OPTICAL AND MAGNETO-OPTICAL MEASUREMENTS ON MAGNETIC RARE EARTH-COBALT ALLOY FILMS

DR. AXEL M. STOFFEL
DR. KARL J. STRNAT

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RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
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FOREWORD

This report is the result of in-house research performed in the Thermal and Solid State Branch, Materials Physics Division, Air Force Materials Laboratory, Research and Technology Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was done under Project No. 7371, "Applied Research in Electrical, Electronic and Magnetic Materials", Task No. 737103, "Applied Research on Magnetic Materials".

This report is based on a portion of a thesis submitted to the Institute of Technology in Vienna, Austria, by Axel M. Stoffel in partial fulfillment of the requirements for the Degree of Doctor of Science.

The authors are indebted to their Branch Chief, Mr. Jules L. Wittebort, and Prof. Dr. Franz Lihl, Mr. Stoffel’s Thesis Advisor, for their continuing interest, support and encouragement.

This technical report has been reviewed and is approved.

JULES L. WITTEBORT
Chief, Thermal and Solid State Branch
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ABSTRACT

The results of an investigation of the Kerr magneto-optic effect and the complex refractive index for pure cobalt and for four ferromagnetic rare earth-cobalt alloys of the composition RCo₅ are reported. Magnetic data of all films are also given. The thin film samples were prepared by vapor deposition in ultrahigh vacuum and measured in situ. The effect of annealing and atmospheric corrosion on the optical and magneto-optical properties was investigated. The index of refraction, n, was found to decrease considerably after short exposure to air, while the Kerr effect increased. The rare earth alloys show an optical and magneto-optical behavior similar to that of cobalt. Dispersion curves of n, k, δM, 4M, Q, and q for wavelengths of 400 to 615 mµ are presented.
AFML-TR-65-445

CONTENTS

I. INTRODUCTION .......................................................... 1

II. MEASUREMENTS ......................................................... 2

Co - Film ................................................................. 3
YCo$_5$ - Film .......................................................... 4
SmCo$_5$ - Film .......................................................... 4
GdCo$_5$ - Film .......................................................... 4
NdCo$_5$ - Film .......................................................... 5

III. SUMMARY AND CONCLUSIONS ...................................... 6

IV. REFERENCES ............................................................ 8
ILLUSTRATIONS

FIGURE PAGE
1. Hysteresis Curves for Co ................................. 9
2. Hysteresis Curves for YCo<sub>5</sub> ....................... 10
3. Hysteresis Curves for NdCo<sub>5</sub> ....................... 11
4. Hysteresis Curves for GdCo<sub>5</sub> ....................... 12
5. Hysteresis Curves for SmCo<sub>5</sub> ....................... 13
6. The Effects of Annealing and Oxidation on the Magneto-
Optical Properties of Cobalt ............................... 14
7. Comparison of n and k for Cobalt with Reference Data . . 15
8. Comparison of Q<sub>o</sub> and q for Cobalt with Reference Data . 16
9. n and k for YCo<sub>5</sub> ..................................... 17
10. θ<sub>M</sub> and η<sub>M</sub> for YCo<sub>5</sub> ......................... 18
11. n and k for SmCo<sub>5</sub> ................................... 19
12. θ<sub>M</sub> and η<sub>M</sub> for SmCo<sub>5</sub> ......................... 20
13. n and k for GdCo<sub>5</sub> ................................... 21
14. θ<sub>M</sub> and η<sub>M</sub> for GdCo<sub>5</sub> ......................... 22
15. n and k for NdCo<sub>5</sub> ................................... 23
16. θ<sub>M</sub> and η<sub>M</sub> for NdCo<sub>5</sub> ......................... 24
17. n and k for the Annealed Films of Cobalt and Alloys .... 25
18. θ<sub>M</sub> and η<sub>M</sub> for the Annealed Films of Cobalt and Alloys . 26
19. Q<sub>o</sub> and q for Annealed Films of Cobalt and Alloys ... 27
20. 4π<sub>M</sub><sup>0</sup> and Q<sub>0</sub> versus Atomic Number for RCo<sub>5</sub> Alloys 28

