THE TEMPERING OF HIGH CARBON HIGH CHROMIUM STEELS

by

OTTO ZMESKAL

B. S. Armour Institute of Technology

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Signature of Author Otto Zmeskal

Department of Metallurgy, September 1941

Signature of Professor
in Charge of Research Morris Cohen

Signature of Chairman of Department
Committee on Graduate Students Robert S. Williams

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I

INTRODUCTION

Hardened high-carbon high chromium steels contain retained austenite. The transformation of this retained austenite by tempering has a marked effect on the properties of these steels. Since these steels are mostly used for dies, a very important consideration in their tempering is dimensional change. This phenomenon is very markedly affected by the austenite transformation.

The investigation will be divided into two parts. One part will be the study of the austenite transformation as a function of time at tempering temperature by means of the accompanying magnetic changes. The second part will be a study of the kinetics of the transformation using magnetic, dilatometric, microscopic, and X-ray measurements.

II

SUMMARY

The tempering of two hardened high-carbon high-chromium die steels was studied by means of magnetic, dilatometric, X-ray and microscopic measurements.

One steel, to be called henceforth 1% C - 5% Cr, contained 1.00 per cent carbon, 5.31 per cent chromium and 1.13 per cent molybdenum.

The other steel, to be called henceforth the 1.5% C - 11.5% Cr steel, contained 1.60 per cent carbon, 11.95 per cent chromium and 0.79 per cent molybdenum.

These steels have many uses: The 1.5% C - 11.5% Cr steel is used mostly for cold-forming dies. The 1% C - 5% Cr steel is also used for dies, but its applications cover a wider field; gauges, master tools, machine ways, broaches, mandrels, shear blades, slitting cutters, and burnishing tools are typical applications.

A magnetic apparatus based upon the ballistic principle was constructed to allow measurement of the amount of non-magnetics at any temperature up to 1300° F. A dilatometer was introduced into the apparatus to allow simultaneous measurements of length and magnetic changes.

The seemingly different magnetization versus tempering temperature curves of previous investigators have been correlated and shown to be dependent upon the field used. The course of a magnetization versus

tempering temperature curve has been determined in relation to the structural changes appearing in the steel.

Retained-austenite transformation curves have been drawn for the two steels for three cooling rates from three hardening temperatures. These have disclosed a highly reactive range in the vicinity of 500 - 600° F. for retained-austenite transformation in the 1% C - 5% Cr steel. In this temperature range the transformation occurred mainly at temperature. In the higher temperatures the transformation took place mainly during cooling from temperature.

Depending upon the time at temperature, the retained austenite may be made to transform during cooling in one or two stages. The first stage appeared below 600° F. and the other below 250° F. When the retained austenite transformed at 250° F. tetragonal martensite was produced.

The retained austenite content was progressively greater in the oil-, air-, and lime-cooled specimens for the usual hardening temperatures. The austenite in the more slowly-cooled specimens transformed more rapidly in the beginning but did not reach complete transformation as quickly as did the austenite in the more-rapidly-cooled specimens.

The retained austenite increased both in amount and in stability with hardening temperature for the two steels. Above 2000° F. the retained austenite decreased in the 1% C - 5% Cr steel. Above 2000° F. the 1.5% C - 11.5% Cr steel became non-magnetic, and was composed of austenite and carbides.

The processes that the hardened steels were found to undergo during a heat to the tempering temperature at the rate of 5° F. per minute are:

1. Tempering of the tetragonal martensite. This started at 212° F., but was most rapid at 350° F. It was not completed until a temperature of 500° F. was attained.

2. Micro-stress relief. This started at 350° F. and was completed at 500° F.

3. Precipitation of complex carbides from martensite. This started at 450° F. and was completed at 550° F.

The above processes were common to both steels for all quenches except that which produced a martensite-free condition.

If only the 1% C - 5% Cr steel was considered in the states as-hardened from 1700 to 1800° F., one more phenomenon ensued during tempering. That was:

4. Transformation of retained austenite - this started at 550° F. and was complete at 650° F.

The stability of the austenite primarily influenced its behavior upon tempering. The 1700° F. and the 1800° F. hardening treatments for the 1% C 5% Cr steel resulted in austenite that was highly reactive at 500 - 600° F. The 2000° F. treatment for the same steel resulted in austenite that could only transform to a low extent in this range. The 1700° F. hardening treatment for the 1.5% C - 11.5% Cr steel produced some reactive austenite. The 1800° F. and 2000° F. treatments for this steel resulted in very stable austenite, which required temperatures above 900° F. for transformation.

III

DISCUSSION OF PREVIOUS WORK

A. Chromium Steels

Despite the fact that two books(69)(89) have been written about chromium steels, the information on high-carbon high-chromium steels is relatively slight in extent. Bain(8) determined the relations existing between the hardness values and the quenching and tempering temperatures of four steels, ranging from 1.70 to 2.03 per cent carbon and 1.37 to 15.65 per cent chromium. He found marked secondary hardening in the higher-chromium steels. Gill(21) carried out a similar investigation for a greater variety of steels. Further data was presented by Wills(57)(71). He found that long time (3 hours) heating at the quenching temperature resulted in lower impact values.

The phase diagrams of the iron-chromium-carbon system have been worked out by Tofaute, Sponheuer and Bennek(54) and by Westgren, Phragmen and Negresco(18). The former were also able, by their combination of data on magnetic saturation and hardness, to distinguish in these steels between precipitation hardening and hardening due to austenite transformation.

The transformations occuring during air-cooling from the hardening temperature were followed magnetically by Murakami and Hatta(53) for steel containing from 1.91 to 2.71 per cent carbon and from 1.56 to

14.35 per cent chromium. The lower-chromium steels displayed both Ar' and Ar" transformations, while only the Ar" transformation was found in the high-chromium steels. Similar results were obtained by Murakami and Kishimoto(62).

Groz(80) claimed that the hardening produced by tempering a cast steel containing 1.47 per cent carbon and 11.81 per cent chromium was due mainly to precipitation hardening. A 24-hour temper at 870° C. (1598° F.) resulted in a two-stage transformation of the austenite upon cooling, while shorter times resulted only in an Ar" transformation.

The parameter relationships in quenched steels of medium-chromium contents have been determined by Nishiyama(61). The magnetic properties have been summarized by Von Auwers(72).

Austenite transformation occurring during the hot-quenching of chromium steels have been elucidated by the work of Steinberg and his coworkers(70)(67)(49). The tempering of these steels received only slight attention.

The steels used as the subject of this thesis investigation have been comparatively recently developed. The dimensional changes on hardening and tempering have been investigated by Scott and Gray(90). White(93) has also investigated the dimensional changes in hardening and tempering a steel of the 1.5% C - 11.5% Cr (with 1% Mo) type.

B. The Transformation of Retained Austenite by Tempering

The phenomenon of secondary hardness being developed upon tempering quenched high-alloy steels is one that has been the object of considerable study. The explanations proposed by Houdremont, Bennek, and Schröder(40) suggested that although the increase in hardness on the first draw of quenched high-carbon vanadium-bearing steels was due to austenite decomposition, the retention of hardness on repeated draws could only be attributable to the extreme difficulty of carbide precipitation in these steels. They measured the magnetic saturation, at room temperature, of low-carbon steels containing vanadium and found no increase in magnetization with tempering although an increase in hardness was observed.

Eilender, Klinar and Carnelius(39) studied a series of high-speed steels by means of magnetic and hardness measurements with the purpose of determining the cause of secondary hardness in these steels. All of the steels showed an increase in hardness upon reheating to the range of 500 - 600° C. (932 - 1112° F.) after quenching--only the high-carbon ones, however, (1 per cent compared to 0.3 per cent C) showed corresponding increases in the magnetic saturation upon tempering in the range 500 - 600° C. (932 - 1112° F.) after quenching. These steels were then studied in a magnetic apparatus designed by Esser and Oberhoffer(12) which allowed measurements of the magnetization as the samples were heated-to, held-at, and cooled-from the tempering temperature.

Two of their curves are reproduced in Figures 1 and 2.

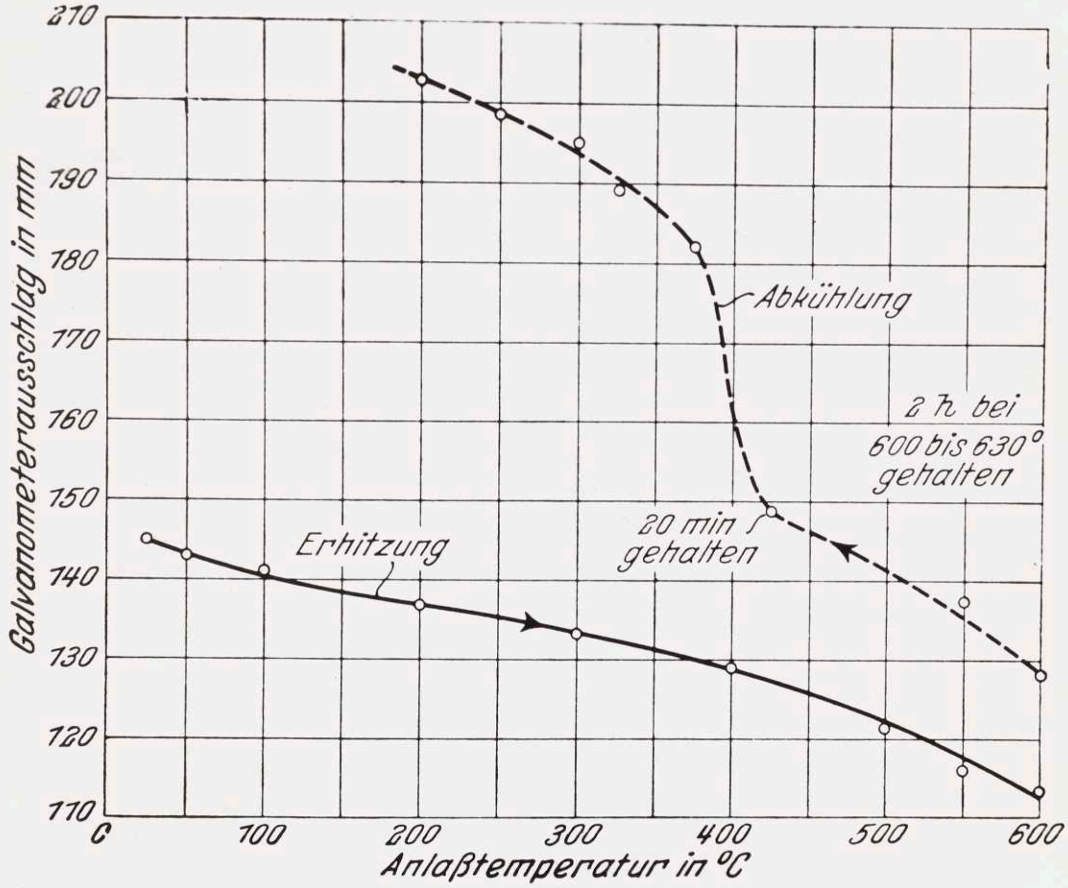

FIGURE 1. Heating and cooling (magnetization) curves of hardened high-speed steel containing 1% C.

Applied Field - 10,000 gauss.

(Eilender, Klinar and Carnelius)

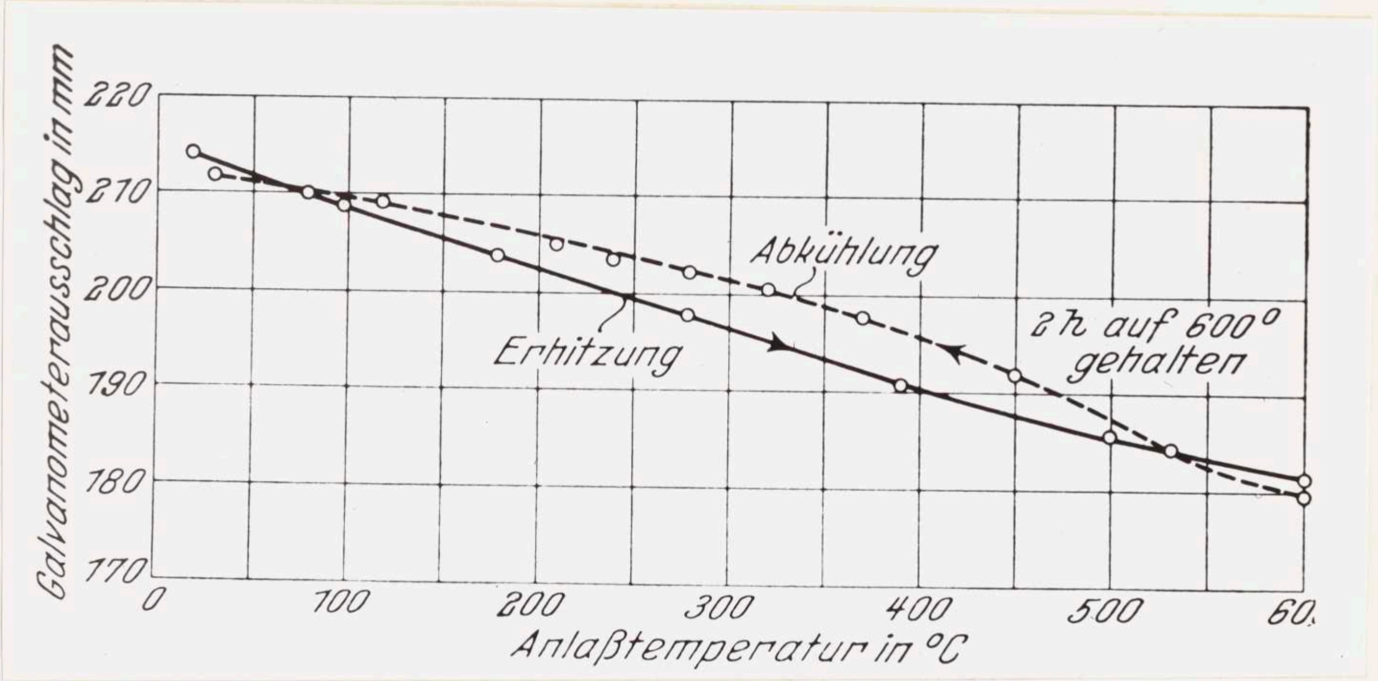

FIGURE 2. Heating and cooling (magnetization) curves of hardened high-speed steel containing 0.3% C.

Applied Field - 10,000 gauss.

(Eilender, Klinar and Cornelius)

These curves show that one steel (Figure 1) had retained austenite which was transformed by the tempering treatment and that the hardness increase upon tempering was due to this transformation. The low-carbon high-speed steel (Figure 2) experienced no change in magnetization due to the tempering process. The investigators concluded that hardness in the low-carbon steel must have been produced by a dispersion-hardening process.

It is to be noted in Figure 1 that some of the austenite transformed isothermally (not quite, since there was a 30° C. (86° F.) variation in the holding temperature) and a considerable amount transformed during cooling, in the range from 300 - 450° C. (572 - 842° F.). The explanation given was that precipitation of carbides at the elevated tempering temperature so reduced the stability of the retained austenite that the suppressed Ar" transformation reasserted itself during the subsequent cooling. Similar results were reported by Ehmcke(29). He studied the tempering of a high-speed steel, containing 3 per cent nickel, with the aid of a dilatometer. His results showed that in this steel the transformation of the retained austenite occurred during cooling.

No evidence was given in the paper by Eilender, Klinar, and Cornelius concerning the completeness of the transformation, that is, whether a retempering would have caused additional austenite transformation. It is likely, that if the steel had been continuously cooled and had not been held for the 20-minute period at 425° C. (897° F.), the transformation on cooling would not have been as marked as the curve of Figure 1 indicated.

They also included a dilatometer curve of a quenched high-speed steel that had been held at 600° C. (1112° F.) for two hours and then cooled. No transformation was indicated until the cooling ensued, and it occurred in the range 300 - 350° C. (572 - 662° F.).

It is interesting to compare the results of similar magnetization versus temperature measurements made on plain-carbon steels with the above. Such information is available in a paper by Esser and Momm(50)*. The apparatus used was a modification of the apparatus used by Eilender, Klinar and Cornelius(39) for their measurements of the intensity of magnetization at room temperature and was originally designed by Stäblein and Schroeter(17). The modification allowed measurements to be made at temperature. Saturation-magnetization measurements were made since the magnet applied a field of 8000 gauss. A curve from their paper is reproduced in Figure 3.

The evidence presented indicated that all the retained austenite transformed on the first heating, in the range from 230 - 280° C. (446 - 536° F.).

It is also to be noted that a pronounced carbide curie point was not evident on the first cooling, but became apparent on the second cooling from 600° C. (1112° F.). This was attributed to the coagulation of the carbides, produced by the second heating, to the size where upon their magnetization intensity became large enough to be measured by the instruement used for the investigation.

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* Additional data are available in papers by Mikami and by Ellinger, which will be discussed in the next section.

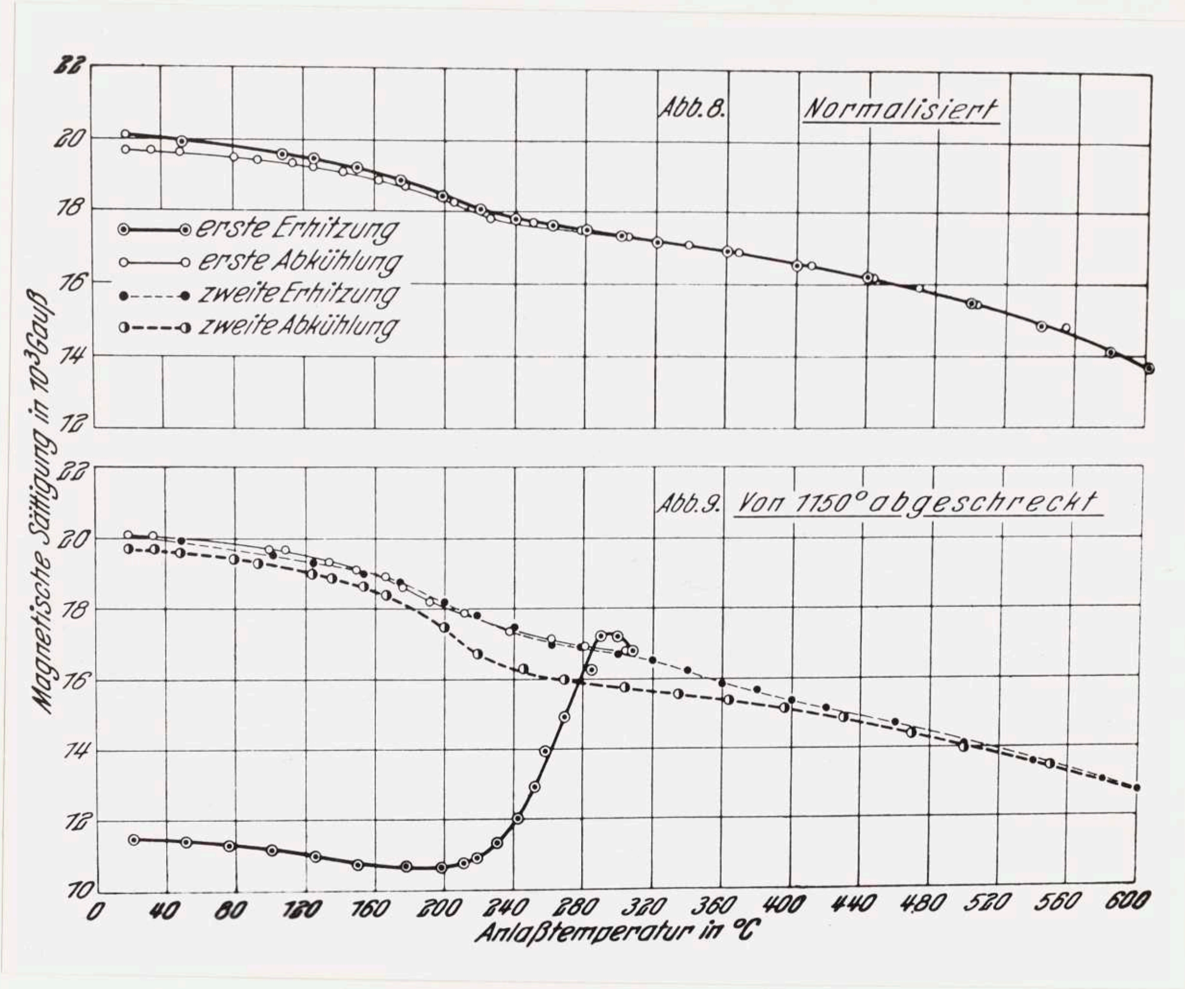


FIGURE 3. Heating and cooling (magnetization) curves of 1.75 % C steel heat-treated as shown.

Applied Field - 8000 gauss.

(Esser and Momm)

That transformation of retained austenite upon reheating is not restricted to plain-carbon steels is evident from the curves shown in Figure 4 from the same paper. The heating and cooling (magnetization) curves of a steel containing 2 per cent carbon and 2 per cent manganese water-quenched from 1120° C. (2048° F.) are given. The transformation existed over a much smaller range of temperatures in this steel than in the plain-carbon steel.

A point to be observed here is that the magnetization values of the specimens were higher immediately after the transformation had been completed on heating than they were at the same temperature during subsequent cooling.

From the data presented above the conclusion may be drawn that high-alloy contents, such as present in high-speed steel, are necessary to stabilize the retained austenite during the heating period to the tempering temperature.

The fact that during tempering the austenite in high-alloy steel transformed only upon cooling, provided that the steel was not held a long time at the tempering temperature and that the latter was not too high, has also been shown by other investigators. Sasagawa(11) found that a 15-minute temper of hardened high-speed steel at 600° C. (1112° F.) produced no transformation at temperature and that it ensued only after cooling. He found that the presence of cobalt made this transformation much more marked.

A detailed dilatometric study of the tempering phenomena in high-speed steel was made by Michel and Benazet(38). The results of

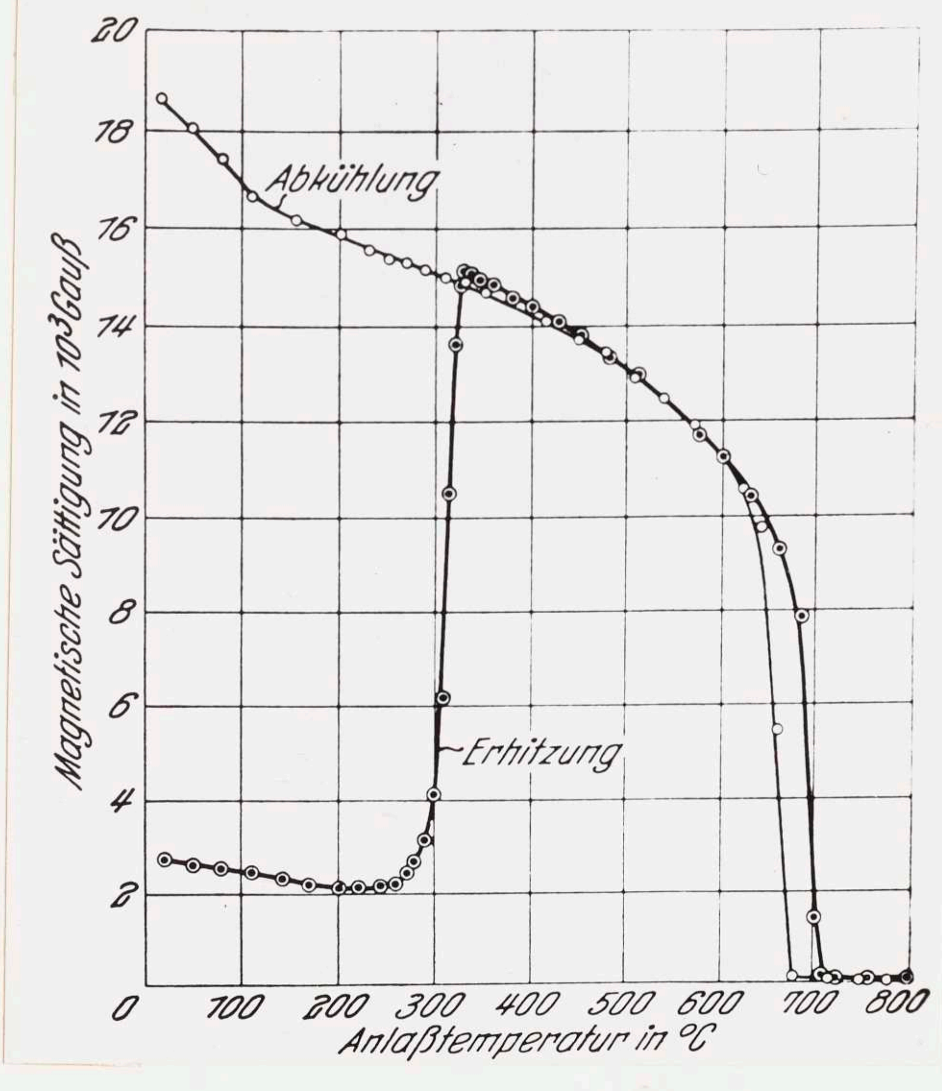


FIGURE 4. Heating and cooling (magnetization) curves of a 2% C - 2% Mn steel, water-quenched from 1120° C. (2048° F.)

Applied Field - 8000 gauss.

(Esser and Momm)

Sasagawa were substantiated. They found that multiple tempering produced more transformation than a single temper for the cumulative time of the multiple tempers. This has been refuted by Cohen and Koh(81).

Chevenard and Portevin(30) quenched high-carbon steels to retain upwards of 50 per cent austenite and then studied their tempering by means of the dilatometer. They found that transformation could be made to occur during heating, at temperature, upon cooling, and in all possible combinations of these ways depending upon the time and temperature used for the tempering.

A very interesting region of austenite instability was found by the same authors in a study of steels containing 1.5 per cent carbon and 2 per cent chromium(27). They quenched the steel in water from 1180° C. (2156° F.) so as to render it austenitic and then tempered it at varying temperatures for periods of 7 hours each. The subsequent coolings of the specimen were followed in a dilatometer and in a magnetometer. It was found that in the range of temperature between 330° C. (626° F.) and 500° C. (932° F.) the austenite transformed only during cooling, either at Ar" or at both Ar' and Ar".

The entire subject of the transformation of retained austenite by tempering has been given much attention by Russian investigators(49)(48)(70)(19)(64)(79)(67)(82). Gulyaev(64) has formulated the following laws governing the structural transformations in high-speed steel.

1. The greater the carbon content of the retained austenite the lower the transformation temperature (M point) on cooling subsequent to tempering.

2. The transformation points are independent of the rate of cooling.

3. The austenite-to-martensite transformation occurs only during cooling.

It has been shown that the austenite may be made to transform isothermally upon interruption of the cooling. Cohen and Koh(81) found that these isothermal products were not the same as martensite. These investigators proved conclusively that in commercial high-speed steels the secondary hardness produced by tempering the quenched steel was attributable to retained-austenite transformation.

C. The Interpretation of Magnetic Data

The literature on this subject may be divided along nationalistic lines, Japanese, German, French, Russian, and American.

The magnetometer method of magnetic analysis is the one used virtually exclusively by the Japanese(15). Their school proposes the existence of two forms of martensite--alpha and beta. In both, the carbon is in solution, but the distinction lies in the different distribution of iron atoms in the lattice. The alpha phase has the tetragonal lattice and the beta phase has the body-centered-cubic lattice. Honda first proposed this after a magnetic study of the tempering of quenched steels(22); the magnetization versus tempering temperature curve showed discontinuities at 130° C. (266° F.) and at 280° C. (536° F.). Nishiyama corroborated the existence of cubic martensite by X-ray investigations(43)(60). He aged quenched steels at 59° C. (138° F.) and at room temperature. An age-hardening occurred but there were no changes in the (111) and (200) austenite lines. He ascribed the changes in other interference lines to the transformation of tetragonal to cubic martensite. Honda and Nishiyama(36) have shown that alpha martensite tempers in a narrow range, but that beta martensite tempers gradually over the range 120 to 400° C. (248 - 752° F.).

The problem of carbides and their magnetic behavior has received much attention by these workers. Honda was the first to relate the drop in magnetization of plain-carbon steel at the temperature of 215° C. (419° F.) to the loss in magnetism of the carbide(2). Through numerous experiments, he and his coworkers haveestablished the facts that the carbide may be deoomposed by heating to a sufficiently-high temperature and that in alloy steels containing carbide-forming elements redistribution of the elements composing the carbides took place as a result of high-temperature treatments(5)(1). Honda has investigated the magnetic behaviors at all of the phase-change temperatures in steel(4)(6).

The problem of the interpretation of magnetization versus temperature curves has several factors; these are:

1. Transformation of austenite.
2. Tempering of martensite.
3. Relief of stress.
4. Curie points.
    - a. Particle size.
    - b. Decomposition and redistribution of constituents.

In a study of the tempering of quenched-plain-carbon steels, Mikami(52) found that the magnetization, measured under the low field of 265 gauss, had the following trends with temperature (Figure 5):

1. Small increase in magnetization up to 200° C. (392° F.) attributed to the tempering of martensite.

2. Large increase in magnetization from 235° C. (435° F.) to 400° C. (752° F.) attributed to austenite transformation.

The possibility of stress relief entering into the scene was not considered. The large increase in magnetization was unquestionably due to austenite transformation, but it is doubtful whether the first small increase should rightfully be attributed to martensite tempering.

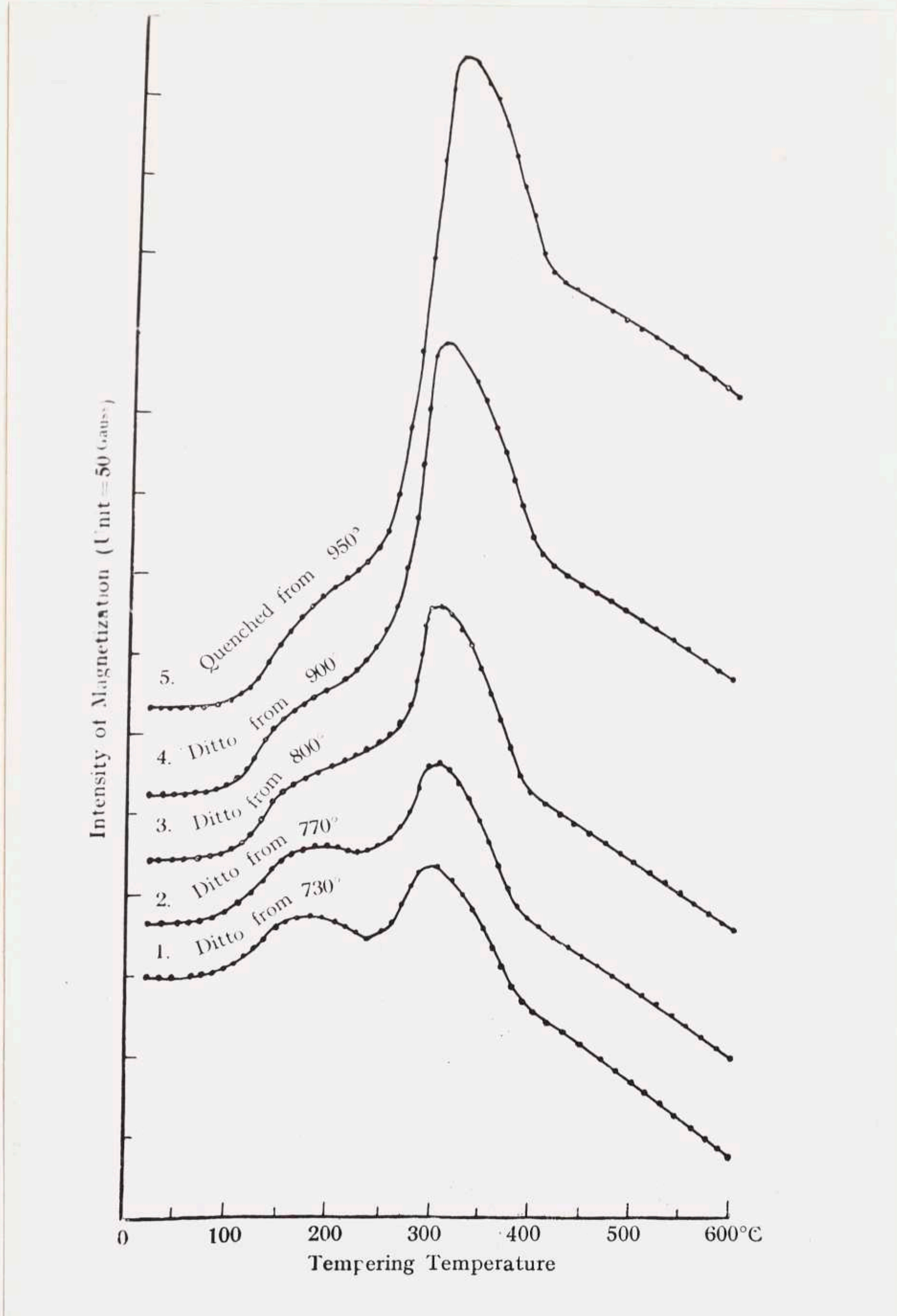


FIGURE 5. Heating (magnetization) curves of 1.25% C steel quenched in water from the temperatures shown.

