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PHYSICAL PROPERTIES OF RARE EARTH-COBALT MAGNET MATERIALS

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Army Materials and Mechanics Research Center

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ABSTRACT

To further define the various types of time dependences noted previously, attempts were made to return a sample to its original state. Because of the failure of these, a series of samples with nearly identical properties is being investigated. Some preliminary results are given. High temperature X-ray measurements were made of prism plane reflections. These yielded thermal expansion values in the basal plane showing no anomaly at the Curie Temperature. The widths of the X-ray lines were a function of temperature and also of previous heat treatments. The width variations are reported in some detail and attributed both to bulk and surface effects. The temperature factor was measured and can be interpreted through a Debye temperature of 281. (Author)

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FOREWORD

This report covers work done in the period Julyl, 1972, to March 31, 1973, under the general title "Physical Properties of Rare Earth-Cobalt Magnet Materials". The work is sponsored by the Advanced Research Projects Agency under ARPA Order No. 1914, Program Code No. 2D10. The work was carried out at the Army Materials and Mechanics Research Center, Watertown, Massachusetts, 02172, by the principal investigators, D. R. Chipman and L. D. Jennings, Jr. (Phone: 617-926-1900, Ext. 386 or 375).

This report, together with technical reports AMMRC TR 72-5 and TR 72-30, concludes the investigations carried out on this program.

CONTENTS

TIME DEPENDENCE OF MAGNETIZATION

As detailed in our previous reports;[1] the time dependence of the magnetization under various conditions can give helpful characterization of $SmCo_5$-based magnets. For example, a single sample might be examined at various applied fields and temperatures. This information would only be valuable, however, if the sample were restored to its original condition before each measurement; unfortunately, temperatures as low as 200 C altered the sample in an irreversible way. It has been suggested that samples can be returned to substantially their original condition by annealing at slightly below the sintering temperature and then repeating the original heat treatment. We carried out such a procedure on our samples, but we did not regain the original time dependences. Because of this failure, we have turned to the other alternative: examination of a number of different samples prepared in as nearly the same way as possible. Such a set of samples was not available until the very end of the period covered by this report. Therefore, only some preliminary data are given here. We also report on some rearrangement of the measuring apparatus which may be of interest to workers using oscillating sample magnetometers.

Measuring Apparatus

The simple integrator-detector unit outlined in our first report was adequate for the experiments on slowly changing magnetization. However, its slow response, nonlinearity and poor resolution for small signals were disadvantages for other applications. These shortcomings were obviated with the circuit of Figure 1. This design was especially tailored to suit the waveform produced by our 1-Hz oscillating sample unit. The purpose of the extra feedback loop around the integrating operational amplifier was to give zero restoration, either after a change in the sample condition or after an inevitable zero drift. The design of this stage proceeded as follows. Using a typical permanent magnet sample, the gain was adjusted to give a convenient level (10 volts, peak to peak) at the input to the integrator. R_I and C_I were adjusted to give a convenient output (also 10 volts). The d-c feedback ratio was adjusted so that the zero drift of the output is within a range that can be tolerated by the peak readers, about 0.2 volt for the present design. The frequency dependence of the feedback was then arranged so as to give rapid zero restoration without seriously degrading the integration performance. For example, C_1 and C_2 values of 35 μf gave approximate critical damping and allowed high accuracy measurements to be made about 9 seconds after a major zero shift. Under these conditions, the peak-to-peak output of the integrator was increased by about 1% over the case with no zero restoration. There was thus a slight dependence of the output on the waveform, but this was so small as to be inconsequential. (Note that the waveform would only change as a result of an undesired effect, such as a shift in the average sample position.) This dependence could be reduced, if required, through use of larger values of C_1 and C_2, but at the expense of underdamped zero restoration.

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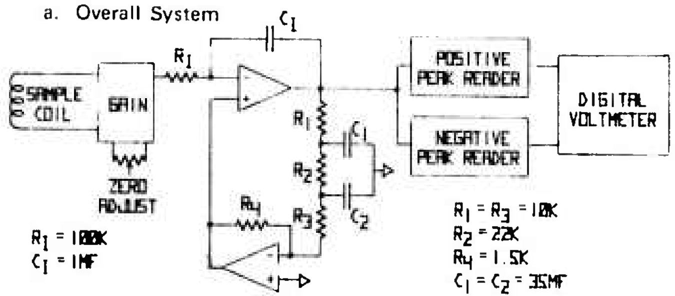
[1] CHIPMAN, D. R., and JENNINGS, L. D. *Physical Properties of Rare Earth-Cobalt Magnet Materials.* Army Materials and Mechanics Research Center AMMRC TR 72-5, February 1972, and AMMRC TR 72-30, September 1972.

a. Overall System



b. Negative peak reader. The positive peak reader has the diodes reversed.

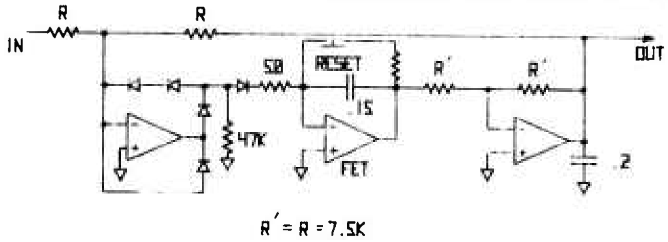


Figure 1. Schematic of the detecting circuitry.

The peak readers use conventional circuitry. The components chosen gave a leakage rate of about 10 mV/sec which was adequate to follow normal drift without introducing appreciable nonlinearity. After large shifts it was necessary to reset the peak readers. Routine precision was established within one second after such a reset, and the ultimate precision of 0.02% within three seconds.