TABLES

TABLE
I  Magnetic Data .................................................. 29
II X-Ray Data ...................................................... 30
III Cobalt Film .................................................... 31
IV Vapor Pressures of RE Metals ................................. 32
SECTION I
INTRODUCTION

In the last few years, the potential advantages of using the magneto-optical Kerr effect as readout for magnetic computer memory elements have been discussed, primarily by D.O. Smith (Reference 1). This scheme would allow good decoupling of drive and sense circuitry and permit higher bit density. The theoretical considerations were limited to iron, cobalt, nickel, and permalloy, all materials whose magnetic properties in thin film form have been the subject of intense investigation and which have been proven feasible for use in memory elements.

Another proposed magneto-optical memory (Reference 2) would employ the Faraday effect in a transparent ferrimagnetic garnet near its compensation point. Further recent work has shown MnBi (Reference 3) and EuSiO$_4$ (Reference 4) to exhibit large Faraday rotations, the latter only at very low temperatures. These or similar transparent ferrimagnets may find an application in memory cells for Faraday effect readout. Additional device applications have been suggested for the Kerr magneto-optical effect. They include high frequency light switches (Reference 5) and magnetic bolometers (Reference 6).

All potential applications of the Kerr effect suffer from the small magnitude of the Kerr rotation of materials otherwise suited for the use (less than five minutes angular rotation in permalloy at saturation) (Reference 7). It is possible to enhance the Kerr effect by dielectric overlayers and this may well prove the most practical approach. However, the search for new ferro- or ferrimagnetic substances which combine a large Kerr effect with favorable magnetic properties should not be neglected, and the work described here is a contribution to this effort. It was found recently (References 9 and 10) that several intermetallic compounds of the formula RCo$_5$ (R = yttrium or a rare earth) have high saturation magnetizations ($4\pi M_s$ up to 10,960 Gauss) and Curie points (up to 850°C). During studies of the domain structure of these compounds, indications were obtained that some of them exhibit extraordinarily large polar Kerr rotations (about 2° for SmCo$_5$ at saturation) (References 10 and 12). Since our laboratory is presently engaged in an investigation of such intermetallics, it was decided to prepare them in film form and measure the Kerr parameters along with the refraction and absorption indices. First results of this work were presented at the 1965 International Conference on Magnetism. In this report, a more detailed account of the experimental procedure is given and the analysis of the data is carried a step farther by making use of magnetization measurements and X-ray diffraction results obtained by A. Baltz at the Franklin Institute Laboratories.

A vacuum evaporator specially constructed for use in this work was previously described in ML-TDR-64-130 (Reference 14) and the polarimeter used in ML-TDR-64-419, Part II (Reference 15). Part I of the latter report discussed the optical measuring procedure and the underlying theory.

The present report and the preceding ones in this series are based on the doctoral thesis of one of the authors, (Reference 16).
SECTION II
MEASUREMENTS

GENERAL CONSIDERATIONS

The rare earths and many of their alloys are very reactive. At high temperatures they combine readily with the atmosphere and in the molten state, with most refractory materials. Even at room temperature many show surface corrosion after a short time. Optical properties are often severely changed by even a thin surface layer, and if one wishes to obtain data for a meaningful interpretation in terms of the theory of metals, the measurements must be made on very clean sample surfaces.

Holmes and Clung (Reference 17) failed to find a magneto-optical Kerr effect on gadolinium films prepared and measured in a vacuum of 10^{-6} Torr and attributed this to surface corrosion. Schuler (Reference 18) was later able to measure the Kerr effect on gadolinium films made in a vacuum of 10^{-5} Torr and could show that the surface remained free from detectable contamination for long periods at this low pressure. The RCo_{5} compounds are much more stable than the pure rare earth metals, especially in thin film form. Samples which were kept in air for over a year still retained their original luster. Even so, their index of refraction is different from that of a clean surface. Contamination of the interior and the surface of the sample by reaction with residual gases can of course occur during the film preparation and heat treatment.