Applied field - 265 gauss.

(Mikami)

The Japanese have also contributed considerably to the solution of the problem of the carbide curie point. Saito(7) found a rise in magnetization upon holding quenched steels at a temperature of 280° C. (536° F.), which rise increased with the carbon content of the steel. Inasmuch as the carbide curie point noticed upon cooling was very slight and increased upon subsequent reheating to higher temperatures he concluded that the very fine particle size of the cementite precipitated at 280° C. (536° F.) was responsible for the slight curie point first noticed. Mikami(52) transformed the austenite in a 0.85 per cent carbon steel quenched from 900° C. (1652° F.) by heating to 235° C. (455° F.); he then observed the drop in magnetization with time as the specimen was held at temperatures from 315° C. (599° F.) to 350° C. (662° F.). He credited the drop to the formation of a crystal lattice by the cementite molecules. Upon cooling the steel from these temperatures the curie points became successively more intense.

It would at first seem that the data of these two investigators are contradictory. The explanation of the differences is that Saito observed a combination of retained-austenite transformation and of carbide formation while Mikami observed carbide formation only.

This work demonstrated that the carbide must be of a certain particle size before it will exhibit a curie point, and that up to a certain limit the intensity of the curie point increases with the size. This was substantiated by the work of Esser and Momm(50) on 1.75 per cent carbon steel quenched from 1150° C. (2102° F.). They obtained an A_0 only when the cementite had been coalesced to a certain point, that is, two tempers at 600° C. (1112° F.) were necessary before the steel exhibited a distinct A_o inflection.

Beyond a limiting particle size the cementite will exhibit a curie point of diminished intensity. This has been conclusively shown by Sanford and Ellinger(32). They established a certain particle size of the carbide by tempering the quenched steel at 1065° F. Variations in this size were then produced by heating for different periods of time at 1255° F. (15 minutes to 64 hours). As the particle size increased, both the intensity and the temperature of the carbide curie point were lowered. Rao(45) has shown that the magnetization of nickel in the colloidal state is less than that for large-particle-size states. He used Bitter's(68) interpretation of the block-structure theory to explain his results.

The quantitative determination of the carbide particle size necessary to allow the carbide to exhibit a curie point would open a whole field of investigation on the relation of magnetic properties to particle size.

The literature of the German school on the general matter of magnetic behavior in relation to tempering phenomena is copious. Esser and his coworkers have conducted investigations at or close to magnetic saturation. They used the ballistic method. Esser and Momm(50) followed the changes in magnetization with temperature under a field of 8000 gauss for quenched steels containing 0.74 per cent carbon and 1.75 per cent carbon. In both steels the magnetization gradually decreased with temperature up to the point where the austenite began to transform, starting at 200° C. (392° F.), and reaching a maximum at 250 - 260° C. (482 - 500° F.). This clearly demonstrated the fact that the tempering of martensite will have no effect on the magnetization-temperature curve when the measurements are made under high field (10,000 gauss). The study of Mikami(52) should be reviewed here as it demonstrates the different magnetic behavior exhibited under low field (265 gauss) by a steel undergoing the tempering process.

The use of the magnetic method for the study of isothermal transformation has been extensive in Germany. Esser and Momm(50) followed the isothermal tempering of a 1.75 per cent carbon steel, water quenched, by measuring the magnetization, under a field of 8000 gauss, of specimens heated-to and cooled-from tempering temperature. Döpfer and Wiester(55) studied the isothermal transformation at temperature of various steels using a magnetic method. Although the field used in this investigation was below that needed to saturate the specimens, results were similar to those obtained by using a saturation field, according to a communication from H. Lange. Messkin(26) measured the magnetization at room temperature of specimens tempered at varying temperatures and found the same relations at 300 gauss as he did at saturation fields. Schottky(35) also used the ballistic method to follow the austenite transformation produced by hot-quenching.

Wide use has been made at the Kaiser Wilhelm Institute at Düsseldorf(86)(78)(28)(84) of the magnetic balance developed by H. Lange(77) for the study of isothermal transformation during hot-quenching.

One of the foundation studies of this entire field was made by Maurer and Schroeter(24). In this paper they evolved the widely-used formula for evaluation of the austenite content of a steel by the magnetic method.

This formula is based on the fact that a steel in the entirely-austenitic state has zero saturation and on the proposition that in the austenite-free state it has maximum saturation. They calculated what the saturation value of a hypothetical completely-martensitic steel would be, based on the assumption that all the carbide was dissolved and this ineffective on the magnetization; the result was greater than that measured for the annealed state (11 per cent deviation). That the annealed state does not possess the greatest magnetization is more than a result of calculation, as it has been also observed by Steinberg and Zyuzin(67) and by Minkevitch, Ivanov, and Dovgalevski(79). Because of the lack of knowledge as to which state of a steel possesses the greatest magnetization, and considering the accuracy of the work as a whole, Maurer and Schroeter adjudged the formula to be suitable for comparative purposes. It is:

$$\%\ \text{austenite} = \frac{a-b}{a} \times 100$$

where

a = saturation value of the annealed specimen

b = saturation value of the specimen under study

Eilender, Klinar and Cornelius(39) used the ballistic apparatus of Esser and Oberhoffer(12) to study the tempering of high-speed steel. They found no discontinuities in the heating curve which demonstrated again the insensibility of magnetization at high field (8000 gauss) to martensite tempering. The changes in magnetization, measured at room temperature, of a 0.78 per cent carbon steel water-quenched from 770° C. (1418° F.) were measured by Messkin(26). He proposed that the drop in the curve of magnetization versus temperature which occurred up to a temperature of 150° C. (302° F.) was due to precipitation of iron carbide from the martensite, a view which coincided with Maurer and Schroeter's(24) calculation. The rise which was observed in the range from 150° C. (302° F.) to 250° C. (482° F.) was attributed to austenite transformation. A similar interpretation was given by Tofaute, Sponheuer and Bennek(54) to their results of the tempering study on a 1 per cent carbon and 5 per cent chromium steel, water quenched from 950° C. (1742° F.).

The transformation of retained austenite may be greatly accelerated by cold-working before tempering. Bollenrath(58) found that by cold-reducing an austenitic silico-manganese steel 20 per cent only 3 per cent of the austenite transformed, but that upon subsequent tempering to 560° C. (1040° F.) the transformation was completed.

The work of the French is characterized by considerable ingenuity. Portevin and Chevenard(9) were the first to observe the dilation that accompanies the carbide curie point. They(66) corroborated the views of Honda(5)(1) as to the changes produced by various tempering treatments in the curie points of steels containing carbide-forming elements. In another paper(66) they developed a method of determining the amount of carbide produced by any treatment from the shape of the magnetization versus temperature curve.

The work in this country on the relation between magnetization and tempering phenomena has been done mainly by Sanford, and by Ellinger. In their work they used an astatic magnetometer developed by Sanford(25).

Their work on the relation of particle size to the intensity of the carbide curie point(32) has been mentioned before. The Japanese(52)(22) attributed the rise in the magnetization versus temperature curves at low temperatures of quenched-plain-carbon steels to the tempering of martensite. Ellinger(31) and Ellinger and Sanford(36), using an applied field of 277 gauss, observed the same phenomena but attributed it to stress relief. Figure 6 shows the observations they made on the tempering of electrolytic iron, 0.75 per cent carbon steel quenched from 825° C. (1517° F.) and 0.75 per cent carbon steel annealed at 825° C. (1517° F.). The electrolytic iron displayed no changes. The quenched steel displayed a small rise up to 120° C. (248° F.), a sharper rise to 240° C. (464° F.) and a very sharp rise to 280° C. (536° F.). Upon cooling this steel displayed the carbide curie point at 200° C. (392° F.) as did the annealed steel. Duell(20) also observed this increase of magnetization in steels quenched to martensite. He found a drop in steels quenched to troostite or softer structures.

Cohen and Koh(81) followed the tempering of quenched, high-speed steel magnetically by means of the torsion balance of Buehl and Wulff(73) and found no effect of the tempering of martensite upon the course of the curves.

The Russian investigators have made wide use of the ballistic method(67)(49)(48)(70)(19)(82)(64)(79)(87). The absence of variation in the magnetization versus temperature curves at high fields for martensite tempering is illustrated in a paper by Zyuzin, Sadovski and Baranchuk(87).

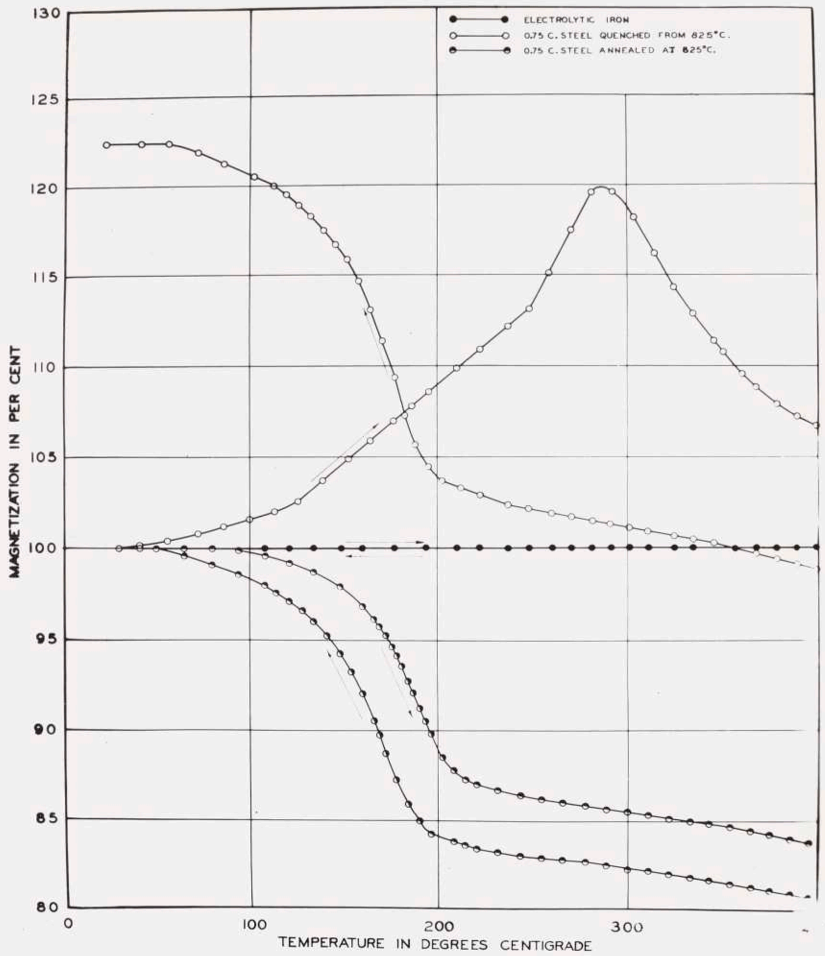


FIGURE 6. Heating and cooling (magnetization) curves of heat-treated 0.75% C steel.

Applied Field - 277 gauss.

(Ellinger)

Chevenard and Portevin(30) published a chart in which they listed the reactions that were possible in a tempering process and the accompanying changes in dilation and magnetization. This chart has been revised and is presented as Table I.

TABLE I

MAGNETIZATION AND DILATION PHENOMENA ACCOMPANYING POSSIBLE TEMPERING REACTIONS

Possible Reactions During Course of Tempering	Accompanying Phenomena	
	Dilation	Magnetization
Austenite to cementite and austenite less rich in carbon.	Contraction	Magnetization of cementite if reaction occurred below curie point.
Austenite to ferrite and carbide and austenite less rich in carbon.	Expansion	Strong magnetization of ferrite.
Austenite to ferrite and carbide.	Expansion	Strong magnetization of ferrite.
Austenite to martensite.	Expansion	Strong magnetization of martensite.
Tetragonal martensite to cubic martensite.	Contraction	Decrease in magnetization at low field. No changes at high fields.
Martensite to ferrite and carbide.	Contraction	No change at high fields.
Stress relief.	No change	Increase at low fields. No change at high fields.
Magnetic transformation in the carbide - evident during cooling from the tempering temperature	Slight Expansion	Increase at high and low fields - more marked at low fields.

IV

RELATION OF PREVIOUS WORK TO THE PRESENT INVESTIGATION

The work that has been done on high-carbon high-chromium steels has been concentrated on hardness and dimensional changes produced by tempering. Retained-austenite transformation was assigned as the cause of these changes. The first of the three objects of this investigation is to determine the characteristics of the austenite transformation in order that the phenomena resulting from the transformation may be controlled.

The work of previous investigators relative to the interpretation of magnetic-tempering data has been shown to be apparently inconsistent. The second object of this investigation is to coordinate this work and to present the explanation for the differences.

Prior work on the transformation of retained austenite in high-speed steels has shown that the austenite transforms during cooling into martensite. An incontestable proof that the transformation product was martensite has not been made. The final object of this thesis is to determine the laws governing austenite transformation and to compare them with those already developed for high-speed steel.

V

OUTLINE OF PROCEDURE

- I. Review of Literature
- II. Preparation of Steels for Tempering Study
    - A. Removal of decarburized surface
    - B. Heat treatment
- III. Construction of Magneto-Dilatometer
    - A. Construction of furnace circuit
    - B. Construction of magnet circuit
    - C. Adaptation of dilatometer to magnet
- IV. Tempering Studies at Various Temperatures
    - A. Selection of suitable tempering baths
    - B. Magnetic measurements
- V. Tempering Studies During Heating-to, Holding-at and Cooling-from the Tempering Temperature
    - A. Magnetic measurements
    - B. Dilatometric measurements
- VI. Determination of Amount of Retained Austenite
    - A. Magnetic Method
    - B. X-ray method
- VII. Microscopic Studies
- VIII. X-ray Studies to Give Fundamental Proofs
- IX. Correlation and Interpretation of Results
    - A. Conclusions

VI

EXPERIMENTAL PROCEDURE

A. Preparation of the Specimens

The steels used as the subjects of this investigation were supplied by Mr. Norman I. Stotz of the Universal-Cyclops Steel Corporation.

At first, the investigation was to be carried out on pure iron-carbon-chromium alloys, but it was impressed upon the writer that the data obtained might have no relation to the steels of this type used in practice. It was decided to use two commercial steels of the high-carbon high-chromium type. Mr. Stotz suggested the study of a high-carbon medium-chromium steel and the study of a high-carbon high-chromium steel. The steels supplied are known in commerce as Sparta and Ultradie Number 2.

The steels were furnished in two shapes - 5/8 in. square stock and 1/4 in. round stock. They were in the annealed states. Although these steels are complex, as is evident from their analyses that follow, they will be designated through the thesis as 1% C - 5% Cr and 1.5% C - 11.5% Cr, respectively.

The analyses and designations of the steels are given in Table II.

TABLE II

DESIGNATION AND ANALYSES OF THE STEELS USED IN THE THESIS

Commercial Designation	Ultradie 2	Sparta
Heat Number	C 16254	C 16530
Thesis Designation	1.5% C - 11.5% Cr	1% C - 5% Cr
Carbon	1.60	1.00
Manganese	0.33	0.61
Silicon	0.32	0.17
Sulphur	0.010	0.013
Phosphorus	0.018	0.014
Chromium	11.95	5.31
Vanadium	0.25	0.27
Molybdenum	0.79	1.13
Nickel	0.13	0.09
Copper	0.04	0.06

The microstructures of the annealed specimens are shown in Figure 91. The carbides could not be completely dissolved in either steel by the heat treatments used. The micro examination also disclosed the presence of decarburized surfaces on both the round and the square stock. These decarburized surfaces were milled off the square stock and were ground off the round stock on a centerless grinder. This operation was carried out on equipment of the United Shoe Machinery Corporation through the courtesy of Mr. John Paine. It was sufficient to grind the round stock from 1/4 in. to 0.24 in. in diameter in order to remove the decarburized surface. The square stock was milled from 5/8 in. to 7/16 in.

The hardening of the steels was carried out in the Sentry laboratory furnace (globar type). The specimens were protected from decarburization during hardening by the use of a carbonaceous muffle. The calibration of the furnace was made with a standard platinum thermocouple. The temperatures were held to ± 3° F.

Three cooling media were used, oil, air and lime. The specimens were removed from the furnace by tongs and cooled either in Gulf Number 2 Heat-treating Oil, in still air on a refractory support, or in lime.

B. The Magnet as an Instrument for Measuring Austenite Decomposition

1. Introduction

Since austenite is paramagnetic and martensite is ferromagnetic, a measurement of magnetic properties suggests itself as being a means of studying the austenite-to-martensite transformation. The magnetic properties that can be considered are induction, permeability, intensity of magnetization, coercivity, remanence, and the curie point.

There are two methods of measurement used (1) the ballistic method and (2) the magnetometer method. Both methods are capable of high accuracy, but the magnetometer has been perhaps more frequently used for precision determinations of the magnetization-intensity's dependency upon temperature. Unless a special type of suspension is used(28) heavy masses of iron in the immediate vicinity cause difficulty. A method usually used for paramagnetic and diamagnetic substances is Faraday's method(15) and has been useful in determining magnetization-temperature dependency(88) for ferromagnetics.

The ballistic method has been most used for the study of magnetic properties at room temperature. The closed-magnetic-circuit methods(37), the air-gap method(17), and the permeameters(33) are examples of the general ballistic type of measurement.

Nicolau(10) has described a variation of the A. C. method wherein the rectifier is dispensed with in the secondary circuit by the use of a double coil in the secondary arranged in opposition. Esser and Oberhoffer(12) have devised a modification of the ballistic method using alternating current with a cathode tube rectifier in the secondary circuit. Although a quantitative determination of the magnetization intensity could not be obtained the curie points were accurately determinable. Furthermore, by using a Saladin apparatus, he was able to eliminate the factor of personal attention.

A similar sort of apparatus, with a millivoltmeter substituted for the Saladin double-reflecting galvanometer, was used by Döpfer and Wiester(55). A similar apparatus has been described by Pfaffenberger(44). Kinsley(76) has described a dynamometer method wherein a Fourier analysis is made of the E. M. F. in the secondary of a transformer whose core is the specimen to be examined. The use of an electrodynamometer has been decried by Bricout(63) as liable to serious error.

McCarthy(92) adapted the yoke method to a study of the temperature dependency of the permeability of high-speed steel.

The simple ballistic method can be used with accuracy if a calibration has been carried out. By using a specimen of hydrogen-annealed Wemco iron of exactly similar size and shape to the specimens under study, the effects of demagnetization and of the air gap between the secondary and the specimen can be grouped together into a constant.

2. Theory of the Ballistic Method(3)

Consider a secondary circuit, the coil of which is linked with a changing field. The relation between

i, the current in the circuit,

E, the voltage drop across the circuit, and

r, the total resistance of the circuit, is given by Ohm's law as

$$i = \frac{E}{r}$$

Now, $i = \frac{dQ}{dt}$ and

$$E = \frac{dN}{dt}$$

where Q = quantity of electricity in the secondary circuit,

N = total change in linkings of flux and wire turns.

Substituting and integrating:

$$\frac{dQ}{dt} = \frac{1}{r}\left(\frac{dN}{dt}\right)$$

$$\int_0^Q dQ = \frac{1}{r}\int_{N_1}^{N_2} dN$$

$$Q = \frac{\Delta N}{r} \qquad (1)$$

Now $N = \phi S$

$$= BAS$$

where ϕ = total flux

S = number of turns linking with lines of force

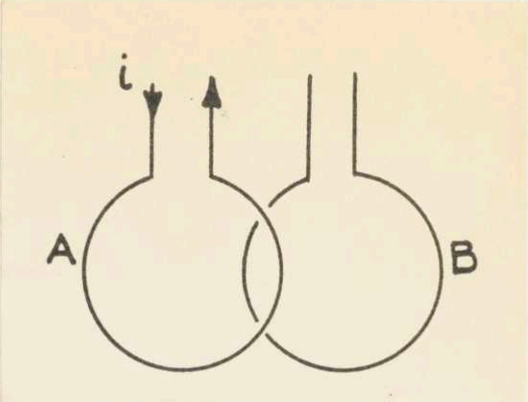
B = induction density

A = cross-sectional area of the coil.

Whereupon

$$Q = \frac{\Delta(BAS)}{r} \qquad (2)$$

Consider two coils



The current flowing in A is i and the resultant field interlocks coil B. The number of linkings is proportional to the current flowing in A, or

$$N = Mi \qquad (3)$$

where M is a geometric constant, independent of the current. Now, for a change in current, di,

$$M = \frac{dN}{di}$$

For a given time interval for the change, dt,

$$M = \frac{dN}{dt} \Big/\!\!\Big/ \frac{di}{dt}$$
$$= E \Big/\!\!\Big/ \frac{di}{dt}$$

This equation affords a definition of M: The mutual inductance is the voltage induced in B when the current in A is changing at unit rate.

The unit of mutual inductance is the <u>henry</u> and is defined as follows: An electro-motive force of one volt is induced in a circuit of one henry when the current is changing at the rate of one ampere per second. The practical unit is the millihenry.

Since (equation 1)

$$Q = \frac{\Delta N}{R}$$

$$= \frac{N_2 - N_1}{R}$$

Introduction of M (equation 2) gives

$$Q = \frac{Mi_2 - Mi_1}{R}$$

For a change in current from $i_1 = 0$ to i_2

$$Q = \frac{Mi_2}{R} \tag{4a}$$

Likewise for opening a circuit, from i_1 to $i_2 = 0$

$$Q = -\frac{Mi_1}{R} \tag{4b}$$

For a reversal of the current

$$Q = \frac{2Mi}{R} \tag{4c}$$

Now, the throw of a ballistic galvanometer is proportional to the quantity of current discharged in the secondary circuit.

For d = galvanometer deflection

G = galvanometer constant

$$Q = Gd \tag{5}$$

In standardizing the galvanometer with a mutual inductance, the throw was observed for only one closing of the circuit. From equations 4a and 5

$$Gd = \frac{Mi}{r}$$

$$G = \frac{M}{r}\left(\frac{i}{d}\right) \qquad (6)$$

In making a run on a specimen, the method of current reversal was used. For an observed deflection D , equation 5 becomes

$$Q = GD$$

From equation 2

$$Q = \frac{\Delta(BAS)}{r}$$

$$\frac{\Delta(BAS)}{r} = GD$$

$$G = \frac{\Delta(BAS)}{Dr}$$

From equation 6

$$G = \frac{M}{r}\left(\frac{i}{d}\right)$$

$$\frac{\Delta(BAS)}{Dr} = \frac{M}{r}\left(\frac{i}{d}\right)$$

$$\Delta B = \frac{M}{AS}\left(\frac{i}{d}\right)D$$

If M is in henrys, i in amperes, and current reversal is used with the specimens, that is, $\Delta B = 2B$ and current break is used in standardizing, the following working equation is obtained

$$B = \frac{M}{2AS}\left(\frac{i}{d}\right) D \times 10^8 \qquad (7)$$

If air is the only core material, $B = H$, and

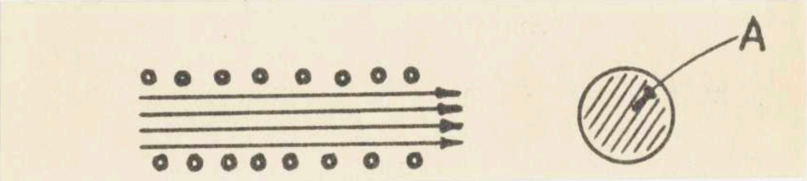
$$H = \frac{M}{2AS}\left(\frac{i}{d}\right) D \times 10^8 \qquad (8)$$

Consider, now, the modification of these equations (7 and 8) for use in the present study.

Returning to the fundamental equation,

$$\frac{\Delta N}{n} = GD$$

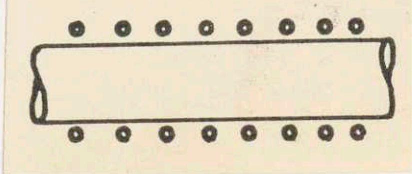
For a <u>coil alone</u>, of cross sectional area A



and for a reversal of current,

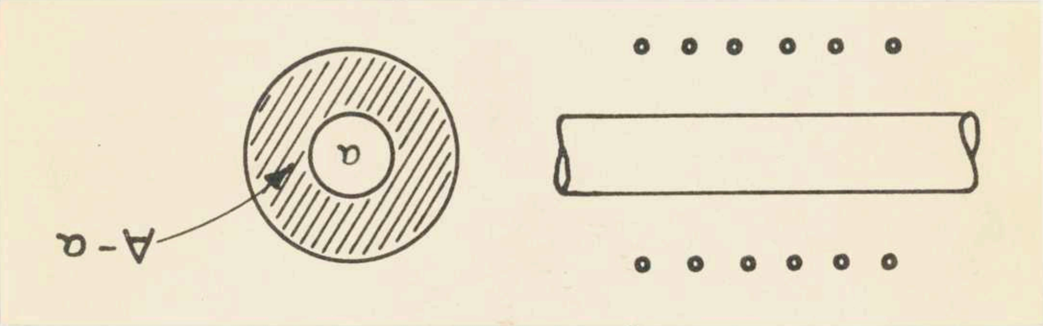
$$\Delta N_1 = 2HAS$$

For a coil with an inserted specimen, demagnetization not considered, and with equal cross-sectional areas of coil and specimen



$$\Delta N_2 = 2(H + 4\pi J)AS$$
$$= 2BAS$$

For the above, but with the more practical condition of different areas of coil and specimen,



$$\Delta N_3 = 2(HA + 4\pi Ja)S$$

Since $Ba = Ha + 4\pi Ja$

$$\Delta N_3 = 2\{Ba + H(A-a)\}S$$
$$= 2BaS + 2H(A-a)S$$

Now $\dfrac{\Delta N_1}{r} = GD_1$ $\qquad\qquad$ $\dfrac{\Delta N_3}{r} = GD_3$

$$\Delta N_1 = GrD_1 \qquad\qquad \Delta N_3 = GrD_3$$

$$\Delta N_3 - \Delta N_1 = Gr(D_3 - D_1)$$

$$D_3 - D_1 = \frac{1}{Gr}\left[2BaS + 2H(A-a)S - 2HAS\right]$$
$$= \frac{1}{Gr}\left[2BaS - 2HaS\right]$$
$$= \frac{1}{Gr}\left[2(4\pi J)\,aS\right]$$

The dimensions of the specimens used in the study 4-1/4 in. long, 1/4 in. diameter rods, make demagnetization a factor that can not be neglected. The field actually effective is less than the field produced by the primary by an amount which is a function of the shape of the specimen(65).

If H_0 = field produced by primary

η = demagnetization factor

the effective field is $(H_0 - \eta J)$.

The previous equations can then be modified as follows:

$$\Delta N_3 = 2\left[(H_0 - \eta J)A + 4\pi J a\right]S$$

Of course, without the specimen, demagnetization is not encountered, or $H = H_0$.

$$\Delta N_1 = 2 H_0 A S$$

$$\Delta N_3 - \Delta N_1 = 2\left[4\pi J a - \eta J A\right]S$$

Since

$$\Delta N_3 - \Delta N_1 = Gn(D_3 - D_1)$$

$$D_3 - D_1 = \frac{2J}{Gn}(4\pi a - \eta A)S$$

$$= 4\pi J\left(\frac{2S}{Gn}\right)\left(a - \frac{\eta A}{4\pi}\right)$$

$$4\pi J = \frac{Gn}{2S}\left(\frac{D_3 - D_1}{a - \frac{\eta A}{4\pi}}\right)$$

Since

$$G = \frac{M}{n}\left(\frac{i}{d}\right)$$

$$4\pi J = \frac{M}{S}\left(\frac{i}{d}\right)\frac{D_3 - D_1}{2\left(a - \frac{\eta A}{4\pi}\right)}$$

The expression $2\left(a - \frac{\eta A}{4\pi}\right)$ could be evaluated from the constituent terms, but the value for η taken from a table had an element of error in it. A safer way was to determine this constant by using a material of known saturation magnetic intensity.

A 5/8 in. rod of Wemco iron was supplied by Professor Kaufmann. This was swaged down to a 1/4 in. diameter and was hydrogen annealed at 800° C. (1472° F.). The value used for $4\pi J$ was 21,580(83).

The mutual inductance used for standardizing was 50 millihenrys. The other values are tabulated

$$\frac{i}{d} = 1.51 \times 10^{-3}$$

$$D_3 - D_1 = 217$$

$$S = 114$$

Using these values

$$2\left(a - \frac{\eta A}{4\pi}\right) = 0.666$$

Then,

$$4\pi J = \left(\frac{M}{S}\right)\left(\frac{i}{d}\right)\left(\frac{D_3 - D_1}{0.666}\right) \times 10^{8}$$

For, $(D_3 - D_1) \sim 180,$ $\quad \frac{i}{d} = 1.52 \times 10^{-3}$

Using as before

$$M = 50 \times 10^{-3}$$

$$S = 114$$

$$4\pi J = (D_3 - D_1)\,10^{2}$$

In remanence measurements, the circuit is opened from a fixed value of the current. The relationships given below may be derived from Figure 7. The changes in the deflection produced by changes in the field are shown for the coil alone (bottom), the coil and the specimen (top), and the specimen alone (middle).

When the field is broken, a throw, D_4 is observed, it consists of:

$$D_4 = D_{S_4} + \frac{D_1}{2}$$

D_{S4} = throw produced by specimen only

D_1 = throw produced by coil only

Reversal of the field determines $\frac{D_3}{2}$ which consists of

$$\frac{D_3}{2} = \frac{D_S}{2} + \frac{D_1}{2}$$

The remanence, D_1 , is given by

$$D_1 = \frac{D_S}{2} - D_{S4}$$

From the previous equations:

$$\begin{aligned} D_1 &= \frac{D_3}{2} - \frac{D_1}{2} - \left(D_4 - \frac{D_1}{2}\right) \\ &= \frac{D_3}{2} - D_4 \end{aligned}$$

The throw corresponding to a "reversal measurement" of remanence would be $2D_1$

Then $\Delta N_1 = 2B_1 a S$

$= 2 G\Omega D_1$

$$G\Omega D_1 = B_1 a S$$

$$B_1 = \frac{G\Omega D_1}{aS}$$

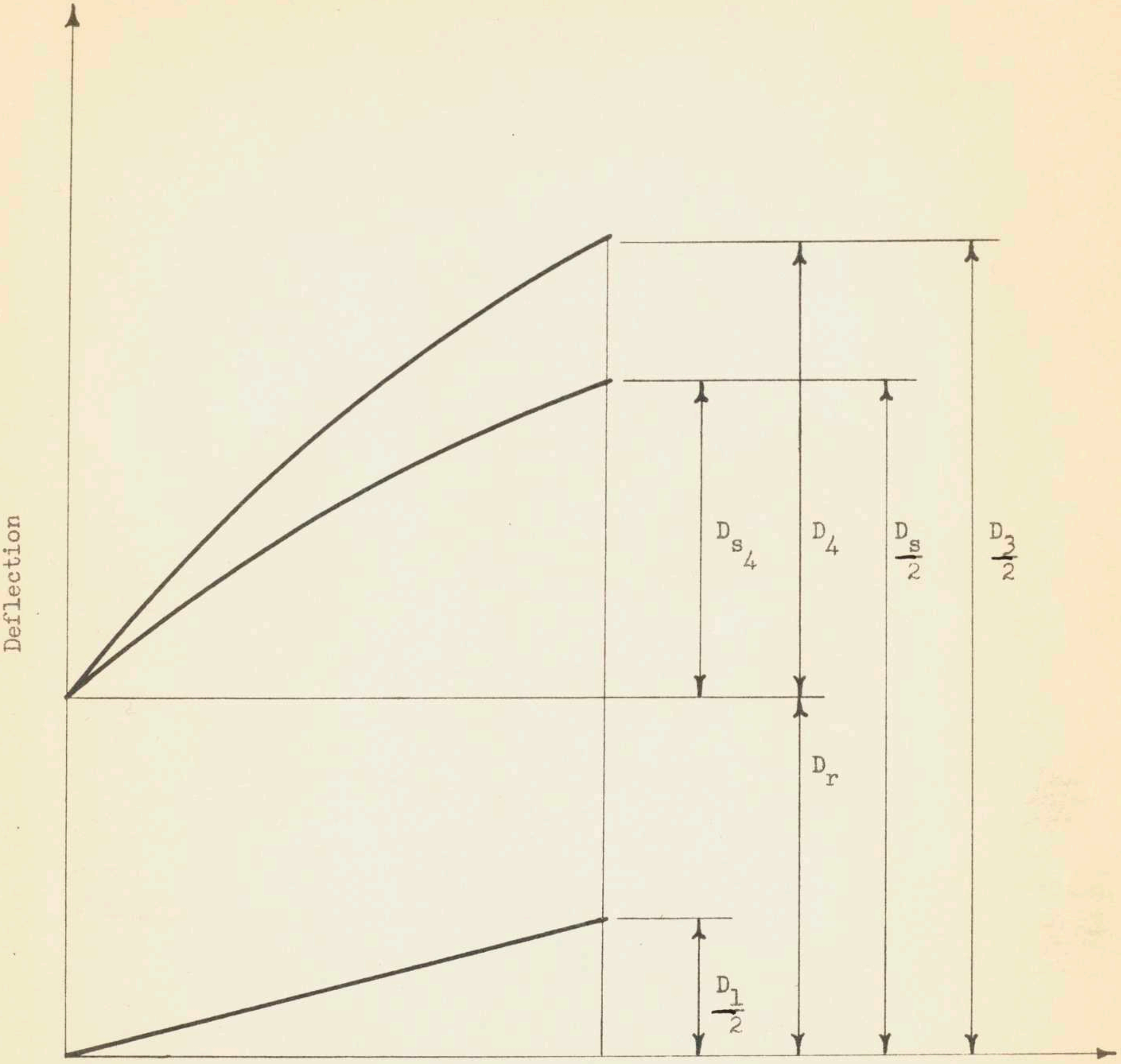


FIGURE 7.

