A spherical section could be obtained on one end of the rod by using a lathe tool whose cutting edge was in the form of a hemisphere; this produced practically a complete sphere, with only a thin connection remaining between the sphere and the rod. This connection was severed with a jeweler's saw, and the sphere then hand-finished with 300 grit abrasive paper.

The alloys were analyzed spectrographically after preparation from the constituent metals, and the upper limits of metallic impurities present are shown in Table 1.

Table 1. Spectrographic analyses of the alloys investigated
(concentrations given in parts per million)

Element	La - Gd	Nd - Tm	Nd - Y	Ce - Y
Ca	< 50	<200	< 50	< 50
Cr	<200	<200	<200	----
Cu	<100	<500	<100	<100
Dy	----	<100	----	----
Er	----	<500	----	----
Fe	500	<500	<500	<200
Mn	<200	<200	<200	----
Nd	----	----	----	<200
Si	<200	<200	200	< 50
Ta	----	<800	----	<800

In addition, the alloys were analyzed for Ce, Co, Cu, Gd, Ho, La, Lu, Ni, Pr, Sc, Sm, Tb, Ti, Tm, Y, and Yb, none of which were present in detectable amounts.

RESULTS

Samples for X-ray analysis were taken from both ends and the center of each rod in order to confirm the homogeneity of the alloys. For annealing purposes, the filings were sealed under helium in a small container made from 1/8 in. tantalum tubing which had previously been outgassed by heating in vacuo at an elevated temperature (ca. 1500°C). The container was then placed inside a quartz tube, and the tube sealed off while a vacuum was being pumped on it. The tube was then placed in an annealing furnace, usually at about 500°C for 8 hr. After annealing, the filings were placed in Pyrex capillaries, and the X-ray diffraction patterns obtained with a Debye-Scherrer powder camera, using Ni-filtered copper radiation. While the La-Gd and the Nd-Tm alloys exhibited the samarium structure at the same compositions as reported by Valletta et al., it was found that the Nd-Y and Ce-Y alloys did not. This discrepancy can possibly be traced to the different methods of preparation employed, Valletta et al. having allowed the molten alloys to equilibrate for periods of about 1 hr in a tantalum crucible. Since yttrium picks up tantalum more readily than any of the other rare earths except scandium, it is possible that tantalum contamination of the yttrium-containing alloys stabilized the δ phase at different compositions. Table 2 compares the compositions and lattice parameters obtained in this study with those of Valletta et al.

Table 2. Comparison between compositions and lattice parameters of the δ -phase alloys obtained in the present study with those of Valletta et al. (11)

<u>Composition, atomic per cent</u>		<u>Lattice parameters, in Å</u>	
Valletta	this study	Valletta	this study
44.5 Ce-55.5 Y	55.0 Ce-45.0 Y	a= 3.653 c=26.55	a= 3.648 c=26.35
53.0 Nd-47.0 Y	70.0 Nd-30.0 Y	a= 3.665 c=26.45	a= 3.644 c=26.28
30.1 La-69.9 Gd	30.1 La-69.9 Gd	a= 3.667 c=26.482	a= 3.667 c=26.44
63.0 Nd-37.0 Tm	63.0 Nd-37.0 Tm	a= 3.656 c=26.36	a= 3.628 c=26.13

The results of the magnetic investigations are shown in Figs. 2-5, in which the inverse of the paramagnetic susceptibility per gram of alloy (corrected for the diamagnetism of the core electrons) is plotted versus the absolute temperature. The dashed lines in the figures represent the theoretical values for a system of non-interacting tripositive ions, obtained from the Van Vleck equation:

$$\chi_g = \left(\frac{N}{m}\right) \frac{g^2 \beta^2 J(J+1)}{3kT}$$

Field dependency measurements carried out on all of the alloys showed that the susceptibilities remained constant as the field strength was varied.

Fig. 2. Inverse paramagnetic susceptibility versus temperature curve for La-Gd δ -phase alloy, from 4.2-300°K.

Fig. 3. Inverse paramagnetic susceptibility versus temperature curve for Nd-Y δ -phase alloy, from 4.2-300°K.

Fig. 4. Inverse paramagnetic susceptibility versus temperature curve for Nd-Tm δ -phase alloy, from 4.2-300°K.

Fig. 5. Inverse paramagnetic susceptibility versus temperature curve for Ce-Y δ -phase alloy, from 4.2-300°K.

