Mechanism of Mechanical and Magnetic Hardening of a 10% Vanadium - Cobalt - Iron Alloy.

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MECHANISM OF MECHANICAL AND MAGNETIC
HARDENING OF A 10% VANADIUM-COBALT-IRON ALLOY

by

Richard William Fountain

A DISSERTATION
Presented to the Graduate Faculty
of Lehigh University
in Candidacy for the Degree of
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1952

Approved and recommended for acceptance as a dissertation in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

12 Jan. 1952 — Date

Joseph F. Libsch — Professor in Charge

Accepted, 8 Jan. 1952
Date

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INTRODUCTION

The properties of permanent magnet alloys are of interest metallurgically because they depend upon a transformation in a solid solution. It can be shown, for all practical purposes, that these properties can be reduced to the variation of a single quantity, internal strain. Therefore, in describing the various permanent magnet materials, they are generally classified in accordance with the manner in which the internal strain is introduced.

The oldest permanent magnets are those belonging to the class known as martensitic steels. These magnets are magnetically strong and mechanically hard because of the martensitic structure developed when these steels are quenched. A more recent group of permanent magnet alloys depend upon the precipitation of a second phase or an order-disorder transformation in a solid solution for good magnetic properties.

In all three types of permanent magnet materials, the enhanced magnetic properties are produced by a change in structure which introduces an internal strain in the system (1-3)*. The amount of improvement produced by this technique is, of course, dependent upon the

---

*The figures appearing in parentheses pertain to the references appended to this thesis.

particular alloy system and the degree and distribution of the strain. In general one may draw a parallel between magnetic and mechanical hardness. This correlation, however, is by no means complete. While both these properties depend upon the internal strain, the former is more explicitly related to the steric variation of the strain rather than to the number of strain centers present (4). On the other hand, in the case of mechanical hardness, there is good reason to believe that the number of strain centers is the critical factor. This difference gives rise to the lack of complete correlation that is observed in some of the newer alloys. However, it is generally known that the addition of cobalt to iron raises the magnetostriction and magnetic induction at saturation. The residual induction is related directly to the saturation induction, while the coercive force and strain are related through magnetostriction.

In permanent magnet design, the selection of the appropriate material and the shape of the part so that the minimum amount of material is used to produce the desired field strength in a specific gap is of paramount importance. This result is accomplished when the BH product everywhere in the material has its maximum. Therefore, the quality of a permanent magnet is determined not only by B_r* and H_c*, but also by the energy product, the product of B and H for various points on the demagnetization curve.

Formerly, most research and development work on permanent magnets, concerned with increasing the energy product, has been directed toward increasing the coercive force. Recently, however, methods have been developed for increasing the residual induction. As mentioned above, one such method is the addition of cobalt which increases the saturation induction and thereby allows for increases in the residual induction. One of the newer techniques of raising the residual induction is that of heat treating in a magnetic field. Certain alloys, such as Alnico V, when cooled in a magnetic field from the annealing temperature to room temperature have shown as much as 50% increase in residual induction, the coercive force being virtually unchanged. This technique was first suggested by Oliver and Shedden (5). In this alloy, the forces of magnetostriction cause elastic deformation which results in preferential growth of nuclei of the precipitate phase of desired orientation.

---

*Residual induction is defined as the value of magnetic induction for a ferromagnetic body that has been magnetized to saturation and removed from the field and designated as B_r. Coercive force is that value of magnetizing field required to reduce the magnetic induction to zero and is designated as H_c.

This elastic deformation is due to the magnetostriction of the strongly magnetic precipitate (6).

By increasing the residual induction in one of the manners mentioned, it not only increases the energy product but also increases the maximum operating flux density of the magnet. In certain applications even a slightly lower energy product with an increased B_r would be advantageous.

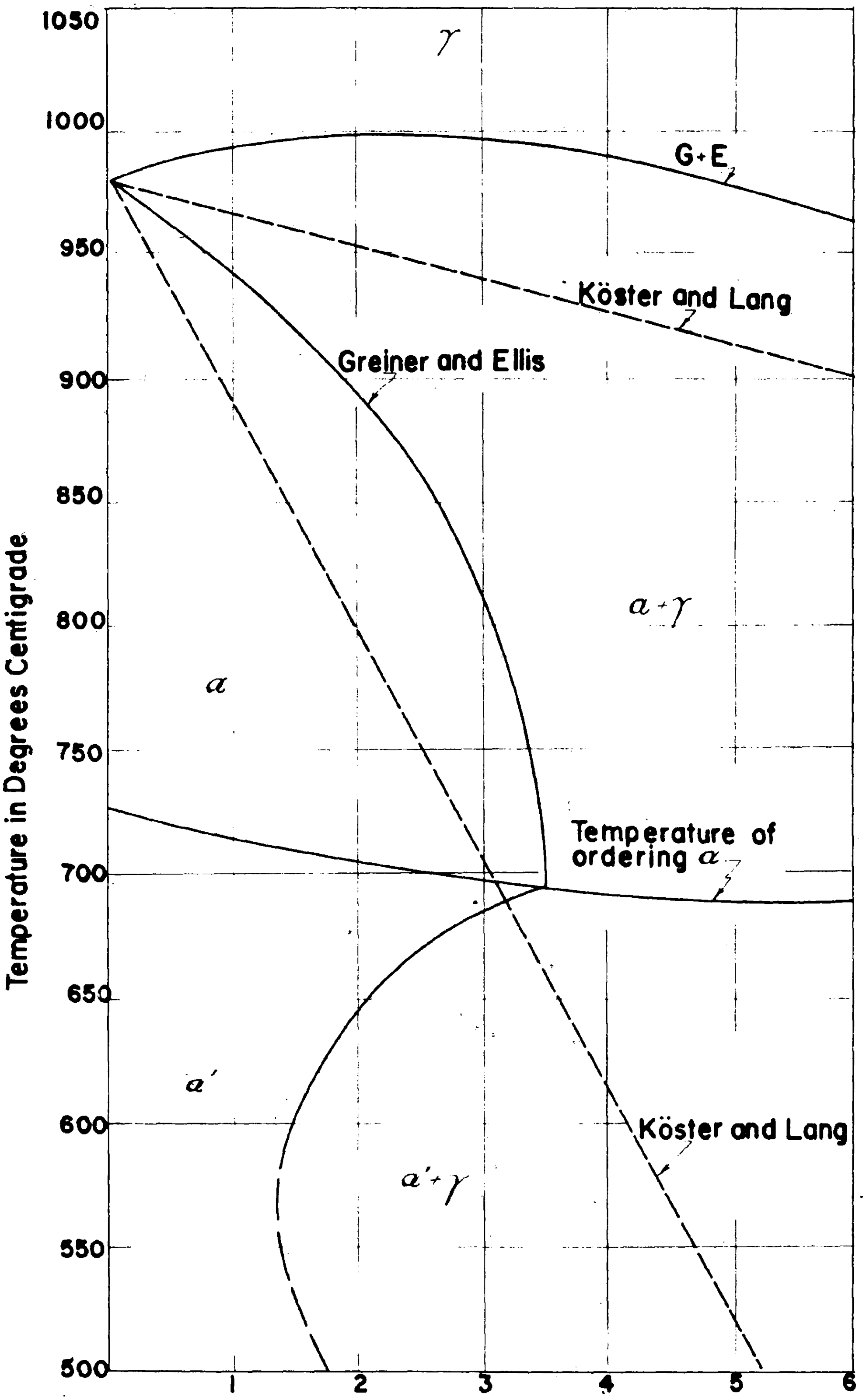
The objectives of the present investigation have been 1) to study the mechanism of mechanical and magnetic hardening in a 10% vanadium-cobalt-iron alloy and to correlate the results of this study with other magnetic properties; 2) to investigate the response of this commercial Co-Fe-V alloy (Vicalloy I) to heat treatment in a magnetic field; and 3) in an effort to supplement the results of 1) above, to determine the influence of increasing vanadium additions on the transformations of Co-Fe alloys.

The ternary Co-Fe-V alloys are an interesting group of magnetic materials. The low vanadium alloys (about 2%) are characterized by a high saturation induction and a low coercive force (7), while the higher vanadium alloys (about 10%) possess attractive permanent magnet qualities (8, 9). From a metallurgical point of view these alloys are of interest for several reasons. First, they are based on the high saturation Fe-Co alloys which are little understood; second, the constitution diagram for the system has not been completely established; and third, the mechanism of mechanical and magnetic hardening has not been definitely established.

Literature Review

Early work on Co-Fe-V alloys was undertaken by Nesbitt (10) who studied a number of alloys in the composition range of 30-52 per cent of iron, 36 to 62 per cent cobalt, and 4 to 16 per cent vanadium. From this early study, two alloys were proposed for commercial application. Vicalloy I, an alloy of approximately 10% V, 52% Co, and 38% Fe, and Vicalloy II, an alloy of approximately 14% V, 52% Co and 34% Fe. Nesbitt attributed the permanent magnet properties of these alloys to the precipitation of a high temperature gamma phase in the matrix low temperature phase during aging in the range of 600°C. This conclusion was based upon the then available phase diagram of Köster and Lang (11), Figure 1, which showed that increasing additions of vanadium to Fe-Co lowered the alpha to gamma transformation to temperatures in the same range as those used in the aging of this alloy by Nesbitt. Although x-ray diffraction data confirm the precipitation of the gamma phase on aging, a more recent constitution diagram by Greiner and Ellis (12) shows that the order-disorder reaction inherent in the Co-Fe binary

Figure 1. Influence of Vanadium on
Constitution Diagram for 50%
Iron-Cobalt Alloy


Temperature in Degrees Centigrade
1050
1000
950
900
850
800
750
700
650
600
550
500
1
2
3
4
5
6
γ
G+E
Köster and Lang
Greiner and Ellis
α+γ
α
Temperature of ordering α
α′
α′+γ
Köster and Lang

system (13) is also present in the ternary Co-Fe-V alloys.

Geisler (14) and Geisler and Martin (15) have recently confirmed the existence of the order-disorder reaction in the Co-Fe-V alloys and have suggested that this reaction may also be responsible for the permanent magnet properties of these alloys. They propose, on the basis of the fine particle theory of coercive force, that the high coercive force of these materials could be attributed to the formation of fine particles of the ordered phase. They further suggest that the effect of the vanadium on the coercive force of Co-Fe alloys may originate either through its influence on some characteristic of the ordering process such as coherency strain or particle size and shape or through its influence on a property of the ordered phase such as magnetostriction or magnetocrystalline anisotropy. However, whether the origin of mechanical and magnetic hardening is due to the precipitation of gamma or ordering in the alpha phase has not been definitely established. Accordingly, an investigation of the constitution and structure of Co-Fe-V alloys was undertaken and the effect of heat treatments on hardness and magnetic properties was determined.

The present investigation was undertaken approximately two years ago independently and without knowledge of the recently published work of Geisler and Martin (15). Although the two investigations are very similar, the results of the two are not completely compatible. Thus, the present investigation was continued with minor modification.

PART I - MECHANISM OF HARDENING

EXPERIMENTAL PROCEDURE

Ternary Co-Fe-V alloys with vanadium ranging from 0% to 14%, 52% cobalt, and varying amounts of iron were prepared for this investigation by powder metallurgy. Electrolytic iron, commercial cobalt, and ferrovanadium powders were used. The processing cycle was as follows:

1. Elemental powders were weighed and mechanically mixed for 24 hours.
2. Mixed powders were pressed at 30 tsi into the form of bars 5/8" x 5/8" x 5".
3. Bars were sintered in an atmosphere of purified hydrogen for 24 hours at temperatures ranging from 1380°C to 1340°C, depending upon the vanadium content.

The as-sintered bars were cut in half and one-half was machined to 7/16 inch diameter for thermal analysis. The remainder of the bar was hot forged to approximately 0.050 inch for x-ray diffraction analysis. All other tests were performed on cast Vicalloy I. Further details of sample preparation will be presented in the section of the report according to the type of test performed.

Thermal Analysis Measurements

One of the most useful techniques for determining phase relations as they vary with temperature and composition is to study the rate of change of temperature of a material as heat is supplied or extracted at a constant rate. The thermal analysis method used in this investigation is the inverse-rate method described by Smith (16). The method consists of plotting the inverse heating or cooling rate versus the temperature as heat is supplied to or extracted from the specimen. The transformation temperatures are readily discernible for alloys that reach equilibrium rapidly.

The apparatus used for these analyses consisted of a cylindrical refractory container which holds a cylindrical specimen. The container is centered in a small vertical tube furnace whose outer jacket is wound with a copper cooling coil in order to provide sufficiently rapid extraction of heat when the cooling curve is being determined. A differential thermocouple, operating across the wall of the refractory container, is the control element for a potentiometer-type controller that is set to maintain a constant temperature gradient across the wall of the specimen container. The constant gradient causes heat to be supplied or extracted from the specimen at an approximately constant rate. A second thermocouple, in the specimen, is used to measure the actual temperature of the specimen. A Leeds and Northrup Type "K" potentiometer in conjunction with a wall type galvanometer, lamp, and scale assembly, is used to measure the thermal e.m.f. Uncalibrated chromel-alumel thermocouples, from the same lot of wire, were used and a cold junction of 0°C was maintained. Measurements made of the alpha to gamma transformation temperature and of the critical temperature of order in the 50% Fe-50% Co alloy are in good agreement with those of Ellis and Greiner (13).