Illustration of method for obtaining remanence.

When demagnetizing fields must be considered, the equation can be revised as follows:

$$\Delta N_1 = 2[(H_0 - \eta J)A + 4\pi Ja]S$$

Now

$$B_1 a = (H_0 - \eta J)\, a + 4\pi J a$$

Then,

$$\begin{aligned}
\Delta N_1 &= 2[B_1 a + (H_0 - \eta J)(A - a)]S \\
&= 2\, G n D_1 \\
2 G n D_1 &= 2 B_1 a S + 2(H_0 - \eta J)(A - a)S \\
2 B_1 a S &= 2\, G n D_1 - 2(H_0 - \eta J)(A - a)S \\
B_1 &= \frac{G n D_1}{a S} - (H_0 - \eta J)\left(\frac{A - a}{a}\right)
\end{aligned}$$

Since $G = \frac{M}{n}\left(\frac{i}{d}\right)$

$$B_1 = \frac{M}{aS}\left(\frac{i}{d}\right) D_1 - (H_0 - \eta J)\left(\frac{A - a}{a}\right)$$

The property of coercivity is one of the properties possessed by a ferromagnetic substance that are used as criteria for the applicability of the substance as a permanent magnet. The coercive force is given by the length, C , in the accompanying Figure 8; it is the field in gauss which has to be applied in the negative direction to the substance under consideration in order that its intensity of magnetization may be reduced to zero.

With a ballistic apparatus the coercivity may be determined as follows:

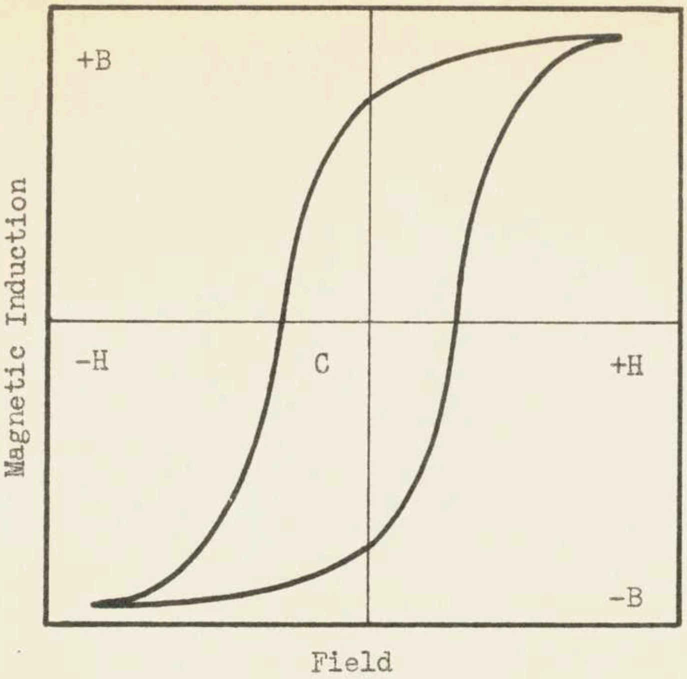


FIGURE 8.

Hypothetical hysteresis loop.

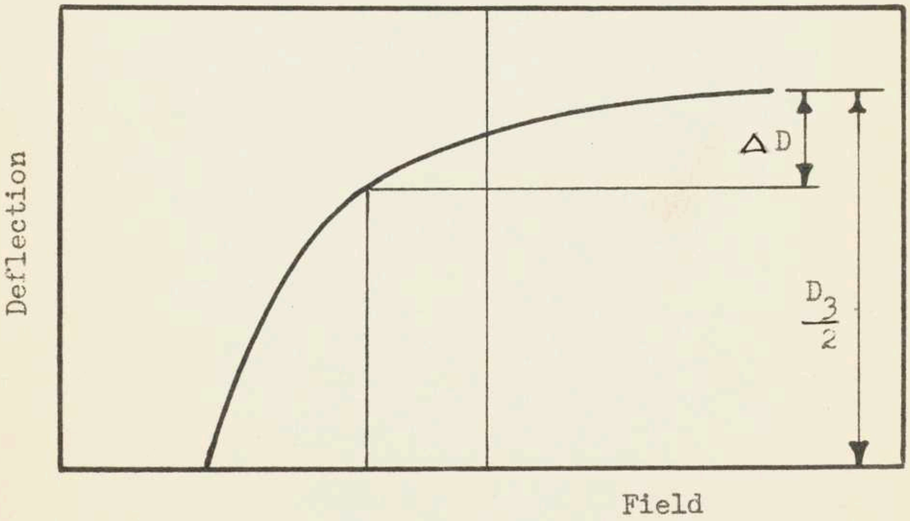


FIGURE 9.

Portion of hypothetical hysteresis loop.

1. Using some established field, in this study 1000 gauss, determine the deflection, D_3 , for a reversal of the field (Figure 9).

2. By breaking the field and immediately making the negative direction to some lower value, $-H_1$, determine ΔD .

3. Repeat (2) with increasing values of H in the negative direction until $\Delta D \sim \frac{D_3}{2}$.

The fact that no compensation is provided for the secondary coil complicates the situation somewhat. The state is described by the diagram of Figure 10.

The lower curve is that for the specimen alone, the upper curve is for the specimen and the coil together.

Fundamentally, it is desired to equate:

$$\Delta D_3 = \frac{D_s}{2}$$

The quantities measured are:

$$\Delta D \quad \text{and} \quad \frac{D_3}{2}$$

The relationships between these quantities are evident from the Figure 9.

$$\frac{D_3}{2} = \frac{D_s}{2} + \frac{D_1}{2} \qquad \Delta D = \frac{D_s}{2} + \frac{D_1}{2} - a$$

$$\frac{D_s}{2} = \frac{D_3}{2} - \frac{D_1}{2} \qquad \frac{D_s}{2} = \Delta D - \frac{D_1}{2} + a$$

Then, equating $\frac{D_3}{2} - \frac{D_1}{2} = \Delta D - \frac{D_1}{2} + a$

$$\frac{D_3}{2} = \Delta D + a$$

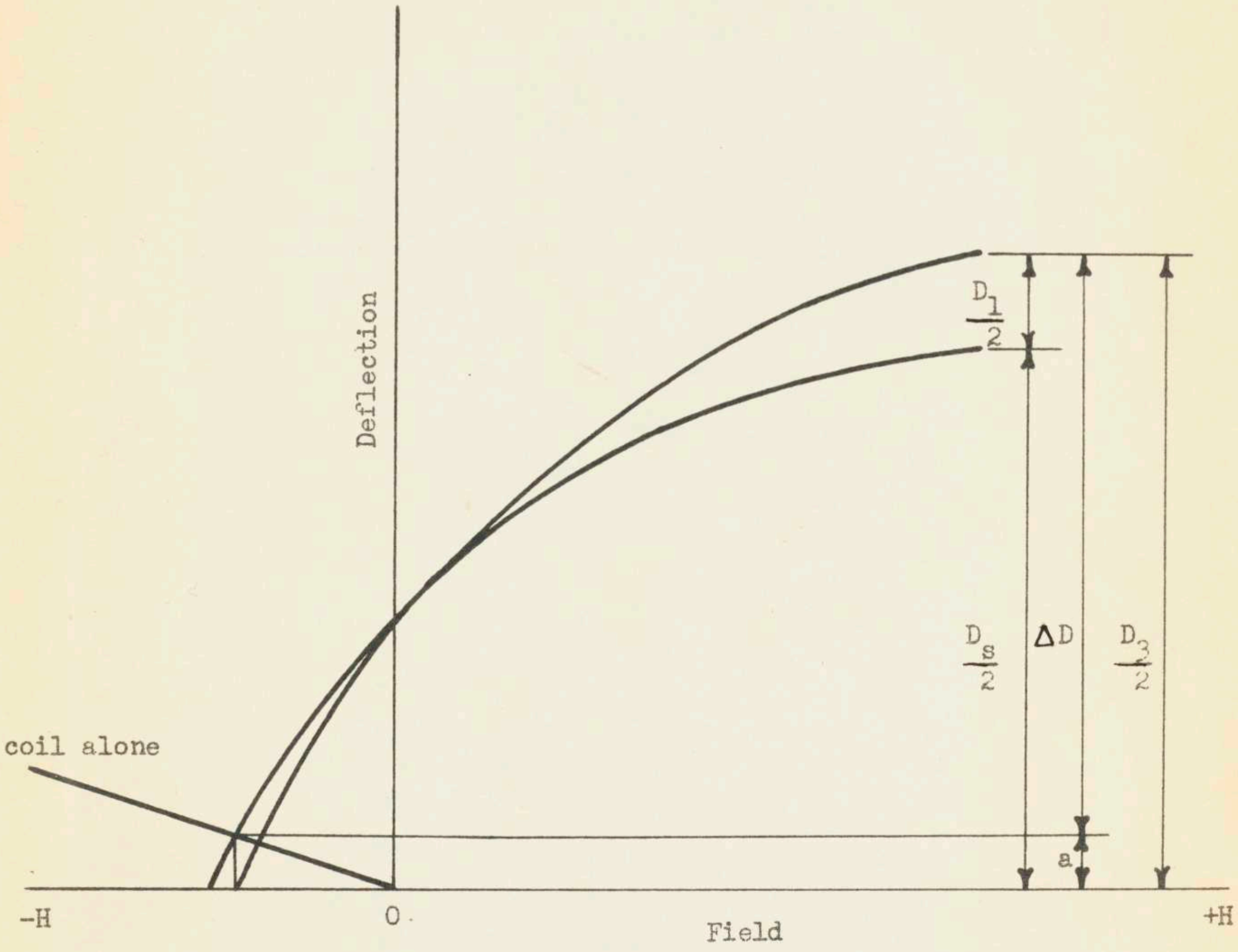


FIGURE 10.

Illustration of method for obtaining coercivity.

The coercivities to be expected range from 40 to 100 gauss. Thus the negative fields to be applied are selected in this range. By first determining $\frac{D_3}{2}$, the proper values to use for the negative fields may be estimated.

To the values of ΔD obtained must be added the values of <u>a</u> corresponding to the fields used in the negative direction. The resultant values are plotted against their respective values of the field, expressed in amperes. From these curves, they should be straight lines if the negative fields have been chosen properly, the currents corresponding to the values of $\frac{D_3}{2}$ can be selected. These values may then be evaluated in terms of gauss from the field-current curve of the magnet.

3. Quantitative Treatment of Magnetization-Temperature Curves

When a ferromagnetic two-phase alloy is subjected to a magnetization versus temperature study a curve similar to that in Figure 11 is obtained.

The dotted line represents an extrapolation of the magnetization of one phase to room temperature. The other phase is not ferromagnetic until the temperature of the inflection is reached. Its magnetization is then added to that of the other phase and produces the solid-line curve.

If saturation-magnetization values are measured, a calculation can be made from this curve of the amounts of the constituent phases. Since the magnetization values obtained are volume measurements, they must be converted to a weight basis. This is done by dividing by the densities.

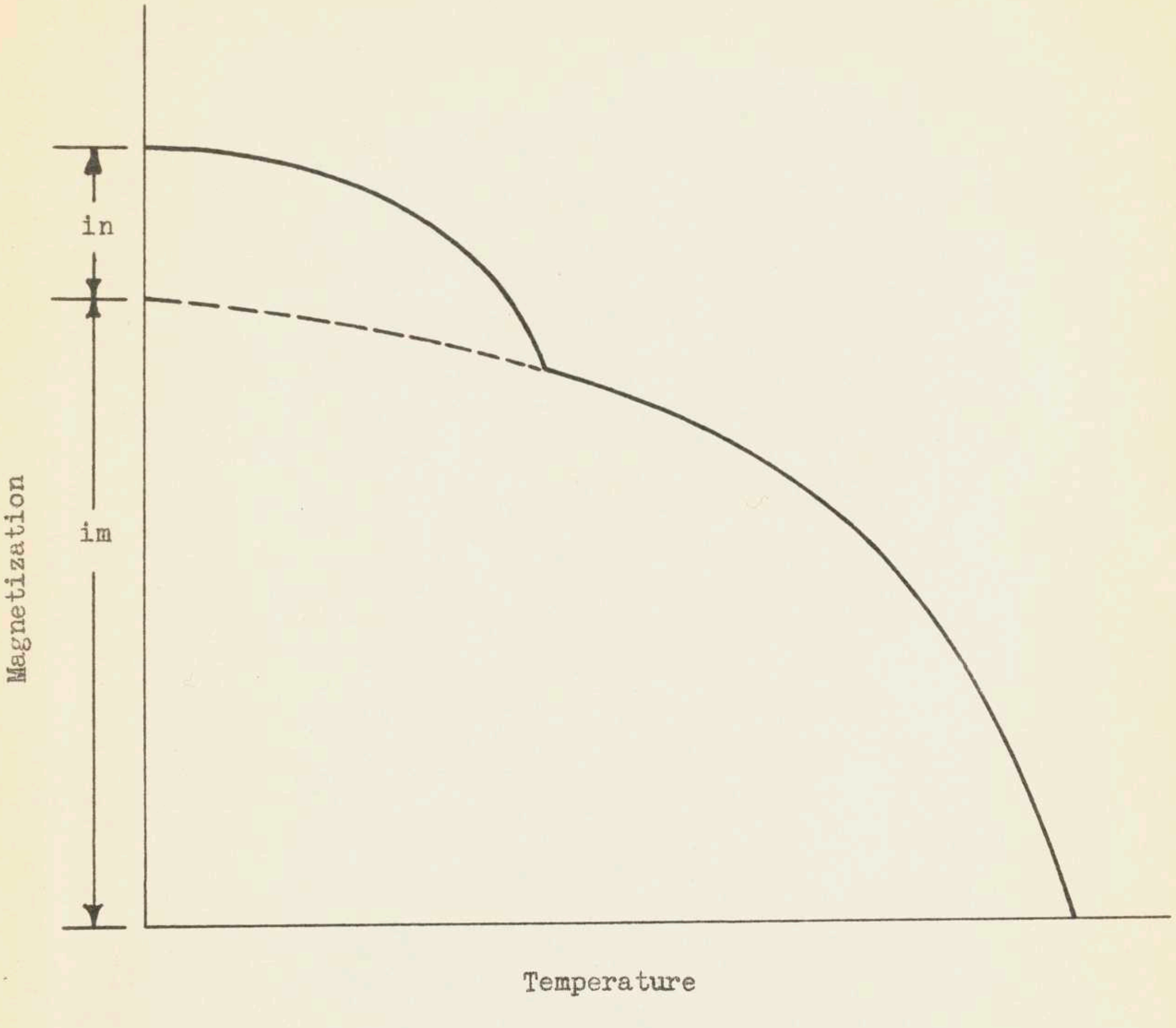


FIGURE 11. Magnetization versus temperature for an hypothetical binary ferromagnetic alloy.

As an example, consider a steel containing a carbide phase. Let the specific saturation magnetization (magnetic saturation divided by the density) value for ferrite is I_m and that for carbide is I_n.

For the alloy studied i_m will be the specific magnetization of the ferrite present and i_n will be the specific magnetization of the carbide.

The amount of ferrite is, then $\frac{i_m}{I_m}$ and of carbide, $\frac{i_n}{I_n}$.

An example of the application of this work may be found in a paper by Wever and Lange(86). For a steel containing 1.0 per cent vanadium and 0.80 per cent carbon they found $i_n = 12.2$ and $i_m = 193.6$. Then the amount of ferrite ($I_m = 218$) was given by the ratio,

$$\frac{193.6}{218} \times 100 = 88.7 \text{ per cent.}$$

The amount of iron carbide ($I_n = 139.3$) was given by the ratio

$$\frac{12.2}{139.3} \times 100 = 8.76 \text{ per cent.}$$

From this they reasoned that the remainder of the carbon was combined with vanadium and manganese.

This assumption was verified by chemical analysis.

It is necessary to apply this method in the determination of the amount of retained austenite by Maurer and Schroeter's method when carbide is present. This was not done in the present investigation because the saturation values of the carbides were not known.

4. Description of the Apparatus

The instrument used for this study consisted of four parts--the furnace, the magnet, the dilatometer, and the measuring and control accessories. The description that follows has been made detailed to facilitate use of the apparatus. It has been made part of the permanent equipment of the laboratory.

The furnace was made with a bifilar winding so as to eliminate the effect of the magnetic field of the furnace winding; constituting the chamber of the furnace was an alundum tube 18 in. long, 1/2 in. bore, and 1/8 in. wall. The winding was composed of 20 gauge Chromel A wire and it was placed on the tube for 17 in. with an average of 4 wound turns per inch distributed along the length.

It was not without some difficulty, however, before the final furnace was evolved. Realizing the considerable temperature drop that would exist in the apparatus, the designer decided upon a dense winding spacing. Eighteen gauge wire was first chosen, would 12 and 13 turns per inch. An attempt was made to wind the wire in the as-received condition--this resulted in a broken tube. Hereafter, the wires were always annealed by connecting them across the 220 volt line and passing current through then until they glowed red. This was done after any kind of cold work was experienced by the wire.

After the furnace had been wound with 12 to 13 turns per inch of 18 gauge wire, it was coated with a mixture of alundum cement (low temperature grade), water glass, and water. At the temperature of the winding necessary to bring the contents of the furnace to 1000° F., the alundum and the sodium silicate reacted, producing a fusible slag. This became conducting and permitted the windings to arc across, which resulted in the destruction of the furnace.

Even after the silicate bond was abandoned, trouble was experienced from the arcing-across of adjacent windings. Of course in an ordinary furnace winding no harm is experienced from the contact of adjacent windings as they are at practically the same potential. In a bifilar winding, however, it is very necessary to insulate completely each turn from its neighbors. Asbestos twine was utilized for this purpose, but it was virtually impossible to keep the heavy 18 gauge wire, wound at the considerable density of 12 to 13 turns per inch, separate.

Still desirous of maintaining the 18 gauge wire and the winding density it afforded, the designer built a furnace with two layers of windings. It was not possible by this method to completely nullify the field of one winding by the field of the other.

Finally, the tube was wound with a wire of smaller diameter whose resistance would permit a one layer winding and a low winding density (20 gauge wire, wound at 4 turns per inch).

Several kinds of insulating material were tried during the assembly of the apparatus. The use of asbestos string for electrical insulation of the wires has already been mentioned. The coating finally used to secure the wound wires in place was high-temperature alundum cement. A mixture of the cement and water of cream-like consistency was applied with a paint brush. Just enough was used to present a smooth surface, that is, so the wires did not show. To separate the insulation of the furnace from the copper pipe into which the unit was inserted a thin sheet of asbestos paper was used. Sil-o-Cel was tried but it was not possible to distribute it properly. Asbestos yarn was used as the insulation for the furnace. It was wound over the cement coating after being dampened to allow it to be made into a unit.

The furnace served to contain the core of the magnet. Serving as the shell of the furnace chamber and as the support for the magnet wire was a copper pipe of standard size, two inches in diameter and 17-3/4 in. long. Two large brass plates, 1/4 in. thick, served as supports for the entire unit. A hole was cut and threaded for a two-inch pipe in each plate. It was found that the threaded units when assembled were not square. Consequently, the threads were reamed out and lugs were attached which enabled the use of set screws as the means of fixing the pipe and plate positions.

The cooling of the magnet was provided for by a copper coil placed over the copper pipe which contained the furnace. Cooling was also expected from the brass support plates. The copper coil was flattened by passing it through rolls. It was thought that the flattened tubing (originally 5/8 in. round) would coil more neatly on the copper pipe. Considerable trouble was experienced in coiling the flattened tubing and it is believed that the flattening was not necessary. Over this tubing were placed several sheets of copper to act as a base for winding the primary. Sixty-five pounds of number 14 magnet wire were wound into the primary, close to a mile of wire, The brass plates between which the primary was wound had to be stayed by three tie rods. Water was run continuously through these coils. In summer the base temperature of the magnet was 70° F., in winter it was 45° F.

It was thought that cooling of the magnet would be facilitated by having the heavy brass plates in immediate contact with the wire of the primary, except for the thickness of the single cotton strand insulation. This practice has placed the magnet above ground potential when connected to the 220 volt D. C. line.

After continuous use the magnet became quite warm. At this stage, small arcs would form from the winding to the brass plates. This was rectified by inserting gasket material as insulation between the winding and the plates. (Not shown in the drawing of Figure 13.) The magnet as a unit is still above ground potential when connected to the 220 volt direct current line because the first layer of winding is wound over a copper sheet which in turn is wound over the copper cooling coil. The single cotton strand used as insulation is not sufficient to prevent electrical contact between the first layer of winding and the copper sheet.

The magnet unit is shown in the accompanying photograph, Figure 12. It was felt that more information could be obtained about the specimen under study in the time available during a magnetic run. Dilation data was to be required for interpretation purposes and it was decided to obtain this data simultaneously with the magnetic data.

Accordingly, a dilatometer was designed to serve the dual purpose of allowing the recording of length changes and the magnetic changes in the specimen. The body of the dilatometer was made of fused-quartz

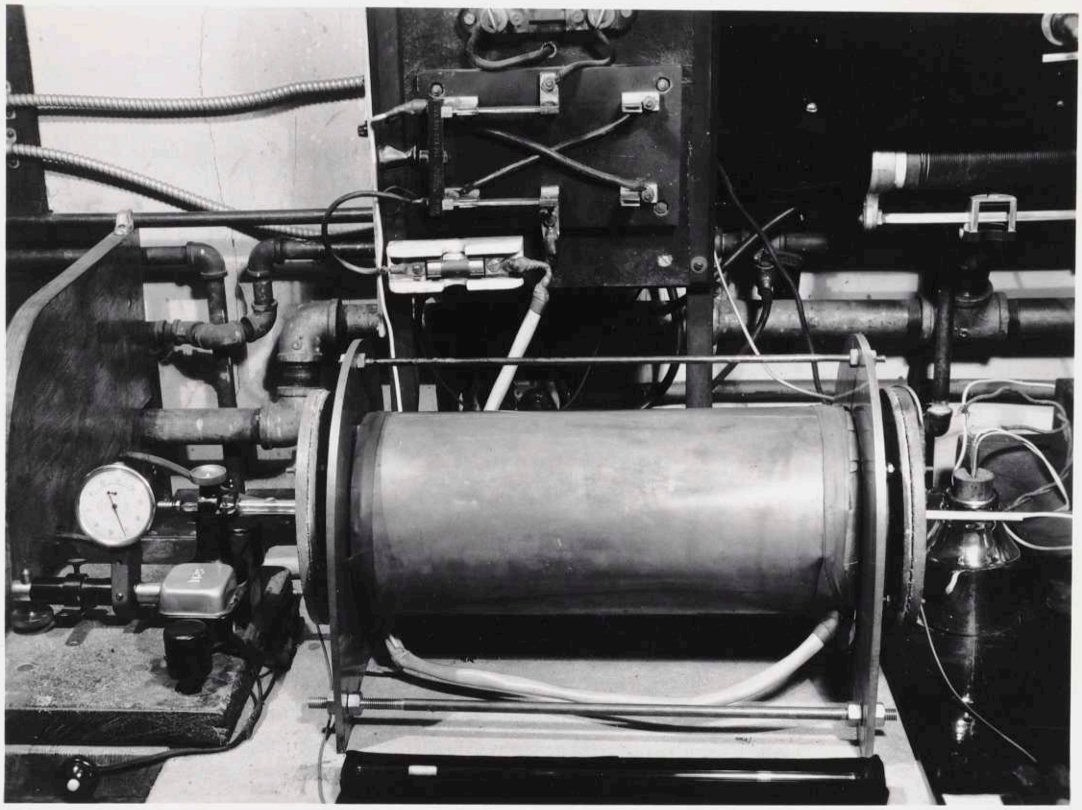


FIGURE 12. Photograph of the magnet unit.

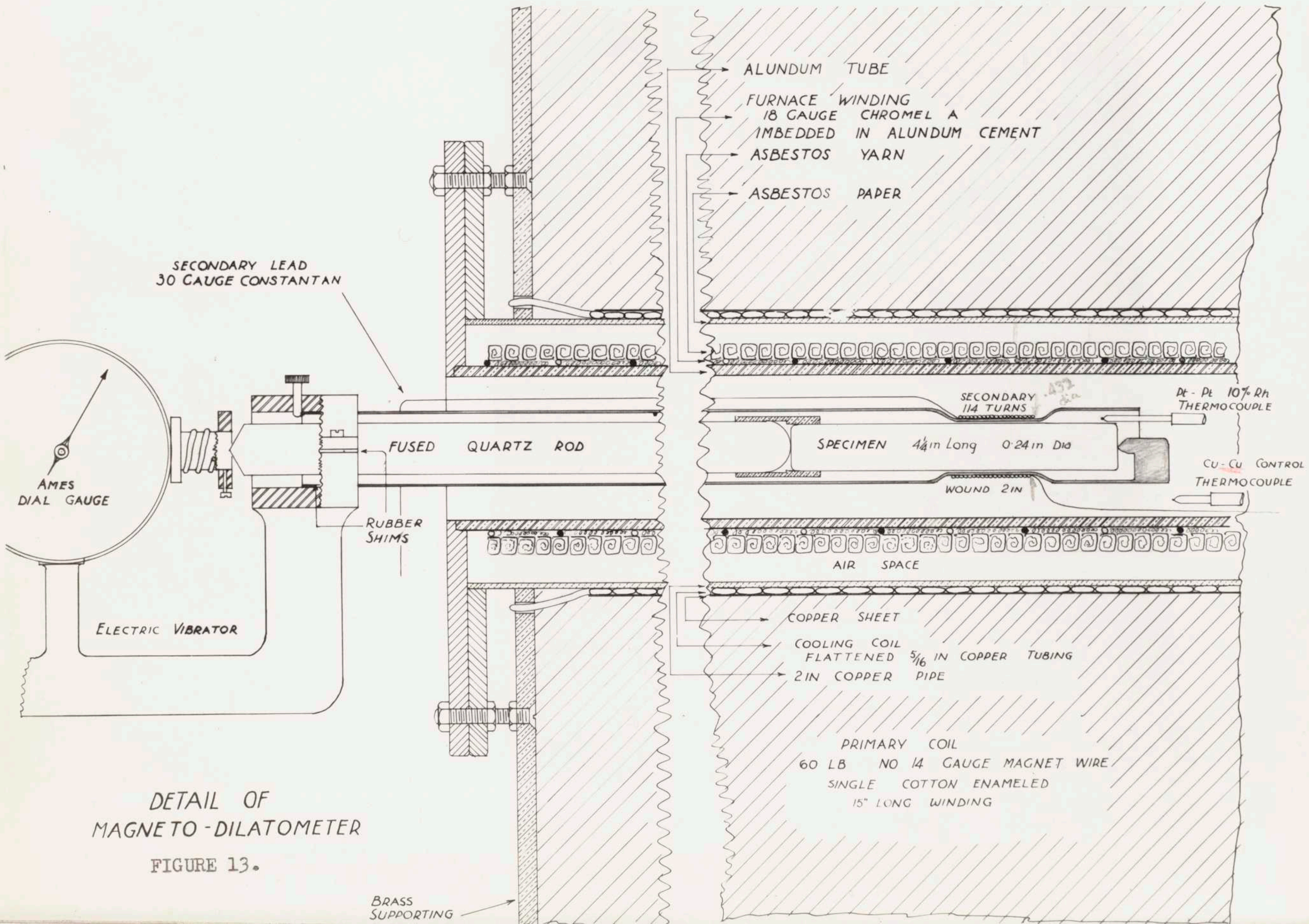
tubing. The outer diameter was 3/8 in. and the inner diameter was 5/16 in. To enable allocation of the specimen in a consistent manner in the numerous runs to be made, and also to fix the position of the secondary, a constriction was made in the quartz tube. The inner diameter of the constricted portion was just sufficient to admit a specimen 0.25 in. in diameter. The length of the constricted portion was two inches. To the end of the tube that was to be inside the furnace chamber was affixed a fused-quartz centering nib. This was sealed to the tube over a third of the periphery. The other two-thirds were left open to admit a thermocouple.

The specimen was supported by this nib. The communication of the length changes in the specimen to the Aimes dial gauge was made by a quartz rod. One end was made spherical and abutted against the specimen. The other end was made conical and fitted into the gauge. To avoid relative slipping of the rod and the specimen a thin brass tube was machined to fit both ends.

To isolate the movement of the rod to only the length changes in the specimen the quartz tube and the dial gauge were fastened to the same support.

The detail of the magneto-dilatometer is shown in the drawing of Figure 13. The dimensions have been purposely distorted to emphasize the important parts of the apparatus.

The detail of the supporting system for the dilatometer was not shown in the diagram of Figure 13, in its actual form because of its complexity. The detail of this stand can be readily grasped from the photograph shown in Figure 14.



DETAIL OF
MAGNETO-DILATOMETER

FIGURE 13.