EXPERIMENTAL PROCEDURES

For these reasons, the samples were prepared in ultra-high vacua near 10^{-9} Torr and annealed and measured in situ. (See ML-TDR-64-130) (Reference 14). All films whose properties are reported here were deposited onto fused quartz substrates at normal incidence. In the case of the alloy films, a small amount of the premelted alloy or lumps of the constituent metals in the proper ratio were placed in a tungsten basket and heated above the melting point until all material was evaporated to assure the proper 1:5 stoichiometry. In all cases, however, some reaction of the melt with the W-basket was noticed. During the evaporation time the pressure rose to 5 \times 10^{-8} Torr and fell again to near 10^{-9} Torr after the vapor source was turned off. The substrate was kept at room temperature. After the optical measurements on the “as deposited” film were completed it was heated to 300°C and annealed at this temperature for one hour (pure Co) or five hours (alloys). A magnetic field of 550 Oe was applied parallel to the film plane during the heat treatment to induce a magnetic easy axis. Measurements of a hysteresis loop for the Kerr rotation of a Co film showed that indeed a high squareness was obtained (Figure 1) and that the maximum field of 550 Oe sufficed to saturate the annealed sample. The optical measurements required a polarimeter of extremely high resolution which was built by us for this investigation and described in a previous report (Reference 15). All measurements with this instrument were made with a fixed angle of incidence of 60 degrees. The films were magnetized in a field of 550 Oe, the field turned off, and the data taken in the remanence point. This was done to avoid corrections for the Faraday effect in the windows. Magnetic measurements on the same films done at the Franklin Institute in Philadelphia showed that this procedure did not yield saturation values. To take this into account, all the magneto-optical data were revised by multiplying them by the factor M_{r}/M_{s}, the ratio of remanence to saturation obtained from the magnetization curves. The magnetic measurements were made on all films before and after an additional annealing. Table I shows the magnetic data obtained. Figures 1 through 5 show graphs of magnetization curves obtained with a vibrating sample magnetrometer. The unusual shape of the curves, that is, the fact that they do not level off horizontally at saturation, is due to the effects of the sample substrates and positioning in the magnetometer which have not been subtracted out. However, one can see that saturation is approached closely when the high field “tail” of the hysteresis loop becomes a straight line.
A value for $M_s$ can be obtained by extrapolating this straight line portion to the intersect with the ordinate axis. Remanence and coercive force may be obtained correctly from these curves by the intersects with the ordinate and abscissa axes respectively. All magnetization values are relative since the total sample magnetization is plotted in arbitrary units. Absolute $M$ values are not required if only the shape of the curve, the remanence ratio and the coercive force are of interest.

The structural analysis was done by X-ray diffraction. Before the optical measurements the films had been annealed at 300°C. In this condition they did not yield any X-ray diffraction patterns. This would indicate an extremely small crystallite size. After additional annealing at 400°C for 12 hours a number of diffraction rings were obtained which were typical of the ingot compositions for cobalt, YCo$_5$, NdCo$_5$, and GdCo$_5$. The SmCo$_5$ had to be subjected to an additional annealing treatment at about 450°C for twelve hours before X-ray diffraction patterns could be obtained. Table II lists the lattice spacings for the films and for comparison those calculated from powder X-ray data found in the literature (Reference 19) for samples prepared in bulk by induction melting. No such reference data were available for SmCo$_5$. Only our experimental values for this composition are shown. It should be noted, however, that the values do not coincide with d-spacings of either samarium or cobalt. The results of the optical and magneto-optical measurements on these films as well as the influence of oxidation and annealing are discussed in the following sections. Differences in the preparation technique are indicated for the individual film when applicable.

The magneto-optical data are given in terms of $\theta_m$, $\epsilon_m$, and $Q_0$, $\theta_m$ and $\epsilon_m$ are the double Kerr rotation and ellipticity, respectively. Both quantities are directly measured as changes in the state of polarization due to a magnetization reversal from $+M_s$ to $-M_s$. They are dependent on the azimuth, the angle of incidence and the color of the incident light. $Q_0$ and $q$ are the Voigt parameters, which are calculated from $n$, $k$, $\theta_m$, $\epsilon_m$ and the angle of incidence. Both these parameters are wavelength dependent, as are $\theta_m$ and $\epsilon_m$. $Q_0$ is dependent on the magnetization while $q$ is not. For each individual film the effect of atmospheric corrosion on $n$, $k$, $\theta_m$ and $\epsilon_m$ was measured. Figures 12 through 14 give a summary of all measured and calculated values for all films in the annealed, unoxidized condition. All measured data were corrected by the factor $M_s/M_s$ taken from Table I, to yield saturation values. Table III for Co is given as an example of a typical summary sheet of all measured and calculated data.