Samples

A number of $SmCo_5$ magnets in the form of rods about 8 mm in diameter and 14 mm long were made available to us through the courtesy of Paul Weihrauch of the Raytheon Co. These had been pressed, sintered, and heat treated with a view toward making them as uniform as possible, rather than producing the best possible magnets. We machined four magnets, 0.100" high by 0.200" diameter, from each rod. Figure 2 shows demagnetization curves for two extreme cases; the other 18 magnets examined lay between these. These two cases, however, were for magnets cut from the same rod, showing the difficulty of preparing a number of well-matched samples. Nevertheless, it is hoped that these samples are adequately matched to display the field and temperature dependence of the effects discussed in our previous reports. Figure 3 shows typical time dependences, hardenings, and cross-over under open circuit conditions.

EXPERIMENTS ON $PrCo_5$ AND YCo_5

The need for high temperature X-ray studies of rare earth-cobalt magnet material, and also the difficulties which we encountered in trying to carry out such measurements on samarium-cobalt, were discussed in our last report. Our principal troubles resulted from an apparent loss of samarium in the surface layers of the alloy, either from vaporization or oxidation, or both, which occurred quite rapidly at high temperatures. Since the X-rays "see" only this thin layer on the surface, measurements had to be taken quickly

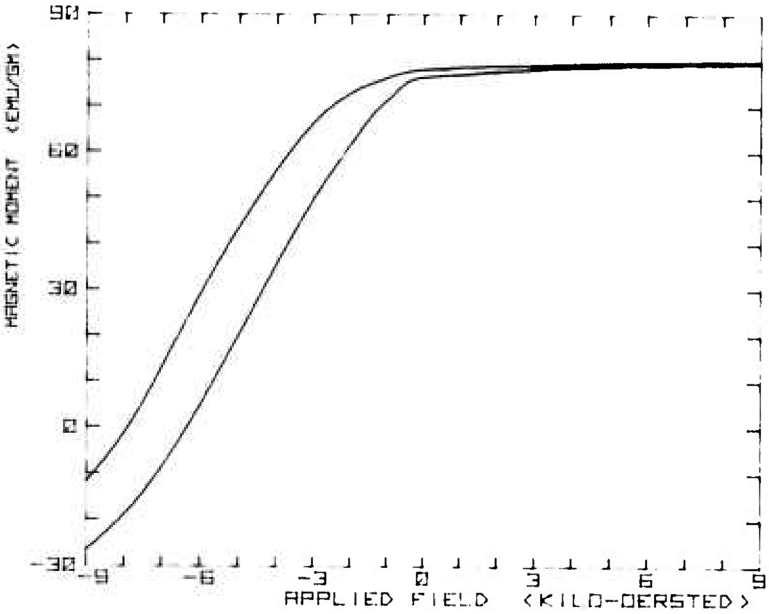


Figure 2. Demagnetization curves showing the range covered by the available samples.

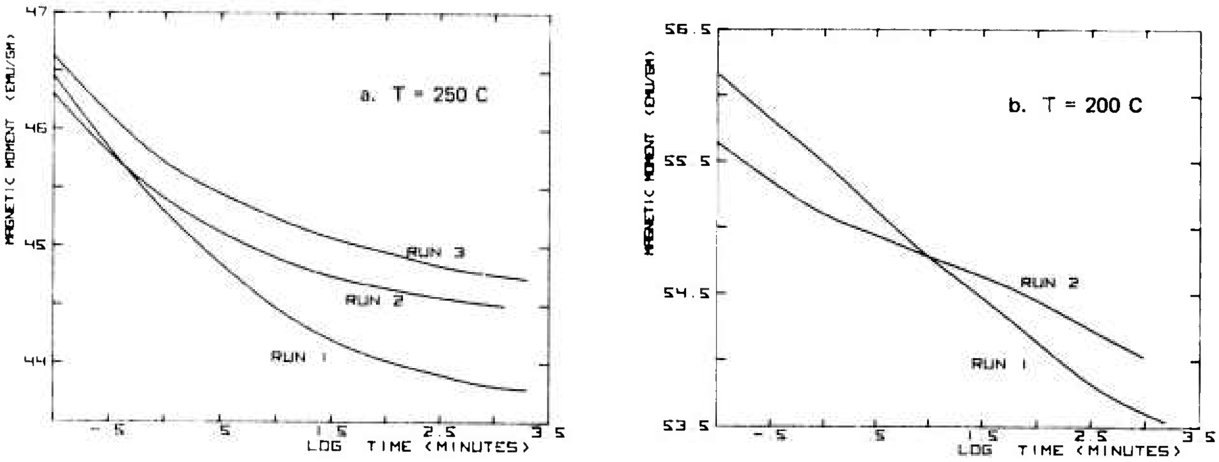


Figure 3. Open circuit time dependence of magnetization.

before complete deterioration occurred, which severely limited both the accuracy of the results and the scale of the investigation which could be undertaken. Because of these difficulties, we began a program of studies on praseodymium-cobalt and yttrium-cobalt, both of which have much lower vapor pressure components than samarium. This section of the report describes the work done with these materials.