The measurements were taken as follows: two stopwatches, operating in "relay" fashion, were used to determine the time required for the thermal e.m.f. to change some specified interval. This was usually 0.10 millivolt. This time was then plotted versus the thermal e.m.f. at the end of the interval, producing a curve similar to that shown in Figure 2. The curves were taken using heating and cooling rates of 3-5°C per minute while maintaining a temperature gradient of 70°C.

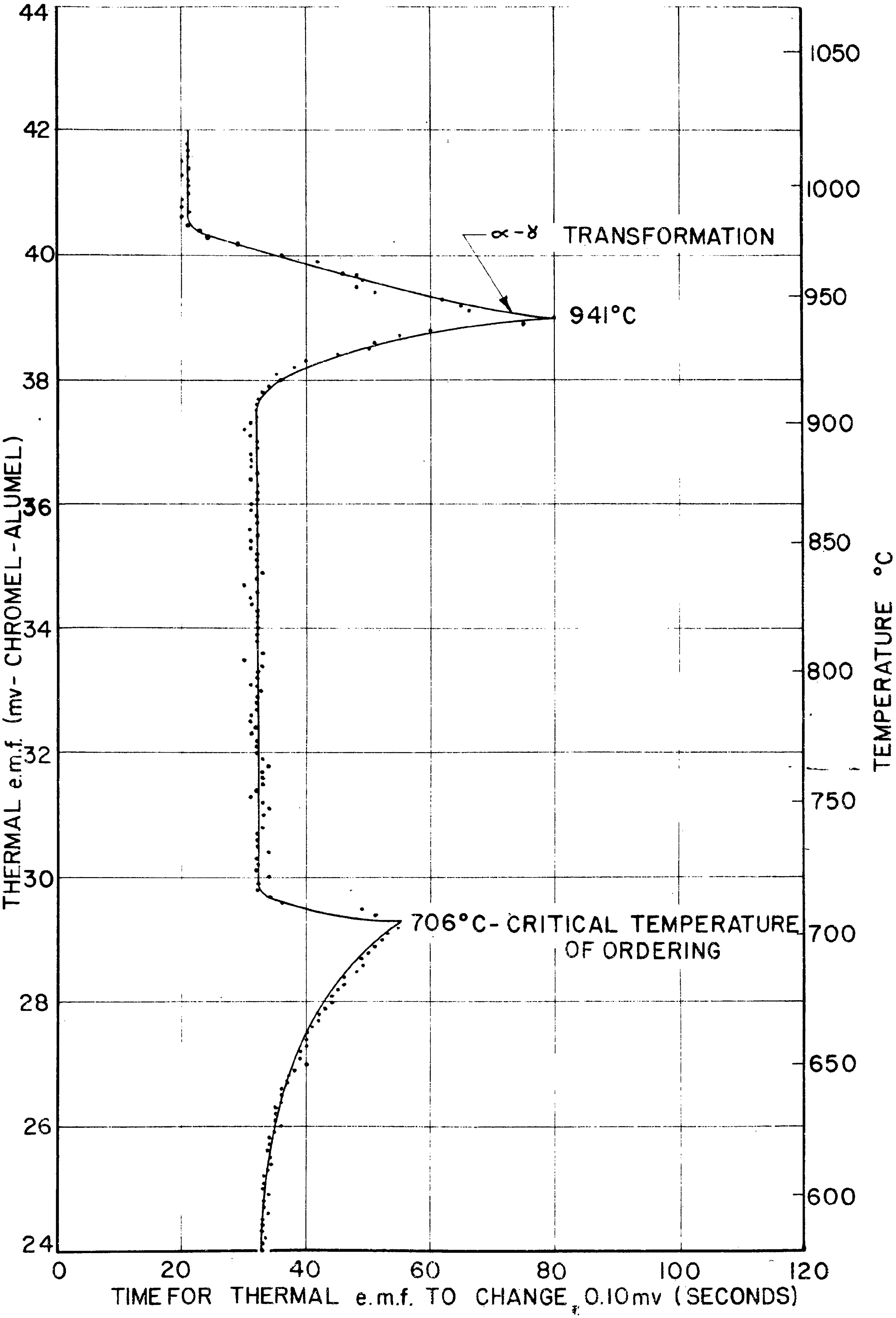
The specimens for thermal analysis were solution treated at 1100°C for one hour followed by oil and liquid nitrogen quenching. The liquid nitrogen quench was used in an effort to transform any retained gamma phase, thus providing a single phase alloy at the start of the analysis.

Figure 2 is the thermal analysis curve obtained on an alloy containing 52% Co, 46% Fe, and 2% V. The discontinuities in the curve at the critical temperature of order and at the alpha-gamme transformation temperature are clearly shown in this example. Although the alpha-gamma transformation is discussed as if it occurred at a single temperature, it can be seen from Figure 2 that it actually occurs over a narrow range of temperature. What is reported as the alpha-gamma transformation on this curve is actually the temperature at which the longest thermal arrest occurred.

On heating, the onset of the ordering reaction is manifested by a gradual increase in the inverse heating rate (the abscissa of the thermal analysis curve). For the alloy shown in Figure 2, this effect becomes evident at about 615°C. At the critical temperature of order (706°C in Figure 2), the inverse heating rate reaches a maximum and then falls off precipitously. Above this temperature, the long-range order in this alloy is considered to be zero.

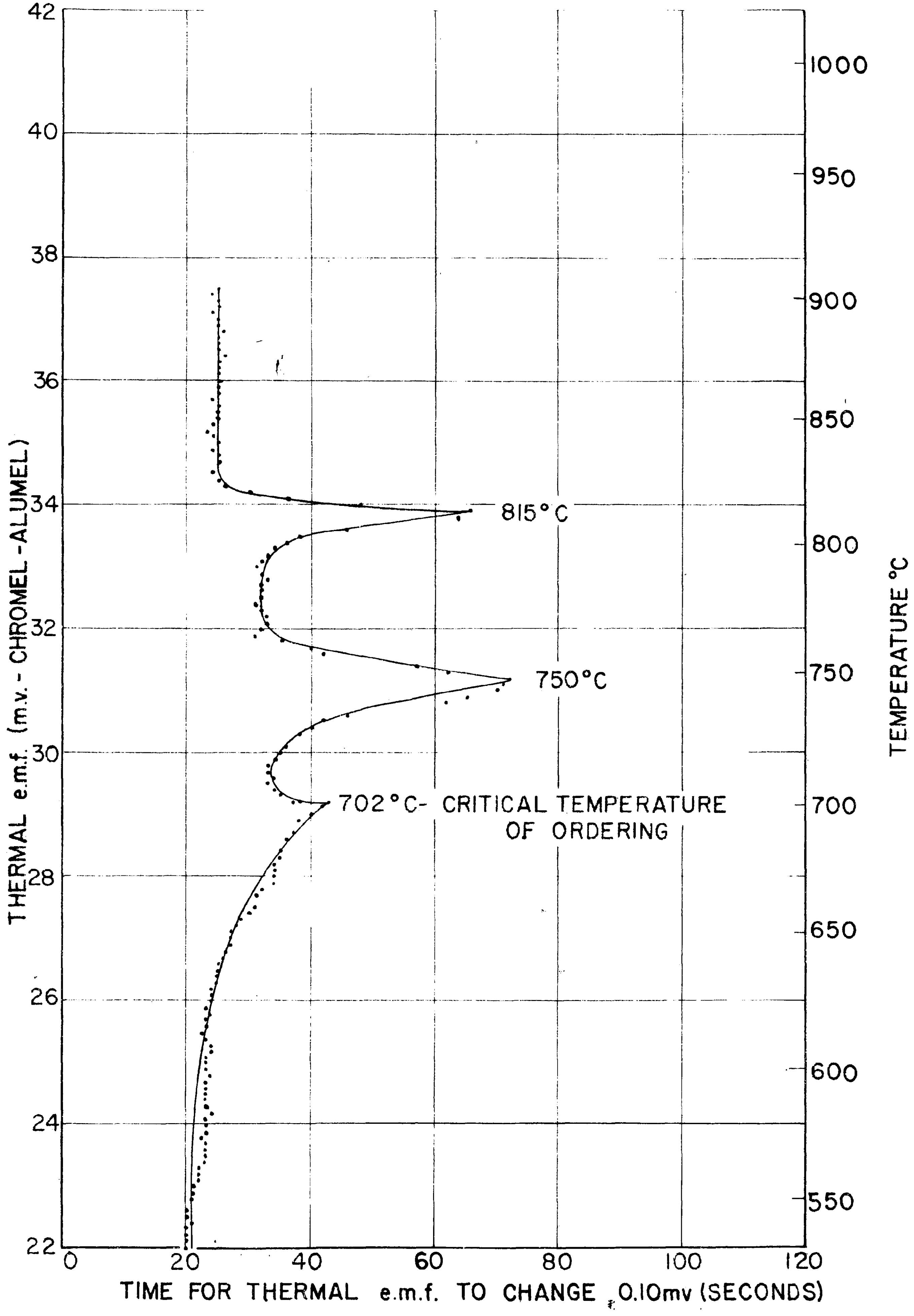
Thermal analysis curves similar to that of Figure 2 were obtained until the vanadium content of the alloy reached 6%. At this point, a curve similar to that of Figure 3 was obtained. The striking difference between the two curves is at once observed as an additional peak lying between the critical temperature of order and the alpha-gamma transformation temperature of the 2% vanadium alloy. At first sight, this additional peak may lead one to believe that some error was made in the course of the thermal analysis of this sample. However, reference to Figures 4 and 5 will show that the additional peak is present in all the curves for specimens ranging from 6% to 12% vanadium. There is no doubt that this peak is

Figure 2. Portion of Thermal Analysis Curve Obtained in Heating an Alloy Containing 52% Co - 46% Fe - 2% V



α-γ TRANSFORMATION
941°C
706°C- CRITICAL TEMPERATURE OF ORDERING
THERMAL e.m.f. (mv- CHROMEL-ALUMEL)
TEMPERATURE °C
TIME FOR THERMAL e.m.f. TO CHANGE 0.10mv (SECONDS)
24
26
28
30
32
34
36
38
40
42
44
0
20
40
60
80
100
120
600
650
700
750
800
850
900
950
1000
1050

Figure 3. Portion of Thermal Analysis Curve Obtained in Heating an Alloy Containing 52% Co - 38% Fe - 10% V



42
40
38
36
34
32
30
28
26
24
22
THERMAL e.m.f. (m.v. - CHROMEL - ALUMEL)
1000
950
900
850
800
750
700
650
600
550
TEMPERATURE °C
0
20
40
60
80
100
120
TIME FOR THERMAL e.m.f. TO CHANGE 0.10mv (SECONDS)
815°C
750°C
702°C- CRITICAL TEMPERATURE
OF ORDERING

Figure 4. Portion of Thermal Analysis
Curves Obtained in Heating
Alloys Containing 52%,
2% - 8% V, Bal. Fe