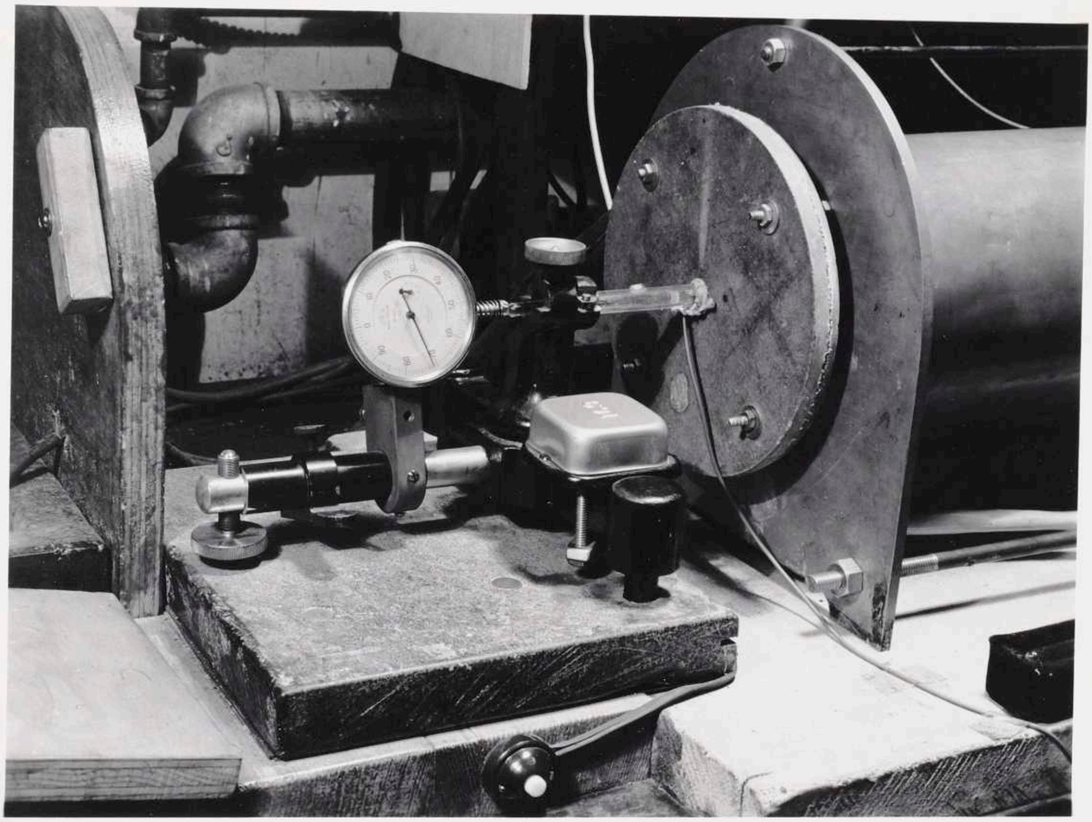


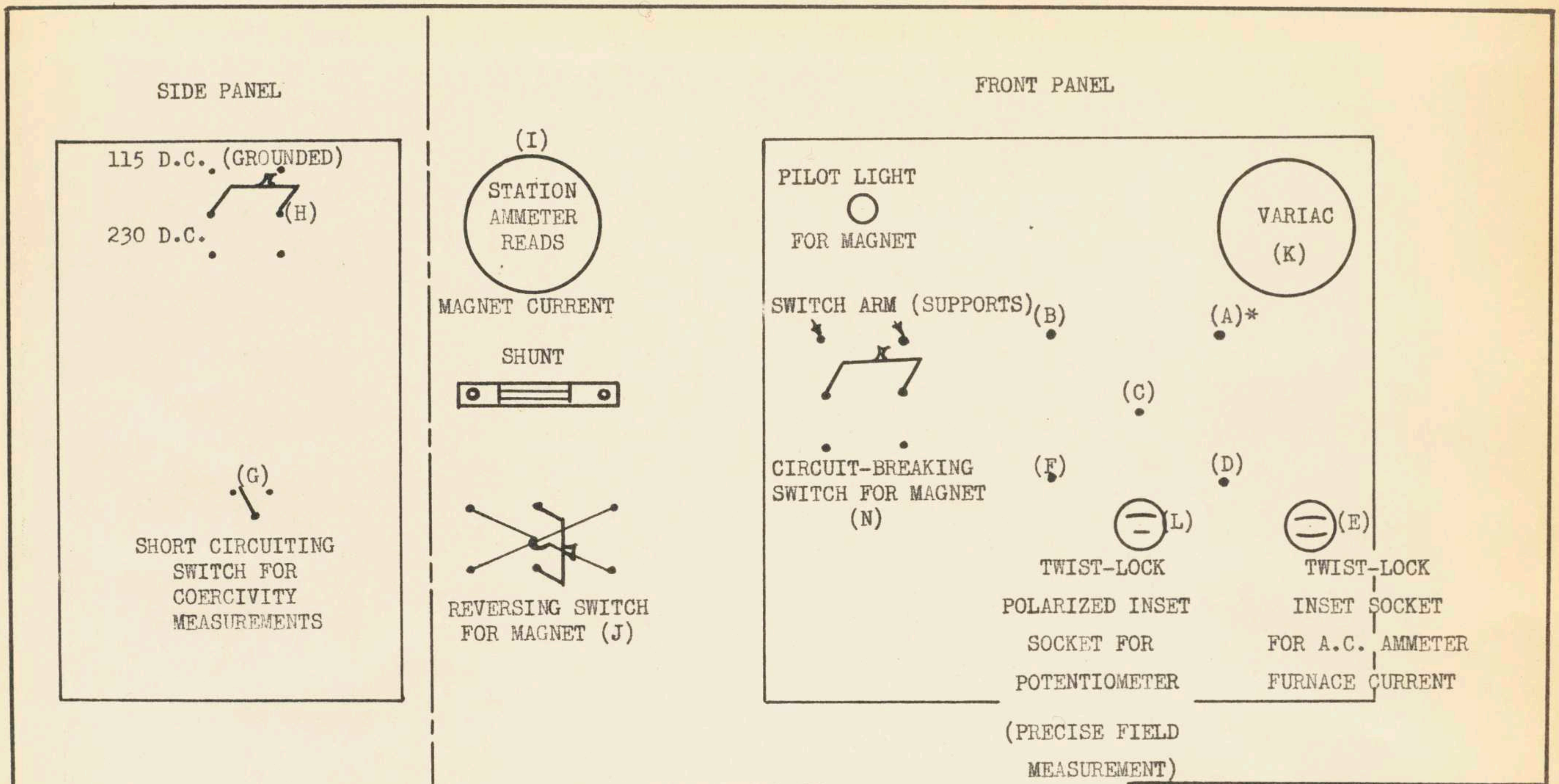
FIGURE 14. Photograph of dilatometer support.

In Figure 15 is shown a schematic detail of the front and side control panels of the apparatus. The capital letters used to designate the various parts may be identified as to their significance from an examination of the circuit diagrams of Figures 18, 19, 22, and 23. A photograph of the front control panel is shown in Figure 16. The side control panel may be seen in the photograph of Figure 17.

In Figure 18 is shown the diagram of the furnace circuit. The winding of the furnace was designed to allow a full-tempering range with 110 volt alternating current as power. The supply was fed into a variac from which the proper voltabe could be chosen to obtain the desired furnace current. The reading of the variac is given in per cent of line voltage and it has been found that any chosen percentage is approximately equal to the furnace current in amperes (It is actually slightly greater.).

The control unit used was a Brown potentiometer controller equipped with an Eagle Signal Corporation synchronous interrupter and a Microflex timer. The synchronous interrupter was substantially a synchronous-motor powered, variable-time drive on the controller setting. The travel of the drive was controlled in two ways: first, the gear ratio which controlled the time of revolution of a cam, and second, the cam setting which controlled the time during which the controller setting was being driven during any revolution of the cam. The minimum revolution time of the cam was one minute and the maximum was six minutes. The on-time could be varied at will below 100 per cent.

It is important to note that the speed of the drive while in motion is constant, since the drive is propelled by a synchronous motor.



* NOTE: THE LETTERS REFER TO ARTICLES DESCRIBED IN THE CIRCUIT DIAGRAMS

DETAILS OF FRONT AND SIDE CONTROL PANELS

FIGURE 15.

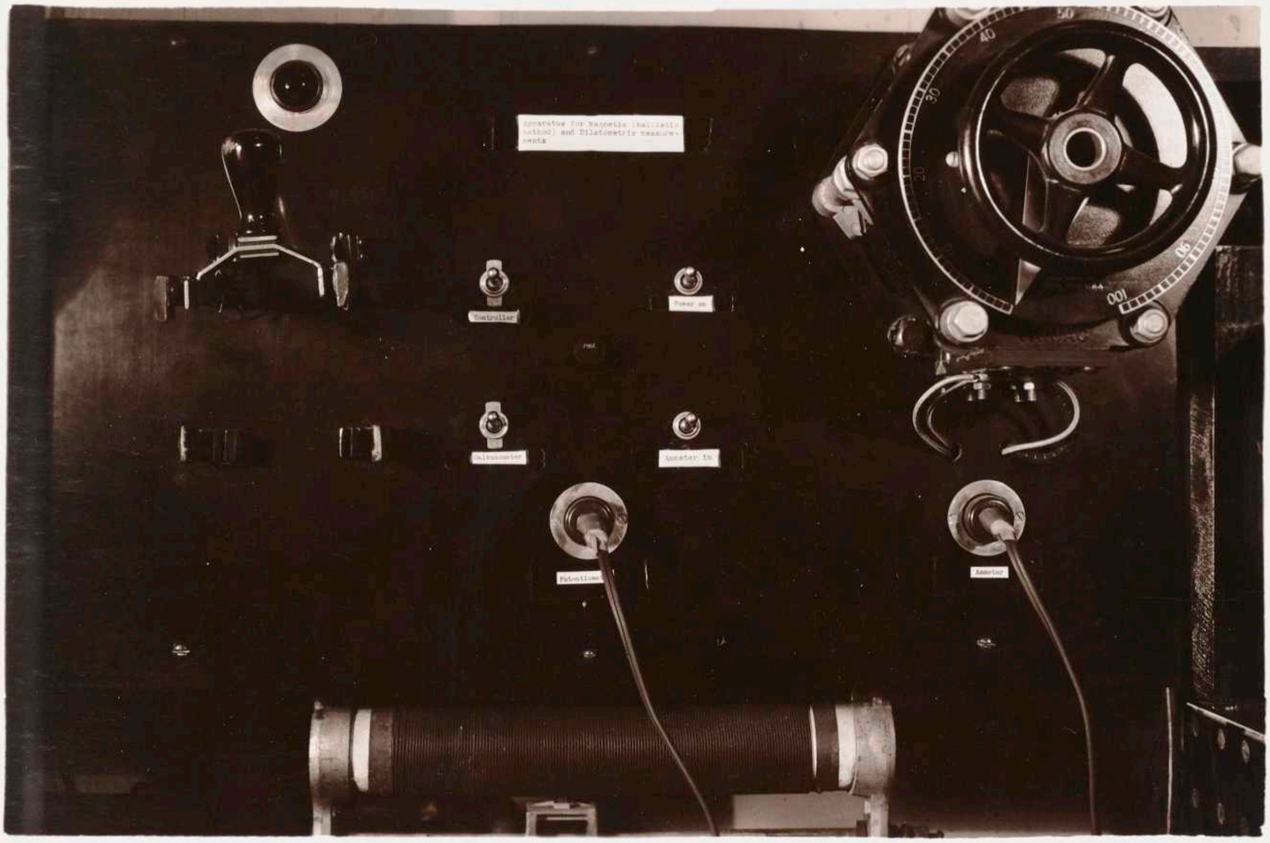


FIGURE 16. Photograph of the front control panel.

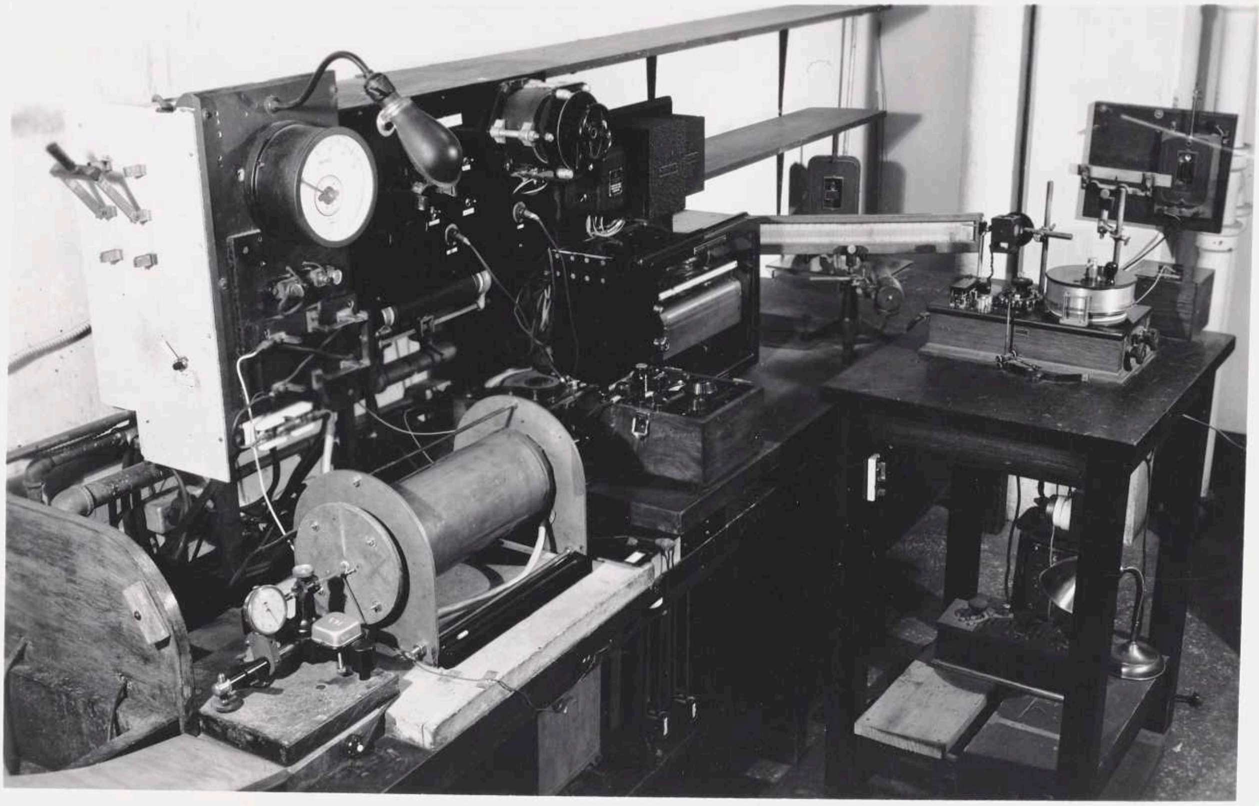
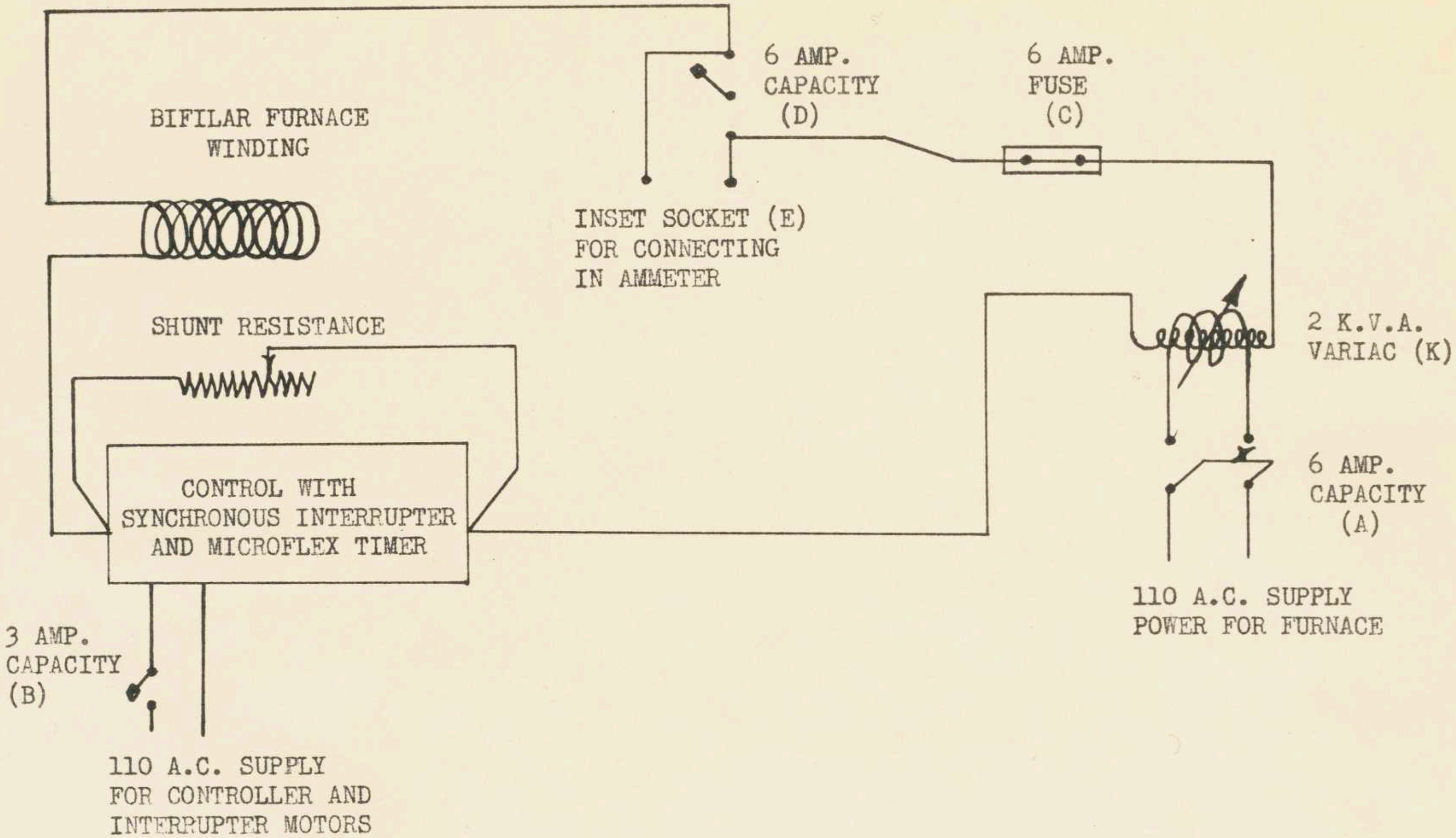


FIGURE 17. General view of the apparatus showing side control panel.



CIRCUIT DIAGRAM
-FURNACE CIRCUIT-

FIGURE 18.

Thus, if the on-time of the cam system is 100 per cent, that is, if the motor is in operation 100 per cent of the time of the cam's revolution, the drive will be a constant, regardless of the gear ratio or cycle setting, and will occur at a rate of 11.8° F. per minute. The gear ratio will only permit adjustment of time between on and off positions of the cam. These on and off positions can be adjusted relative to each other. By using the minimum on-time of 1 per cent and setting the gear ratio to the maximum cam revolution cycle of six minutes the minimum speed of the drive is obtained--a value of .114° F. per minute.

The speed used for the experiments recorded in this thesis, unless otherwise stated, was 5° F. per minute. This was true for both heating and cooling. This is actually an average speed as the drive-temperature relationship is based on a chromel-alumel thermocouple. Since a copper-constantan thermocouple was used the actual speed was somewhat greater than 5° F. per minute at temperatures below 500° F. and somewhat less at temperatures above 500° F. In order to have a continuous and not a step-wise rise in the temperature with time, two things were necessary. First, the variac had to be gradually increased with the temperature in order that the furnace should not pass beyond the driven controller setting, and second, a shunt resistance had to be used to carry some current when the controller was off. This had been installed in the apparatus. In this way, close control of temperature was possible in spite of the high temperature gradient between the furnace and the water-cooled jacket. The microflex timer was not properly connected to enable utilization of its full usefulness. However, it was used to start the controller setting on the cooling cycle.

A copper-constantan thermocouple was used for the control circuit because it was non-magnetic and absolute accuracy of temperature measurement was not necessary.

The necessity of insuring that all magnetic changes picked up by the secondary were due to the changes in the specimen only required that no other magnetic material be close to the coil of the secondary. The thermocouple used for temperature measurement thus had to be either of copper-constantan or of platinum-platinum-rhodium.

The latter was chosen because it could be relied upon after having been calibrated once. The temperature-measuring circuit diagram is shown in Figure 19. The copper-constantan thermocouple changed its thermoelectric-power as its time of use increased. It was renewed every 100 hours. The bond of this thermocouple was made with silver solder. The bead on the platinum thermocouple was made in the carbon arc. The low thermoelectric-power of this thermocouple required the use of a potentiometer that would read to a tenth of a microvolt. This potentiometer may be seen in the photograph of Figure 20.

Calibration of the platinum thermocouple was made by the use of standard melting-point samples supplied by the Bureau of Standards. They are listed below:

Aluminum - National Bureau of Standards, Standard Sample Number 44, Melting Point 658.7° C. (1217.7° F.).

Zinc - National Bureau of Standards, Standard Sample Number 43d, Melting Point 419.5_2° C. (787.1° F.)

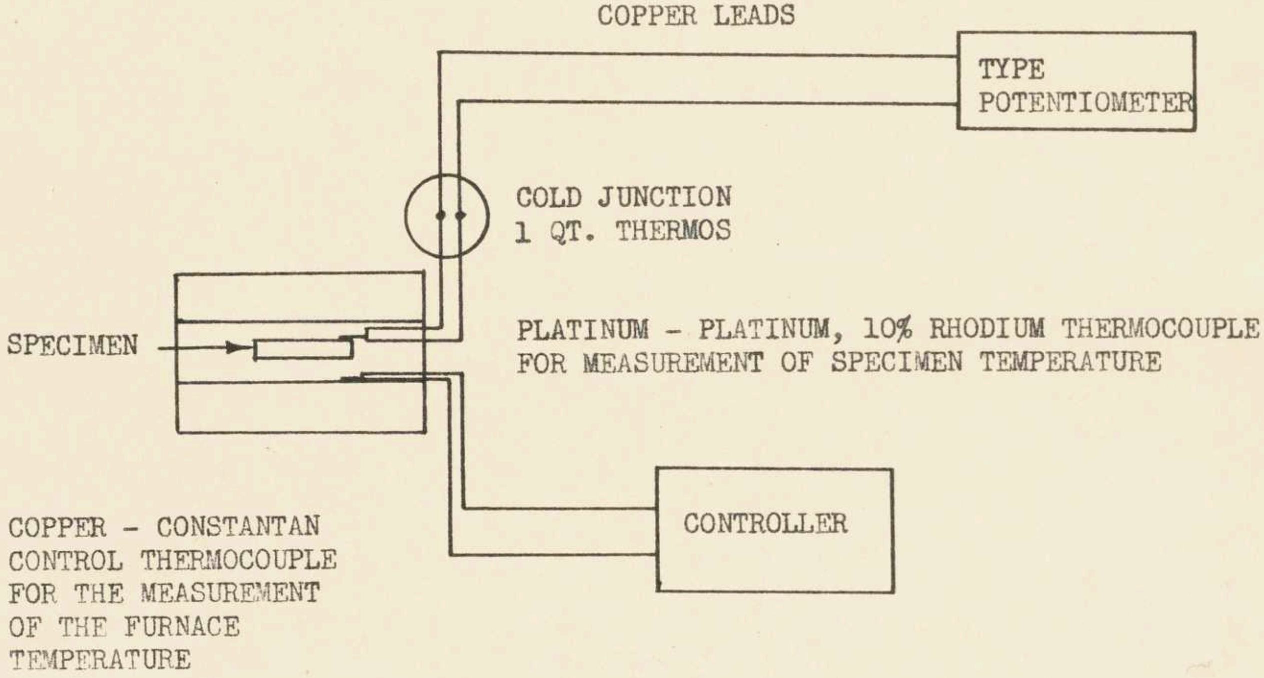


FIGURE 19.

CIRCUIT DIAGRAM

-THERMOCOUPLE CIRCUITS-

FIGURE 20. Photograph of ballistic galvanometer and of apparatus for temperature measurement.

Tin - National Bureau of Standards, Standard Sample Number 42a, Melting Point 231.9° C. (449.4° F.).

To insure reducing conditions during the melting of the standard samples, crucibles, drilled out of carbon-electrodes, were used to hold the metals, and graphite flakes were used to cover the surfaces. The platinium thermocouple was protected by a silica tube.

The size of the samples used for melting-point determinations was approximately 500 grams. The crucible and its contents were introduced into an electric furnace provided with external resistance. After the sample had melted it was allowed to cool. The cooling rates were made slow through the critical zone by gradually increasing the resistance in the furnace circuit. The electro-motive force of the thermocouple was determined by a type K potentiometer.

In this way, millivolt versus temperature curves were obtained for the three standard samples. From them, the values in millivolts corresponding to the horizontal portions of the three curves were obtained. A deviation chart permits easy and accurate determination of the temperature equivalent of a millivolt reading. One was made for the thermocouple used from the data given by Roeser and Wenzel(46). This is shown in Figure 21. From this curve and from the standard curve Table III was prepared giving the millivolts corresponding to temperatures from 500° F. to 1200° F. in 12.5° F. intervals.

It should be mentioned here that the cold junction is a very important source of error in this work. The mercury used for contacting the platinum thermocouple wires and the copper leads should have a dry surface or else

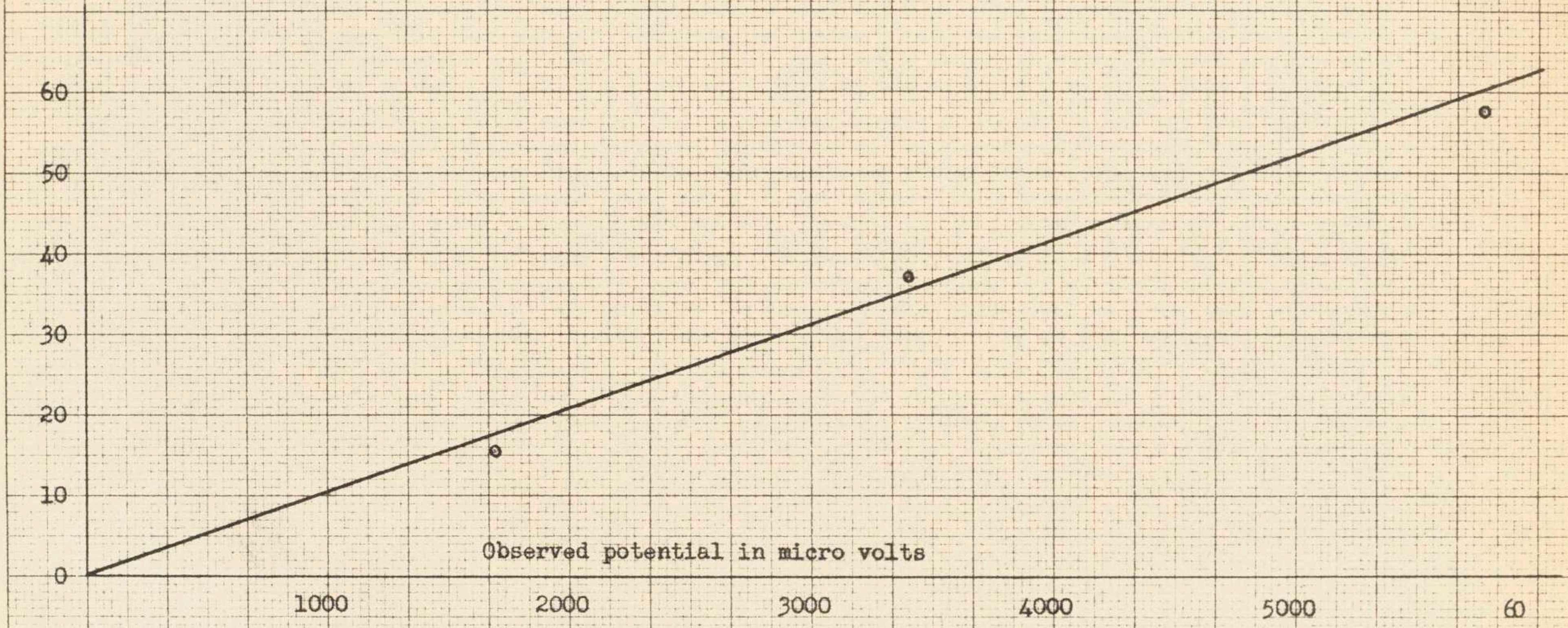


FIGURE 21 Deviation curve of the P t 10% Pt-Rh thermocouple used in the apparatus.
Standard couple-potentials given in B.S. Jrnl. Res. vol. 10, 1933, p. 275

TEMPERATURE Degrees Fahrenheit	VOLTAGE Micro-Volts	TEMPERATURE Degrees Fahrenheit	VOLTAGE Micro-Volts
50	56	650	2687
62.5	96	662.5	2751
75	136	675	2816
87.5	178	687.5	2880
100	220	700	2945
112.5	262	712.5	3010
125	306	725	3076
137.5	352	737.5	3141
150	397	750	3207
162.5	444	762.5	3273
175	492	775	3339
187.5	539	787.5	3404
200	586	800	3470
212.5	636	812.5	3536
225	687	825	3602
237.5	739	837.5	3669
250	791	850	3736
262.5	844	862.5	3802
275	896	875	3868
287.5	950	887.5	3935
300	1005	900	4002
312.5	1061	912.5	4070
325	1114	925	4138
337.5	1169	937.5	4205
350	1229	950	4273
362.5	1285	962.5	4340
375	1343	975	4408
387.5	1403	989.5	4477
400	1463	1000	4546
412.5	1516	1012.5	4614
425	1575	1025	4684
437.5	1635	1037.5	4752
450	1693	1050	4823
462.5	1753	1062.5	4893
475	1815	1075	4964
487.5	1875	1087.5	5034
500	1928	1100	5104
512.5	1996	1112.5	5174
525	2058	1125	5242
537.5	2115	1137.5	5313
550	2182	1150	5382
562.5	2246	1162.5	5453
575	2306	1175	5524
587.5	2368	1187.5	5596
600	2431	1200	5667
612.5	2495		
625	2560		
637.5	2623		

TABLE III

Table of temperatures and corresponding voltages developed by the platinum, platinum - 10 per cent rhodium thermocouple used in the apparatus.

the insulation on the wires should project below the surface of the mercury. The amount of mercury should be small, a one-half inch depth was used in 7-millimeter glass tubes, and should be well in the ice-water bath. Small thermos containers (pint size) are worthless as cold junctions for the temperature gradient between the mercury and the top of the ice both may be as much as 7.2° F. A tall size, one-quart narrow-mouth thermos bottle was used for this purpose.

There are several precautions that should be taken in working with a high precision potentiometer. Three galvanometer K? ~~d~~eys were provided. They allowed insertion of different resistances into the galvanometer circuit. The high resistance key was always depressed first, adjustment of the battery resistance or of the slide wire made and then the lower resistance keys were depressed to secure the final adjustment.

These keys were independent of the electrical circuit of the potentiometer, itself. The rocking switch determined whether the circuit was open or the standard cell was in the circuit or if the thermocouple was in the circuit. A very sensitive standard cell was placed in the apparatus. The rocking switch should be depressed on the standard cell side only when a resetting of the battery resistance is to be made.

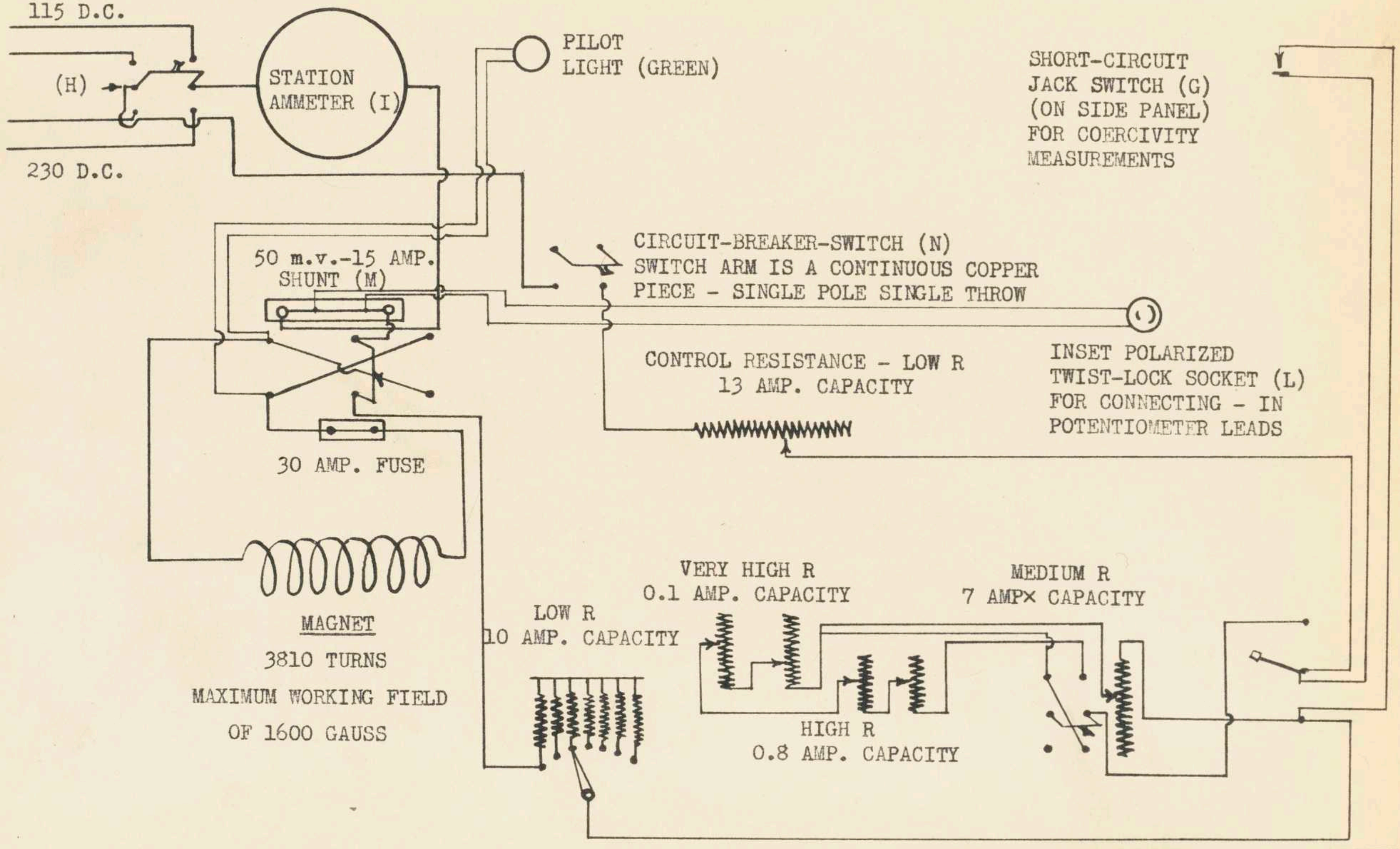
The battery used was a two-volt storage cell of 40 ampere-hours capacity. When the battery was charged resistance external to that present in the potentiometer had to be used.