Ordinary oriented magnet material makes a satisfactory sample for the X-ray powder diffraction work. Orientation gives additional intensity for an appropriately cut sample, the grain size is about right, and sintering provides a closed pore structure, probably desirable at high temperatures. We therefore began by attempting to make magnets of $PrCo_5$ and YCo_5 in the usual way. Powders were ground in a shaker mill, oriented in a static field of about 10 kG (or occasionally in a pulsed field of 100 kG), pressed uniaxially at various pressures up to 50,000 psi, and some were sintered in argon in sealed quartz tubes. Although this technique led to usable samples of YCo_5, we were unable to make even a marginally useful sample of $PrCo_5$ from the starting material at hand. This was chill cast multiphase material which homogenized (1120 C for three days) to single-phase $PrCo_5$ by X-ray determination. However, the powdered material oriented very little in the magnetic field, and after sintering always showed lines of the 2-17 phase. All X-ray lines were very weak which we attributed partly to poor orientation and partly to high absorption of the FeKα incident beam (not a good choice for this alloy). Magnetic measurements on the sintered samples showed the absence of the high anisotropy expected, probably indicating the presence of an appreciable amount of the 2-17 phase, thus precluding magnetic measurements of the orientation ratio. It would have been interesting to try a starting material richer in praseodymium, but since we have not set up to make our own alloys, we turned our attention at this point to yttrium-cobalt.

The yttrium-cobalt starting material was granular single phase (1-5), which we ground and oriented as described above. Although we were unable to make as highly oriented a sample as desired, sufficient orientation was achieved. The X-ray rocking curve had a full width at half maximum of about 15° compared to commercial $SmCo_5$ widths of about half this amount. Magnetic measurements showed that eh projection of easy directions along the alignment axis was about 75 to 80% and perpendicular, about 35 to 40%. A sample of this sort was mounted in the furnace on the X-ray spectrometer as described earlier,[1] and as an indication of possible improvement over $SmCo_5$, we observed the (002) and the (111) reflections at 770°C over a two-hour period. The results are given in Figure 4, which shows the integrated intensity of the (002) as a function of time. The figure also gives the results of the same measurement made on $SmCo_5$. Contrary to expectation, the YCo_5 material seems to deteriorate about as rapidly as the $SmCo_5$. The vapor pressure of pure Sm is higher than that of pure Y by a factor of over 10^9 at 770 C. Although it is possible that the partial pressures of Y and Sm over the 1-5 compounds are more nearly equal to one another, still it now seems very unlikely that the deterioration observed in Figure 4 is primarily a result of vaporization of the rare earth component. There are, however, differences between the two materials. In each case the heating at 770 C led to some new very weak X-ray reflections, probably mostly oxides, and to a line which we have attributed to metallic cobalt in the cubic state, which is very strong in the $SmCo_5$ sample and quite weak in the YCo_5 sample. Thus there may be a difference in the mechanism of the deterioration which might, for example, lead to significantly different rates at temperatures appreciably above 770 C.

Since there seems to be little to be gained from further studies on YCo_5 versus $SmCo_5$, however, we chose to return at this point to the samarium compounds since these are of more direct commercial interest.

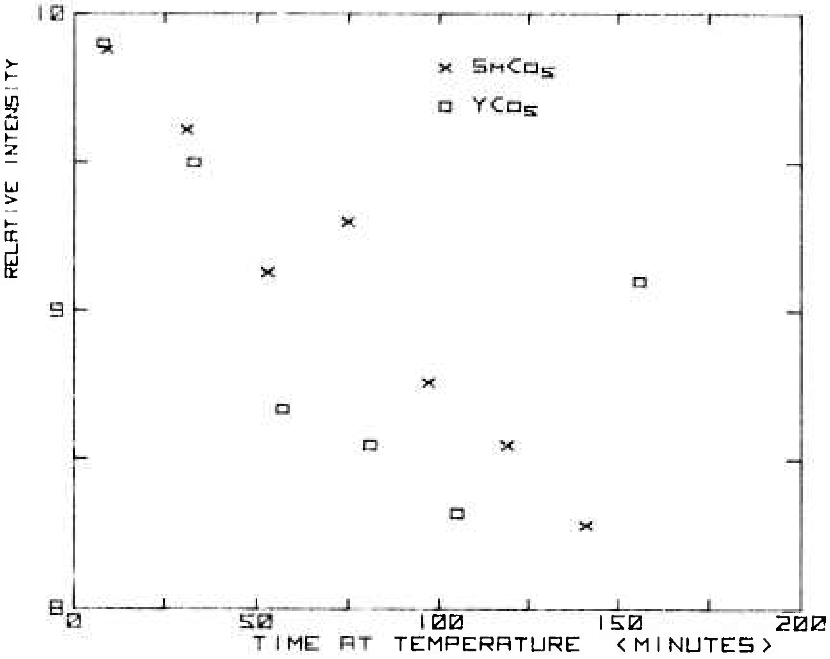


Figure 4. Comparison of time loss of X-ray intensity for $SmCo_5$ and YCo_5 at 770 C.

LATTICE EXPANSION IN $SmCo_5$

Our previous report describes experimental measurements of the lattice parameter perpendicular to the basal planes in $SmCo_5$. In this report we give the results of similar measurements made within the basal plane. A sample was cut from the same material which was used previously but cut perpendicular to the original sample such that the (h00) reflections were enhanced and (00ℓ) diminished. After cutting, by spark machine, the samples were polished on polishing paper and etched in an acid etch described by Becker et al.[2] A sample was then mounted in the X-ray furnace as before, with a 0.005-inch-thick Grafoil sheet clamped over the face, and examined in a purified helium atmosphere at temperatures up to 930 C. The (400) reflection was selected for study, and the (200) was also followed in order to correct for translation of the sample which occurs during heating in our furnace. FeK^{α} radiation was employed and these reflections had 2θ values near 127° and 53° at room temperature. Because of the rapid deterioration of the X-ray reflection with time at higher temperatures, it is necessary to limit as much as possible the time spent in taking the data. We measured the angular positions of the peaks by observing the intensities of three (later five) angles near the peak position (as shown in Figure 6), and fitting these points to a parabola (later to the actual shape of the X-ray peak itself) to obtain a measure of the peak position. As will be described later in the report, it was soon observed that the width of the reflection was quite dependent on temperature, and also on previous history, which necessitates comparison with a set of standard curves to determine peak position, and also requires that the overlapping of $K\alpha_1$ and $K\alpha_2$ wavelengths be properly