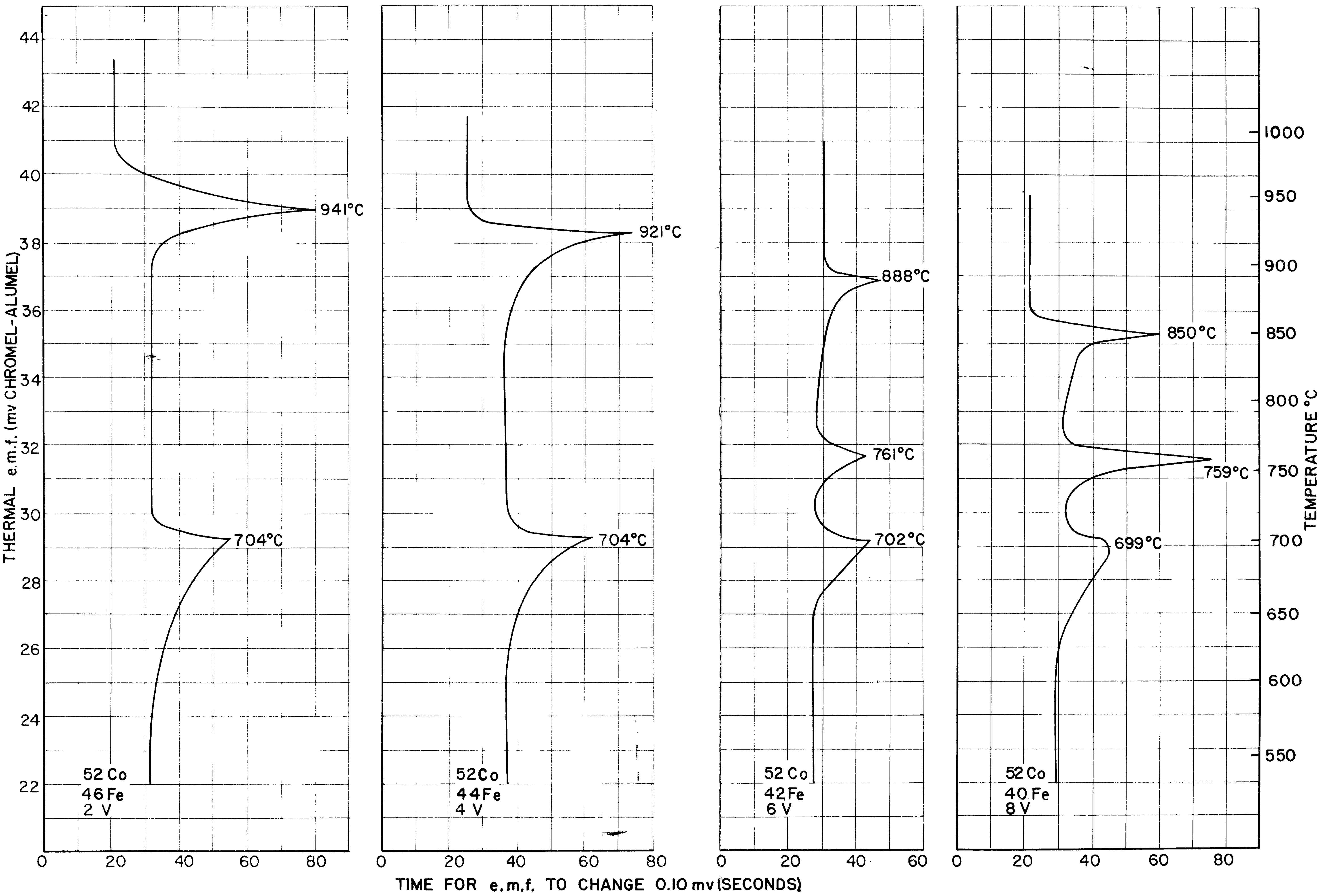
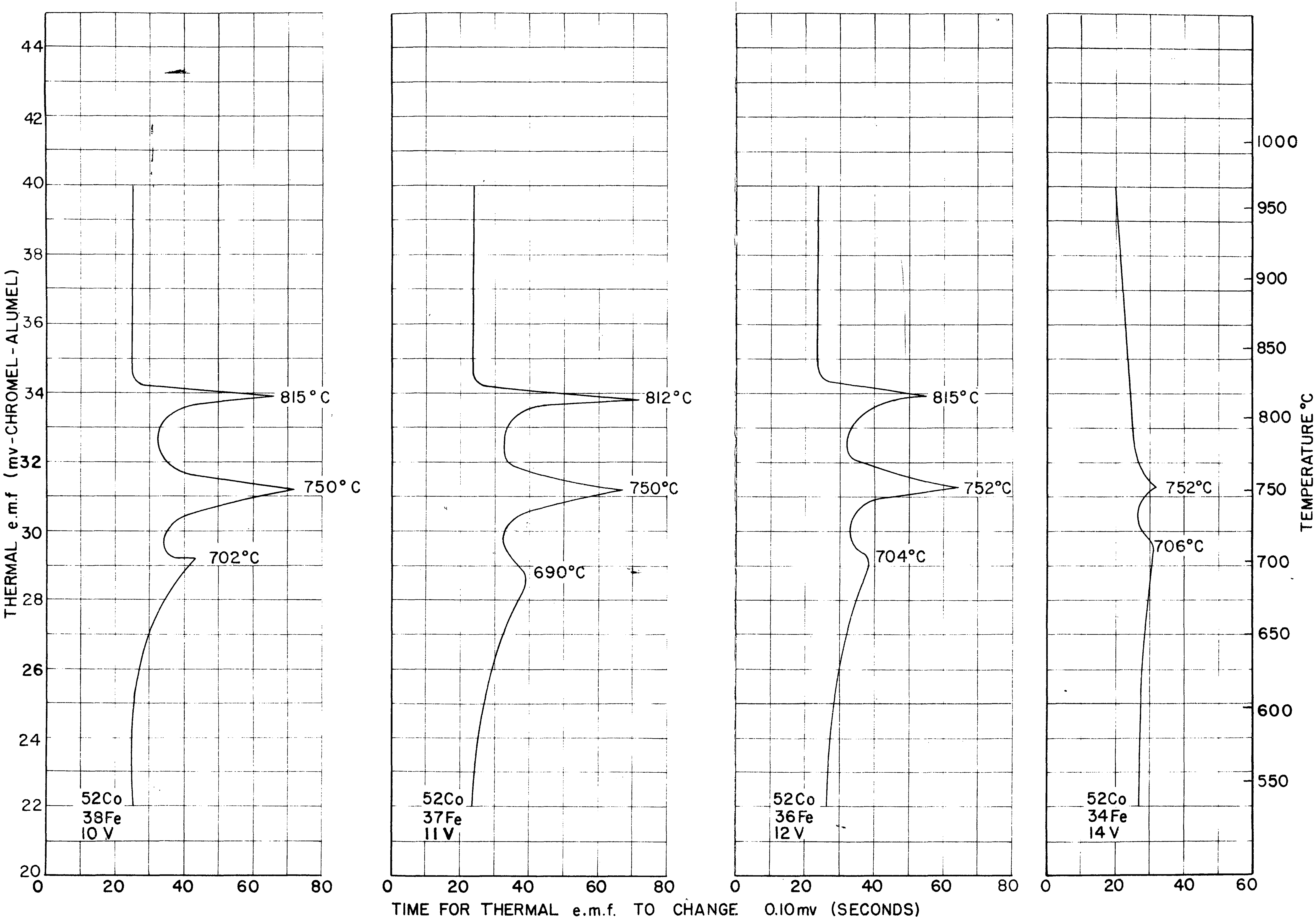

THERMAL e.m.f. (mv CHROMEL-ALUMEL)
44
42
40
38
36
34
32
30
28
26
24
22
941°C
704°C
52 Co
46 Fe
2 V
921°C
704°C
52 Co
44 Fe
4 V
888°C
761°C
702°C
52 Co
42 Fe
6 V
850°C
759°C
699°C
52 Co
40 Fe
8 V
TEMPERATURE °C
1000
950
900
850
800
750
700
650
600
550
0
20
40
60
80
0
20
40
60
80
0
20
40
60
0
20
40
60
80
TIME FOR e.m.f. TO CHANGE 0.10 mv (SECONDS)

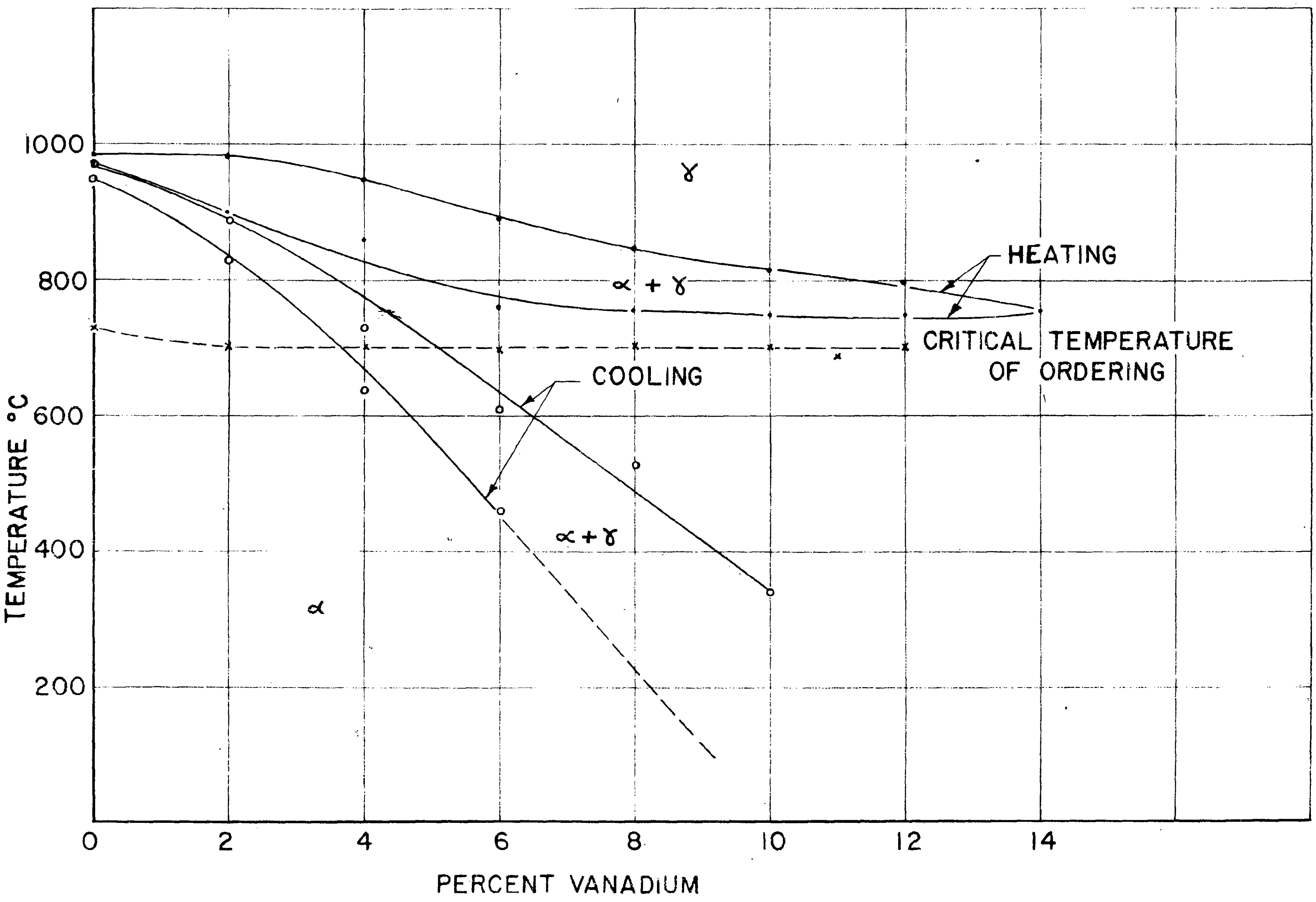
Figure 5. Portion of Thermal Analysis
Curves Obtained in Heating
Alloys Containing 52% Co,
10% - 14% V, Bal. Fe


THERMAL e.m.f (mv-CHROMEL-ALUMEL)
44
42
40
38
36
34
32
30
28
26
24
22
20
815 °C
750 °C
702°C
52Co
38Fe
10 V
812°C
750°C
690°C
52Co
37Fe
11 V
815°C
752°C
704°C
52Co
36Fe
12 V
752°C
706°C
52Co
34Fe
14 V
0
20
40
60
80
0
20
40
60
80
0
20
40
60
80
0
20
40
60
TIME FOR THERMAL e.m.f. TO CHANGE 0.10mv (SECONDS)
TEMPERATURE °C
1000
950
900
850
800
750
700
650
600
550

indicative of a phase transformation and has been interpreted as the beginning of the alpha to gamma transformation in the present work. This interpretation is based upon the work on order-disorder reactions presented by Rhines (17). From the experimental evidence presented, it is concluded that the order-disorder transformation proceeds as a classical Gibbsian phase change and ordering is believed to occur by a process of nucleation and growth. Thus the ordered and disordered structures should be considered as distinct phases in accordance with the requirements of the Phase Rule. Thus, if the ordered and disordered structures are considered as separate phases, it is not unreasonable to expect them to have different transformation temperatures. Therefore, the first peak above the critical temperature of ordering was interpreted as the beginning of the alpha-gamma transformation (most probably the ordered alpha transforming, as short-range order can persist above the critical temperature of ordering) and the second peak as the completion of the alpha-gamma transformation. Another significant feature of these curves is the continued decrease in intensity of the ordering peak as the vanadium content is increased. Although no quantitative relation can be obtained from these curves, the qualitative implications are two. First, the increased vanadium content may directly prevent the formation of the ordered structure by blocking diffusion processes, or second, it may enter into a second phase with the iron and cobalt and thus may disrupt the necessary ratio of Fe/Co to form the ordered structure. The second possibility seems to be more likely as the continued additions of vanadium lower the alpha to gamma transformation indicating that the vanadium is promoting the formation of the gamma phase, until at 14% vanadium the alloy shows a very small transformation peak, indicating that very small amounts of the alpha phase are present.

The transformation temperatures for the Co-Fe-V alloys as determined from the heating and cooling curves are presented in Figure 6. The reactions in the higher vanadium alloys were found to be rather sluggish. For this reason, the heating curves gave the more complete data as the samples consisted of the low temperature phase at the start and the transformations were not suppressed. However, as can be seen from Figure 6, the transformation on cooling is greatly suppressed, and after 8% of vanadium, the alpha to gamma transformation could not be detected with the apparatus available. However, from the heating curves, it can be seen that at 12% vanadium considerable amounts of the alpha phase are still present, but the 14% vanadium alloy is essentially all gamma phase.

Figure 6. Influence of Vanadium on the
Transformation Temperatures of
Co - Fe Alloys


HEATING
CRITICAL TEMPERATURE
OF ORDERING
COOLING
γ
α + γ
α + γ
α
TEMPERATURE °C
1000
800
600
400
200
PERCENT VANADIUM
0
2
4
6
8
10
12
14

X-Ray Diffraction Analysis

X-ray diffraction analysis was used mainly to determine the phase relations of Co-Fe-V alloys heat treated to produce non-equilibrium conditions, in the hope that the results would provide more information regarding the actual mechanism of mechanical and magnetic hardening.