A battery of this capacity enabled continuous measurement of the specimen temperature during a run without frequent standardization against the standard cell.

The slide wire after a period of idleness was noticed to have spots of faulty contact. A good practice is to turn the drum back and forth a few times at each setting. Since an accuracy of one degree was entirely sufficient for this work, the slide wire setting was only made to a microvolt.

In Figure 22 is shown a diagram of the magnetic circuit. A double-pole, double-throw switch on the side panel allowed quick charge of the voltage supply to the magnet. This was very convenient when runs were conducted in which measurements were made at alternate fields, 265 and 1000 gauss, every 12.5° F. The station ammeter served for coarse estimation of the magnet current. Accurate measurement of the field was made by the use of an ordinary external ammeter shunt. For a current of 15 amperes through this shunt a drop of 50 millivolts existed between the potential terminals. A Brown semi-precision portable potentiometer was used to read the drop across the shunt. This instrument allowed reading to .01 millivolt. Expressed in amperes this is 0.003, which at the currents normally used, 7 - 8 amperes, meant a precision better than 0.01 per cent.

In order to obtain a wide variation in the field for magnetization-field-relationship work, a wide variety of resistances had to be used. For currents below 0.8 amperes a single setting of the resistances would suffice during the time of a reading. The Institute power varied but this was a variation of minutes rather than of seconds, which was the time of reading. For higher currents, where the heating of the resistances became considerable, the control panel was used to secure the



CIRCUIT DIAGRAM

- MAGNETIC CIRCUIT -

FIGURE 22.

exact value of current desired. The field was thus held constant to one part in ten thousand at the normally-used field of 1000 gauss.

When the magnet was cold its resistance was low enough to allow a field of 1600 gauss to be produced. The maximum field that could be obtained after the magnet had been in use several hours was 1500 gauss.

At no time should the magnet draw current for a continuous period beyond two minutes. It is very unlikely that the condition requiring longer use of the magnet without interrupting the current will arise. The warning has been stated to prevent carelessness in this regard. A green-colored pilot light was inserted in the front control panel as an additional precaution.

The resistances in the magnet circuit were separated by appropriate switches, as shown in the circuit diagram of Figure 22, to allow short circuiting of the low-current-capacity resistances when desired. The short-circuiting jack switch on the side panel was introduced for the special purpose of coercivity measurements. By its use quick change from high to very low field was afforded. Ordinarily it should be open.

This switch is supplementary to another resistance-short-circuiting switch on the resistance bank. The latter is capable of carrying much heavier currents for long periods of time than the jack switch is.

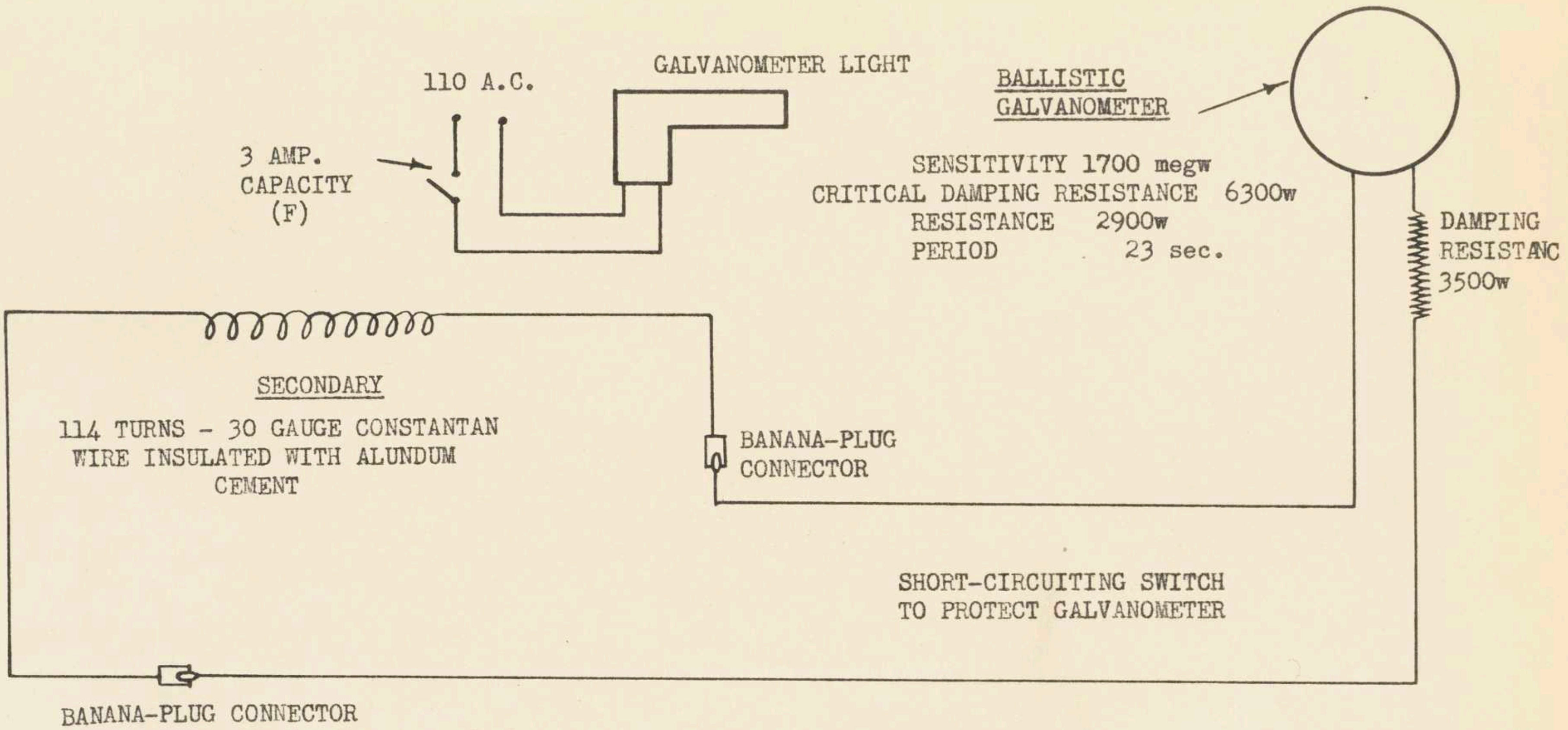
Current reversal of the currents normally used could not be made for any period of time with the reversing switch shown. The arcing would soon burn out the contacts. A circuit-breaking switch was installed. This switch was opened before the reversing switch was thrown to the other side. Having heavy graphite contacts, the switch was able to break the current of 13 amperes continually.

In Figure 23 is shown the circuit diagram of the secondary circuit. The first construction of this circuit employed a much more complicated arrangement. It included a variable damping resistance, a compensating resistance for the mutual inductance and extensive carry-back leads to the control panel. Probably due to a poor contact in this extensive circuit, erratic behavior was sometimes exhibited by the galvanometer.

The simplified circuit shown in Figure 23 was finally evolved. The secondary leads were brought clear of the magnet. Banana-plug connectors were used to enable insertion of mutual inductance for standardization purposes. Since the total resistance of the circuit was over 7000 ohms, the few ohms difference in the resistances of the secondary coil and of the damping resistance, composed of fixed one-half watt resistors, was chosen at 3500 ohms. The total resistance was slightly greater than the critical-damping resistance given as one of the galvanometer characteristics. The extent of under-damping existant, however, enabled successive readings to be made more rapidly. A short-circuiting switch was included to protect the galvanometer during the times when the circuit was opened and closed.

Several considerations entered into the construction of the secondary coil. The most important consideration was the number of turns to be used as this determined the scale deflection of the galvanometer.

It was found that 114 turns gave a full-scale deflection for the most magnetic state of the steels studied. To insert the number of turns in the two-inch space provided on the quartz tube required the use of 30 gauge wire. The coil was wound with insulated wire. The insulation was then impregnated with a suspension of alundum cement. This was necessary in order that the turns would not be short circuited at elevated temperatures.



THE BANANA-PLUG CONNECTORS ALLOW THE INSERTION

OF THE MUTUAL INDUCTANCE FOR STANDARDIZATION PURPOSES.

USE MUTUAL INDUCTANCE OF RESISTANCE CLOSE TO 100w.

CIRCUIT DIAGRAM

-SECONDARY CIRCUIT-

FIGURE 23.

The need for good contacts in the secondary circuit cannot be overemphasized. This is evident from the magnitude of the current that is discharged through the circuit. The ballistic galvanometer used had a sensitivity of 1700 megohms. This means that the galvanometer would give a deflection of one millimeter on a scale one meter distant from the mirror if current of 0.6×10^{-9} amperes existed in its circuit. For the average deflection obtained this circuit current would be 0.6×10^{-7} amperes.

The primary was standardized by observing the deflections produced by the secondary coil alone under the application of various fields. From the equations derived in the first part of this thesis the curve shown in Figure 24 was obtained. This gives the field in gauss produced by any given current in the primary. The curve which shows the relation between the deflection produced by the coil alone and the field is shown in Figure 25. The field versus current curve is shown magnified in the lower-field portion in Figure 26, and the coil-deflection versus field curve is shown magnified in the lower-field portion in Figure 27. These latter two curves were used for coercivity measurements.

The operation of the apparatus may be summarized by the following directions. (See Figure 28 for a general view of the apparatus.)

1. Prepare the specimen in form of a rod, 4-1/4 in. long and not over 0.25 in. in diameter. Scale and decarburization should be removed.
2. Insert the specimen into the quartz tube and push the specimen gently to the back of the tube with the glass rod. This is important,

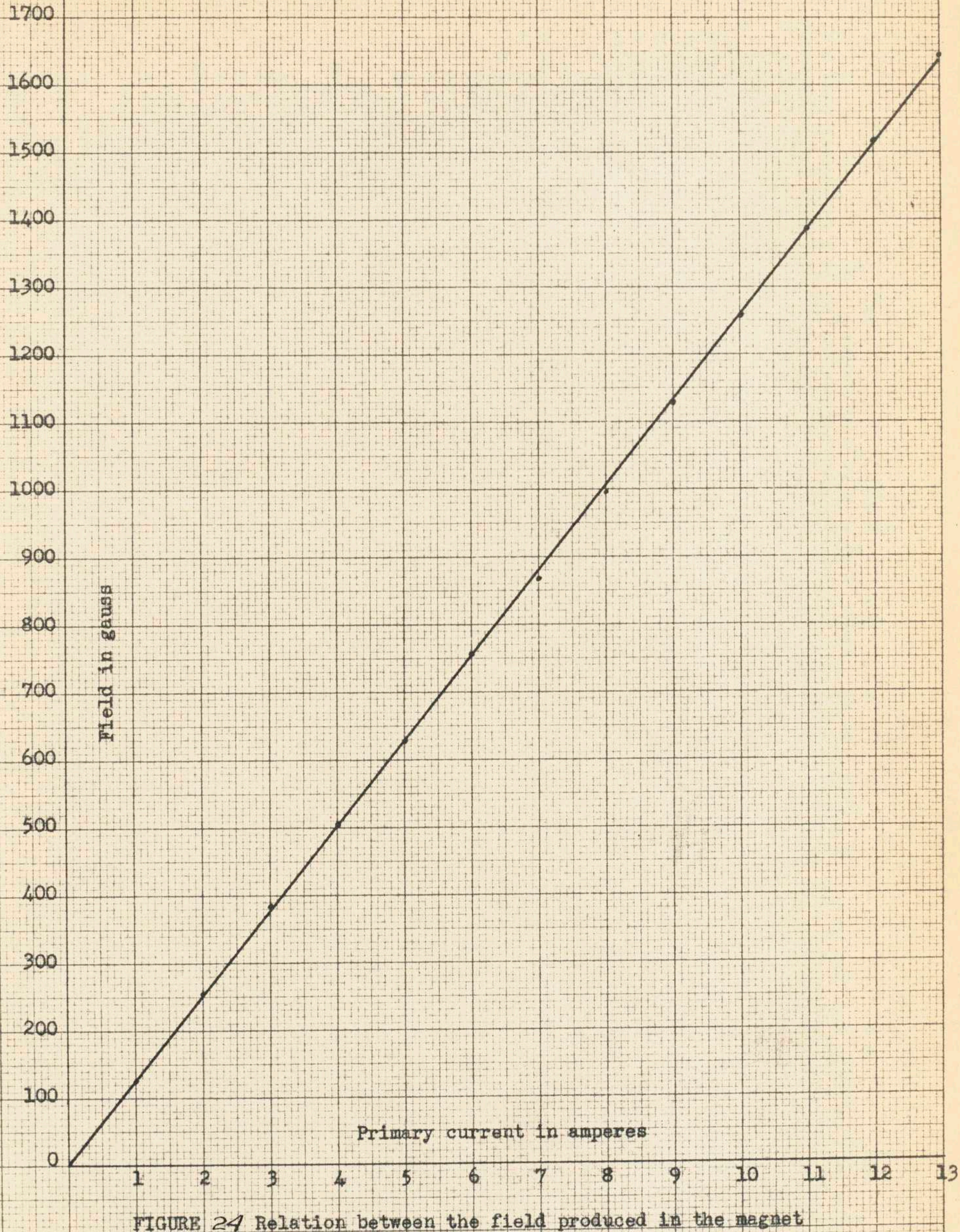


FIGURE 24 Relation between the field produced in the magnet and the current in the primary coil.

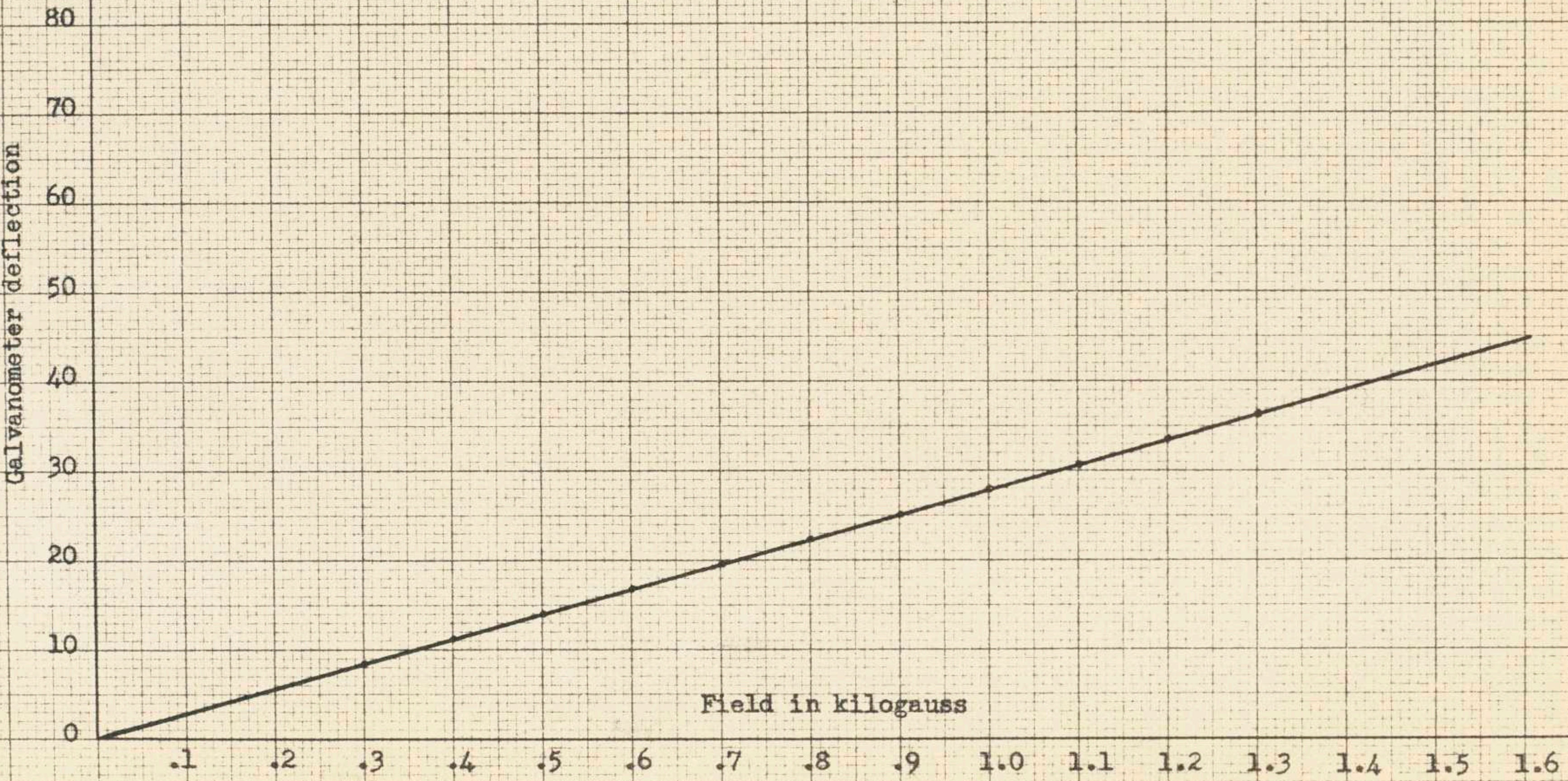


FIGURE 25 Galvanometer deflection produced by the secondary coil under the working range of the magnet.

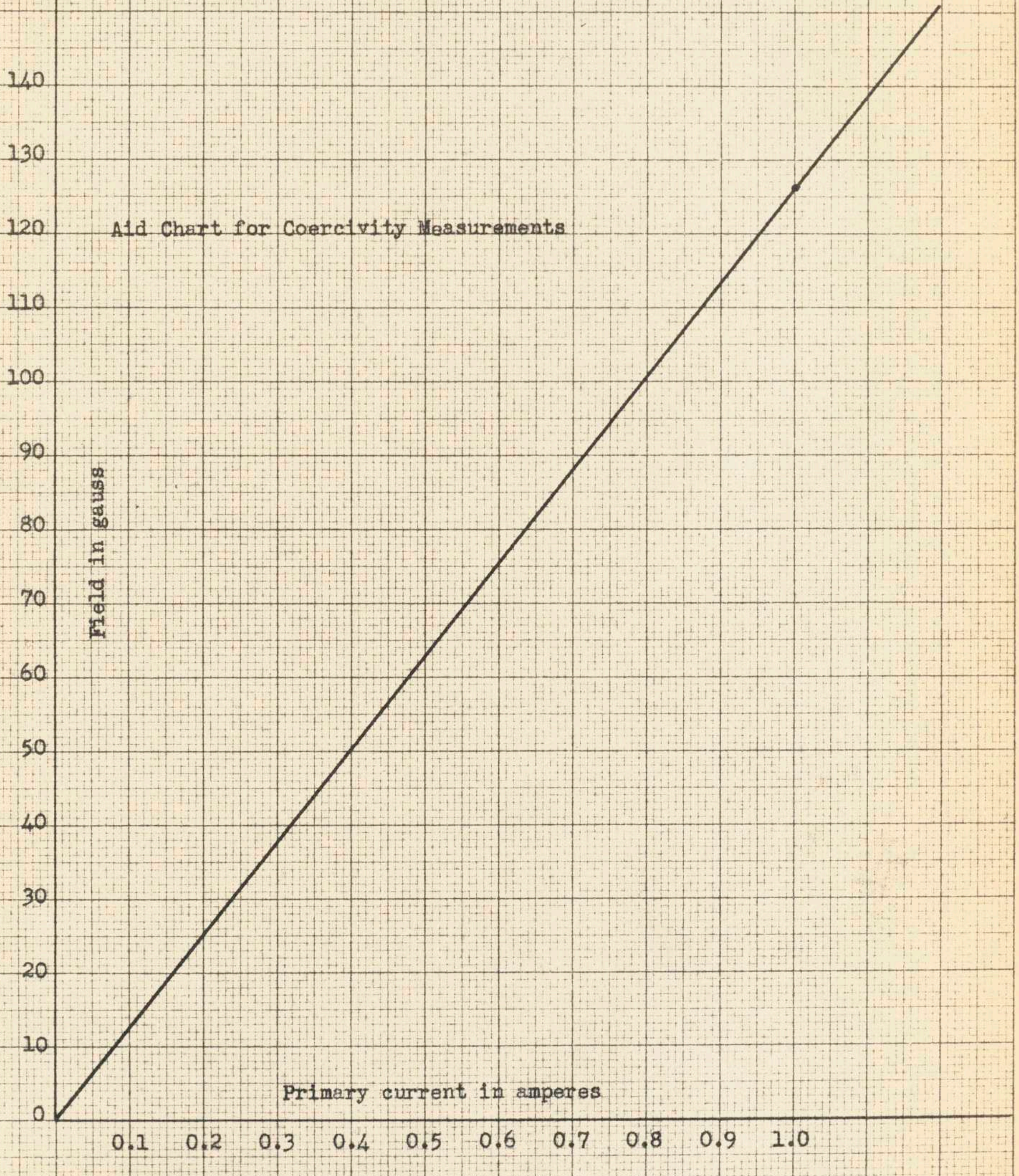


FIGURE 26 Relation between the field produced in the magnet and the current in the primary coil for low values of the current.

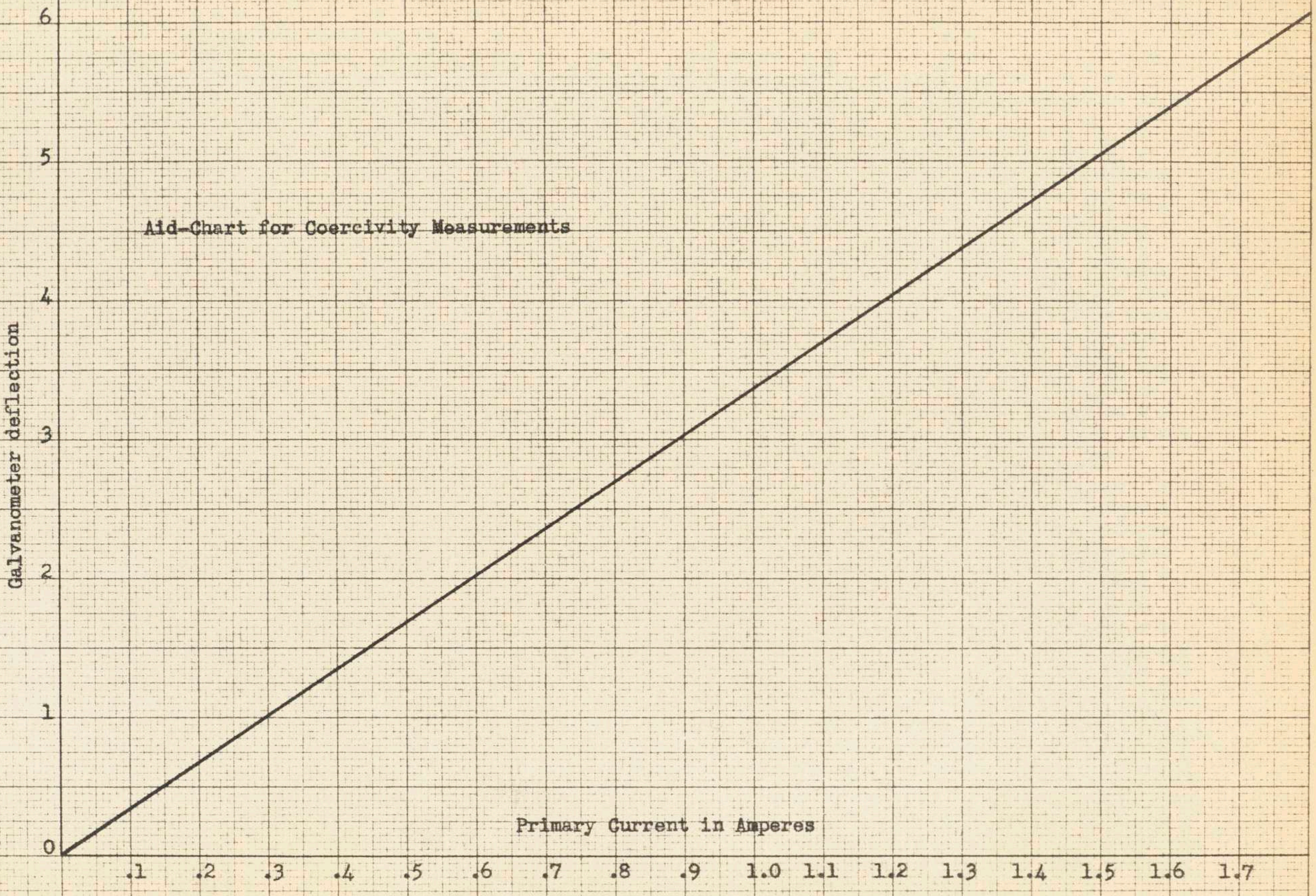


FIGURE 27 Galvanometer deflection produced by the secondary coil for low fields.

FIGURE 28. General view of the apparatus.

for if the specimen were not seated, it would fly into and beyond its proper position, carrying the end of the quartz tube with it, when the current was switched on.

3. Adjust the current to the value required to produce the desired field with the galvanometer short circuited. Multiply current by three to obtain the proper millivolt value.

4. Close the circuit-breaking switch, open, throw the reversing switch to the opposite side, close the circuit-breaking switch, open, throw the reversing switch, close--repeat this cycle as many times as is necessary to put the specimen in the cyclic condition. At fields of the order of a few gauss this should be done at least fifteen times; at fields of several hundred gauss once is sufficient. The fundamental test is to increase the number of reversals until no change is produced by further reversals. During these operations the short-circuit switch on the galvanometer should be closed.

5. Open the galvanometer short-circuit switch, check the value of the field.

6. Read the position of the hair line on the scale.

7. Quickly open the circuit, throw the reversing switch and close the circuit.

8. Watch the scale for the maximum deflection.

9. Close short-circuiting switch and open circuit.

10. Obtain deflections as difference between (8) and (6).

If a temperature run is to be made the following directions should be added to the above.

11. Insert the dilatometer rod and secure the Aimes dial gauge in position. Have enough starting on the spring compression to enable the dilatometer to follow the contractions with sensitivity. Four to six revolutions of the dial have been found to be sufficient.

12. Set the synchronous interrupter to a minute cycle (mesh the two large gears) and use a 65 per cent on-time. This will give a heating or cooling rate of 5° F. per minute.

13. Start the furnace with the variac set at 18. Increase every 25° F. by an additional setting of 2. Do not go beyond a setting of 60.

14. After the desired temperature has been reached, set the variac back to 2 to 5 divisions depending upon the temperature. Set off-resistance to one-eighth of the total length.

15. The potentiometer key may be kept depressed to enable a continuous representation of the temperature to be made.

For coercivity measurements:

16. Establish the temperature at which the coercivity is desired to be obtained.

17. Obtain the deflection, as described above, for a field of 1000 gauss.

18. Open the side panel short-circuiting switch and increase the resistance of the magnet circuit until the current is approximately 0.1 ampere.

19. Close the short-circuiting switch and procede as if a deflection at 1000 gauss were to be made.

20. Open the circuit-breaking switch, throw the reversing switch, but before closing the circuit, open the short-circuiting switch. This must be done in rapid succession.

21. Obtain the throw and the actual deflection.

22. Compare with half the deflection found in (17).

23. Repeat the procedure using a greater of smaller lower field. Obtain three points.

24. Follow directions given in first part of the thesis under theory.

a. Sensitivity of the Magnet

The sensitivity of the apparatus can best be gauged by the carbon content of the steel in which the carbide curie point, A_o, can just be detected. Ellinger(31) stated that the unpublished records of the Magnetic Section of the National Bureau of Standards showed that a definite A_o (of the order of 3 per cent) was found in an annealed steel containing 0.14 per cent carbon. The magnometer method was used here, and has been described by Sanford(25). Buehl and Wulff(73) claimed that with their torsion balance they could determine iron carbide in stainless steels to as low as 0.01 per cent. Buehl, Hollomon, and Wulff(88), using the same apparatus, claimed to have found a curie point in Armco iron containing 0.04 per cent carbon.

Sanford and Ellinger(32) have shown that the A_o point in an 0.75 per cent carbon-steel wire is completely masked by cold-drawing the wire. These authors have also investigated the effect of carbide particle size upon the A_o point. Honda(1) concluded that the A_o was constant if the carbide was lamellar. Sanford and Ellinger showed that for the spheroidized state the temperature of the beginning and end of the carbide magnetic change depended upon the particle size. The larger were the particles, the narrower was the range. Of more importance is their finding concerning the effect of particle size upon the intensity of the magnetic change occurring at the A_o range. For carbide so finely dispersed as to be barely resolvable under the microscope at 2000 diameters magnification, the intensity of the change was about half that for the lamellar state. As the particle size increased the intensity of the change decreased.

As indicated above for the case of cold drawing, the curie point of the carbide is really a very sensitive phenomenon. The mere existence of carbides is not enough; they must also be in the right state for a curie point to become evident.

Thus, in order to obtain the proper estimate of the sensitivity of the magnet the specimens to be studied were treated so as to have the carbides in the same state. Annealed plain-carbon steel specimens containing 1.25, 0.45, and 0.15 per cent carbon were placed in the apparatus and the magnetization versus temperature curves were determined up to 600° F. The curves are shown in Figure 29. The carbide curie point can be barely noticed in the 0.15 per cent carbon steel. The temperature of the magnetic transformation can be readily selected to be 410° F. for the 1.25 per cent carbon steel; but, as the carbon content was lowered the point became indefinite.

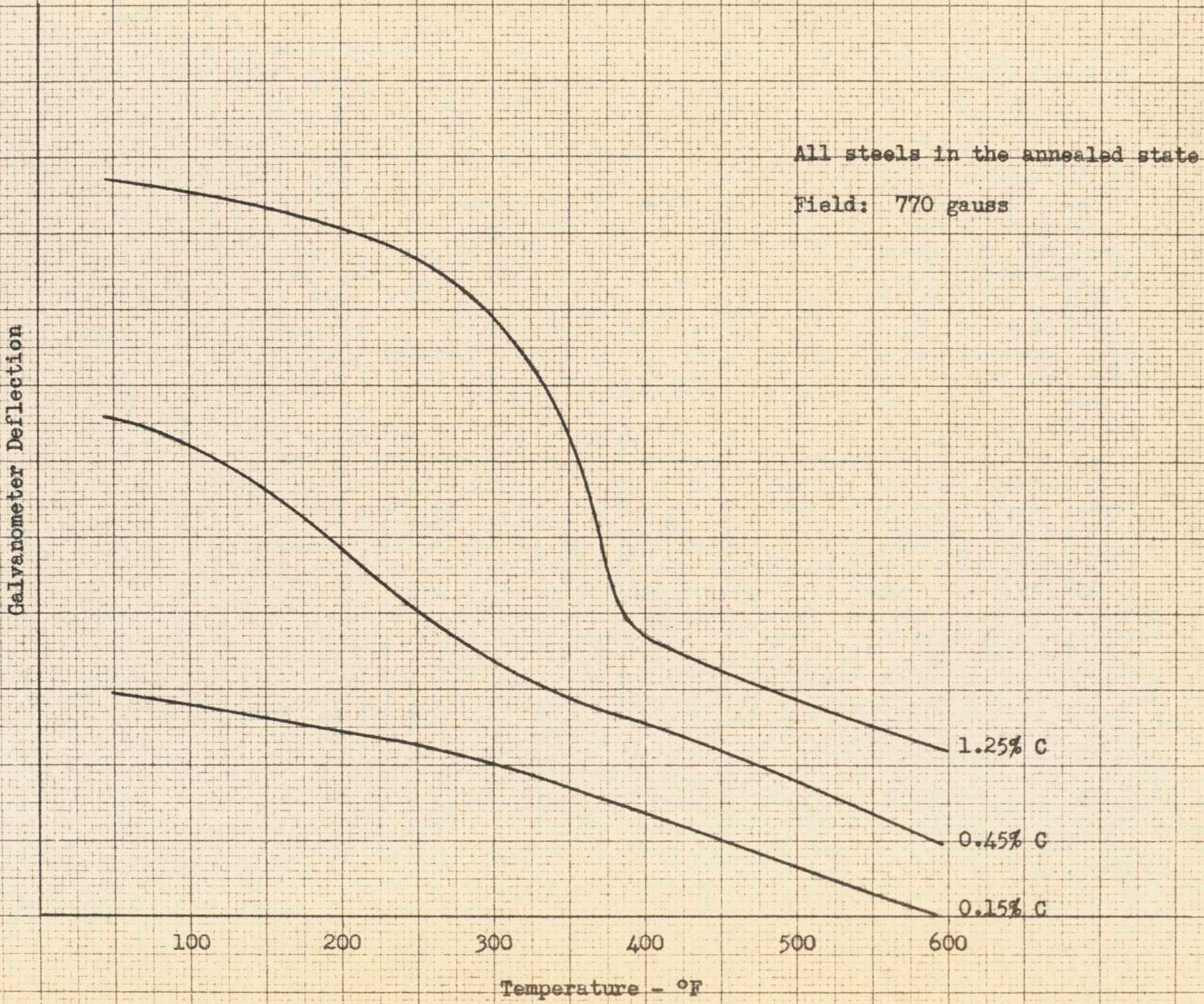


FIGURE 29 Magnetization as a function of temperature for annealed plain carbon steels.