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[2] BECKER, J. J., CECH, R. E., and MARTIN, D. L. *Research to Investigate Fundamental Magnetic Interactions in Selected Materials.* General Electric Research and Development Center, Schenectady, New York, Contract USAF F33615-68-C-1248, Final Report, AFML TR 70-76, April 1970.

treated as the peak width changes. Before attempting to match the data to a set of standard shapes, the data at each temperature were normalized so the peak height above background was unit. For a typical case we estimate the accuracy with which we could measure the angular position of the (400) reflection to be about ±0.01° 2θ at temperatures below 400 C diminishing to about ±0.04° 2θ at the highest temperatures.

Specifically, the deterioration referred to above amounts to an overall loss in integrated intensity, and a somewhat inconsistent broadening and shift in the room temperature peak shape as well as in the shape at elevated temperature. The deterioration, of course, proceeds faster as the temperature is raised. We usually tolerated this loss until the intensity remaining was only about 20% of the starting value or until strong interferences developed. The sample was then removed from the furnace and repolished and etched. This treatment restored the sample to its original peak size and position, showing that this so-called deterioration is a surface phenomenon only. The shift in the room temperature peak after a high temperature run was not well correlated with the exact history of the sample. Shifts of as much as 0.2° 2θ upward in angle, accompanied by broadening, may result from heating at about 900 C, and subsequent heating at about 800 C seems to tend to return the most shifted of the peaks toward their former values, but not all of our data support these conclusions. The line broadening observed under these conditions will be discussed more fully in a later section.

The deterioration of the $SmCo_5$ peaks as discussed above is accompanied by the appearance of other diffraction peaks in the pattern. After prolonged heating we found strong peaks of Sm_2Co_{17}, of samarium oxide (Sm_2O_3) both A and B forms, and of beta cobalt, as well as weak reflections which remain unidentified. Compared to the sample which was cut parallel to the basal planes, the present sample has an increased tendency to develop lines of the 2-17 phase and although additional reflections of beta cobalt are observed, the strong (111) reflection is much weaker in the present sample.

The lattice expansion data determined as described above is presented in Figure 5. Data obtained from three classes of sample preparation are included in the figure. The points plotted as plusses (+) represent data obtained in runs starting with a freshly etched sample surface. The room temperature peak positions after such a run reproduced those of the etched sample well if 800 C was not exceeded during the run, but for higher temperatures the shift and broadening discussed earlier usually resulted. Data from runs showing this shift are not included in the figure. A second class of points plotted as open squares (□) represent data obtained from runs in which the sample had already been heated to a high temperature in a previous run. These points follow those of the first class quite well at the low and intermediate temperatures, although they probably represent a samarium-depleted surface, and because of this and because they do not experience the room temperature shift they are included in the figure up to the highest temperatures. The third class plotted as crosses (x) represent data from two individual runs which each began with a freshly etched sample, but during which the temperatures were cycled back and forth

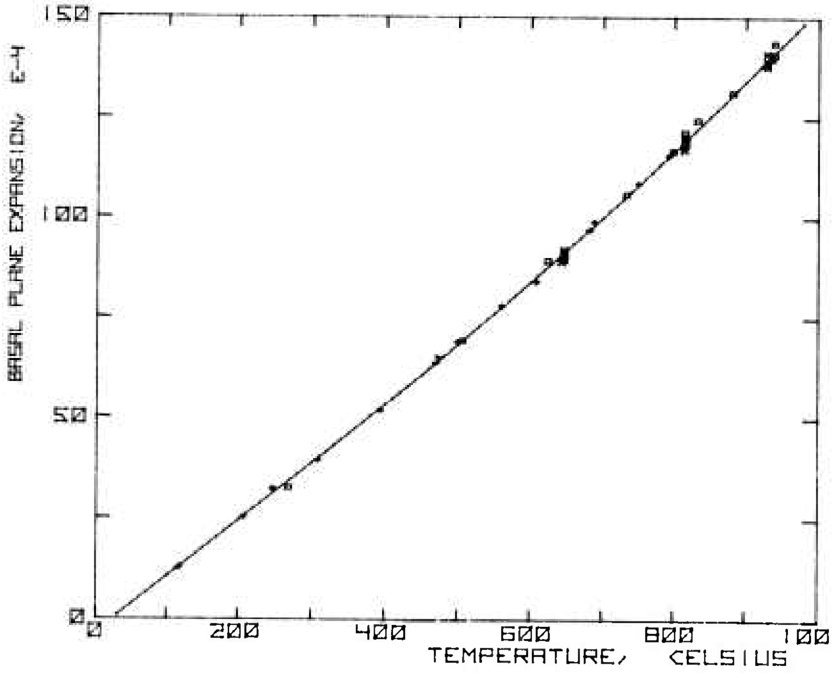

Figure 5. Fractional change in basal plane lattice parameter for SmCo as a function of temperature. The units are 10^{-4} and the significance of the various symbols is explained in the text.