The diffraction studies were carried out using the new General Electric XRD-3 x-ray diffraction apparatus for direct measurement of diffracted intensities. This machine provides a wide range of operating conditions with extremely accurate control of x-ray current and voltage. The x-ray tube is held in a fixed position and the sample is revolved around the primary beam by means of a spectrogoniometer. The spectrogoniometer is a precision mechanical instrument for the direct measurement of the angular positions of all practical x-ray diffraction effects. The instrument functions according to Brentano parafocusing geometry for recording diffraction data in the maximum Bragg angle range from minus 10° through 0° to 180° two theta. The resolution of the diffraction effects varies with accessories and techniques in the range from 0.02 to 0.2°. An x-ray sensitive Geiger-Müller tube is used to detect the diffracted x-rays and to produce electrical impulses whose repetition rate is a measure of the x-ray intensity. The instrument employs a logarithmic counting-rate meter to give a continuous indication of the x-ray intensity, with provisions made for connecting a strip-chart recorder to the rate-meter output, for continuous plotting of the diffraction effects.

The advantages of this x-ray technique can best be realized by a comparison of the results obtained with those obtained by use of film techniques. In the initial stages of the investigation, x-ray patterns were obtained by use of a powder camera (7.16 cm radius) and an unsymmetrical focusing camera. The focusing camera has a radius of 10.03 cm, and thus excellent resolving power. By the use of the spectrogoniometer, complete resolution of the K alpha doublet was obtained in alloys of low vanadium contents, while this resolution could not be obtained by film techniques. In addition, a separation of the (111) gamma and the (110) alpha lines in a 10% vanadium alloy was made only by the spectrogoniometer and not by either of the film techniques. This point is interesting, as the angular separation of these two lines is approximately 0.5 degree.

As was mentioned earlier, the as-sintered bars were forged to 0.050 inch for x-ray diffraction analysis. The samples were solution treated individually by placing them in a stainless steel tube and providing a purified hydrogen atmosphere. The specimens were held for one hour at 1100°C and quenched in oil. When the sample had reached room temperature, the atmosphere was discontinued and the sample was removed from the tube and quenched in liquid nitrogen. Aging was carried out in lead and the specimens were water quenched from the aging temperature. The use of filtered cobalt x-radiation was essential in this investigation.

In the early stages of the diffraction study, it was found that due to the sluggishness of gamma-alpha transformation in the higher vanadium alloys, many samples would appear to be entirely alpha phase, but when etched to remove the surface layers, traces of gamma phase were detected. For this reason, all subsequent samples were etched until no change in the diffraction pattern occurred.

The results of the x-ray diffraction study are reported in Table I. Two main conclusions can be drawn from these data. First, a face centered cubic gamma phase can be precipitated from the body centered cubic alpha phase at temperatures as low as 400°C. Second, ordering in the alpha phase can take place in the temperature range of 600°C to 700°C. This fact was also established by the thermal analysis data. However, the detection of the ordered phase was very difficult and for this reason any change in lattice parameter due to ordering could not be determined.

A portion of the diffraction data of Table I is presented in Table II which also includes the amounts of

TABLE I. RESULTS OF THE X-RAY DIFFRACTION ANALYSES ON Co-Fe-V ALLOYS.

X-Ray Data:

Filtered Co radiation (Fe_2O_3filter). Tube operated at 35 kvp and 9 ma. Spectrogoniometer speed 0.2°/min. - 1° x-ray defining slit - 0.2° x-ray detector slit.

TABLE I

Weight %		Aging Temp. °C	Aging Time Hrs.	Phases	Parameters	
V	Fe				α	γ
0	48	As quenched	0	α	2.858	
		400	8	α	2.857	
		600	8	α	2.858	
2	46	As quenched	0	α	2.858	
		400	8	α	2.856	
		600	8	Ord.* α	2.859	
4	44	As quenched	0	α	2.861	
		400	8	α	2.860	
		600	8	Ord.* $\alpha + \gamma$	2.857	3.572
8	40	As quenched	0	α	2.863	
		400	8	α	2.862	
		600	8	Ord.* $\alpha + \gamma$	2.856	3.568
10	38	As quenched	0	α	2.864	
		400	4	$\alpha + \gamma$	2.860	3.567
		400	8	$\alpha + \gamma$	2.861	3.581
		500	8	$\alpha + \gamma$	2.861	3.567
		600	8	Ord.* $\alpha + \gamma$	2.857	3.575
		700	8	Ord.* $\alpha + \gamma$	2.856	3.573
12	36	As quenched	0	$\alpha + \gamma$	2.862	3.574
		400	8	$\alpha + \gamma$	2.860	3.577
		600	8	$\gamma + \alpha$	2.856	3.574
14	34	As quenched	0	$\gamma + \alpha$	-	3.577
		600	8	$\gamma + \alpha$	2.861	3.578

*Ordered alpha phase

the alpha and gamma phase present as a function of vanadium content and aging temperature. For purposes of discussion these data are also presented in graphical form in Figure 7. From Table II one can see that the additions of vanadium up to 10% to the Co-Fe alloys cause a gradual increase in the lattice constant of the alpha phase in both the as-quenched alloys and those aged at 400°C. This increase is observed to take place until the gamma phase begins to precipitate at which point the lattice constant decreases. This increase of the lattice constant would, of course, be expected, but the sudden decrease of the constant due to the precipitation of gamma indicates that the vanadium is going primarily into the gamma phase. In addition to the change of the lattice constant due to gamma precipitation, additional broadening of the peaks on the diffraction patterns occurred indicating that considerable strain was being introduced into the system due to the gamma precipitate. This broadening was observed to take place even before the gamma phase could be detected. Although no such trend in the lattice constants was observed at the 600°C aging temperature, additional broadening of the diffraction peaks was observed. From Figure 7 the formation of the gamma phase can be followed as it varies with vanadium content and temperature. The presence of more gamma phase in the 12% vanadium alloys

TABLE II. INFLUENCE OF VANADIUM UPON THE PHASE RELATIONS OF Co-Fe ALLOYS AS DETERMINED BY X-RAY DIFFRACTION.

X-Ray Data:

Filtered Co radiation (Fe_2O_3filter). Tube operated at 35 kvp and 9 ma. Spectrogoniometer speed 0.2°/min. - 1° x-ray defining slit - 0.2° x-ray detector slit.

TABLE II

As Quenched

Weight %				Parameters	
V	Fe	% α	% γ	α	γ
0	48	100	0	2.858	-
2	46	100	0	2.858	-
4	44	100	0	2.861	-
8	40	100	0	2.863	-
10	38	100	0	2.864	-
12	36	50.7	49.3	2.862	3.574
14	34	12.3	87.7	-	3.577

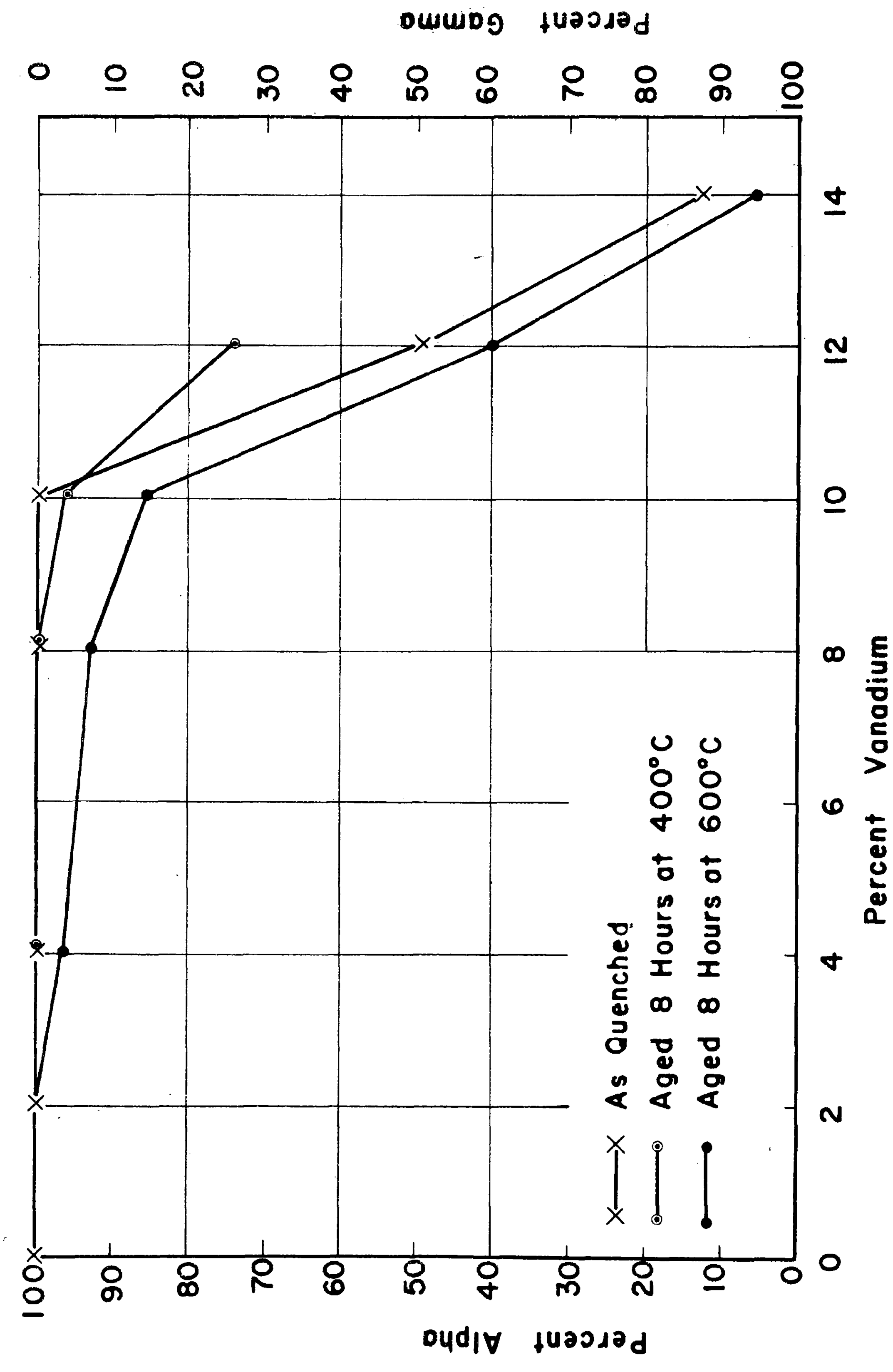
Aged 8 hrs. @ 400°C

0	48	100	0	2.857	-
2	46	100	0	2.856	-
4	44	100	0	2.860	-
8	40	100	0	2.862	-
10	38	96.5	3.5	2.861	3.581
12	36	74.5	25.5	2.860	3.577

Aged 8 hrs. @ 600°C

0	48	100	0	2.858	-
2	46	100	0	2.859	-
4	44	96.5	3.5	2.857	3.572
8	40	93	7	2.856	3.568
10	38	86	14	2.857	3.575
12	36	40.5	59.5	2.856	3.574
14	34	6	94	2.861	3.578

Figure 7. Influence of Vanadium Upon the Phase Relations of Co - Fe Alloys as Determined by X-Ray Diffraction



Percent Alpha
100
90
80
70
60
50
40
30
20
10
0
Percent Gamma
0
10
20
30
40
50
60
70
80
90
100
0
2
4
6
8
10
12
14
Percent Vanadium
As Quenched
Aged 8 Hours at 400°C
Aged 8 Hours at 600°C

as quenched than when aged at 400°C is undoubtedly due to the sluggishness of the gamma-alpha transformation on quenching. When the specimen was aged at 400°C, the retained gamma phase was unstable at this temperature and consequently transformed to the alpha phase. This process is very familiar in the case of tempering alloy steels which contain retained austenite after quenching.

Age Hardening

From the results presented thus far, it has been established that suitable heat treatments can cause the precipitation of a gamma phase as well as ordering in the alpha phase. However, the ordering reaction is confined mainly to the temperature range of 600°C to 700°C for practical heat treatments to develop best magnetic properties. Thus, it seems that two mechanisms are available to explain the hardening characteristics and permanent magnet qualities of this alloy. The precipitation of the gamma phase is certainly a possible explanation as many binary (18) and ternary (19) permanent magnet alloys develop their properties by this process. On the other hand, however, an order-disorder reaction is responsible for the permanent magnet qualities of the well known Co-Pt alloys (20). Thus the problem now created is to separate the effects of these two reactions.

Hardness and Electrical Resistivity Measurements

In an effort to provide an answer to the above mentioned problem, an extensive survey of electrical resistivity and hardness was made on a Co-Fe-V alloy of the Vicalloy I composition. The analysis of the alloy used for this purpose was as follows:

C	Mn	P	S	Si	Co	V	bal.
0.034	0.60	.014	.005	0.32	52.06	9.10	Fe

The material for resistivity measurements was received in the form of hot rolled strip 0.055 inch x 5/8 inch x 5 inches. The resitivity samples were solution treated at 1100°C for one hour, first quenched in oil and subsequently into liquid nitrogen. The samples were aged in lead or salt at temperatures ranging from 300°C to 800°C for varying time periods and water quenched from the aging temperature. The resistivity measurements were made using the well known voltmeter-ammeter technique. In this technique the sample to be measured is made part of an electrical circuit and the voltage drop across the sample is measured with an extremely sensitive voltmeter having a high internal resistance. The technique has been checked thoroughly against known standards and a Kelvin Double Bridge and found to be capable of producing results accurate to within 1%.

The hardness measurements were made on 3/8 inch x 3/8 inch samples cut from a 3/4 inch hot forged bar. These samples were also solution treated at 1100°C for one hour and quenched in oil and liquid nitrogen. Subsequent aging was carried out in lead or salt for varying time periods in the temperature range of 300°C to 800°C. The samples were water quenched from the aging temperatures.