DISCUSSION

The pertinent features of the magnetic behavior of the alloys will be discussed as follows:

1. **La-Gd:** A Curie-Weiss law was obeyed over the temperature range 300-155°K, with $\theta_p = +10^\circ$. The susceptibility in this region was markedly higher than that expected from theory, and the effective moment of the gadolinium in the alloy was calculated from the measured susceptibility to be 9.4 Bohr magnetons, whereas the theoretical moment for the Gd^{+3} ion is 7.94. From 130-7°K, the susceptibility remained roughly constant. The high susceptibility values obtained for this alloy are quite difficult to interpret. Thoburn et al. ⁽¹⁵⁾ found, for a La-Gd alloy containing 75 atomic per cent Gd, that $\mu_{eff}(Gd) = 8.38$ Bohr magnetons, and attributed the increased moment to either orbital contributions or transfers from the conduction band electrons. These authors reported that the crystal structure of this alloy was unknown, but since a weak antiferromagnetic maximum was observed at 130°K, in good agreement with the present work, the sample was probably a δ -phase or a two-phase ($\delta + \alpha$ - Gd) alloy. An interesting observation can be made, if one assumes that a conduction electron could somehow be transferred into the 4f shell of gadolinium, resulting in a 7F_6 , or Tb^{+3} electronic configuration. Comparison between the observed susceptibility values from the Gouy measurements and those which would be predicted from theory for such a mechanism shows good agreement, as can be seen in Table 3.:

Table 3. Comparison between observed susceptibilities of the La-Gd δ -phase alloy with those for the Tb^{+3} ion

T, °K	$\chi_g \times 10^6$ (theo. for 69.9 at. % Tb^{+3})	$\chi_g \times 10^6$ (obs.)
300.4	179.4	166.8
273.2	197.3	194.1
251.6	214.2	210.2
225.2	239.3	234.6
199.1	270.7	265.0
173.4	310.8	313.7

Moreover, it should be pointed out that the theoretical value for the effective moment of the Tb^{+3} ion is 9.72 Bohr magnetons.

2. Ce-Y: This alloy followed a Curie-Weiss relationship, from 300- ca. 50°K, with $\theta_p = -57^\circ$. The observed susceptibility of the alloy was lower than the theoretical susceptibility, while the effective moment of the cerium ($2.68 \mu_B$) was slightly higher than the theoretical value ($2.56 \mu_B$). A rather poorly defined region of constant susceptibility was observed from 26-37°K. Gschneidner⁽¹⁶⁾ reports that the valence for γ -Ce, the room-temperature form, is 3.1, while that of α -Ce, the lowest temperature form, is 3.7. In addition, McHargue et al.⁽¹⁷⁾ have found that only α -Ce exists at 4.2 °K. If one assumes that the $Ce^{+3} - Ce^{+4}$ transition on cooling is the cause of the susceptibility decrease, one can calculate the percentage of Ce^{+3} present in the alloy at various temperatures from the susceptibility data obtained. The results are shown in Table 4.

Table 4. The extent of the Ce^{+3} - Ce^{+4} transition in the
Ce-Y δ -phase alloy

T, °K	$\chi_g \times 10^6$ (theo. for Ce^{+3})	$\chi_g \times 10^6$ (obs.)	% Ce^{+3}	Valence
300	13.25	11.81	88.5	3.12
250	15.86	13.81	87.1	3.13
200	19.69	16.71	84.9	3.15
150	26.03	20.49	78.7	3.21
100	38.70	27.41	70.8	3.29

3. Nd-Y: Good agreement was obtained for this alloy between the observed and theoretical values of the susceptibility from 300-ca. 40°K, with $\theta_p = -24^\circ$, although the effective moment of the neodymium in the alloy ($3.82 \mu_B$) was slightly larger than the theoretical value ($3.68 \mu_B$). A susceptibility maximum was observed for this alloy, at 34.5°K.
4. Nd-Tm: Again in this case, fair agreement with theory was obtained. A Curie-Weiss law was obeyed from 300-ca. 30°K, with $\theta_p = -8^\circ$, and the susceptibility was consistently about 7 per cent lower than the theoretical values. The effective moments for this alloy were calculated assuming the Weiss constants for the pure elements, since both alloy constituents are paramagnetic, and the results showed $\mu_{\text{eff}}(\text{Nd}) = 3.42 \mu_B$, $\mu_{\text{eff}}(\text{Tm}) = 6.82 \mu_B$. The theoretical values are 3.68 and 7.56 μ_B , respectively. A susceptibility maximum was also observed in this alloy, at 28.5°K.