**COBALT FILM**

The cobalt films were deposited in the absence of a magnetic field; subsequent magnetic annealing induced a magnetic easy axis. The magneto-optical results for a characteristic sample are summarized in Figure 6. The unannealed films ("as deposited") have the highest index of refraction which is reduced only slightly by annealing. After exposure to air, the refractive index drops 3 to 5 percent. Practically all of this change occurs in the first few minutes. A comparison of the values for the oxidized films with those of Ingersoll (Reference 20) measured on massive materials shows good agreement for $n$ (Figure 7). Ballantyne's (Reference 21) values for cobalt films however are considerably lower (up to 20 percent) which would indicate that those layers were appreciably oxidized. This is very likely because they were deposited in relatively poor vacua. Our values for $k$, however, agree better with those of Ballantyne than those of Ingersoll. This may be explained by the fact that the structure of a film is considerably different from the massive material. These structural differences affect the absorption which takes place in the metal near the surface while the dielectric oxide layer has a dominating influence on the reflection. Annealing increases the extinction coefficient $k$. This is to be expected if one assumes that the film after annealing has an improved crystal structure, (fewer dislocations and grain boundaries), consequently better electrical conductivity. Similar results were obtained in experiments on nickle (Reference 22) in which absorption was measured in dependence of temperature of condensation and annealing.
Figure 8 shows a comparison of the magneto-optic parameters with the values of Ballantyne and Dziwulski (Reference 23). Our values for \( Q_0 \) and \( q \) lie between the values reported by these two authors.

An interesting fact is that both the Kerr rotation and Kerr ellipticity are larger after oxidation. This effect was also found on the compound films and can be explained as follows: The oxide layer acts as an enhancement layer. It increases the Kerr effect by causing multiple reflection on the metal surface, and for proper film thickness also by destructive interference of the ordinary metallic reflection component (polarized parallel to the plane of incidence). The condition for maximum enhancement is an oxide layer thickness of \( d = n \lambda / 2 \).

**YCo\(_5\) FILM**

This film was made by evaporating from a single source a premelted alloy of the composition YCo\(_5\). The results of the optical investigation are shown in Figure 9. Compared to cobalt, YCo\(_5\) shows a higher index of refraction and a smaller extinction coefficient. The values for \( Q_0 \) and \( q \) are somewhat larger than for Co (see Figure 20). The effects of oxidation are similar to those observed on cobalt: The change of \( n \) is larger on the long wavelength end of the spectrum, \( k \) increases more on the short wavelength end. Figure 10 shows the values measured for the Kerr rotation \( \theta_m \) and the Kerr ellipticity \( \epsilon_m \). Both increase slightly as the film oxidizes.

The annealing effect is also similar to that observed on cobalt. While \( n \) changes insignificantly, \( k \) becomes larger than that measured of the unannealed film.

**SmCo\(_5\) FILM**

This film was prepared by co-deposition of the unalloyed constituent metals from one source. A premelted ingot was not available. The large difference in vapor pressure of the two metals makes the preparation of stoichiometric SmCo\(_5\) in the vacuum melting furnaces available in this laboratory very difficult (the boiling point of samarium, about 1570°C is only slightly higher than the melting point of cobalt, 1420°C). Because of the very small amount (0.5 gram) of evaporation used for the preparation of the film, a very careful weighing of the constituents was necessary. After the deposition, the film was annealed for 5 hours at 300°C. The magneto-optical constants determined before and after annealing and after 5 minutes of exposure to air are presented in Figures 11 and 12.