The results of the electrical resistivity and hardness measurements are presented in Figures 8 and 9. These data in conjunction with the x-ray diffraction data permit one to draw some very interesting conclusions. The most striking feature of the resistivity curves is the sudden increase at aging temperatures below 600°C. This occurrence has been observed in many of the more conventional precipitation hardening systems and is associated with the early stages in the nucleation process. As time is increased at temperatures below 600°C, the resistivity begins to decrease, although the hardness is still increasing. These results are not contradictory as a change in electrical resistivity can be caused by slight variation in structure, whereas a macroproperty such as hardness is less subject to slight structural changes. The behavior of the resistivity curves at temperatures of 600°C to 800°C is not quite as clear. For example, the decrease in resistivity at 500°C is undoubtedly due to the coalescence of the gamma precipitate, but at higher temperatures the effect of ordering in the alpha phase may be contributing to the decrease in resistivity. As the temperature is increased to 800°C a sudden reversal is

Figure 8. Influence of Aging Time and Temperature Upon the Electrical Resistivity of Vicalloy I

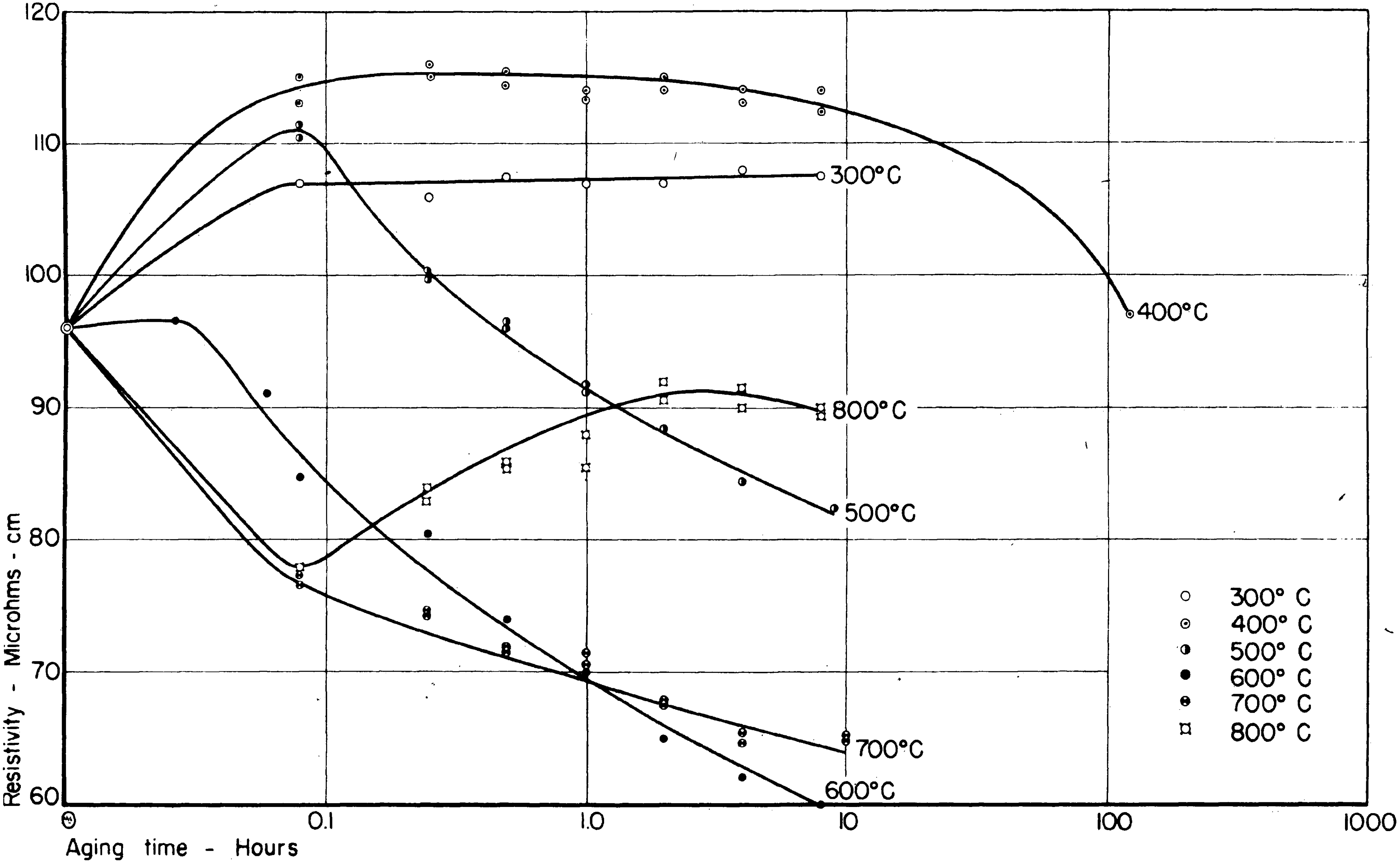
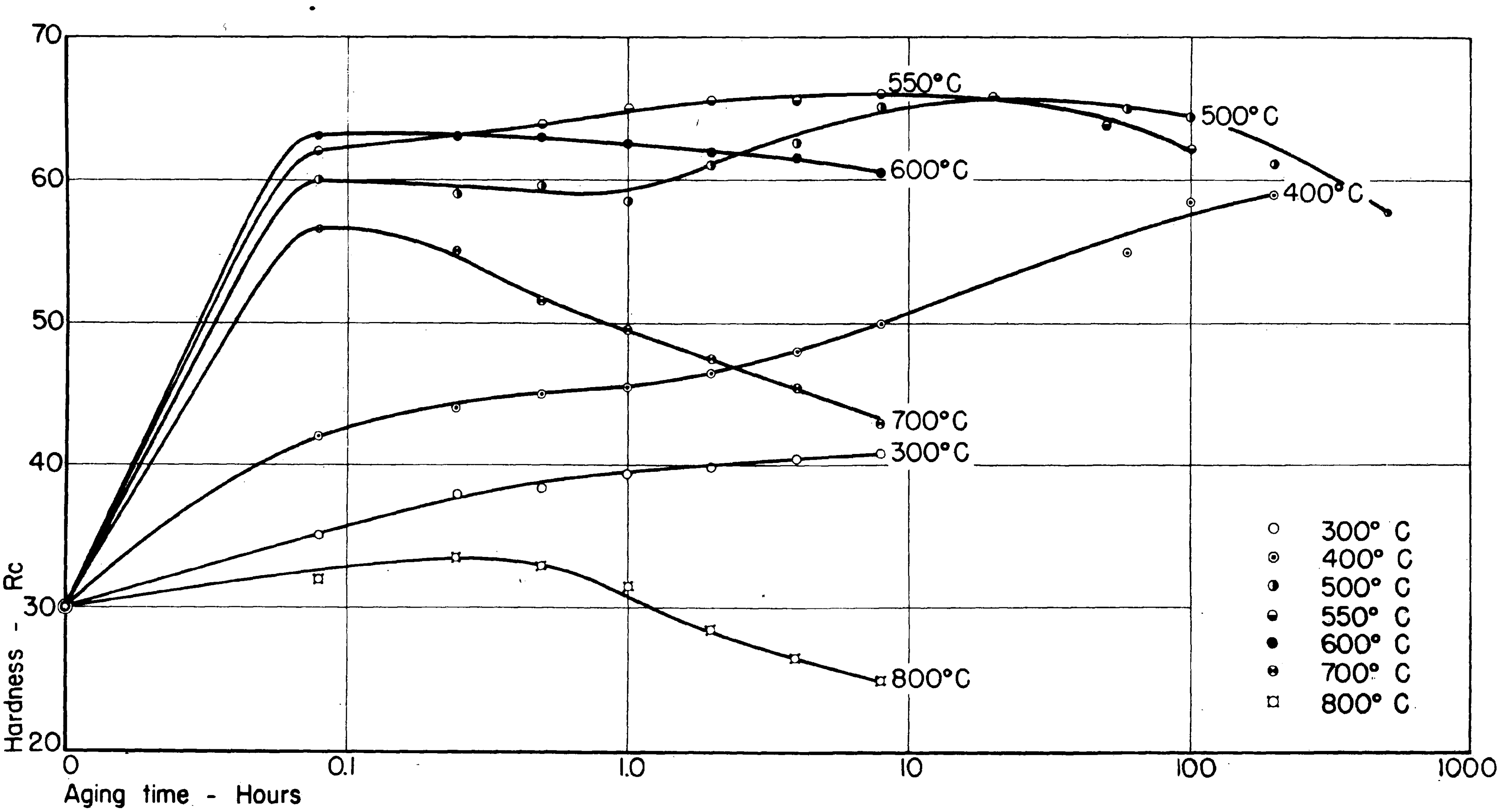

120
110
100
90
80
70
60
Resistivity - Microhms - cm
0
0.1
1.0
10
100
1000
Aging time - Hours
300°C
400°C
800°C
500°C
700°C
600°C
○ 300° C
⊙ 400° C
◐ 500° C
● 600° C
⊕ 700° C
¤ 800° C

Figure 9. Influence of Aging Time and
Temperature Upon the Mechanical
Hardness of Vicalloy I


70
60
50
40
30
20
Hardness - Rc
0
0.1
1.0
10
100
1000
Aging time - Hours
550°C
500°C
600°C
400°C
700°C
300°C
800°C
○ 300° C
⊙ 400° C
◑ 500° C
◒ 550° C
● 600° C
⊕ 700° C
¤ 800° C

observed in the resistivity curve. At temperatures of 800°C, a large quantity of gamma phase will be present and thus one is now obtaining a measure of the resistivity of the gamma phase.

The hardness curves of Figure 9 are very revealing and need little explanation. The sudden rise in hardness at temperatures up to 600°C is almost certainly due to the precipitation of the gamma phase, as at these low temperatures the formation of the ordered phase would not be expected and was not detectable by x-ray diffraction or thermal analysis. Extremely long time periods at these low temperatures are also reported by Geisler and Martin (15) before the ordered phase could be detected by diffraction analysis. These time periods are much longer than the time required to produce the maximum hardness.

The characteristic shape of the curves of 300°C, 400°C, 500°C, and to some extent 550°C suggest the existence of two stages in the precipitation process. The hump of the hardness curves is also observed in many of the common precipitation hardening systems such as Al-Cu. On increasing the aging temperature from 600°C to 800°C the maximum hardness is attained much more rapidly and, as would be expected, the hardness decreases after very short time periods, indicating that overaging is taking place very rapidly.

The change in microstructure of this alloy on aging at 600°C for increasing time periods is shown in Figures 10a - 10d. The metallographic samples were heat treated according to the same cycle used in the hardness tests. All samples were water quenched from the aging temperature. The structure of Figure 10a is for a sample in the as-quenched condition. According to x-ray diffraction analysis, this sample is all alpha phase. The slightly grey areas may be due to a difference in orientation on etching. After eight hours at 600°C (Figure 10b), the block type structure of the as-quenched sample is still basically preserved, although a slight indication of grain boundary precipitation can be observed. On increased times (Figures 10c and 10d) the precipitating phase has completely enveloped the grains and has become quite massive. From Figure 10d, the general intragranular precipitation can be seen. This sample is completely overaged. Thus, from this series of photomicrographs, the course of precipitation of the gamma phase can be observed.

Summary and Conclusions Part I

From the first portion of this investigation several conclusions can be drawn. The existence of a gamma phase precipitate can be detected by x-ray diffraction analysis in samples aged at 400°C to 600°C. At temperatures of 600°C, the first sign of the gamma phase is detected at 4% V. At lower temperatures (400°C) small quantities of the

Figure 10a

Vicalloy I. Solution Treated at 1100°C - 1 hr. Oil and Liquid Nitrogen Quenched. Etched in Aqua Regia + Ferric Chloride + Glycerol. 800X.

Figure 10b

Vicalloy I. Solution Treated at 1100°C - 1 hr. Oil and Liquid Nitrogen Quenched. Aged 8 hrs. at 600°C. Etched in Aqua Regia + Ferric Chloride + Glycerol. 800X.

Figure 10c

Vicalloy I. Solution Treated at 1100°C - 1 hr. Oil and Liquid Nitrogen Quenched. Aged 24 hrs. at 600°C. Etched in Aqua Regia + Ferric Chloride + Glycerol. 800X.

Figure 10d

Vicalloy I. Solution Treated at 1100°C - 1 hr. Oil and Liquid Nitrogen Quenched. Aged 72 hrs. at 600°C. Etched in Aqua Regia + Ferric Chloride + Glycerol. 800X.

gamma phase are detected at 10% V. The existence of ordering in the alpha phase was found to take place in the temperature range of 600°C to 700°C. By electrical resistivity measurements and hardness measurements, it has been established that the gamma phase precipitation is responsible for the mechanical hardening of this alloy in samples aged in the temperature range up to 600°C. In some cases, the maximum hardness obtained is equal to that of fully hardened steel. Above 600°C the precipitation of the gamma phase is thought to be the primary source of mechanical hardening, although a slight contribution may be made by the ordering of the alpha phase. The course of precipitation was also followed by metallographic techniques.