An interesting observation relative to the effect of field on sensitivity can be made from a study discussed in Chapter VII - B - 5 (Figure 83). The inflection of the cooling (magnetization curve upon passing through the curie point was more marked in the 265-gauss curve than in the 1000-gauss curve.

It is evident from the above that the ballistic method is not as sensitive as the magnetometer or the torsion-balance methods.

C. Isothermal Study

The purpose of this study was to determine the tempering characteristics in regard to austenite transformation of the two steels used in the research. Since the effects of the particular temperatures were to be obtained uniquely the specimens had to be heated-to and cooled-from the tempering temperatures quickly. This was accomplished by the use of molten-metal baths, molten-salt baths, and boiling-liquid baths followed by either water- or oil-quenching to room temperature.

A preliminary series of runs was first made upon specimens of Sparta steel air-hardened from 1700° F. Using duplicate samples and different specimens for the hardness and for the magnetic runs, the following measurements were obtained:

1. Magnetization under a field of 1000 gauss.
2. Magnetization under a field of 265 gauss.

The result of this series showed that the most sensitive indication of austenite transformation was given by the changes in magnetization as measured under a field of 1000 gauss. Duplicate specimens were run and showed such good agreement with the "originals" that the duplicate specimens were not used for the systematic study.

Accordingly, a program waslaid out which would yield a comprehensive picture of the transformation of the retained austenite in both the 1% C - 5% Cr and 1.5% C - 11.5% Cr. The outline of the program follows:

STEELS - 1% C - 5% Cr
1.5% C - 11.5% Cr

SIZE - 4-1/4 in. long, 0.24 in. diameter

HARDENING TEMPERATURES AND TIMES - 1700° F. - 50 minutes
1800° F. - 45 minutes
2000° F. - 30 minutes

RATES OF COOLING FROM HARDENING TEMPERATURES -
90° F. per second - oil cool
1° F. per second - air cool
0.5° F. per second - lime cool

TEMPERING TEMPERATURES - Room temperature 212, 300, 400, 500
600, 700, 800, 900
1000, 1100, 1200, °F.

MEASUREMENTS - Galvanometer deflections under an applied field of 1000 gauss.

Since some transformation occurred at room temperature, it was necessary to standardize upon the time between the hardening treatments and the beginnings of the runs. Furthermore, since the rate of cooling was critical, it had to be also standardized for each method of cooling.

The method used in this series was to harden at one time the specimens needed for a run at a particular tempering temperature. This meant six specimens were hardened in one treatment. There were three specimens of each steel; those to be oil-hardened were quenched separately, those to be air-hardened were wired together and withdrawn from the furnace at the same time and cooled together upon the hot piece of asbestos which supported them while in the Diamond Block. The specimens to be lime-cooled were also wired together and were withdrawn from the furnace and inserted into the lime bed as a unit.

Measurements were made on the oil-quenched specimens as soon as possible. The air-hardened specimens were allowed to cool 1-1/2 hours and the lime-hardened specimens were allowed to cool 3 hours before measurements were taken. In all instances the samples were cleaned of the black, tightly-adhering film produced by the hardening treatment. Number one emery cloth, slightly worn, was sufficient. Even in the instances of the air-cooled specimens in which the film was heaviest, the film's removal with the resultant restoring of the original bright surface to the steel did not have any measurable magnetic effect.

The specimens of each type of steel came from the same heat, and were in the shape of forged rods from which the decarburized surfaces had been removed by centerless grinding. The lengths of all the specimens were maintained at 4-1/4 in. They were prepared by cutting with a hacksaw to the approximate size and then grinding to the desired length on an ordinary laboratory belt grinder with the aid of a jig. The sharp corners were rounded off.

The steels in the form of rods were immune to cracking during hardening. This was true even when deep number impressions were made. In the form of cubes or the like, however, the steels would be cracked during hardening of only a very slight impression of a number stamp was left.

An interesting observation relates to the cutting of these steels in the annealed condition. If they were cut with continuous strokes, the saw blade soon dulled, regardless of whether it was high-speed or plain carbon. Using this practice, three or four cuts was the maximum, despite the fact that the steels had a Brinell hardness of 200. However, if only a few continuous strokes were made and oil was applied between times, the plain-carbon blades would cut over twenty pieces and remain sharp. It is evident from this that the heat developed by the frictional resistance of the steel to the blade quickly tempered the teeth to the point where they were too soft to hold their edges.

Tempering temperatures above 700° F. were produced by a molten-lead bath. Molten-salt baths were used for temperatures above 300° F. A boiling, refluxed carbitol-water mixture was used for the 300° F. tempering, and boiling water was used for the 212° F. tempering. In some instances, the temperings beyond 50 hours were continued in wire-wound muffle furnaces.

All the baths were controlled by the usual controlling pyrometers and maintained their temperatures with an individual variation of not more than plus or minus 3° F.

At the proper time, depending upon the method of hardening, the cleaned specimen was inserted into the magnet and the "as-hardened" magnetization value was determined. The specimen was then immersed in the bath working at the temperature under study. The times at temperature varied logarithmically from 7 seconds to several hundreds of hours. In most instances the same time intervals were used for the different specimens.

It had been determined prior to the systematic run that the time of reaction was independent of the number of individual tempering times composing the cumulative time provided the heating-to and cooling-from the tempering temperature were rapid.

After their magnetic values had been determined, the specimens were put back into the tempering baths until the time when they were to be again measured. The specimens tempered in the salt baths were cooled in water upon removal from the bath, and those tempered in the lead bath were cooled in oil.

The combination of two steels, three hardening temperatures, and three cooling rates resulted in eighteen specimens for each tempering temperature. The twelve tempering temperatures required a total of 216 specimens.

D. Kinetic Study

The specimens used for the kinetic study were centerless ground rods, 0.24 in. diameter and 4-1/4 in. long. To obviate the room temperature transformation the specimens were heat treated immediately before the time of the study. The oil-quenched specimens were introduced into the magneto-dilatometer as soon as they were removed from the oil, and were then washed and dried. The air-cooled specimens were allowed to cool for 1-1/2 hours and the lime-cooled specimens were allowed to cool for 3 hours before they were introduced into the apparatus.

A detailed list of instructions for conducting a kinetic study may be found in the chapter on the description of the appratus, pages 79 to 87.

E. X-ray Study

An X-ray study was conducted with a two-fold objective. The first was the clarification of the kinetics of the process. The second was the determination of the amount of retained austenite in quenched specimens of the steels studied.

The method used was that described by Gardner(91). A gas tube of the Hägg type was used with a chromium anticathode. The Phragmen camera used allowed measurement of medium angles—a range of $\sin^2 \theta$ from 0.23 to 0.7. The exposure time was 20 minutes under the tube conditions of 45 kilovolts and 10 milliamperes. Traces of the film were made on a Moll recording micro photometer. Reproductions of these traces are shown in Figures 87 and 88 of the chapter on the results of the X-ray study. Aluminum lines occurred on all of these as the method of Gardner is based upon a comparison of austenite-line, aluminum-line density ratios in an unknown and in a standard specimen of known austenite content.

The specimens had to be ground to the curvature of the camera before hardening in order that sharp lines could be produced and line-shift avoided. After hardening, the surface was etched for 15 - 20 minutes. This etching was done electrolytically at a current of 1.5 amperes and in an aqueous solution of 5 per cent mixed acids, hydrochloric and sulphuric.

In order to study the amount of retained austenite in quenched specimens by the X-ray method, photograms were made so as to have densities in the range 0.6 to 1.2 so that the intensities and densities should be directly proportional to each other. A microphotometer trace was made of each film.

The aluminum- and the austenite- (200) alpha lines were used for the measurements because they were not affected by the lines of the other phases. In order to obtain more accurate measurements modified traces were made. That is, the intensity of the microphotometer light was adjusted to give the maximum-scale deflection of the galvanometer when the beam was focused on the background adjacent to the aluminum- (200) alpha line. This background was always denser than the unexposed portion of the film because of scattered radiation. Thus, the lines in the modified traces were magnified over what they would be in traces made by using the clear portion as the background.

The most probable straight line was drawn through the background immediately adjacent to the austenite-and the aluminum- (200) alpha peaks on each trace. Lines were dropped from the tops of the peaks, perpendicular to the ruled abscissae. The intersections of these lines with the background line determined the lengths to be used in the calculation of the peak densities: The calculation was carried out by the use of the following formula:

$$\text{Density} = \text{logarithm}\,\frac{\text{Galvanometer deflection of background}}{\text{Galvanometer deflection of top of peak}}$$

This was done for the aluminum- (200) alpha line and for the austenite- (200) alpha lines. The ratio of these two densities gave the number that was used in calculating the retained-austenite content.

At the present state of the theory of this method, the austenite content is assumed to be in a linear relation, from 0 to 100 per cent austenite with the density ratio. That is, the density ratio is established for the 100 per cent austenite state of the steel and is assumed to go linearly to zero in the completely transformed state.

Parameter determinations were simplified by the use of the Gardner charts. These charts enabled the ferrite parameters, the austenite parameters, and the tetragonal-martensite axial ratios to be obtained from measurements on the films without any calculations. The Gardner charts were based on the distances of lines from the aluminum line (the tetragonal-doublet displacement was used directly) on a standard trace. The distance between the aluminum-alpha (200) and (111) lines were measured for each film and then divided into the standard chart distance of 114.7 millimeters to obtain the multiplying factor for the other distances in the film.

The precision thus obtained (0.3 per cent) was not great but was all that was necessary for the large changes that were considered.

F. Magnetic Determination of the Amount of Retained Austenite

Maurer and Schroeter's formula(24) for the calculation of the amount of retained austenite in a steel is strictly true if two conditions exist. The first condition is that either austenite be the only non-magnetic constituent present or that if non-magnetic carbides are also present their amount should be known. The second condition is that the saturation-magnetization values should be used for the constituents.

Under the fields available, saturation of the steels used could not be attained. This suggested that an extrapolation should be carried out to infinite field. The austenite contents determined at infinite field by this method were apparently not correct; for, in some cases negative values were obtained.

The magnetization of a steel specimen may be affected by stress and carbide content as well as by austenite content. We desire to know the austenite content. The stress content affects the magnetization only at fields below 350 gauss. The non-magnetic carbide content must be estimated from the micro-structure.

It was hoped that the exact nature of the field-dependency of the retained austenite content could be determined by means of the X-ray method. This was not possible, however, because of the segregation in the steels investigated.

A standard field of 1000 gauss was arbitrarily adopted for retained-austenite-content measurements.

The choices of the standard states for the steels were not immediately apparent. Maurer and Schroeter(24) used the annealed state as the standard state in their formula. They calculated that the completely martensitic state should be more magnetic than the annealed state, but they were not able to attain it.

It has been found in this investigation that the annealed states of the steels used did not have the maximum magnetization values. Magnetization values about one per cent greater were obtained from specimens quenched and tempered to transform all the martensite. This has been noticed for other alloy steels by other investigators(79)(85).

The reason for a higher magnetization value for the quenched-and tempered state than for the annealed state is that the carbides were different in kind and in amount.

The quenched-and-completely-transformed states were chosen as the standard states. The values assigned to these states were obtained by averaging the values obtained from many different specimens quenched and tempered until the austenite was transformed in each.

Figure 30 gives magnetization versus field curves for an air-hardened and completely transformed specimen, and iced-brine hardened and liquid air transformed specimen, and an annealed specimen. It can be seen that if the annealed specimen was used as the basis for calculation of the retained-austenite content the completely transformed state would have a negative value. The stress differences between the tempered and the liquid-air-treated specimens is shown by the spread of the curves below 350 gauss.

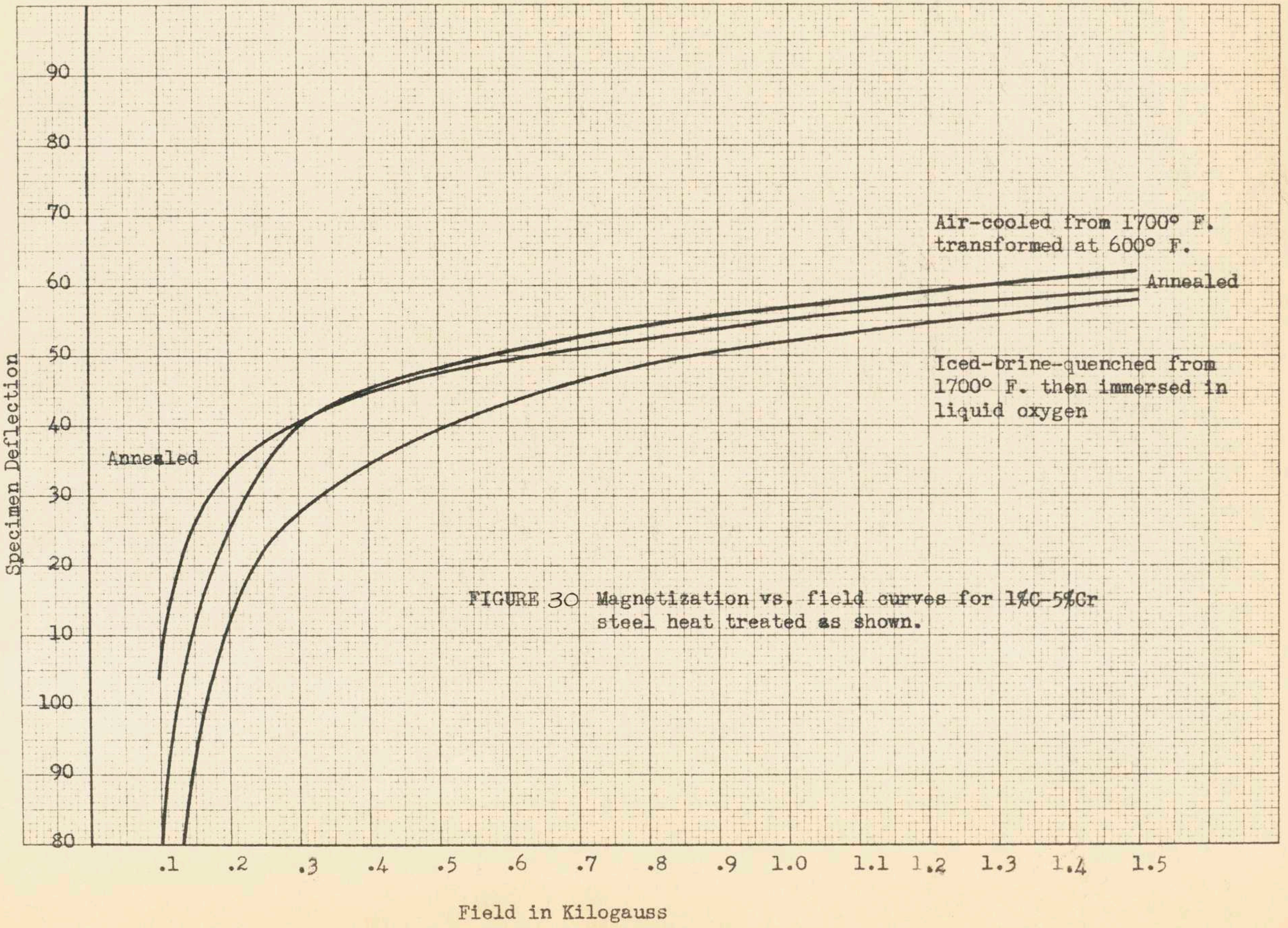


FIGURE 30 Magnetization vs. field curves for 1%C-5%Cr steel heat treated as shown.

VII

EXPERIMENTAL RESULTS

A. Isothermal Transformation

Data consisting of values for the galvanometer deflection (magnetization) at various times of tempering were obtained at twelve different temperatures. They were plotted on semi-logarithmic paper using the galvanometer deflections as the ordinates and times, expressed in hours as the logarithmic abscissae. Typical plots are shown in Figures 31 to 36 inclusive. The six figures give the eighteen aging curves at 900° F.––a total of 216 of these curves were obtained.

In Figures 31, 32, and 33 are shown the aging curves at 900° F. for the 1% C - 5% Cr steel cooled in oil, in air, and in lime from 1700, 1800, and 2000° F., respectively. In Figures 34, 35, and 36 are shown the aging curves at 900° F. for the 1.5% C - 11.5% Cr steel cooled in oil, in air, and in lime from 1700, 1800, and 2000° F., respectively.

The first measurements in all cases were taken after an immersion time in the bath of 7.2 seconds. The as-hardened values are given on the 0.001 hour ordinate, and are connected by broken lines to the rest of the curves. The maximum values of the galvanometer deflection attained averaged 185 for the 1% C - 5% Cr steel and 164 for the 1.5% C - 11.5% Cr steel.

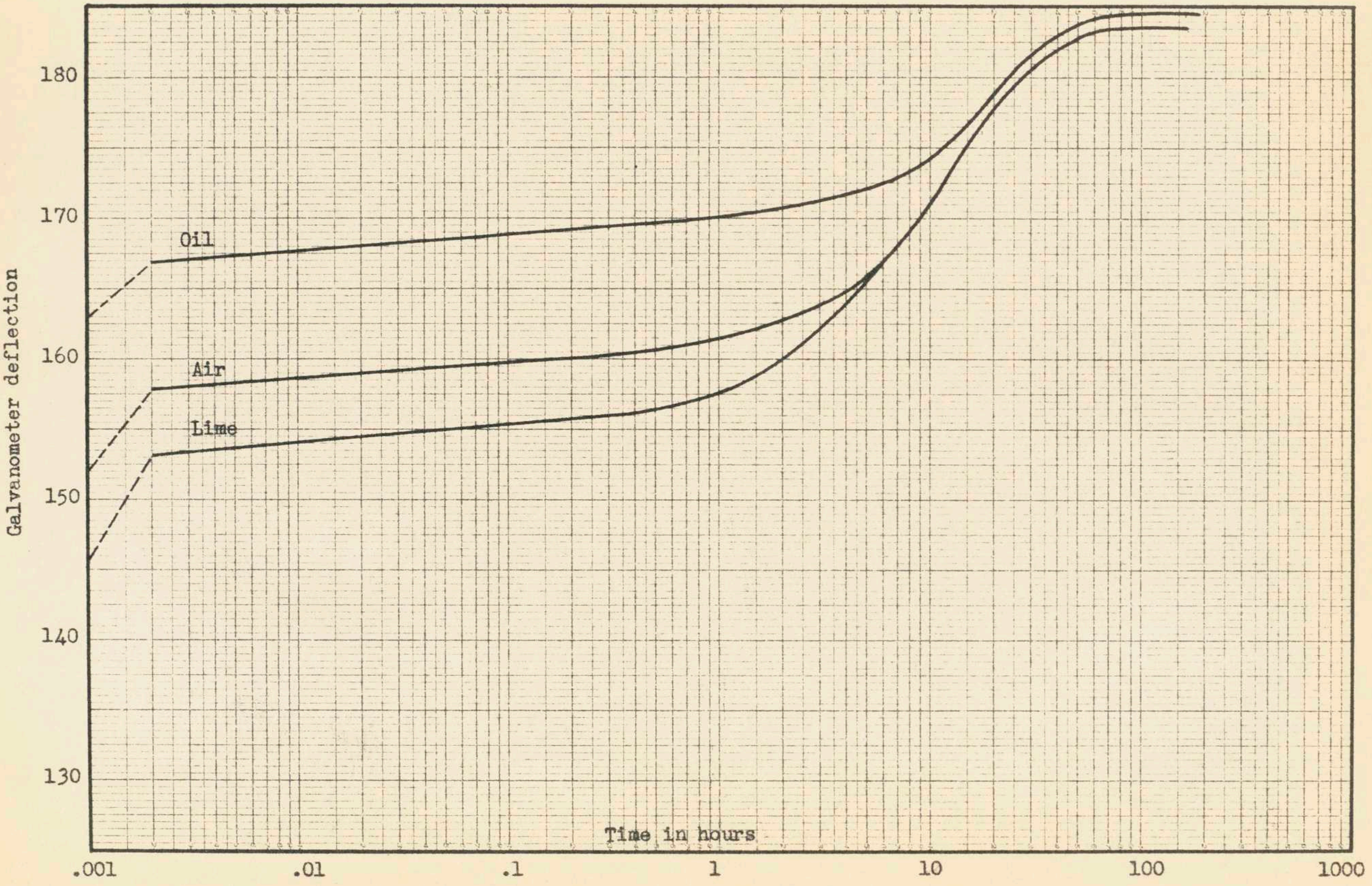


FIGURE 31 Aging curves at 900° F for 1% C 5% Cr steel cooled from 1700° F.

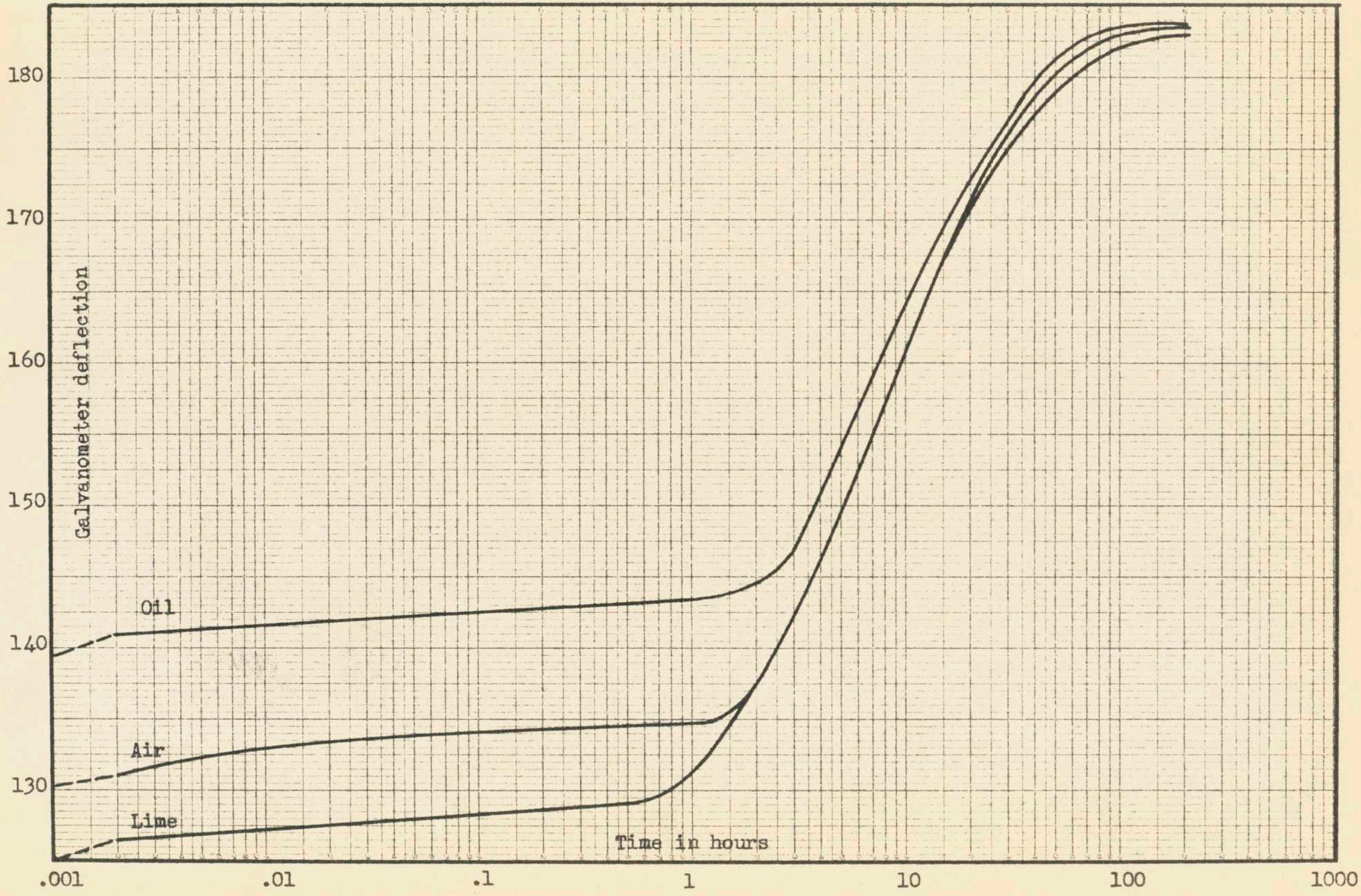


FIGURE 32 Aging curves at 900° F for 1% C 5% Cr steel cooled from 1800° F as shown.

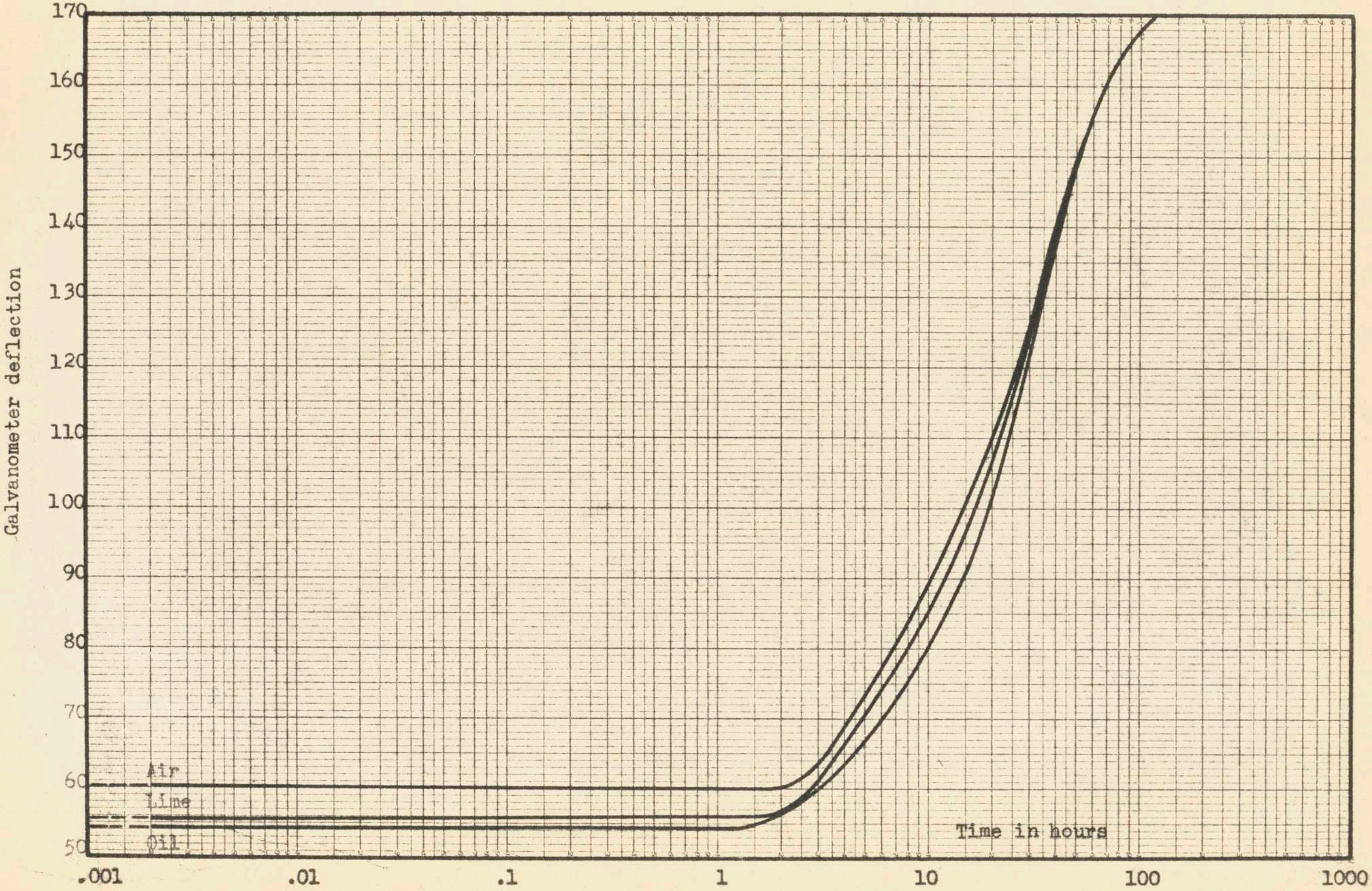


FIGURE 33 Aging curves at 900° F for 1% C 5% Cr steel cooled from 2000° F as shown.

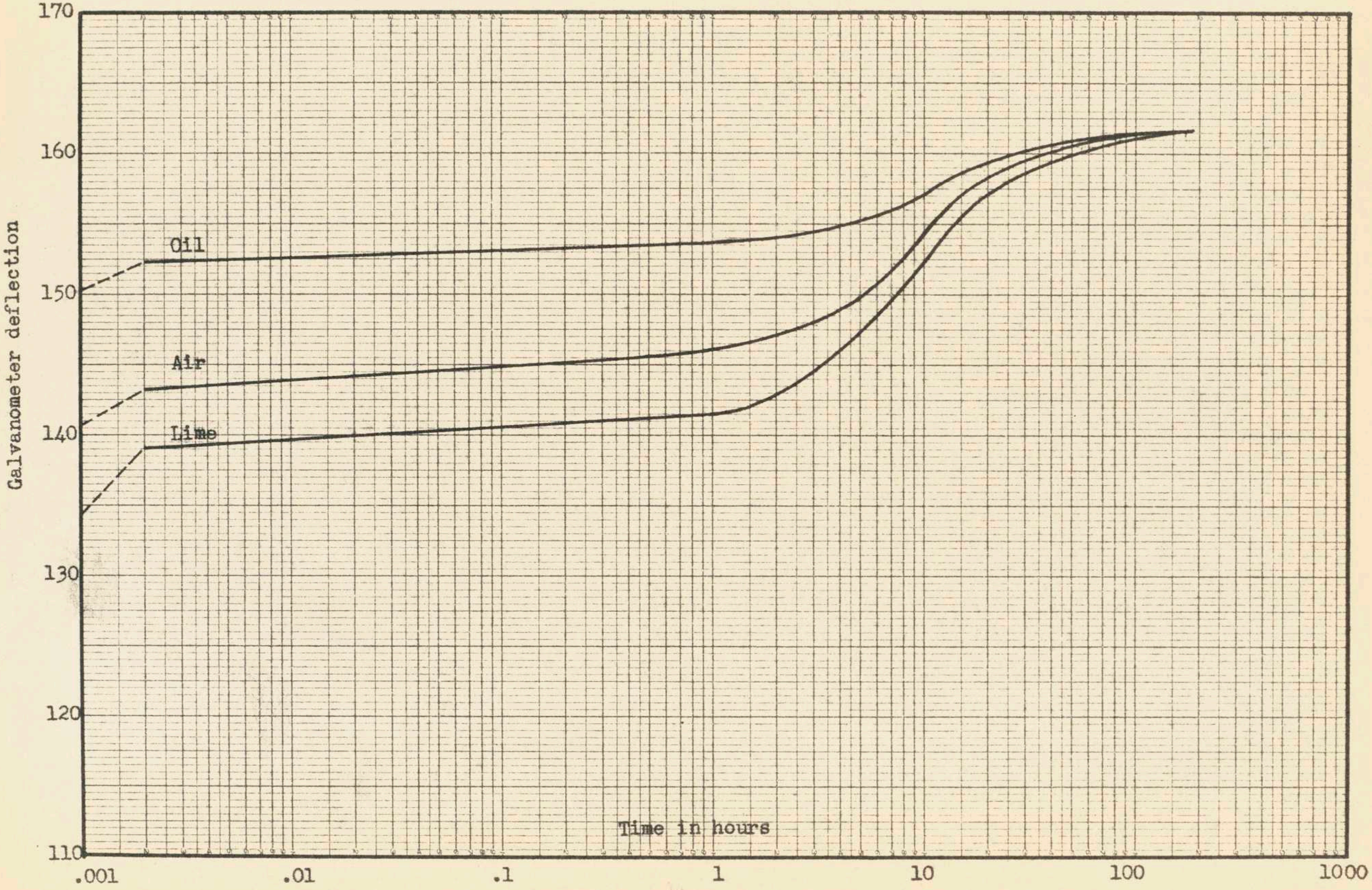


FIGURE 34 Aging curves at 900° F of 1.5% C 11.5% Cr steel cooled from 1700° F as shown.