among the three values shown (approximately 650 C, 815 C, and 930 C). These runs did not exhibit the room temperature shifts in peak position which followed all previous runs to 900 C or above. We attribute this to some kind of a recovery occurring during the 815 C hold which followed in each case the final heating at over 900 C. In these two runs, 20 minutes and 12 minutes were spent at 815 C before final cooling. In previous runs which showed the shift after 900 C heating, the cooling rate through 800 C was about 5 C per second. The smooth curve in Figure 5 represents a least-squares fit to all of the data points shown, using a cubic equation, which seems sufficient to represent the data. Points taken from this curve at equal 100 C increments are given in Table I, as well as our previous results for the "c" axis expansion. It should be noted that there is no observable anomaly in the thermal expansion coefficient in the basal plane as there is in a direction perpendicular to this plane. Near room temperature, our coefficients are about 15% larger than those reported by Martin et al.[3]

X-RAY LINE WIDTHS

As mentioned above, substantial changes in X-ray line widths were observed. Some of these changes were undoubtedly caused by the chemical changes taking place at the surface of the sample, but others were clearly representative of the bulk material. Much more research will be required to sort out these contributions, but a few representative observations are reported here. For convenience, we categorize these observations with appropriate titles, but the effects should not be considered to be valid except in the specific circumstances cited.

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[3] MARTIN, D. L., BENZ, M. G., and ROCKWOOD, A. C. *Cobalt-Samarium Permanent Magnet Alloys: Variation of Lattice Parameters with Composition and Temperature.* AIP Conf. Proc., v. 10, 1973, p. 583.

TABLE I

Values of total thermal expansion from 23 C to the indicated temperature, in the basal plane ($\Delta a/a$) and normal to it ($\Delta c/c$), the latter from Reference 1. Typical room temperature values are a = 4.9980 A and c = 3.9698 A (this value was inadvertently halved in Reference 1). Errors can be estimated from Figure 6.

T, deg C	$\Delta a/a$	$\Delta c/c$
100	.0011	.0005
200	.0025	.0010
300	.0039	.0016
400	.0054	.0021
500	.0069	.0026
600	.0084	.0031
700	.0101	.0036
800	.0118	.0048
900	.0135	.0063
1000	.0153	.0076

Reversible Bulk Narrowing

The solid curve in Figure 6 is the (400) peak of a polished and etched sample at room temperature. The curve is smoothed from point-counted data such as that shown in Figure 7. The curve and also the points, which will be discussed shortly, are normalized as to peak height above background and as to peak position. The shift in peak position can be determined with good accuracy from three or more appropriately chosen points as described previously. As examples, we show a set of points for temperatures near 260 C and near 690 C. In actual fact, the former are a composite of three triplets taken on three different runs. A typical statistical error is shown on one of the points. From these results it is clear that there has been significant narrowing of the X-ray peak at a temperature of 260 C. Inasmuch as it is possible to identically recover the original room temperature curve after a measurement at 260 C, we consider this narrowing to be representative of the bulk magnet material. After the measurements at 690 C, on the other hand, the room temperature intensity was noticeably reduced, although the form of the curve was unchanged. Thus it is clear that some surface changes had taken place during the run. Nevertheless, since the form of the curve was unchanged, it appears almost certain that the results are still representative of the bulk material.

At first sight it may not appear that the amount of the line narrowing involved is very great. In actual fact, the width at 690 C is comparable to the instrumental width as shown by the + symbols. These latter were obtained from a silicon standard which has a very small intrinsic width.

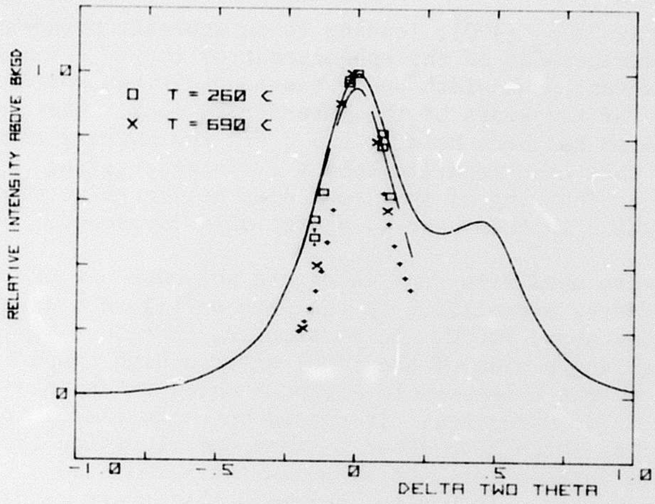

Figure 6. The (400) peak of $SmCo_5$. Results are normalized as to peak position and peak height. The solid curve is for 23 C and the other symbols are for the temperatures indicated. The + symbols refer to a standard of small intrinsic width. The broken curve (not normalized) is for the same conditions as the solid curve, but corrected to α_1 radiation alone.

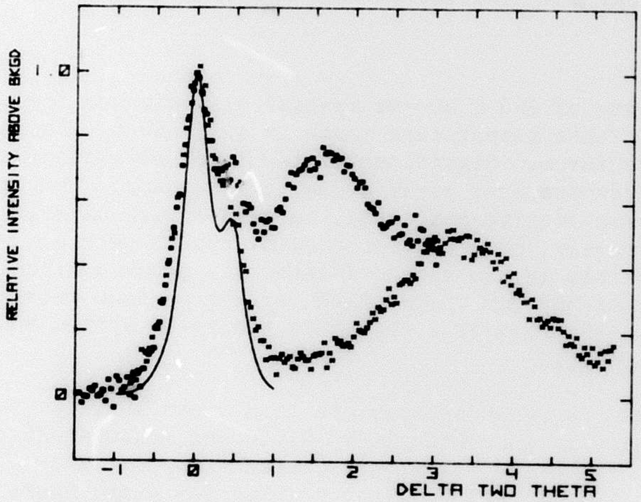

Figure 7. The (400) peak of $SmCo_5$ at room temperature after various treatments. The solid curve is for an etched sample. Open symbols refer to a sample cycled in range 650 to 930 C, with Grafoil protection. Crosses refer to a sample held two hours at 625 C, without Grafoil protection.