The theory of antiferromagnetism as developed by Neel⁽¹⁸⁾ and Van Vleck⁽¹⁹⁾ provides the means for an interpretation of the magnetic transitions observed in these alloys. There exists a general tendency in an antiferromagnetic substance for the atomic moments to line up in an antiparallel array, perpendicular to the direction of the applied field. Such an alignment gives rise to a temperature-independent susceptibility, since the moments are not free to move with respect to each other. This tendency is opposed by the anisotropic crystalline field, which tends to align the moments in a preferred direction in the crystal. When the applied field is parallel to such a direction, the susceptibility should fall as the temperature is lowered, tending to zero at 0°K. In a polycrystalline sample, however, one must assume that this internal field is randomly oriented. Thus it is perpendicular to the applied field 2/3 of the time and parallel to the applied field 1/3 of the time. It is the combination of these two aligning tendencies which gives rise to the maximum in susceptibility normally associated with antiferromagnetism.

As a corollary to this theory, one can consider the behavior of a polycrystalline sample of an antiferromagnetic substance as follows: at temperature close to 0°K, assuming a random distribution of crystal axes, there should be an average susceptibility, given by

$$\bar{\chi}_0 = \frac{2}{3} \chi_{\perp,0} + \frac{1}{3} \chi_{\parallel,0} = \frac{2}{3} \chi_{\perp,0} = \frac{2}{3} \chi_{T_N}$$

since $\chi_{\parallel,0} = 0$. Thus the ratio of the average susceptibility at $T = 0$ to the susceptibility at $T = T_N$ should be 2/3. Applying the theory to the

experimental results of this investigation, we find that maxima occur in the susceptibilities of the Nd-Y and Nd-Tm alloys, suggesting that appreciable anisotropic crystalline fields exist, and that the susceptibility ratios are

$$\frac{\chi_0}{\chi_{T_N}} = \frac{1430 \times 10^{-6}}{2070 \times 10^{-6}} = 0.69 \text{ for Nd-Y}$$

$$= \frac{1540 \times 10^{-6}}{2490 \times 10^{-6}} = 0.62 \text{ for Nd-Tm}$$

which are in quite good agreement with the prediction above. The existence of a temperature-independent susceptibility was exhibited by the La-Gd sample, and to a lesser extent by the Ce-Y alloy. Since these alloys are of the same crystal structure as those of Nd-Y and Nd-Tm, the internal fields should be of the same order of magnitude. However, since no maxima occur in the susceptibilities, one must conclude that the direction of the internal fields in these cases are such as to align the moments perpendicular to the applied field, and that there is little or no tendency for any of the moments to align parallel to the external field. Thus, though antiferromagnetic ordering does occur in these alloys, its direction is free to orient itself perpendicular to the applied field, and hence a temperature-independent susceptibility is exhibited. It should be noted that the same type of behavior exhibited by these δ -phase alloys was observed in lanthanum-cerium alloys by Roberts and Lock⁽²⁰⁾.

The discontinuities in the $1/\chi$ versus T curves at the junction of the Gouy and the mutual inductance data for all but the Nd-Y curves are

attributed to the fact that the inductance bridge requires a calibration procedure that may introduce some uncertainties in the final results, while the Gouy method is an absolute measure of the susceptibility. In this connection, it should be noted that the remarks made above in reference to the susceptibilities of the La-Gd and Ce-Y alloys were based only on Gouy data. Further, even though field dependency measurements carried out on the samples used in the Gouy technique afforded negative results, it should be remembered that for these measurements, the field was varied from approximately 1000 to 7500 oersteds, i.e., a factor of about 7, while the field employed in the inductance measurements was of the order of thousands of times smaller. Since the magnetic data indicate that appreciable crystalline fields exist in these alloys, such a drastic change in external field could alter the susceptibility values.

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FIGURE CAPTIONS

- Fig. 1. Dewar assembly for the Gouy apparatus.
- Fig. 2. Inverse paramagnetic susceptibility versus temperature curve for La-Gd δ -phase alloy, from 4.2-300°K.
- Fig. 3. Inverse paramagnetic susceptibility versus temperature curve for Nd-Y δ -phase alloy, from 4.2-300°K.
- Fig. 4. Inverse paramagnetic susceptibility versus temperature curve for Nd-Tm δ -phase alloy, from 4.2-300°K.
- Fig. 5. Inverse paramagnetic susceptibility versus temperature curve for Ce-Y δ -phase alloy, from 4.2-300°K.

END