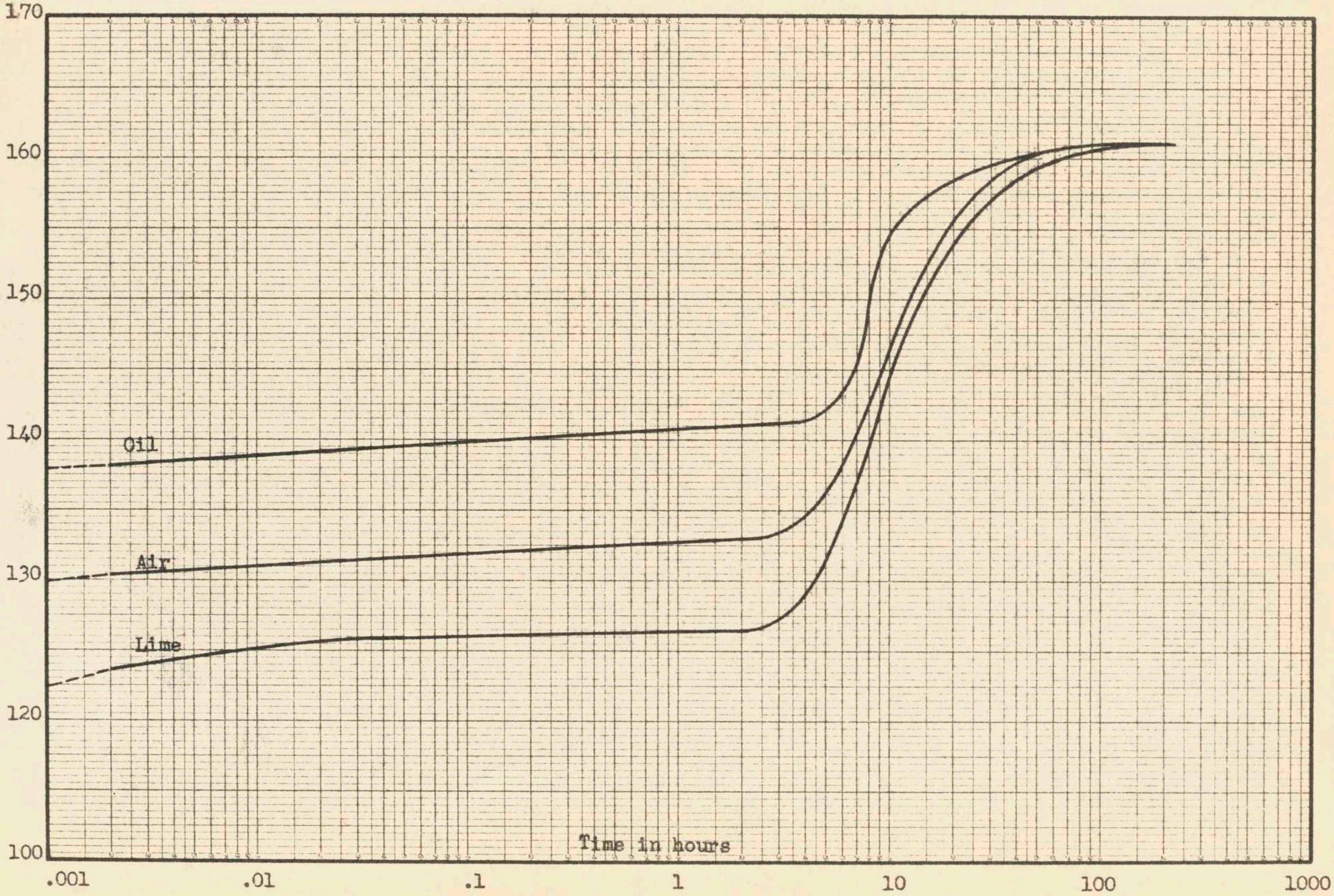


FIGURE 35 Aging curves at 900° F of 1.5% C 11.5% Cr steel cooled from 1800° F as shown.

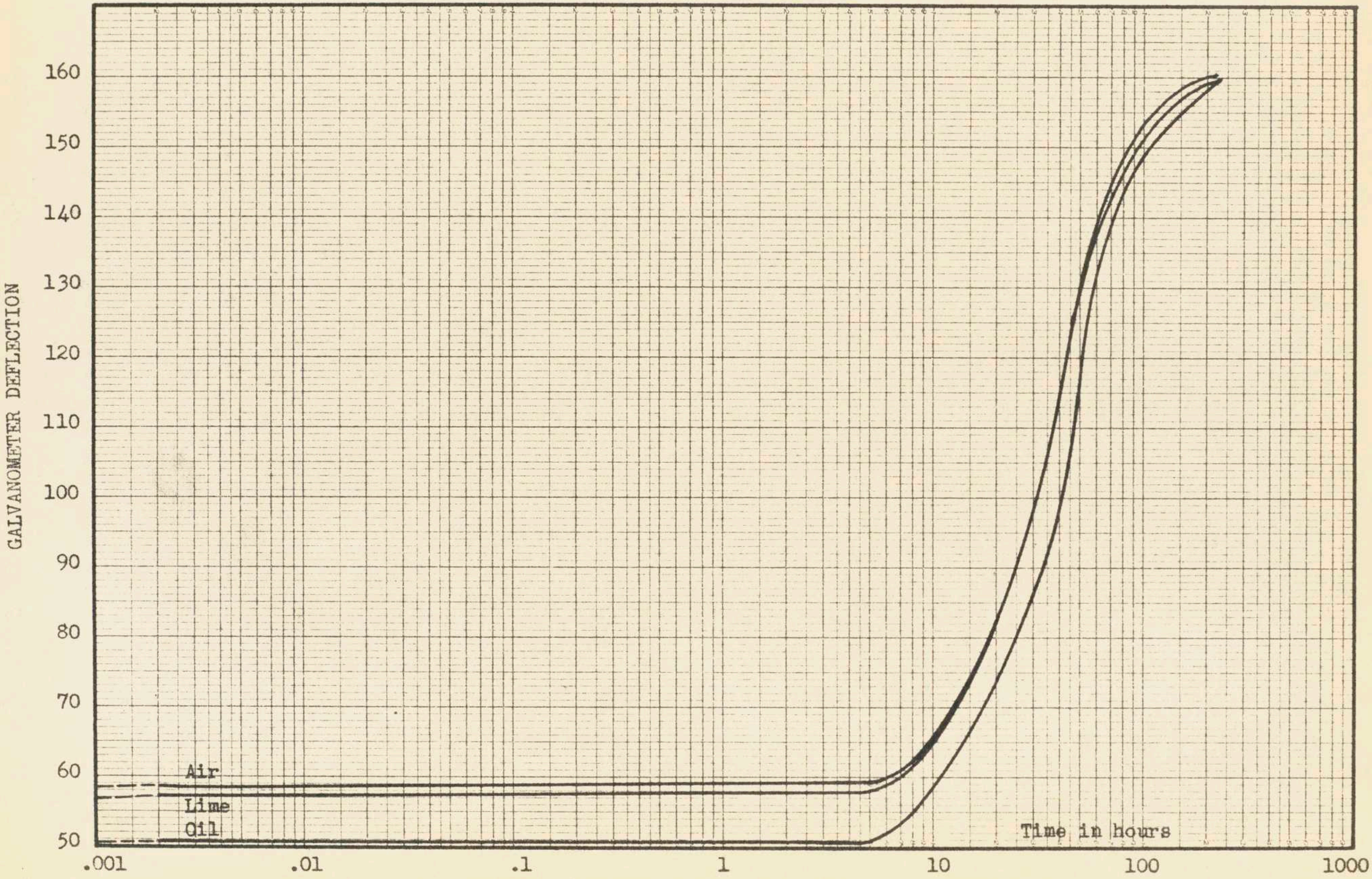


FIGURE 36 Aging curves at 900° F of 1.5% C 11.5% Cr steel cooled from 2000°F as shown.

In all of these curves the following trends were evident:

1. The reaction was composed of two stages. The first stage was one of little or no reaction, and the second stage was one of considerable reaction.

2. The first stage was one of slow reaction for the specimens hardened from 1700° F. and 1800° F., the rate of this slow reaction being greater in the specimens hardened from 1700° F. The first stage was one of no reaction for the specimens hardened from 2000° F.

3. The second stage resulted in a greater change the greater the amount of austenite that was present in the specimen when in the as-hardened state.

4. For the steels hardened from 1700° F. and 1800° F. the slower cools resulted in specimens of higher-austenite content, the order being oil, air, and lime. Hardening from 2000° F. produced specimens in which the austenite contents were highest in the oil cools and least in the air cools.

5. For a given quench, the more austenite the steel retained, the sooner the transformation started, but the later the transformation ended.

6. The retained austenite in the 1% C - 5% Cr steel was slightly more reactive in all states than the retained austenite in the 1.5% C - 11.5% Cr steel. This was true for all tempering temperatures from 900 - 1200° F., inclusive. Below 900° F. the difference in austenite reactivity became more outstanding.

In the following set of illustrations, Figures 37 to 42 inclusive, are shown aging curves at 212, 400, 500, 600, 800, and 900° F. for specimens of the two steels air-cooled from 1700, 1800, and 2000° F. In Figures 37, 38, and 39, are shown the aging curves of 1% C - 5% Cr steel specimens air-cooled from 1700, 1800, and 2000° F., respectively. In Figures 40, 41, and 42 are shown the aging curves of 1.5% C - 11.5% Cr steel specimens air-cooled from 1700, 1800, and 2000° F., respectively.

The following facts may be obtained from these curves:

1. The retained austenite in specimens of the 1% C - 5% Cr steel hardened from 1700° F. was very reactive in the range 500 - 600° F. In the specimens hardened from 1800° F. the austenite was reactive up to a limited extent at 600° F. and was very reactive at 500° F. In the specimens hardened from 2000° F. the austenite was not reactive at 600° F. but was still reactive at 500° F.

2. The retained austenite in specimens of the 1.5% C - 11.5% Cr steel hardened from either 1700, 1800, or 2000° F. was very unreactive at temperatures below 900° F.

From these data, of which specimen-curves have been shown, a picture may be obtained of the transformation characteristics of retained austenite in the two steels, 1% C - 5% Cr and 1.5% C - 11.5% Cr, hardened from 1700, 1800, and 2000° F. in air, in oil, and in lime.

Since there was some variation between the as-hardened values of the specimens for any given treatment a comparison of the absolute amounts of retained austenite calculated from a fixed basis would not give a

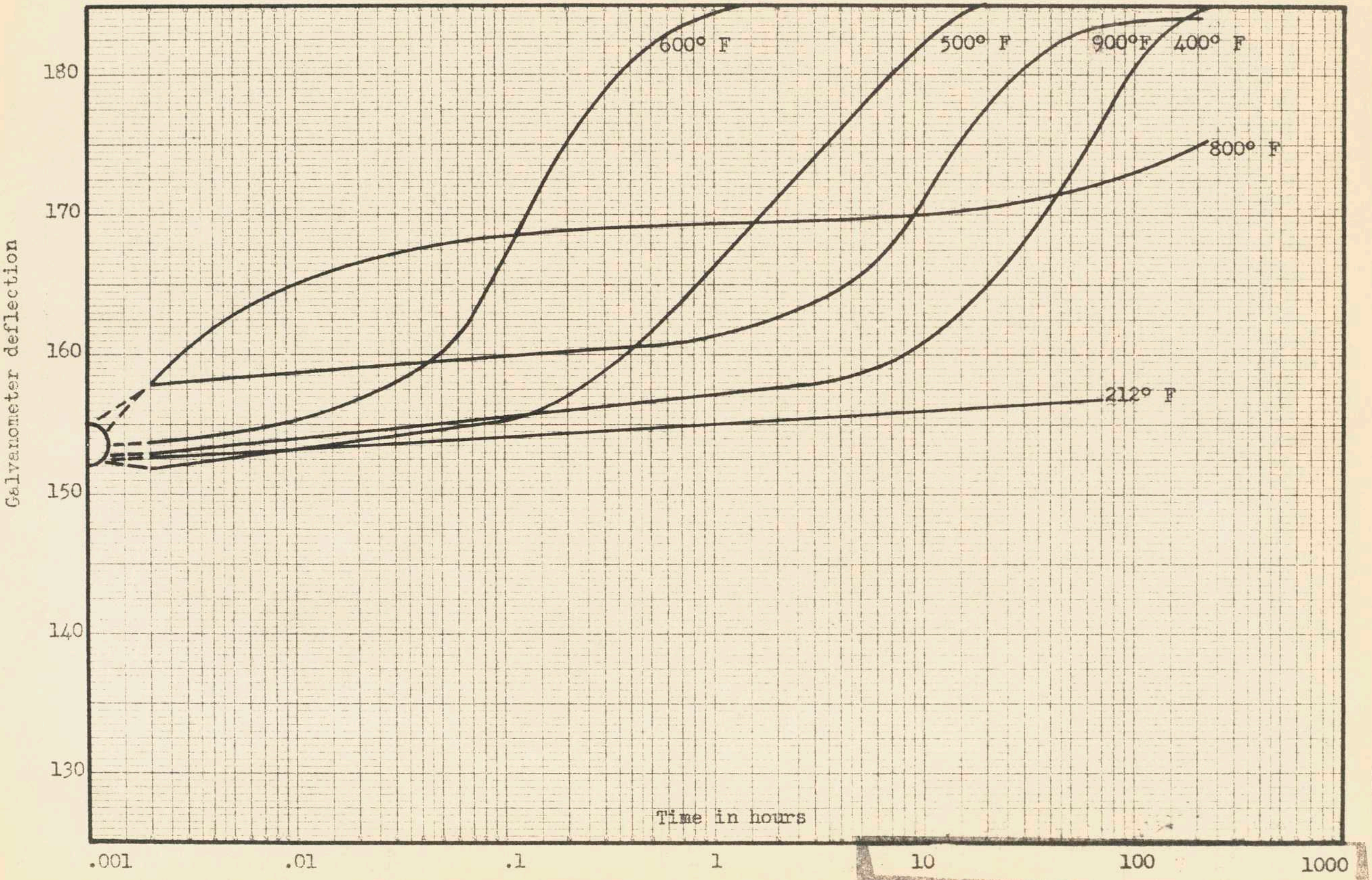


FIGURE 37 Aging curves for 1% C 5% Cr steel air cooled from 1700° F.

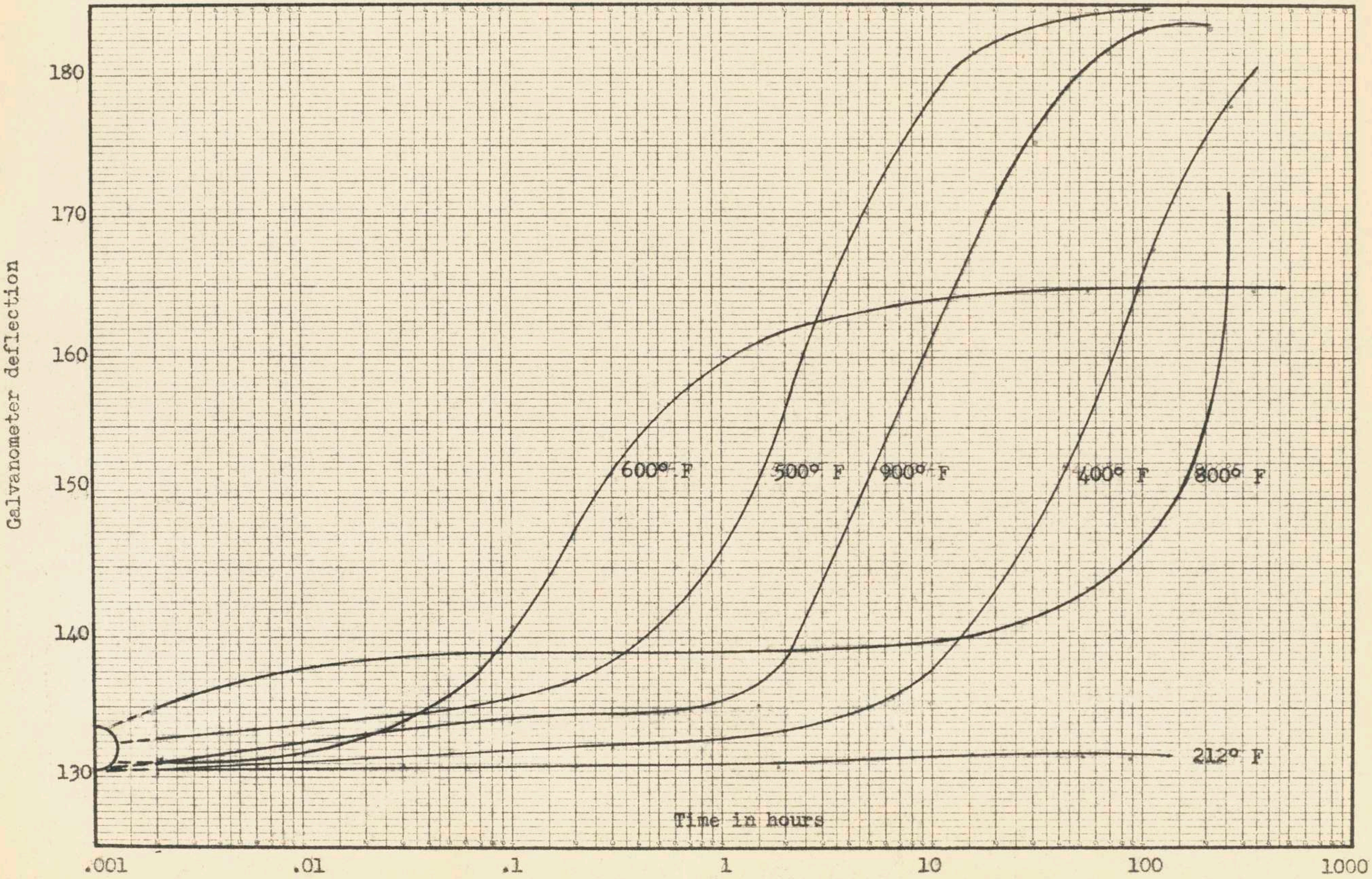


FIGURE 38 Aging curves for 1% C 5% Cr steel air cooled from 1800° F.

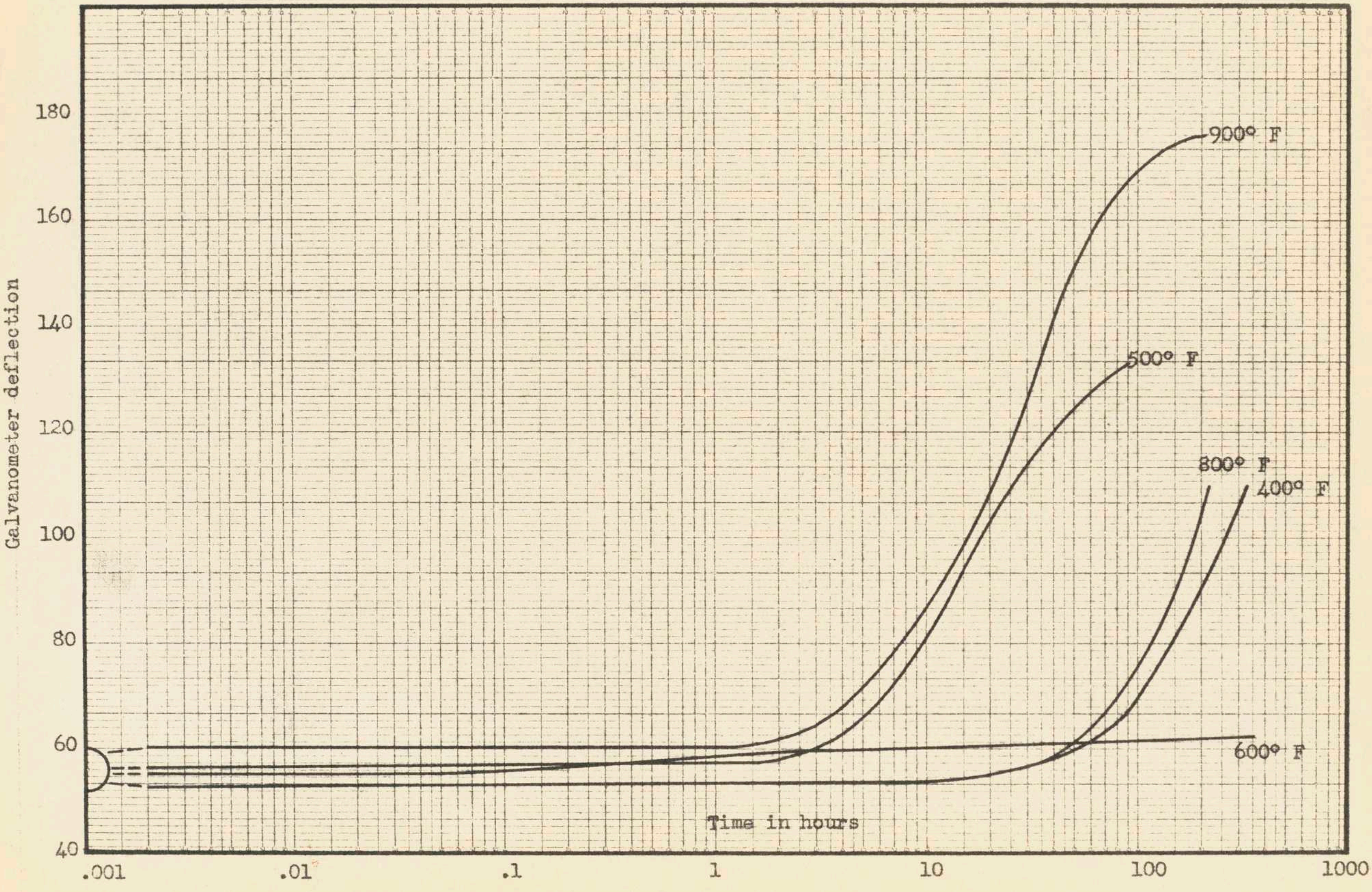


FIGURE 39 Aging curves for 1% C 5% Cr steel air cooled from 2000° F.

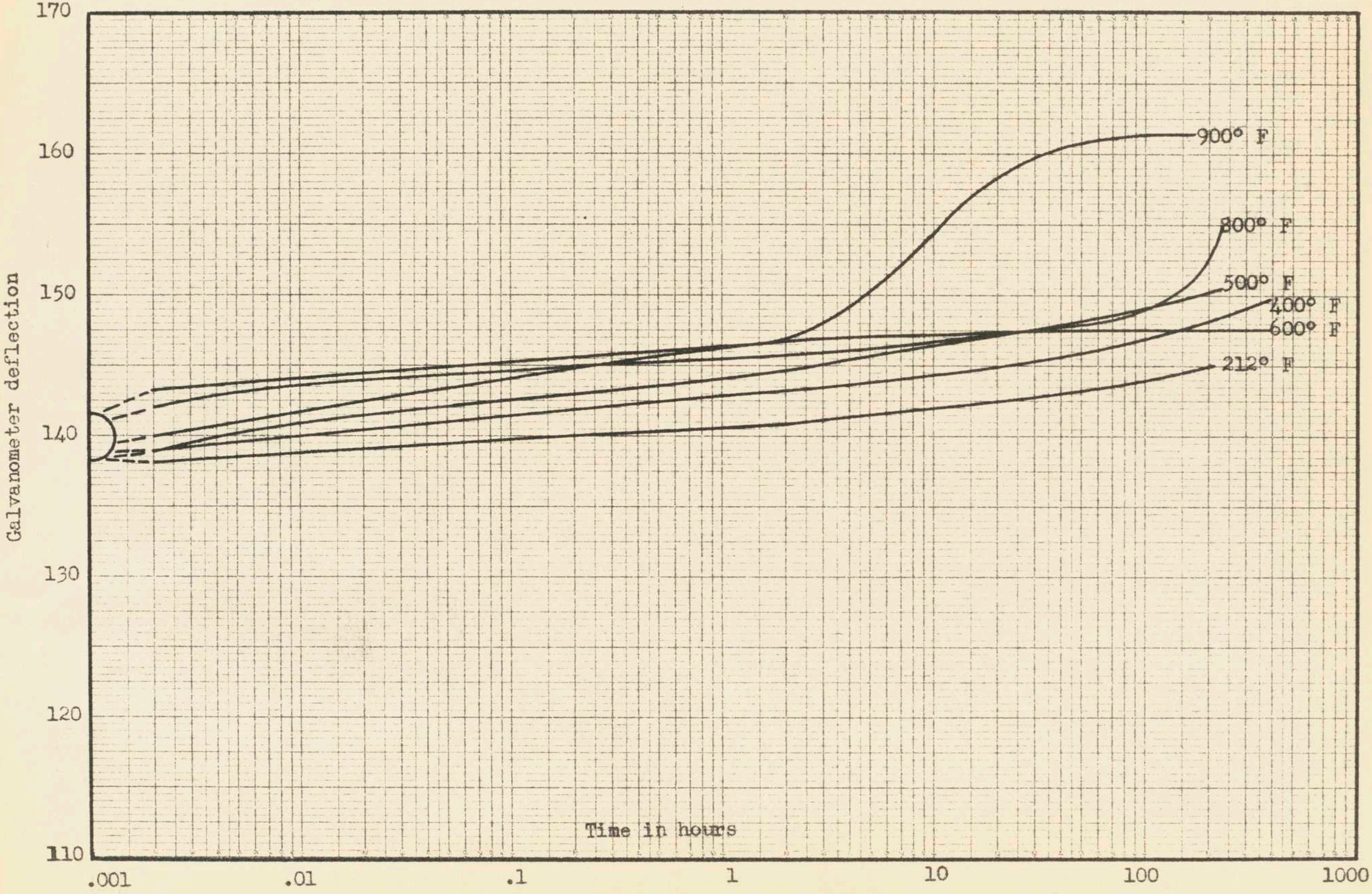


FIGURE *40* Aging curves for 1.5% C 11.5% Cr steel air cooled from 1700° F.

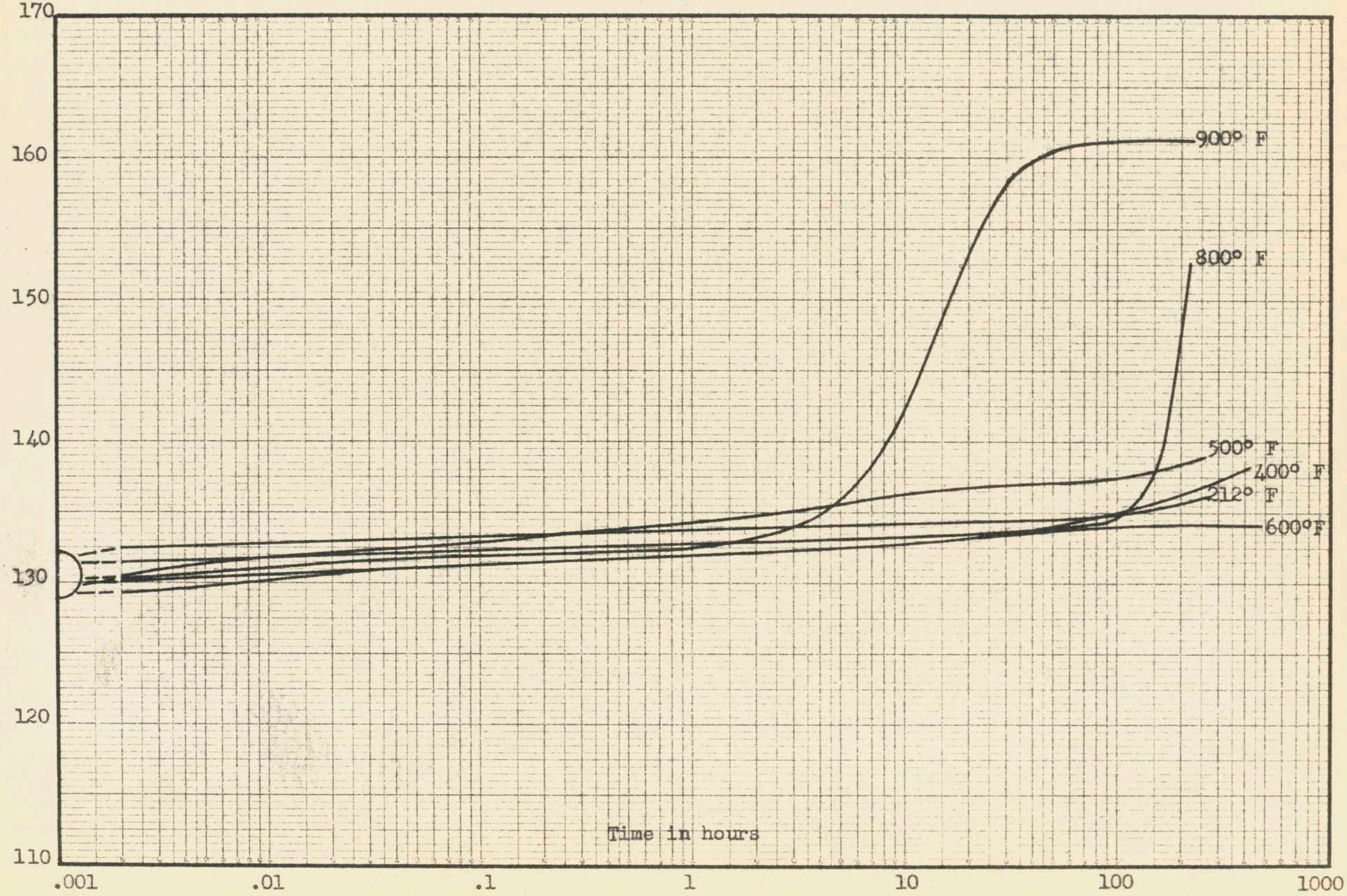


FIGURE 41 Aging curves for 1.5% C 11.5% Cr steel air cooled from 1800° F.

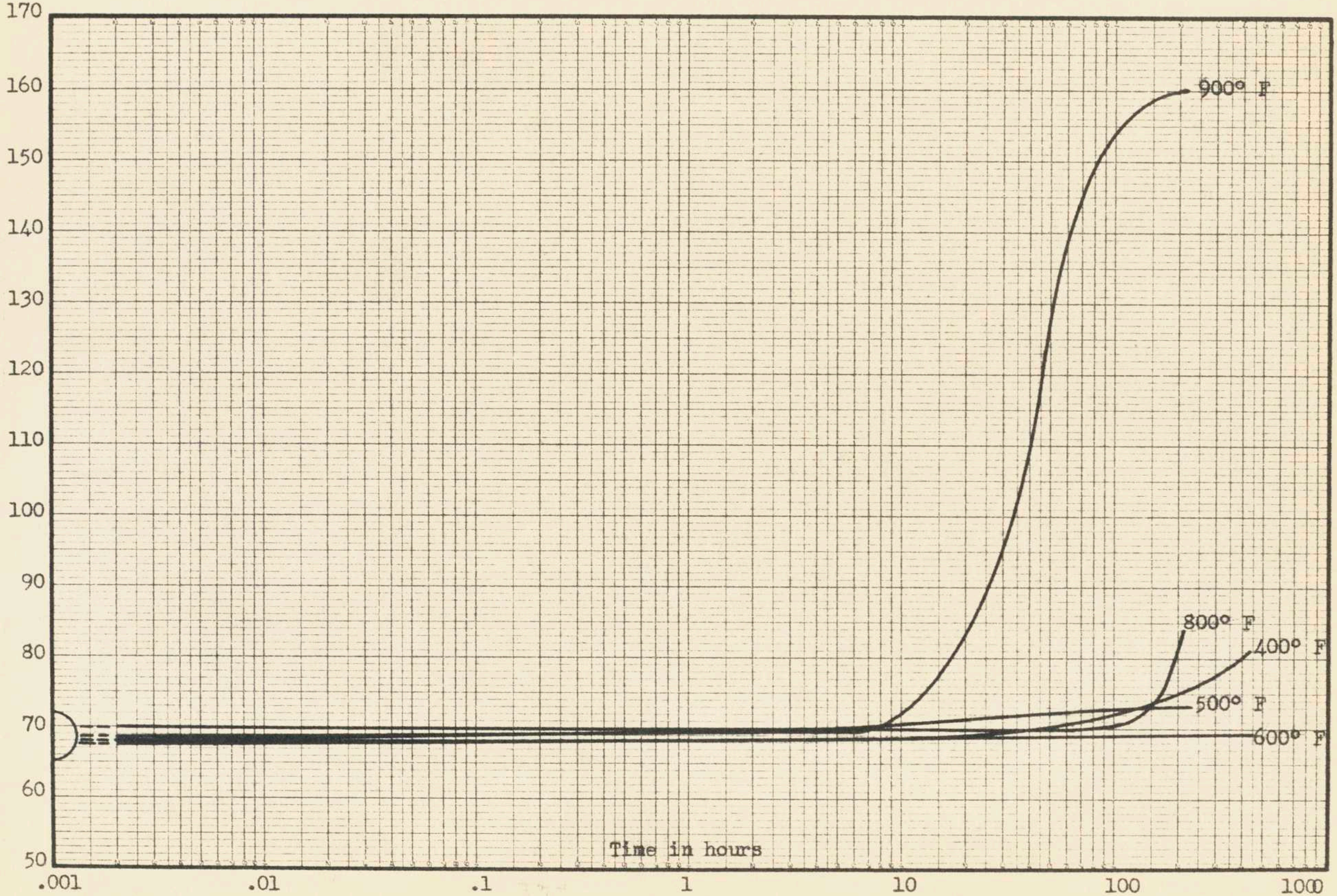


FIGURE 42 Aging curves for 1.5% C 11.5% Cr steel air cooled from 2000° F.

visually clear comparison. It was decided to use each curve as its own basis and to derive from each the time required for definite changes in the as-hardened austenite content. The original values of retained-austenite content were, of course, calculated from the same base values.