All quantities are similar to the corresponding ones measured on pure cobalt. This makes it probable that the surface layer which determines the reflection properties was indeed mostly cobalt. Because of the large difference in vapor pressure of the constituents, it is likely that all the samarium metal vaporized first leaving almost pure cobalt to evaporate and condense toward the end of the deposition time. This must have brought about a sandwich structure of the film of such a nature that next to the substrate there was nearly pure samarium, followed by an alloy layer of varying composition, and finally by cobalt. Whether diffusion during the heat treatment was able to equalize this gross segregation effect or not could not be determined.

**GdCo\(_5\) FILM**

This film was prepared like that of YCo\(_5\), that is, by evaporating a piece of a premelted stoichiometric ingot. The "sandwich effect" just discussed is not expected to occur with either the gadolinium or the yttrium alloys since both metals, Gd and Y, have boiling points and vapor pressures very close to those of cobalt. Simultaneous evaporation of the constituent elements at approximately equal rates can thus be expected. This should have resulted in a very intimate mixture Gd and Co in a nearly homogeneous film, and even if the compound GdCo\(_5\) was not formed immediately during condensation, the reaction probably took place quickly during the annealing in view of the short diffusion distances involved.

The optical parameters of the annealed film (with the exception of \( n \)) are, indeed, radically different from those measured on cobalt (See Figures 13 and 17). All the field dependent Kerr parameters \( \theta_m \), \( \epsilon_m \), and the magneto-optical amplitude \( Q_0 \) (Figures 14, 15, and 19) are much smaller for GdCo\(_5\) than for any of the other investigated substances. \( Q_0 \) is an order of magnitude lower.
than for NdCo$_5$, the compound in the series with the highest values for the saturation magnetization.

GdCo$_5$ behaves unusually in another respect, too: The Kerr rotation and ellipticity are strongly dependent upon the azimuth in which the incident light is polarized. Our measuring technique requires observation of the polarization states of the reflected light for two slightly different azimuthal positions of the primary light beam, namely one degree to either side of the plane of incidence (See Reference 15, Part I). For GdCo$_5$, in contrast to all the other substances, these two measurements yielded appreciably different results for $\epsilon_m$ and $\epsilon_m$ if the center azimuth was not very exactly aligned with the plane of incidence. Since the formula used to calculate $Q_0$ and $q$ from the measured quantities are derived only for the two principal planes of polarization, that is, parallel or perpendicular to the plane of incidence (See Reference 24, p. 289) it was very important in this case to make use of a sensitive procedure of adjusting the polarizing prism to one of the principal planes which was described previously in Reference 15, Part I. In measurements by other authors who employed a similar polarimetric method no attention was apparently paid to this source of error, and this fact may account in part for the large discrepancies in the values for $Q_0$ reported in the literature. It is also important to note that even the values for the refractive index and the absorption coefficient may be falsified by the incorrect alignment if they are determined by this method.

Oxidation affected the GdCo$_5$ film in the same way as the other films; the Kerr effect was enhanced, the index of refraction lowered.

NdCo$_5$ FILM

The NdCo$_5$ film was prepared like the SmCo$_5$ sample by simultaneous evaporation of pure Nd and Co from a single resistance-heated vapor source. The danger of gross metallurgical segregation ("sandwich formation") is present, since the vapor pressure of Nd in the range of 2000 K is an order of magnitude higher than that of cobalt, but it is much less severe than with the Sm-Co alloy (See Table IV). While the refractive index and extinction coefficient are again very similar to those measured on pure cobalt (there is not much variation of these quantities in the entire group of investigated materials!), the magneto-optical parameters show a clear individuality (Figures 15 and 16). It is thus probable that the compound NdCo$_5$ was formed in the film during deposition and ensuing heat treatment.
SECTION III
SUMMARY AND CONCLUSIONS

An investigation of the optical reflection properties of cobalt and four rare earth-cobalt alloys of the nominal composition RCo$_5$ was conducted. These measurements were performed on vapor deposited thin films with and without a magnetizing field. The investigation required sophisticated vacuum equipment, optical instruments, and an extensive theoretical analysis of the measuring technique. Equipment design and the mathematical groundwork were discussed in three previous reports, and the present thesis relies heavily on these reports (References 13, 14, 15, and 16).