High Temperature Surface Deterioration

As discussed previously, we have not found it possible to make X-ray measurements at temperatures above 700 C without incurring surface deterioration. The form of this deterioration can vary, however. Two examples are shown in Figure 7. Both runs are at room temperature and the solid curve is the same one shown in Figure 6. The open symbols in Figure 8 refer to a sample which, with its usual Grafoil protection, was cycled several times through the range 650 to 930 C. The peak at $\Delta 2\theta = 1.5°$ ($2\theta = 128.5°$) is associated with material deposited on the Grafoil sheet. The wing of this peak clearly interferes with the $SmCo_5$ (400), leading to an apparent broadening, and also to an apparent enhancement of the enhancement of the $\alpha_1 - \alpha_2$ resolution. To evaluate the actual peak width under these conditions would require knowledge of the shape of the wings of the interfering peak. The x symbols refer to a sample which had been held at 625 C for two hours without the usual Grafoil shield. In this case metallic cobalt is formed, giving rise to the peak at $\Delta 2\theta = 3.4°$. The wing of this peak does not cause serious interference with the (400), which in this case is appreciably broadened.

In actual fact, no data were used from runs which did not make use of the Grafoil protector. The effects exemplified by the data on Figure 6 were nevertheless present in all runs above 700 C. In particular, there was always an excess intensity on the high angle side of the (400) after a high temperature run. A typical case is shown by the Y symbols in Figure 8 which refer to a sample previously held at 955 C for 8 minutes. It should be emphasized that all these results are for samples which have shown surface deterioration in that the absolute values of the intensities (not shown in the normalized curves of the figures) are diminished. It may be, however, that these results will be helpful in explaining why Sm-poor samples make inferior magnets.

Time-Dependent Narrowing

Temperatures in the vicinity of 800 C are of special significance for $SmCo_5$ magnets. A short hold in this temperature range is sufficient to almost completely destroy the coercive force. Significantly lower temperatures give a less rapid decay and higher temperatures yield magnetic hardening. Temperatures in the 800 C range appeared to give especially interesting results in our experiments also. Unfortunately, our temperature-time cycles were not chosen as well as would be possible in the light of information now available and there is also the question of how our observations of the surface relate to bulk effects. Nevertheless, the results do refer to the Sm-Co system and we give them with no further apology.

In Figure 8 the solid curve is again appropriate to a fresh surface at room temperature. The sample was then brought to 815 C and measurements made at temperature. The open symbols refer to data taken after one hour at 815 C and the x symbols to the same sample after holding for eight hours at 815 C. The additional narrowing during this time is small, but distinct. No further narrowing is possible, for this latter run is within statistics of instrumental width, as may be seen through a comparison with Figure 6.

Broadening at 815 C

The room temperature results for the sample after the long hold at 815 C are shown as the + symbols in Figure 8. The interpretation of these points is complicated by the appearance of the excess high angle scattering discussed above, but it is fairly clear that the line has been broadened by comparison with its original shape (solid curve). It is not nearly so broad, however, as after a higher temperature run such as shown by the Y symbols.

Narrowing at 815 C

One may also consider the behavior of a sample which has already been broadened by a run at high temperature. For example, the open symbols in Figure 9 show the room temperature curve for a sample which has been held at 932 C for 12 minutes. This sample was then taken to 830 C for 6 minutes and then returned to room temperature. The line was of course greatly narrowed at 830 C. It also appeared to be narrowed at room temperature as shown by the x symbols in Figure 9.

Reversible Narrowing of a Deteriorated Surface

We were not sure that a deteriorated surface would yield similar properties as bulk material at moderate temperatures. The reversible narrowing (discussed above for a fresh sample) was therefore studied. The open symbols in Figure 10 are again the room temperature results for a sample which had been held for 8 minutes at 954 C. The x symbols are for the same sample then taken to 505 C. Appreciable narrowing can be noted and the effect was reversible.

All the above effects have been discussed in qualitative terms only. Unfortunately, the modest intensity and limited time available precluded careful investigation of the form of the curves at high temperatures. At lower temperatures a systematic deconvolution is, in principle, possible, but difficulties with sample graininess make the value of such a procedure questionable. Nevertheless, in an attempt to make some quantitative statement of the results, the following procedure was carried out for a typical room temperature run. The form of the peak arising from α_1 radiation alone was calculated making use of the assumption that the α_2 peak is of the same form and one-half as strong. Such a separation is shown by the broken line in Figure 6. This separation was carried out for both the (200) and (400) reflections of $SmCo_5$ and also for the presumably unbroadened Si standard. A deconvolution was then carried out using standard Fourier techniques. In the case of (400) this procedure was reasonably satisfactory and yielded a full width at half maximum of 0.18°. In the case of (200) the resolution was poor and it is only possible to say that the deconvoluted width was less than 0.06°. It is thus possible to state that the broadening may be caused by stress (in which case the widths would differ by a factor of 4) or perhaps by faulting, but not by simple particle size broadening (which would give a factor of only two).