PART II - MAGNETIC PROPERTIES

Introduction

The influence of increasing vanadium content upon the magnetic properties of the 52% Co-Fe alloy prepared by powder metallurgy was determined. These tests were restricted to a single aging temperature of 600°C and an aging time of two hours. However, an extensive survey was made on the influence of aging time and temperature upon the cast Vicalloy I alloy which was used in Part I of this investigation. The effect of aging this alloy in a magnetic field was also determined.

Experimental Procedure

The vanadium series of alloys was prepared using the same processing cycle as that outlined for the thermal analysis samples. In addition, however, the magnetic test samples were hot forged to approximately 0.400 inch square. Subsequently, the samples were solution treated at 1100°C for one hour and quenched in oil and liquid nitrogen. In all cases, the samples were aged in lead at 600°C for two hours and air cooled from the aging temperature. It should also be mentioned that after forging, the density of the powder magnets was 98% of the density of the cast material, and that the magnetic properties were very similar.

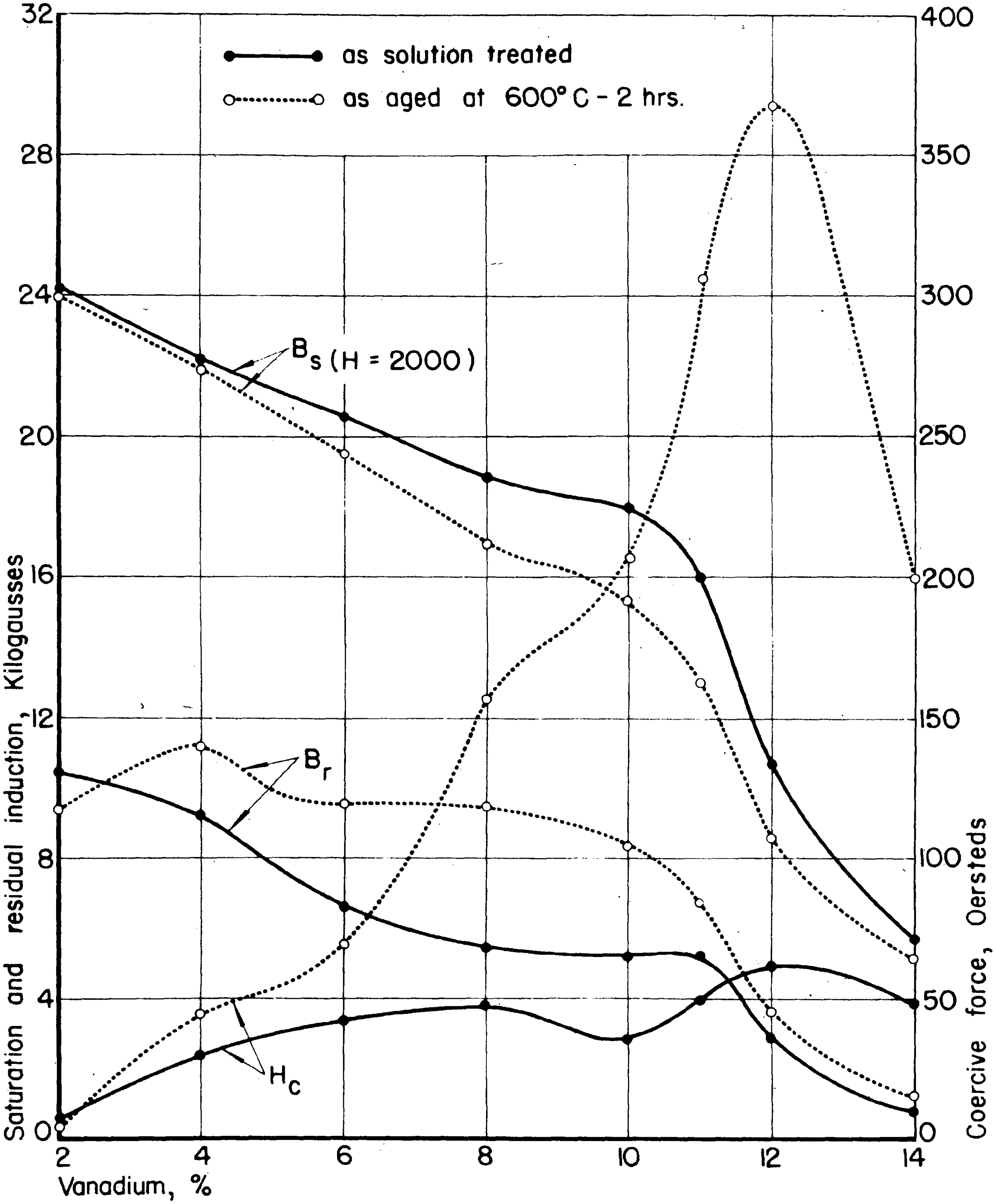
The Vicalloy I material was received in the form of hot forged bars 3/4 inch square. Test samples 1/2 inch square were machined from the as-forged bar and solution treated in the same manner as the powder parts. The test samples were aged at temperatures from 400°C to 800°C for various time periods. The magnetic properties were determined on a General Electric saturation permeameter. All saturation values reported are for H = 2,000 gausses.

Effect of Composition

The influence of increasing vanadium content on the magnetic properties of the 52% Co-Fe alloy in the as-quenched and aged condition is presented in Figure 11. Several interesting conclusions can be drawn from these data. In the as-quenched condition, the saturation induction and residual induction decrease with increasing vanadium content. However, the highest values of saturation and residual induction are observed in the lower vanadium alloys, where the composition is closest to that of the high saturation binary Fe-Co alloys. The coercive force increases slightly with the vanadium addition, and this result would be expected merely from a composition standpoint.

On aging at 600°C, however, a striking change in properties occurs. The coercive force increases rather slowly with increasing vanadium content to 6% vanadium after which it increases very rapidly. The highest values of coercive force are obtained in alloys containing 52% cobalt, 36-38% iron, and 10-12% vanadium. The saturation induction decreases more rapidly in the aged condition then in the quenched condition as vanadium increases. This

Figure 11. Influence of Increasing
Vanadium Content on the
Magnetic Properties of the
52% Co - Fe Alloy


as solution treated
as aged at 600° C - 2 hrs.
B_s (H = 2000)
B_r
H_c
Saturation and residual induction, Kilogausses
0
4
8
12
16
20
24
28
32
Coercive force, Oersteds
0
50
100
150
200
250
300
350
400
2
4
6
8
10
12
14
Vanadium, %

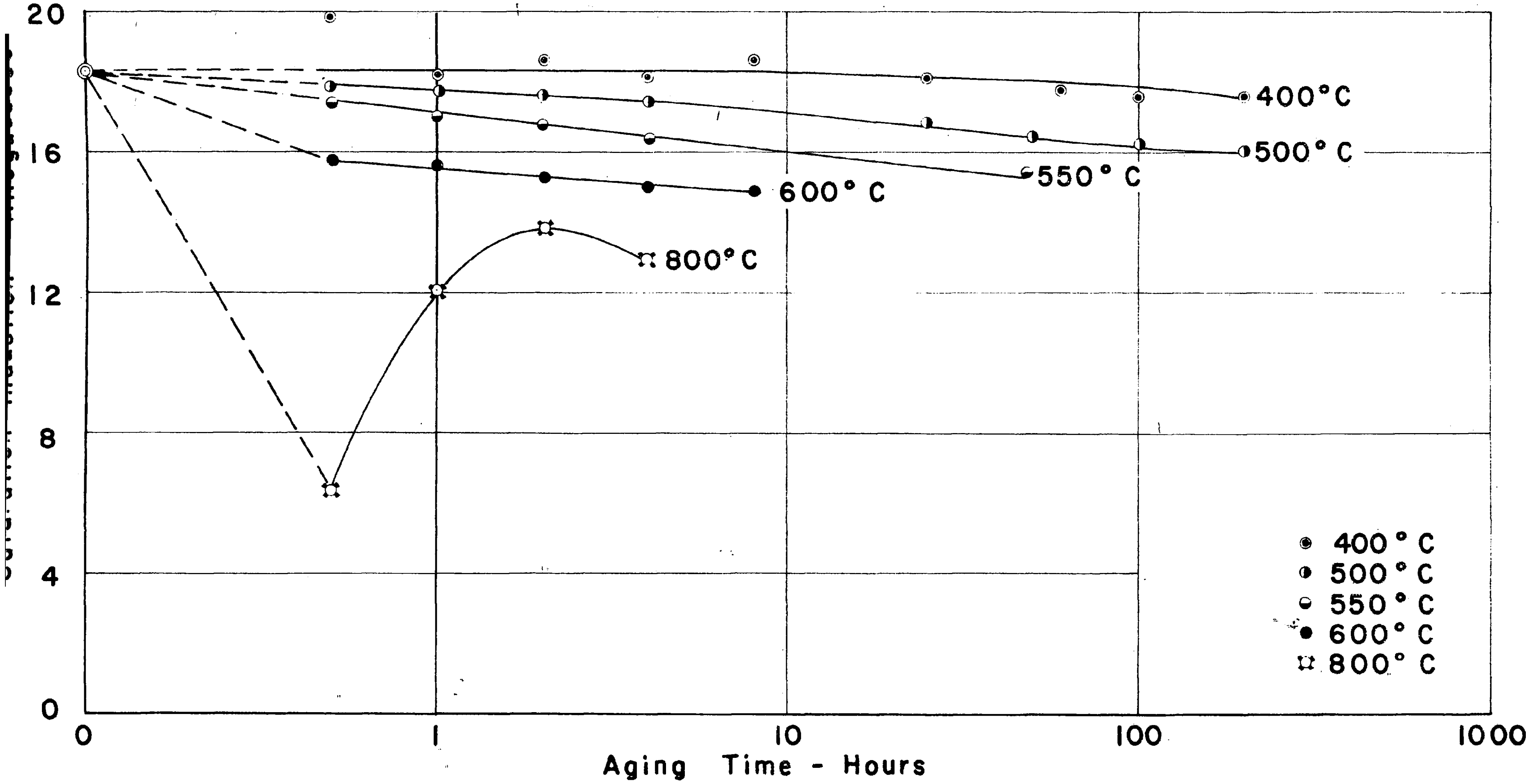
occurrence is to be expected as Nesbitt (10) reports that the gamma phase is non-magnetic, and it was shown in the x-ray diffraction study that the gamma phase began to precipitate at about 4% vanadium. The residual induction values do not follow the same trend after aging as they did in the as-quenched condition. This difference in the values of residual induction is probably associated with a structural change in the alpha phase that will be discussed in greater detail later in the report.

Magnetic Properties of the 10% Vanadium Alloy

The results of the more extensive survey of the magnetic properties of the Vicalloy I alloy are presented in Figures 12-17. The data of Figures 15-17 were obtained on this alloy for aging in the presence of a magnetic field. In all cases the strength of the magnetic field was 1,300 gausses.

The saturation induction values (Figure 12) show a general decrease as a function of time at temperature and aging temperature. The highest values of saturation are obtained at 400°C. This would be expected because at this low temperature the amount of the gamma phase present would be a minimum. The initial low value of saturation at 800°C is probably caused by large quantities of gamma phase precipitated even at these very short aging times. However, the sudden reversal of the saturation induction is unexplainable from the available data. On increasing the

Figure 12. Influence of Aging Time and Temperature Upon the Saturation Induction of Vicalloy I


20
16
12
8
4
0
0
1
10
100
1000
Aging Time - Hours
400°C
500° C
550° C
600° C
800°C
◉ 400° C
◑ 500° C
⊖ 550° C
● 600° C
¤ 800° C

aging time one would expect the saturation to continue to decrease as more and more of the non-magnetic gamma phase formed. One may think at first sight that the reversal is due to some structural change other than the gamma phase formation. However, it must be remembered that saturation induction is one of the magnetic properties that is structure insensitive. The increase may be due to a composition change of the alpha phase.

The data of Figure 13 show the changes in residual induction on aging. The residual induction increases with aging time in the temperature range of 400°C to 550°C. At 600°C the rapid rise and decrease of the residual induction is most probably due to the rapid precipitation of the gamma phase. The influence of ordering in the alpha phase may be playing an important role in the residual induction value for the 600°C aging temperature and for the longer time periods at 500°C and 550°C. This point will be discussed in more detail later. Once again, the behavior of the residual induction on aging at 800°C is not clear on the basis of the existing data.