The work had various objectives. Some were clearly achieved and valid conclusions may be drawn from the results. An example of this is the effects of oxidation on the optical properties of cobalt. Other aspects of the work have yielded only preliminary results which serve, in part, to reveal where experimental difficulties exist and to point out possible solutions of these problems (such as in the preparation of stoichiometric compound films). With respect to the latter, the report in hand must be viewed as a progress report of an incomplete research project which is being continued.

Brief exposure to air or preparation of the sample surfaces in only moderately good vacua brings about considerable changes (up to 30 percent) of the index of refraction and the magneto-optical Kerr effect. This was found true even for such a nonreactive metal as cobalt, even more so for rare earth-containing alloys. If the data is to be used for an interpretation in terms of the theory of metals, one must clearly make in-situ measurements on samples prepared in ultra-high vacuum. Such results are reported for cobalt. They differ considerably from older literature data obtained on less pure samples.

Rare earth-cobalt alloys of the nominal composition 16.7 at. percent rare earth (k), 83.3 at. percent Co, where R represents the elements yttrium, neodymium, samarium and gadolinium, were prepared in thin film form. With Y, Gd and probably Nd, the samples were apparently the desired intermetallic compound RCo$_5$, although final proof of this is still missing. The correct conditions under which films of these compounds may be obtained by vapor deposition were established. The refractive index, $n$, absorption coefficient, $k$, the magneto-optical parameters describing the Kerr effect, and their dispersion in the visible part of the spectrum were measured on these substances for the first time. $n$ and $k$ are not very different from the corresponding values for pure cobalt. The magneto-optical amplitude, $Q_0$, of YCo$_5$ exceeds that of Co by up to 25 percent, while GdCo$_5$ shows a very small Kerr effect (See Figure 19). For all the substances, $Q_0$ is largest on the red side of the spectrum.

There appears to be a roughly linear correlation between the values for the magneto-optical amplitude and the saturation magnetization, $M_S$ (See Figure 20). This might be expected in view of the fact that the RCo$_5$ are all isostructural and resemble one another closely in many respects. The electrical conductivity is one of the factors which determine the magnitude of the Kerr effect. While it has not been measured for the RCo$_5$'s, it is likely to change only insignificantly and monotonously as R is changed in the compound series. Thus, all RCo$_5$ compounds (with the possible exception of CeCo$_5$, EuCo$_5$, and YbCo$_5$ in which the rare earth has a valency substantially different from 3) may be considered like "a single substance" for this argument. In a given ferromagnetic substance, though, the magneto-optical amplitude is proportional to the magnetization, $M$. A proportionality between $Q_0$ and $M_S$ indicated by our incomplete data could be understood in these terms.

One of the stimuli for our investigation was a report of an extra-ordinarily large
Kerr rotation for the compound SmCo₅ (References 10 and 12) which suggested a potential application of this or a similar material in magnetic computer memories with optical readout. The referenced observation was one of the polar Kerr effect. We studied the longitudinal effect because it is better suited for the computer readout scheme, even though it is smaller in magnitude than the closely related polar effect. The magneto-optical amplitude found for YCo₅ is indeed large and comparable to the absolutely highest values measured on some steels. However, the hope for an order-of-magnitude improvement over known materials was not fulfilled. The Q₀ determined for “SmCo₅” is not the highest, and even though the exact composition of this film was questionable, the previously discussed relation between Kerr effect and saturation magnetization lets one expect approximately the Q-value measured.

Our comparative measurements on ultra-pure and air-oxidized films show that the latter have always the larger Kerr rotation. This is believed to be analogous to the well-known Kerr effect enhancement by an over-layer of a dielectric such as ZnS. Since the effects of air exposure also appear to occur only in the first few minutes and a stable state is then reached, one can draw the following conclusion of practical significance: in any application where a large magneto-optical rotation is desired, the films would not have to be protected against air oxidation. The oxidation decreases the refractive index, n, in all cases. This fact is at variance with an opinion published by Lauch (Reference 25) that the purest film should always exhibit the smallest n. Lauch’s films, however, were made by sputtering and were probably largely oxidized, even within the material and not only on the surface.
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11. G. Hoffer, K. Strnat (to be published).
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Figure 1. Hysteresis Curves for Co.
Figure 2. Hysteresis Curves for YCo$_5$