DEBYE-WALLER FACTOR

Because of the surface deterioration discussed above, it was not possible to make detailed line shape studies at high temperatures. If, however, the integrated intensity of a line were known as a function of temperature, the peak intensity alone would give at least some measure of the line width. To afford ourselves the opportunity of making this check and also to give some information about the elastic constants of $SmCo_5$, we measured the temperature factor for the (400) reflection. A brief description of the experimental procedure will give insight not only into the temperature factor

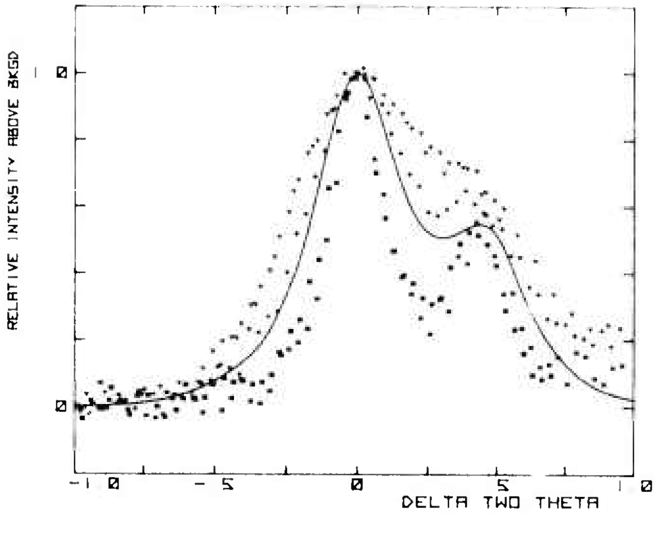

Figure 8. The (400) peak of $SmCo_5$. Solid curve: freshly prepared surface at room temperature. Open symbols: measurements at 815 C after one hour at temperature. X symbols: measurements at 815 C after eight hours at temperature. + symbols: measurement at room temperature after 815 C run. Y symbols: measurement at room temperature after eight minutes at 954 C.

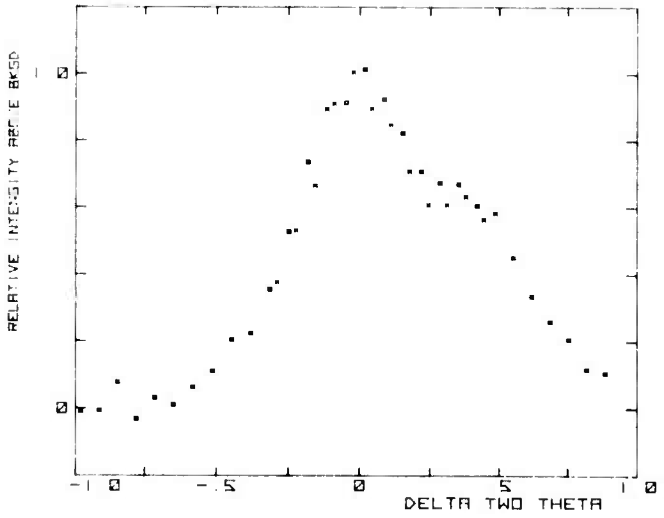

Figure 9. Room temperature measurements of (400) of $SmCo_5$. Open symbols: after twelve minutes at 932 C. X symbols: after six additional minutes at 830 C.

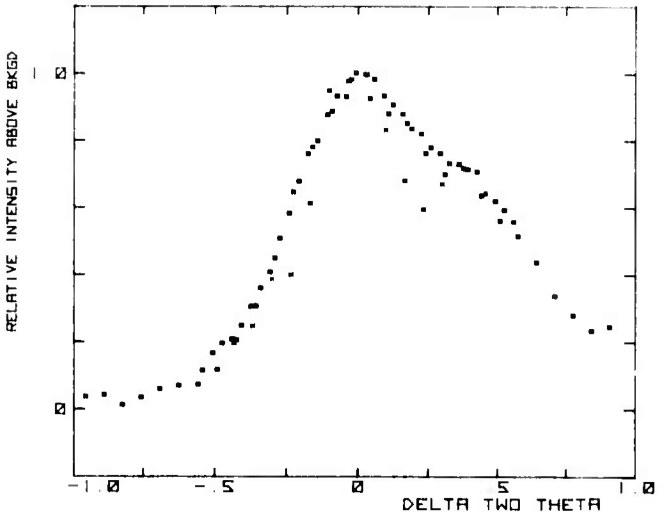

Figure 10. The (400) peak of $SmCo_5$. Open symbols: room temperature measurement after eight minutes at 954 C. X symbols: succeeding run at 505 C.

measurements but also into some of the line width experiments discussed earlier.

To assess the effect of sample graininess, the scattering angle was set for the peak of the (400). The count rate was then determined as a function of the rotation of the powder sample. In some instances, the rate (above background) changed by more than 20% for a sample rotation of less than 0.1°. Operation in such a region was deemed to be unfeasible because of the possibility of such rotation on account of warpage of the sample holder during temperature changes. In fact, in the line width experiments, spectacular results were occasionally obtained which could only be explained as a large grain coming into reflecting position. Unfortunately, we were not able to distinguish whether this came about through a small motion of the sample at temperatures near 900 C, or from grain growth at the surface under these conditions. For the temperature factor measurements it was important to minimize such effects as much as possible. Therefore, a region was found where the intensity was insensitive (to within 1%) to rotations up to 0.1° and temperatures were limited to a suitably low value where neither surface deterioration or holder warpage were significant. The actual measurements were then made using 2.75° scans alternately at room temperature and at 506 C.