The values of coercive force obtained on aging are shown in Figure 14. The general increase in coercive force as the aging time and temperature are increased does not seem to correlate with the mechanical hardness values reported earlier. However, this lack of complete correlation is not surprising, as the direct effect of strain on coercive

Figure 13. Influence of Aging Time and Temperature Upon the Residual Induction of Vicalloy I

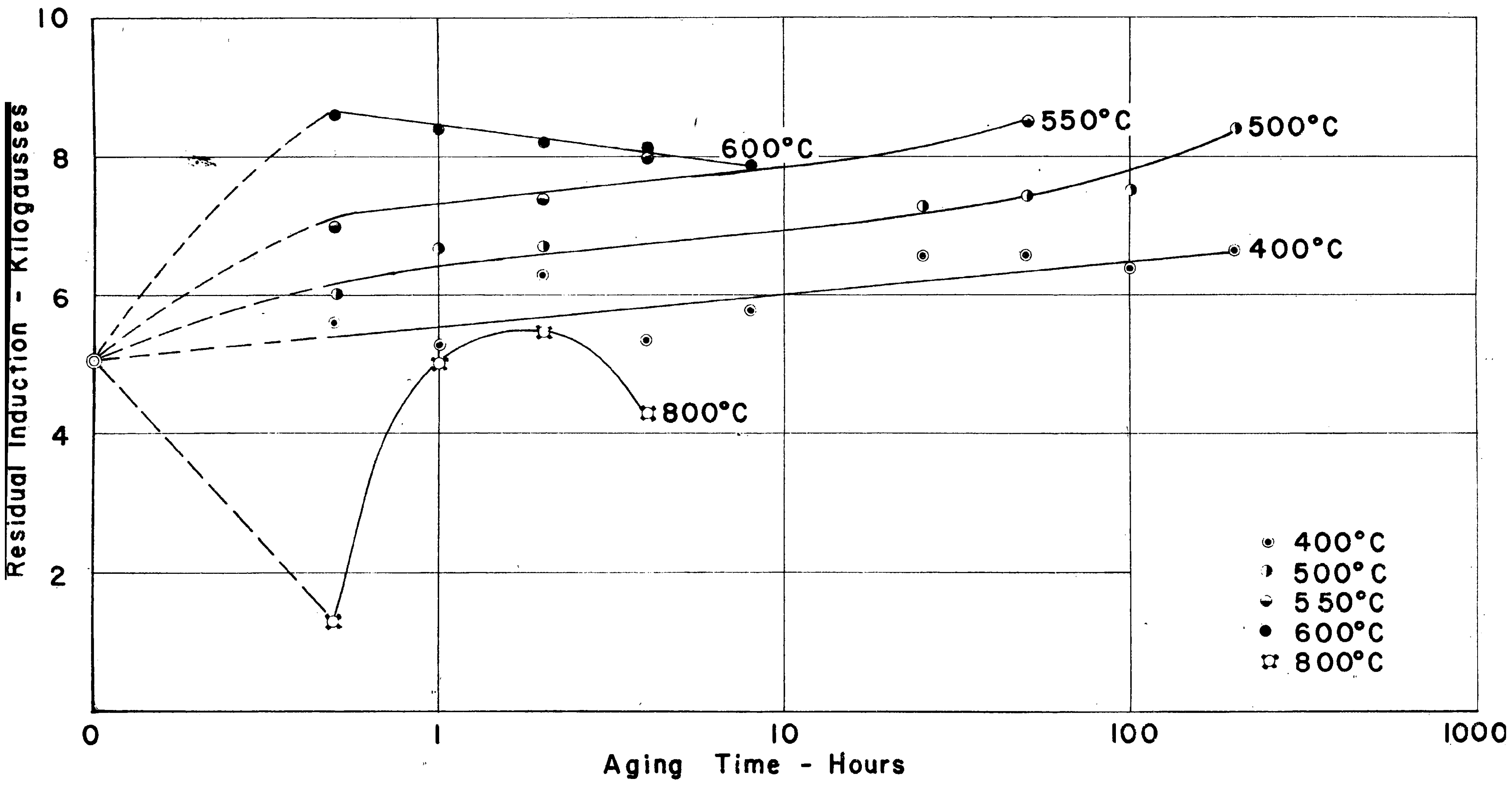
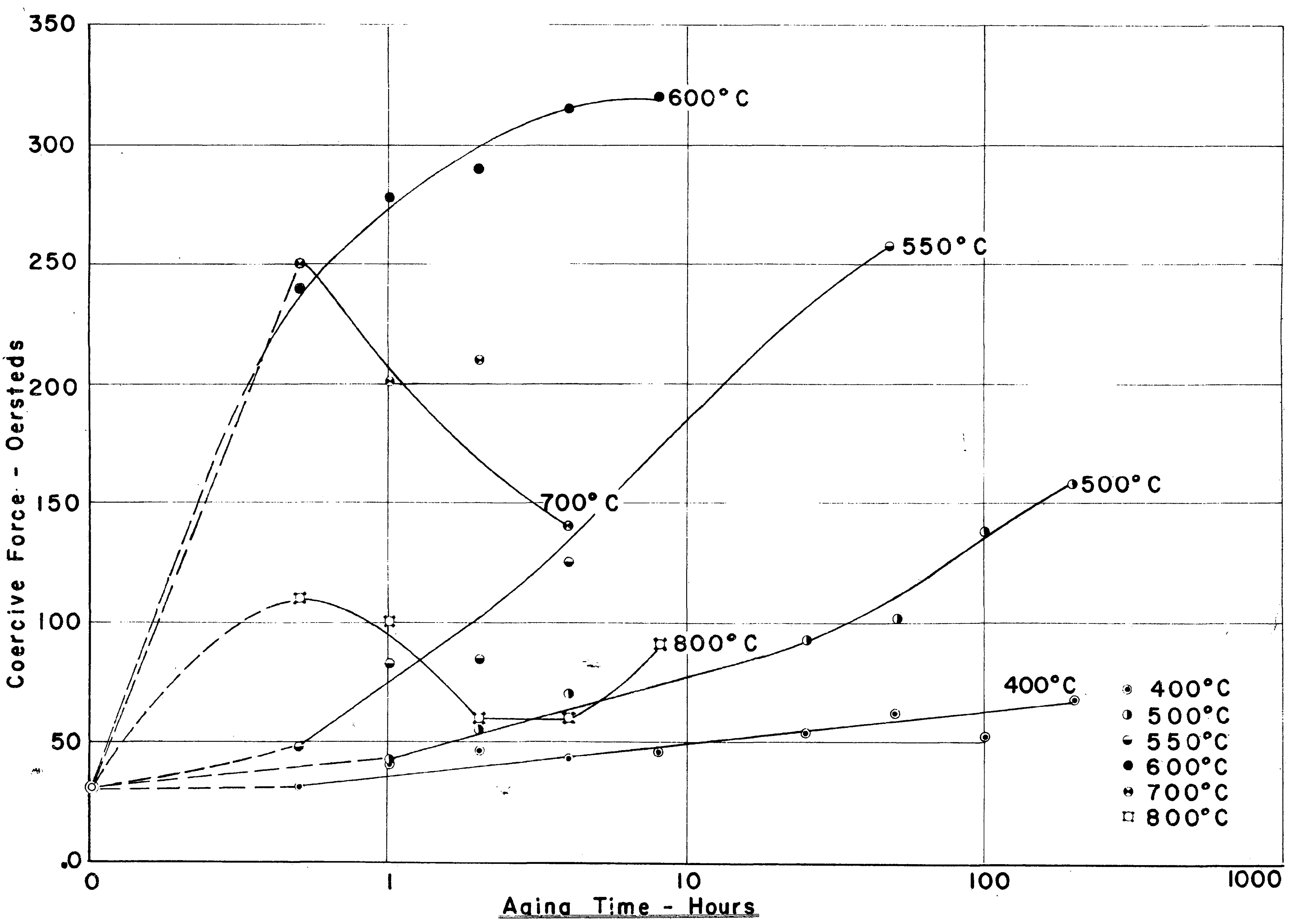

10
8
6
4
2
Residual Induction - Kilogausses
0
1
10
100
1000
Aging Time - Hours
600°C
550°C
500°C
400°C
800°C
400°C
500°C
550°C
600°C
800°C

Figure 14. Influence of Aging Time and Temperature Upon the Coercive Force of Vicalloy I


350
300
250
200
150
100
50
.0
0
1
10
100
1000
Coercive Force - Oersteds
Aging Time - Hours
600° C
550° C
500° C
700° C
800° C
400°C
◉ 400°C
◑ 500°C
⊖ 550°C
● 600°C
⊗ 700°C
⌑ 800°C

force is somewhat different from its effect on mechanical hardness. The general lag of coercive force behind the mechanical hardness is observed in other precipitation hardening systems (19). The most interesting point observed from these data is the increase in the coercivity of the longer aging times at 500°C to 550°C, and, of course, the shorter time periods at 600°C, 700°C, and 800°C. However, the best balance of magnetic properties was obtained on aging at 600°C for time periods of two to four hours.

Magnetic Properties of Vicalloy I Aged in a Magnetic Field

The effect of aging this alloy in the presence of a magnetic field is presented in Figures 15-17. It was hoped that this treatment would result in an increase in the residual induction without any loss in coercive force. However, from the data presented, it can be seen that the magnetic field had very little influence on the residual induction. One significant difference between the results obtained by aging in a magnetic field and those without the field is that the presence of the magnetic field seems to have accelerated the reactions leading to the improvement in the magnetic properties of the alloy. The failure of this alloy to respond favorably to the magnetic heat treatments is probably associated with the presence of the nonmagnetic phase and the rather low magnetostriction of this alloy (21). It has been found quite generally in the case

Figure 15. Influence of Aging Time and Temperature Upon the Saturation Induction of Vicalloy I Aged in a Magnetic Field

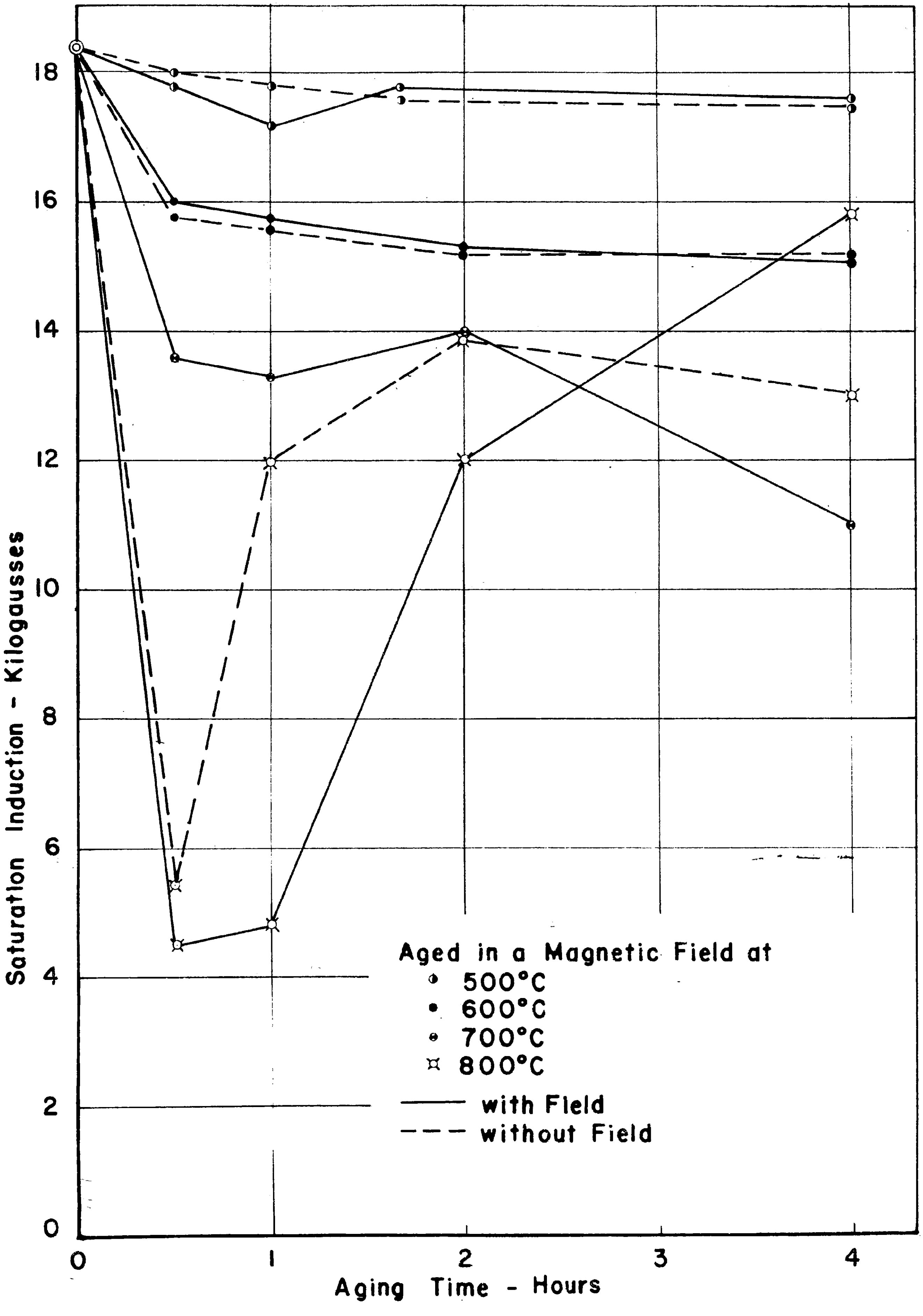

Saturation Induction - Kilogausses
0
2
4
6
8
10
12
14
16
18
Aging Time - Hours
0
1
2
3
4
Aged in a Magnetic Field at
◑ 500°C
● 600°C
⊖ 700°C
¤ 800°C
—— with Field
– – – without Field

Figure 16. Influence of Aging Time and Temperature Upon the Residual Induction of Vicalloy I Aged in a Magnetic Field