YCo$_5$ "AS MEASURED"

YCo$_5$ AFTER ADDITIONAL ANNEALING AT 400°C
Figure 3. Hysteresis Curves for NdCo$_5$
Figure 4. Hysteresis Curves for GdCo$_5$
Figure 5. Hysteresis Curves for SmCo$_5$
Figure 8. The Effects of Annealing and Oxidation on the Magneto-Optical Properties of Cobalt.
Figure 7. Comparison of n and k for Cobalt with Reference Data
Figure 8. Comparison of $Q_0$ and $q$ for Cobalt with Reference Data
Figure 9. $n$ and $k$ for YCo$_5$
Figure 10. $\theta_M$ and $\epsilon_M$ for YCo$_5$. 

- **ANNEALED**
- **OXIDIZED (5 MIN)**
Figure 11. \( n \) and \( k \) for SmCo\(_5\)
Figure 12. $\theta_M$ and $\epsilon_M$ for SmCo$_5$
Figure 13. $\mu$ and $k$ for GdCo$_5$
Figure 14. $Q_M$ and $\epsilon_M$ for GdCo$_5$
Figure 15. \( n \) and \( k \) for \( \text{NdCo}_5 \).
Figure 16. $\theta_M$ and $\epsilon_M$ for NdCo$_5$
Figure 17. $n$ and $k$ for the Annealed Films of Cobalt and Alloys
Figure 18. $\theta_M$ and $\varphi_M$ for the Annealed Films of Cobalt and Alloys
Figure 15. $Q_0$ and $q$ for Annealed Films of Cobalt and Alloys
Figure 30. $4 \pi M_s$ and $Q_0$ versus Atomic Number for RCo$_5$ Alloys
### TABLE I

**MAGNETIC DATA**

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<th>YCo&lt;sub&gt;5&lt;/sub&gt;</th>
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<td>17.3 0.88</td>
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<td>100 2.5x10^-2</td>
<td>126 1.51x10^-2</td>
<td>12 2.8x10^-3</td>
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</table>

- Bef. Ann.: Before Application
- Aft. Ann.: After Application
- H<sub>c</sub>: Oe
- H<sub>s</sub>: Oe
- M<sub>L</sub>/M<sub>s</sub>
- M<sub>s</sub>: emu
## TABLE II

**X-RAY DATA**

**RARE EARTHS - COBALT THIN FILMS**

**COBALT THIN FILM**

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<td></td>
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</tr>
<tr>
<td>( \lambda ) (m/Å)</td>
<td>Film</td>
<td>as deposited</td>
<td>annealed (5 min)</td>
<td>oxidized (24 hrs)</td>
<td></td>
</tr>
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### TABLE IV

VAPOR PRESSURES OF THE RARE EARTH METALS USED IN THIS INVESTIGATION AT 2000°K (1727° C)

<table>
<thead>
<tr>
<th>Metal</th>
<th>Sm</th>
<th>Nd</th>
<th>Gd</th>
<th>Y</th>
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<td>Vapor Pressure (Torr)</td>
<td>245.</td>
<td>0.875</td>
<td>0.052i</td>
<td>0.0495</td>
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**Abstract**

The results of an investigation of the Kerr magneto-optic effect and the complex refractive index for pure cobalt and for four ferromagnetic rare earth-cobalt alloys of the composition RCo₅ are reported. Magnetic data of all films are also given. The thin film samples were prepared by vapor deposition in ultrahigh vacuum and measured in situ. The effect of annealing and atmospheric corrosion on the optical and magneto-optical properties was investigated. The index of refraction, n, was found to decrease considerably after short exposure to air, while the Kerr effect increased. The rare earth alloys show an optical and magneto-optical behavior similar to that of cobalt. Dispersion curves of n, k, \( \theta_M \), \( \epsilon_M \), \( Q_0 \), and q for wavelengths of 400 to 610 m\( \mu \) are presented.
**MAGNETO-OPTICS, KERR EFFECT, INTERMETALLIC COMPOUND FILMS, RCO.**

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