The experimental result was that the ratio of integrated intensities for T = 779 K and T = 295 K is 0.67 ± .03. This result can be cast in a more useful form with the aid of the conventional temperature factor e^{-2M}. In the high temperature limit, M is proportional to temperature and an approximate correction for quantum effects can be made through Debye theory. Our result can then be interpreted through a Debye temperature of 281 assuming a lattice of average atomic weight 74.2. It is then possible to evaluate the temperature factor at all temperatures. It is also possible to make an estimate of an average value of Young's modulus for strains involving atomic motion in the basal plane. Using reasoning discussed, for example, by Herbstein,[4] one can estimate such an average as 14×10^{11} dynes/cm^2.

The information about breadth for a few typical cases, obtained with the aid of the temperature factor, is indicated in Figure 11. At each temperature an estimate was made of the breadth (full width at half maximum) for the α_1 component alone, as indicated in Figure 6, and also of the peak height. In Figure 11, the reduction in breadth from room temperature is plotted as the abscissa whereas the corresponding reciprocal of peak height, corrected by the temperature factor, is plotted as the ordinate. The scatter of the data is caused by statistical error arising from the short times available for counting, from difficulties with the interfering peaks, and perhaps from changes in peak shape with sample condition. In addition to the scatter, however, there are cases where something is clearly wrong. For example, the very low lying point has an extremely high count rate and a width slightly narrower than that of the Si standard. This is one example of the supposed large grain effect alluded to above.

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[4] HERBSTEIN, F. H. *Methods of Measuring Debye Temperatures and Comparison of Results for Some Cubic Crystals.* Phil. Mag. Supplement, v. 10, 1961, p. 313.

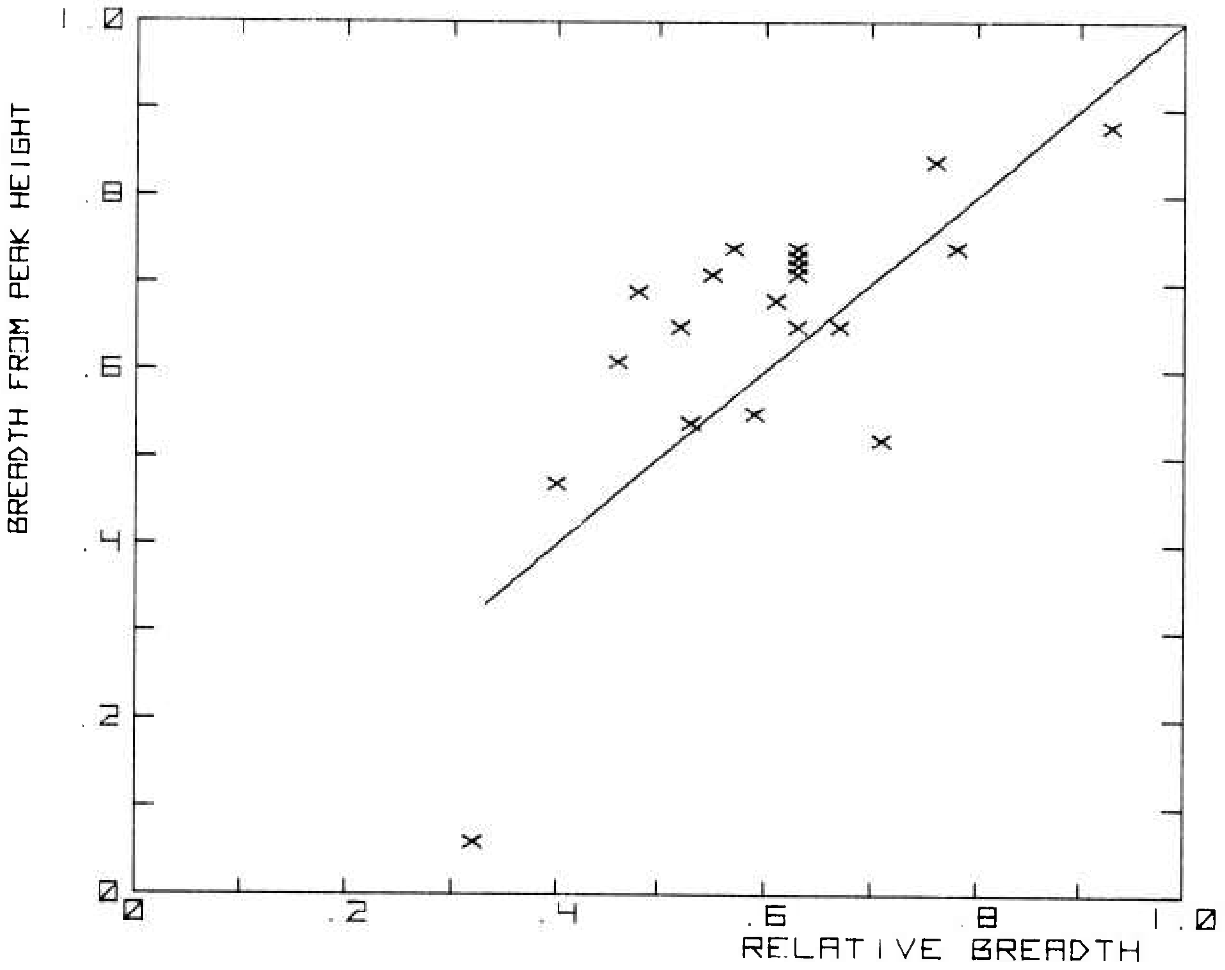

Figure 11. X-ray line breadth determined from peak height, corrected for the temperature factor, plotted against the directly measured breadth. The straight line gives the expected result in the absence of experimental difficulties and higher order effects.

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