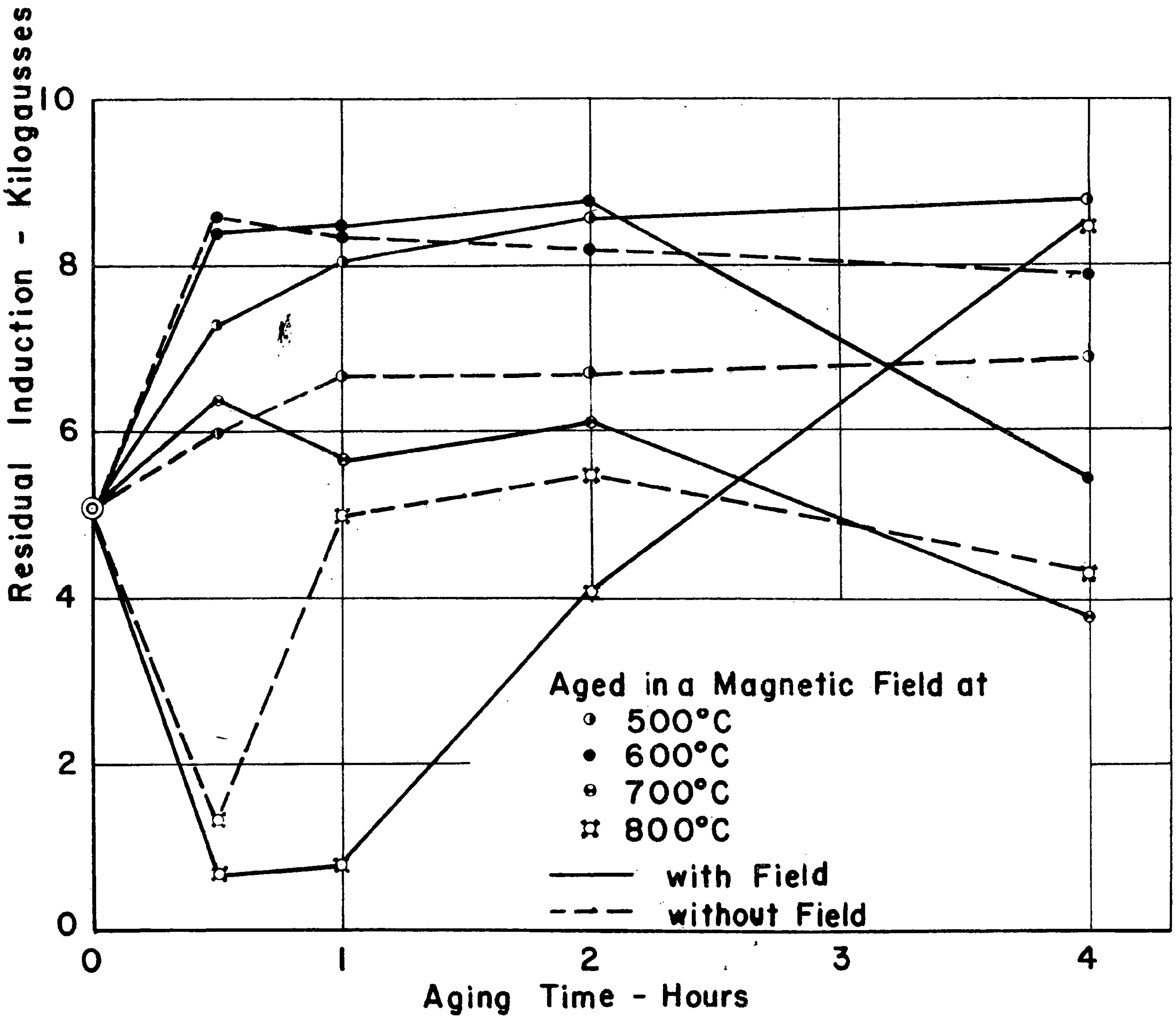
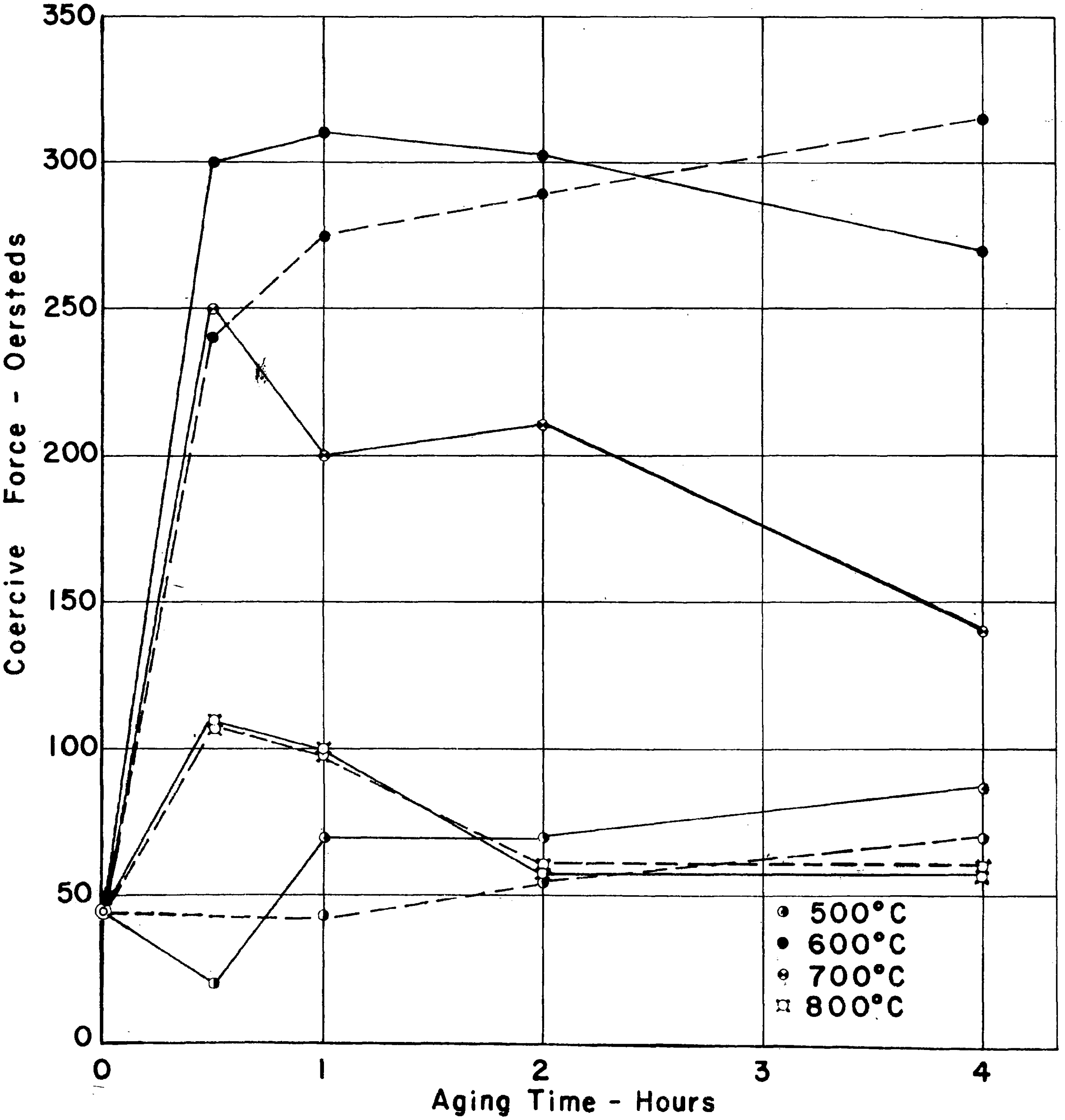

Residual Induction - Kilogausses
10
8
6
4
2
0
0
1
2
3
4
Aging Time - Hours
Aged in a Magnetic Field at
◑ 500°C
● 600°C
⊖ 700°C
¤ 800°C
—— with Field
- - - without Field

Figure 17. Influence of Aging Time and Temperature Upon the Coercive Force of Vicalloy I Aged in a Magnetic Field



350
300
250
200
150
100
50
0
Coercive Force - Oersteds
0
1
2
3
4
Aging Time - Hours
◐ 500°C
● 600°C
⊖ 700°C
¤ 800°C

of soft magnetic materials, that the presence of a second phase inhibits their response to magnetic annealing heat treatments. However, sufficient data are not available to explain why these results are obtained.

Correlation of Structure with Magnetic Properties During Aging

Correlation of the x-ray diffraction and thermal analysis data with the magnetic properties of this alloy should allow the mechanism of magnetic hardening to be identified.

The influence of the formation of gamma phase on the saturation values is quite clear. The presence of the gamma phase results in a decrease in the saturation induction due to its non-magnetic character. It is also possible that the precipitation of this phase may produce a composition change in the magnetic alpha phase, which detracts from its magnetic properties, thus aiding in the decrease in saturation induction.

The increase in residual induction with aging time and temperature is almost certainly due to gamma phase precipitation and possibly ordering in the alpha phase at the higher temperatures. One would expect that the precipitation of the non-magnetic gamma phase would result in a decrease in residual induction similar to the decrease observed in the saturation induction. However, it should be remembered that residual induction is a structure sensitive property. Therefore, it appears that the strain accompanying the gamma phase precipitation is resulting in a more favorable domain structure, so that during magnetization a greater degree of domain orientation in the direction of the applied field is obtained. At the higher aging temperature (600°C), ordering in the alpha phase probably contributes to this mechanism, resulting in the very rapid initial increase in the residual induction. As the aging time at 600°C increases, the gamma phase begins to coalesce and thus lose its efficiency and merely acts to decrease the residual induction by its non-magnetic character.

In regard to the high values of coercive force obtained on aging, the presence of the gamma phase seems to account for this property while ordering in the alpha phase is probably only of secondary importance. The high coercive force may not be a direct result of the gamma phase formation, but a result of its effect on the alpha phase. The precipitation of the gamma phase in the grain boundaries and along certain crystallographic planes of the magnetic alpha phase, may partially break up the alpha phase into small isolated particles. According to a theoretical treatment of this subject by Stoner and Wohlfarth (22), the presence of such particles of the proper size and shape, can result in the production of coercive force values of several thousand oersteds. In this explanation, the term "isolated" is used in the sense that there is sufficient magnetic discontinuity to prevent common boundary formation and movement so that two or more particles do not form a unit. It is not necessary that each particle be completely surrounded by another phase. The precipitation of the non-magnetic phase in the alloy would separate the particles of the alpha phase, in the sense discussed, and partially fulfill this requirement. However, the values of coercive force obtained in this alloy are in the neighborhood of 300 oersteds, indicating that the primary factor involved by the gamma precipitate is strain (strain anisotropy), although the particle shape (shape anisotropy) is almost certainly a contributing factor. In this connection, the strain resulting from ordering of the alpha phase is probably contributing to the coercive force also. Thus, the precipitation of the gamma phase apparently explains the magnetic hardening in this alloy if the fine particle theory of coercive force is considered.

Summary and Conclusions Part II

From the second portion of this investigation several conclusions can be drawn. The addition of vanadium to Co-Fe alloys lowers the saturation and residual induction and raises the coercive force in the as-quenched condition. On aging at 600°C, the residual induction and coercive force are considerably increased. The highest values of coercive force are obtained in alloys containing 52% cobalt, 36-38% Fe, and 10-12% vanadium. The results of the more extensive survey on the Vicalloy I alloy indicate that the precipitation of the gamma phase is primarily responsible for the increases observed in residual induction and coercive force. However, ordering of the alpha phase may be contributing to the improvement in magnetic properties. The presence of the gamma phase also lowers the saturation induction.

Aging the Vicalloy I alloy in a magnetic field had very little influence on the magnetic properties. The only significant result was that the presence of the magnetic field appears to increase the rate of precipitation of gamma phase.

GENERAL CONCLUSIONS

The results of this investigation may be summarized as follows:

1. Increasing vanadium additions to Co-Fe alloys lower the alpha to gamma transformation, thus promoting the formation of the non-magnetic gamma phase. The vanadium additions also suppress the ordering reaction.

2. Precipitation of the gamma phase is primarily responsible for the mechanical hardening in this system. Above 600°C it is felt that ordering in the alpha phase may be contributing to the mechanical hardening.

3. Increasing vanadium additions to Co-Fe alloys lowers the saturation and residual induction and increases the coercive force. The highest values of coercive force were found in alloys containing 52% Co, 36-38% Fe, and 10-12 V.

4. The increase in residual induction of the Vicalloy I alloy on aging was attributed to the production of a more favorable domain structure as a result of the strain accompanying the gamma phase precipitation.

5. On the basis of the fine particle theory of coercive force, the high values of coercive force reported for this alloy were associated with strain anisotropy of the magnetic alpha phase produced by the gamma phase precipitate and ordering in the alpha phase.

6. Heat treatment of Vicalloy I in a magnetic field was found to have very little influence on the magnetic properties.

SUGGESTION FOR ADDITIONAL WORK

From the results of the present investigation, three suggestions can be made for further research. These are as follows:

1. A more extensive study of the Co-Fe-V alloys to develop a more complete and better understanding of the constitution of the system.

2. Further x-ray diffraction analysis to establish, if possible, the chemical composition of the alpha and gamma phases, and the structure of the ordered phase. This work would aid tremendously in providing a better understanding of the magnetic properties of the Co-Fe-V alloys.

3. A more detailed investigation of the Co-Fe alloys containing from 2%-6% of vanadium. In this composition range the alloys still possess reasonably high saturation and residual inductions. The practical possibilities of such an investigation are two. First, in this alloy range a favorable response to magnetic heat treatments may be obtained resulting in the production of permanent magnets with higher residual induction values than are now available; and second, the addition of a fourth element might produce an increase in the coercive force with a minimum loss in residual induction, thus providing an interesting class of new permanent magnet alloys.

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