From each curve was obtained the times necessary to transform 10, 30, 50, 75, 90, and 100 per cent of the retained austenite. The way in which this was done is described below.

Method of Obtaining Points for Transformation Curves

Plot - Time for given percentage transformation versus tempering temperature.

Obtain percentage transformation as follows:

Basis - completely transformed state. Let S represent the deflection for the coil and the specimen and S-28 = R is the deflection for the specimen alone.

The amount of austenite in the as-quenched state - Let the galvanometer deflection = Q for the coil and the specimen and (Q-28) is the deflection for the specimen alone.

$$\text{Then, per cent austenite} = \frac{(S-28) - (Q-28)}{(S-28)} \times 100$$

$$= \frac{S-Q}{R} \times 100$$

We desire to find the throw corresponding to any percentage change, (100)a, in the retained-austenite content. The austenite left after

$(100)a$ per cent change $= \frac{S-Q}{R} \times 100 - a(\frac{S-Q}{R})100.$

From the general formulas

$$\text{per cent austenite} = \frac{(S-X)}{(R)}100$$

$$X = S - \frac{R}{100}(\%A)$$

substituting,

$$X = S - \frac{R}{100} \quad \frac{S-Q}{R} \times 100 - a(\frac{S-Q}{R} \times 100)$$

$$= S - (s-Q) + a(S-Q)$$

$$= Q + a(S-Q)$$

Using U for the maximum magnetization of the Ultradie specimen and the coil,

$$X = Q + a(U-Q).$$

This data was then plotted for each steel, for each hardening rate. The plots are shown in Figures 43 to 60, inclusive.

The ordinates in all cases are the tempering temperatures given in degrees Fahrenheit and the abscissae (6 cycle logarithmic) in all cases are the times at temperature given in hours.

The plots are designated as follows:

Points marked indicate 10 per cent transformation
indicate 30 per cent transformation
indicate 50 per cent transformation
indicate 75 per cent transformation
indicate 90 per cent transformation
indicate 100 per cent transformation

In Figures 43, 44, and 45 are shown the transformation curves for specimens of 1% C - 5% Cr steel, hardened from 1700° F. in oil, air, and lime, respectively. It is to be noted that although transformations to the extent of 75 - 100 per cent occurred rapidly in the 500 - 600° F. range, transformations of less extent, 30 and 50 per cent occurred more rapidly at 700 and 800° F., respectively. Although the times for 10 per cent transformations varied considerably, they illustrated an interesting point, which is that the oil-quenched specimen was not at all transformed by boiling water, while the air- and lime-cooled specimens were transformed to the extent of 10 per cent by this treatment.

In Figures 46, 47, and 48 are shown similar curves for the 1800° F. heat treatments. Here again, the transformations of lesser extent occurred most rapidly at temperatures above the range critical for transformations of greater extent. In both heat treatments, the austenite was singularly unreactive at 900° F. for transformation over 10 per cent until a considerable time had elapsed. Thus a nose was introduced into each of the 30 per cent transformation curves.

In Figures 49, 50, and 51 are shown in like manner the transformation curves for the 2000° F. heat treatments. The only reactive low temperature for an austenite transformation was at 500° F.; the extent of transformation, however, was small. Complete transformation could only be obtained by using a tempering temperature above 900° F.

The transformation data for the 11.5% Cr steel are shown in Figures 52 to 60, inclusive.

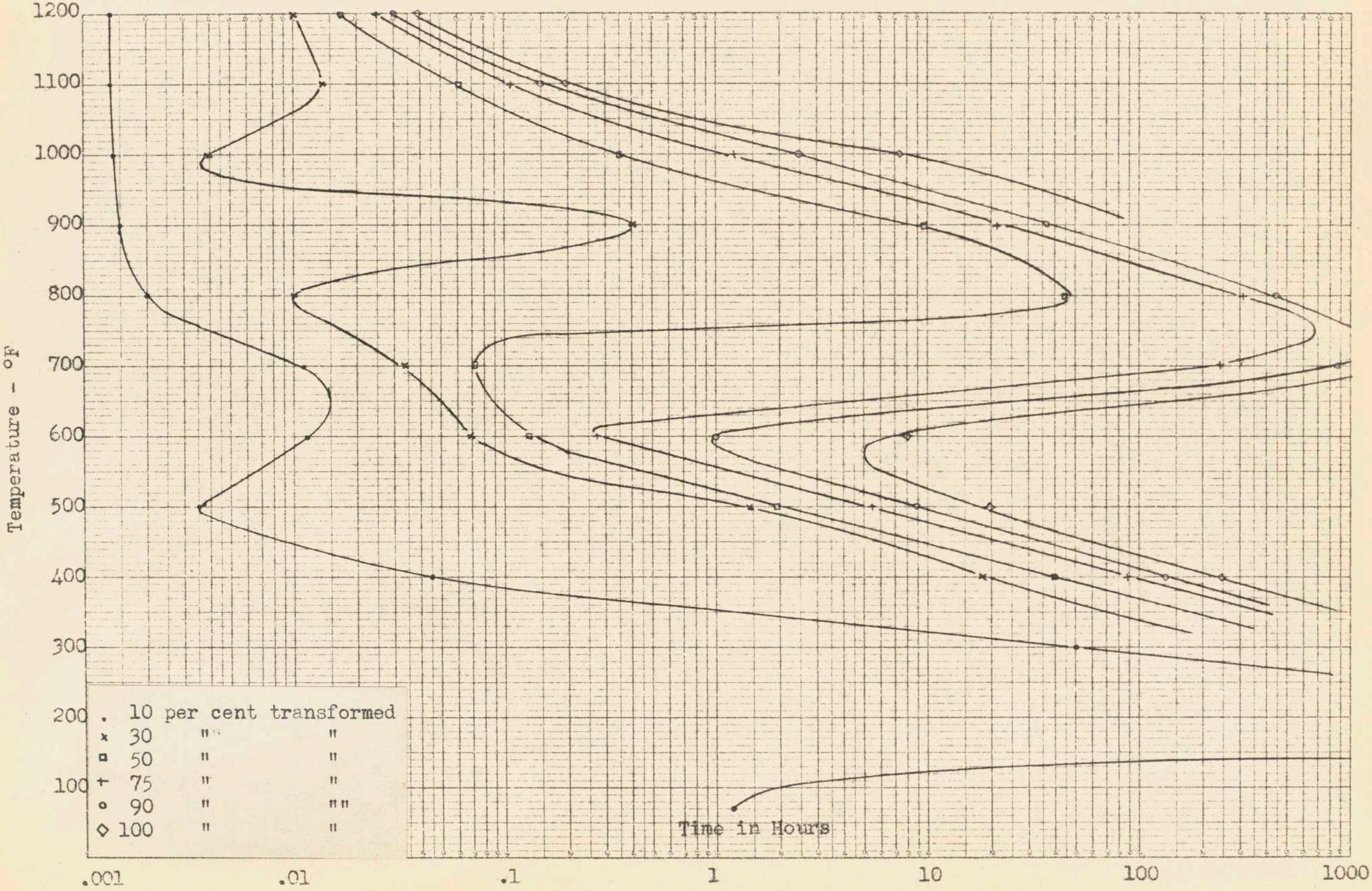


FIGURE 43 Transformation curves of retained austenite for 1% C 5% Cr steel oil cooled from 1700° F.

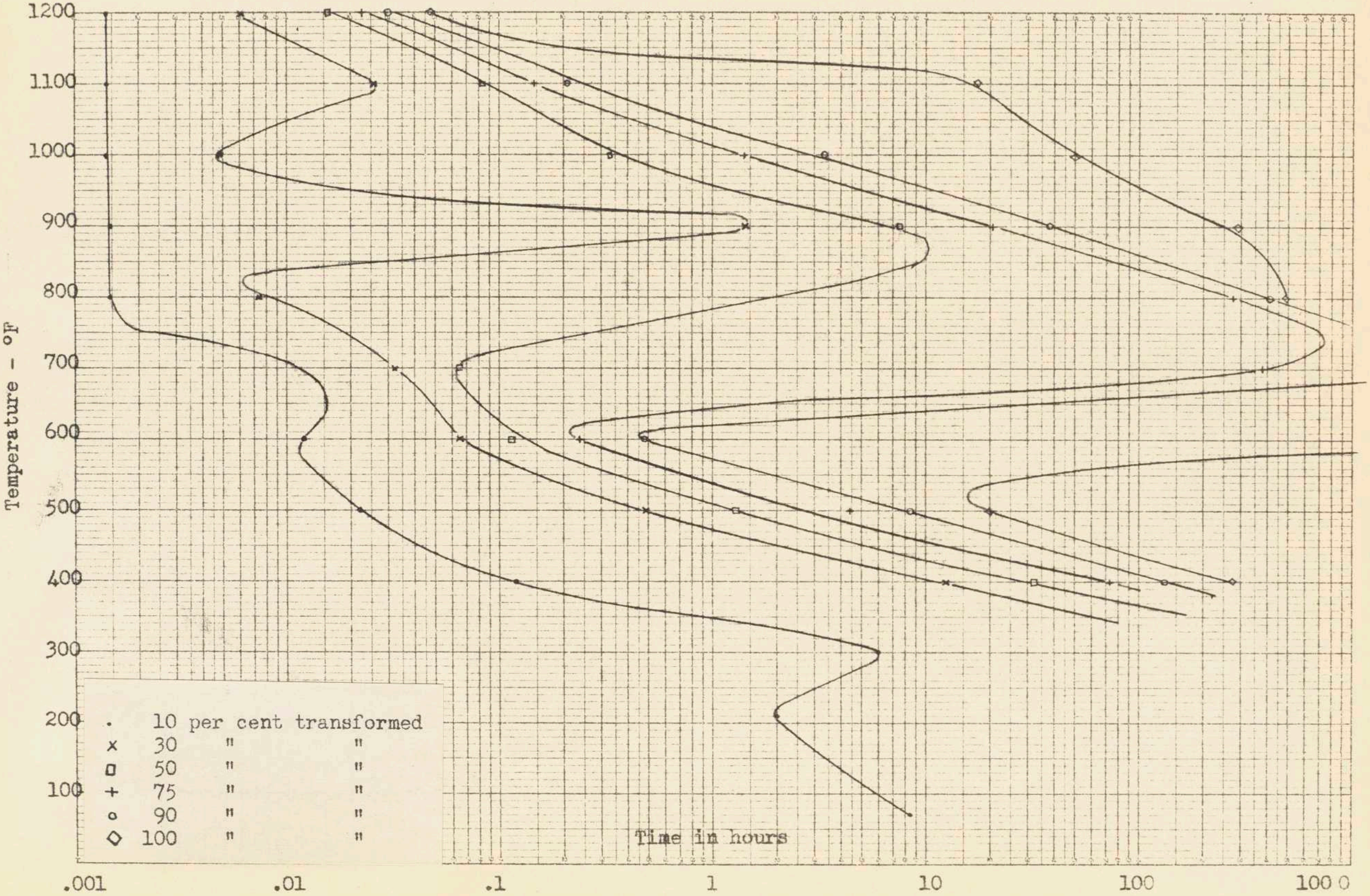


FIGURE 44 Transformation curves of retained austenite for 1% C 5% Cr steel air cooled from 1700° F.

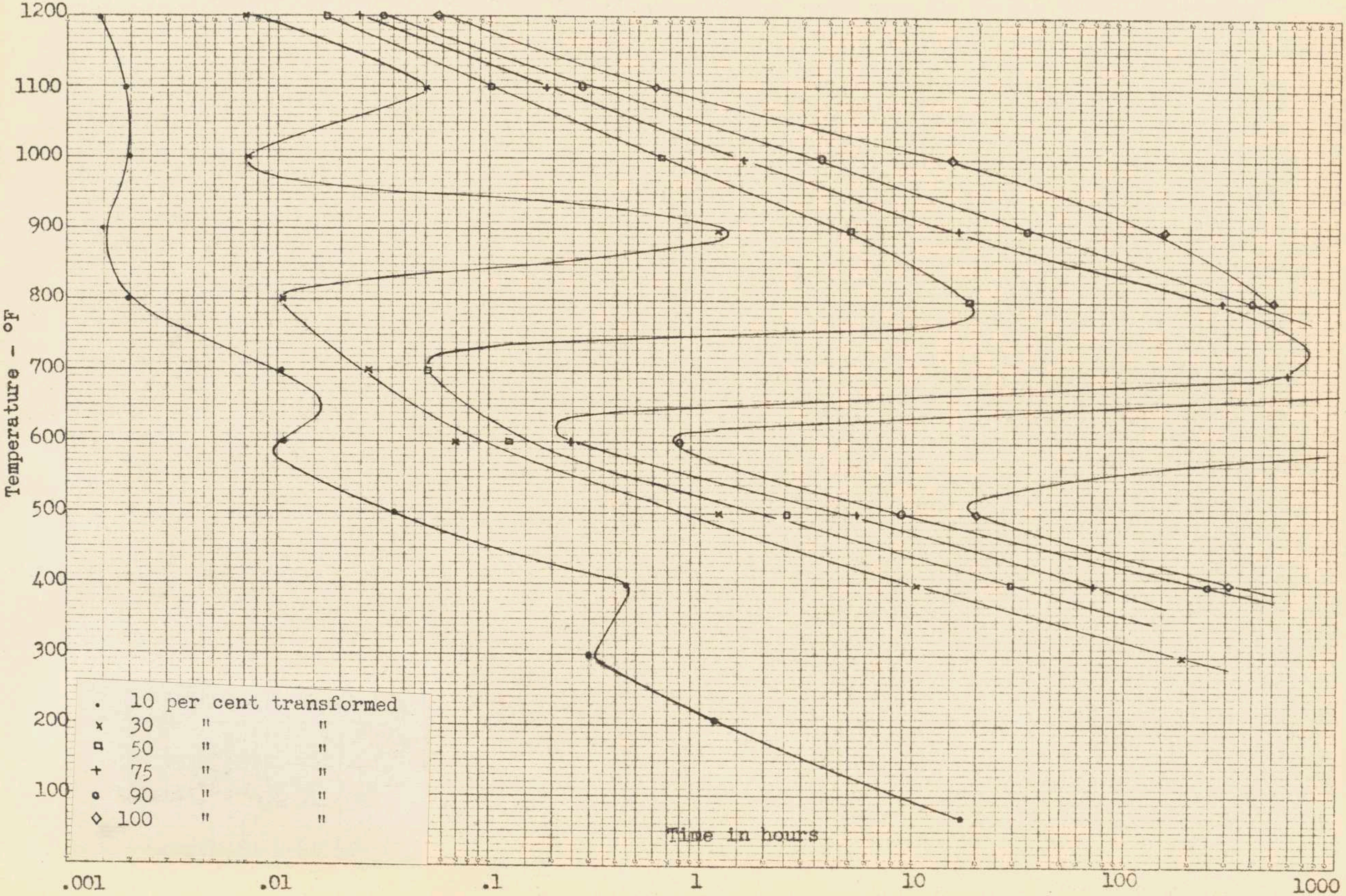


FIGURE 45 Transformation curves of retained austenite for 1% C 5% Cr steel lime cooled from 1700° F.

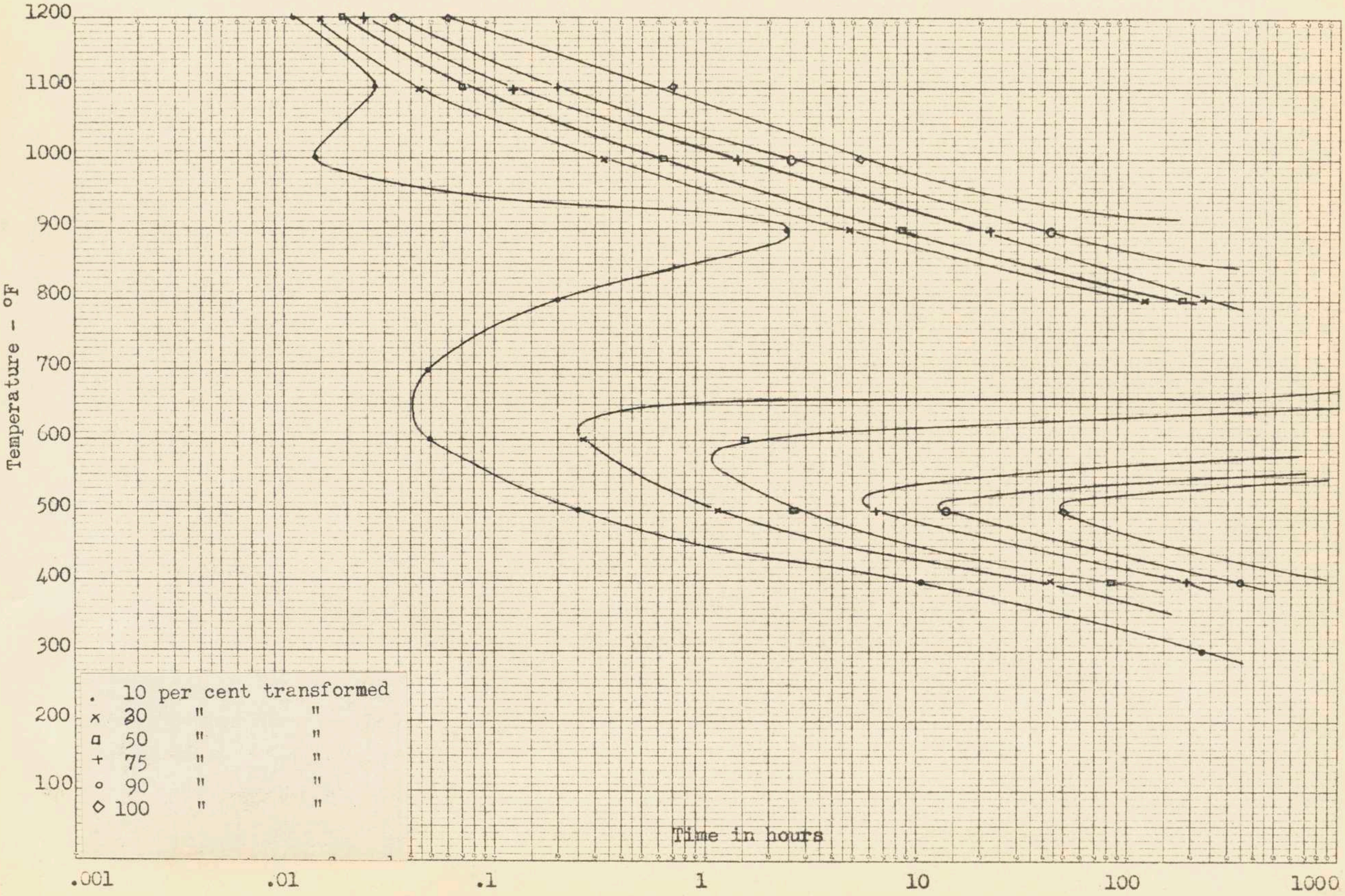


FIGURE 46 Transformation curves of retained austenite for 1% C 5% Cr steel oil cooled from 1800° F.

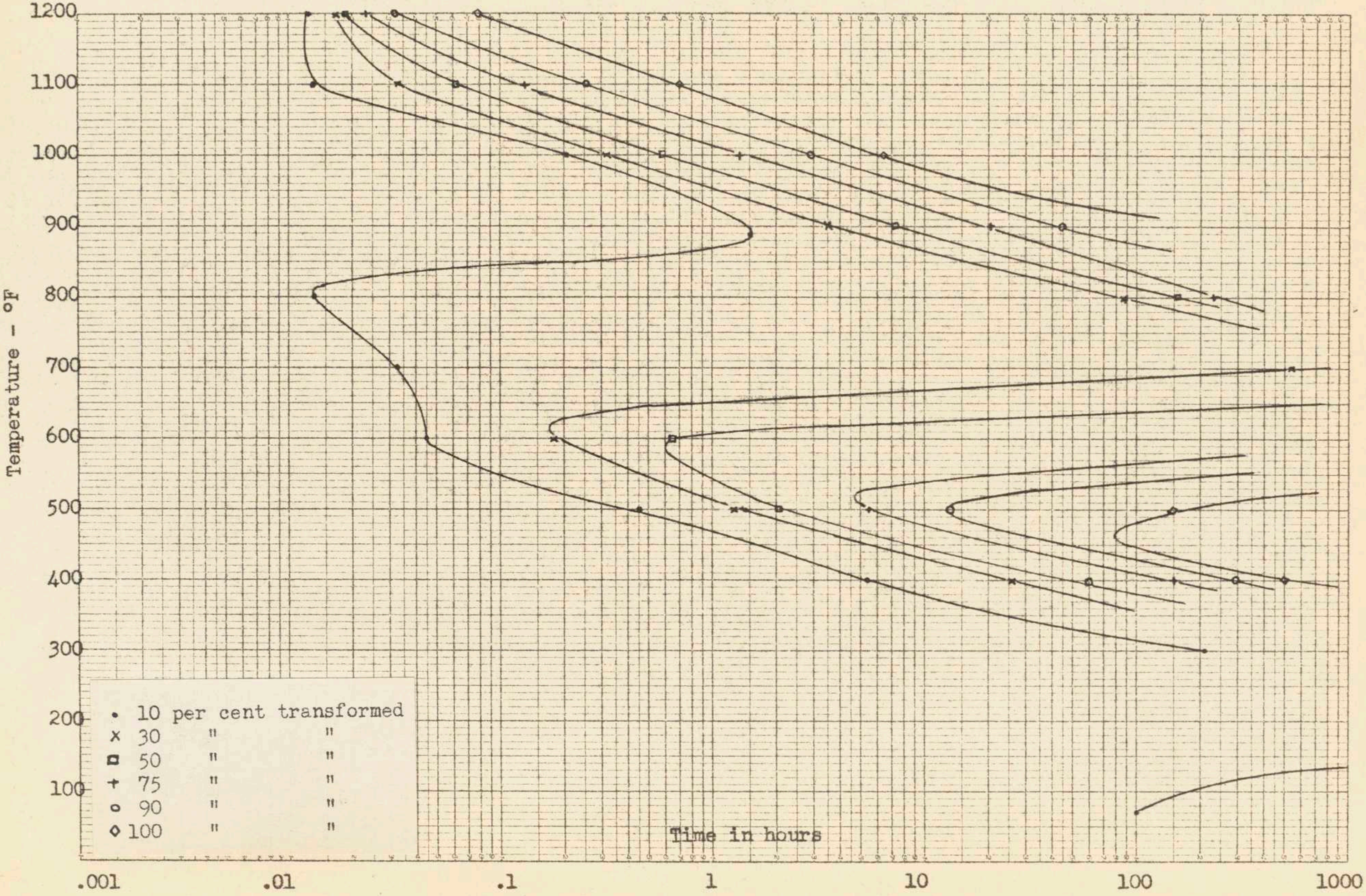


FIGURE 47 Transformation curves of retained austenite for 1% C 5% Cr steel air cooled from 1800° F.

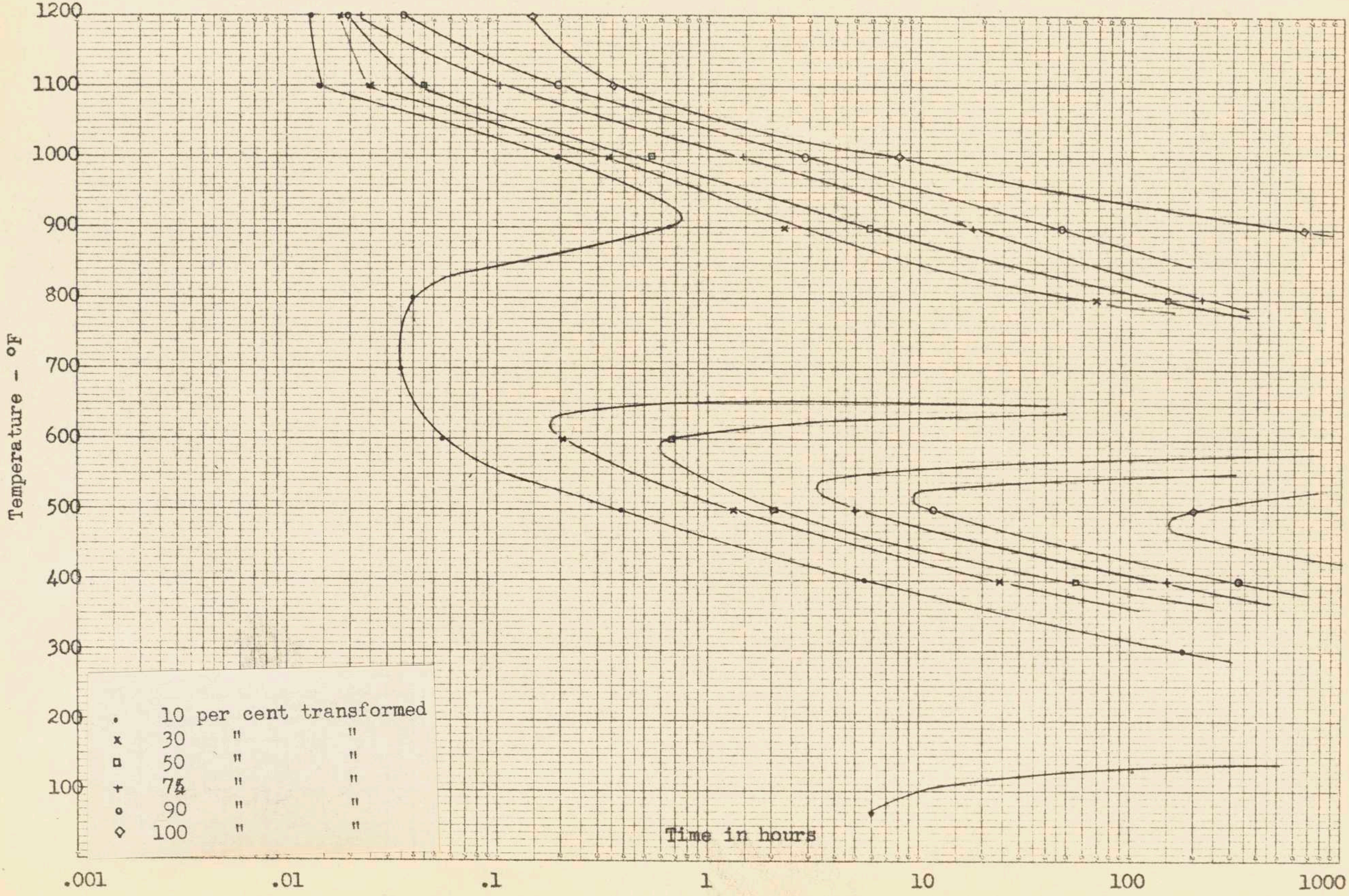


FIGURE 48 Transformation curves of retained austenite for 1% C 5% Cr steel lime cooled from 1800° F.

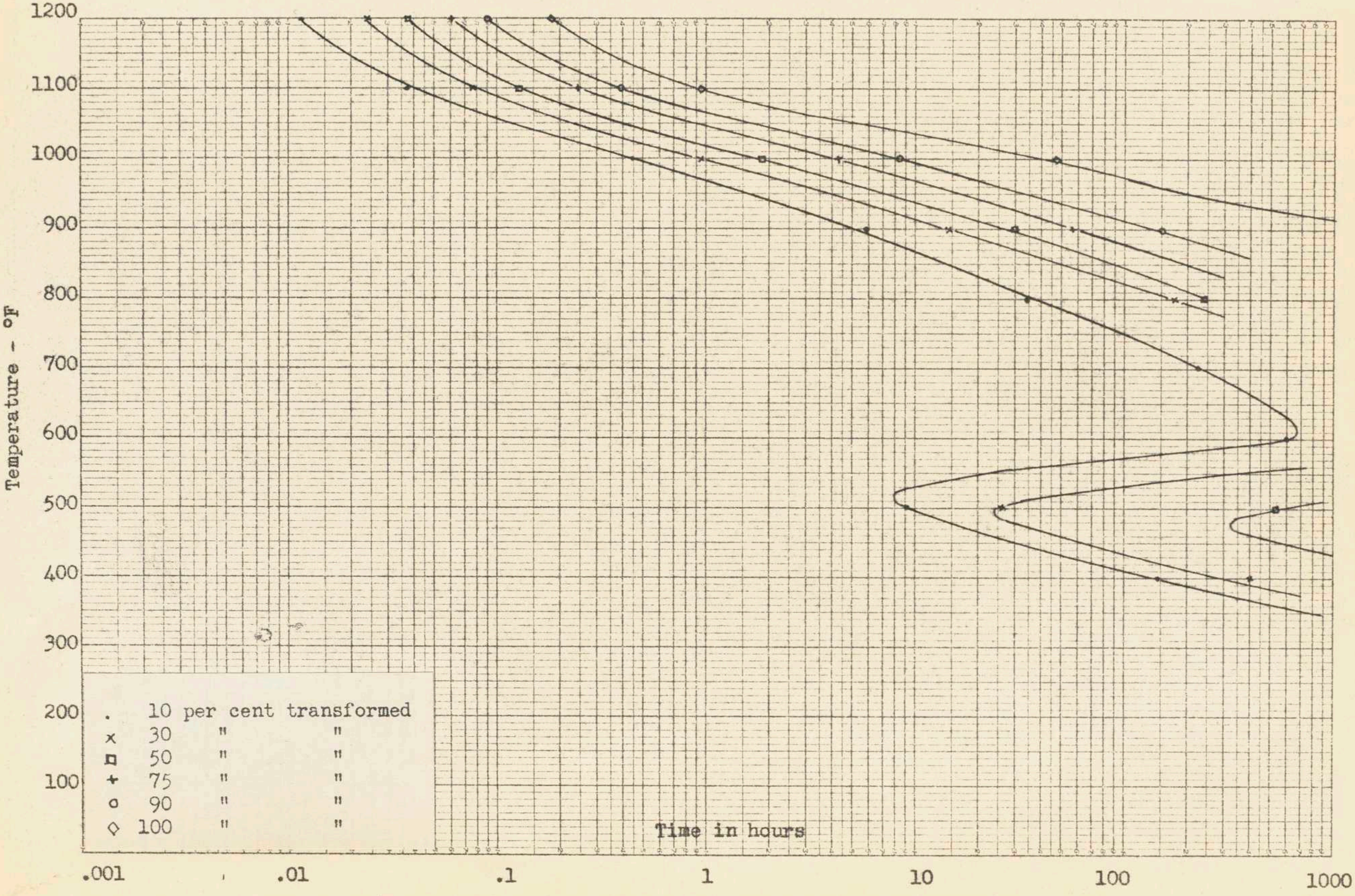


FIGURE 49 Transformation curves of retained austenite for 1% C 5% Cr steel oil cooled from 2000° F.

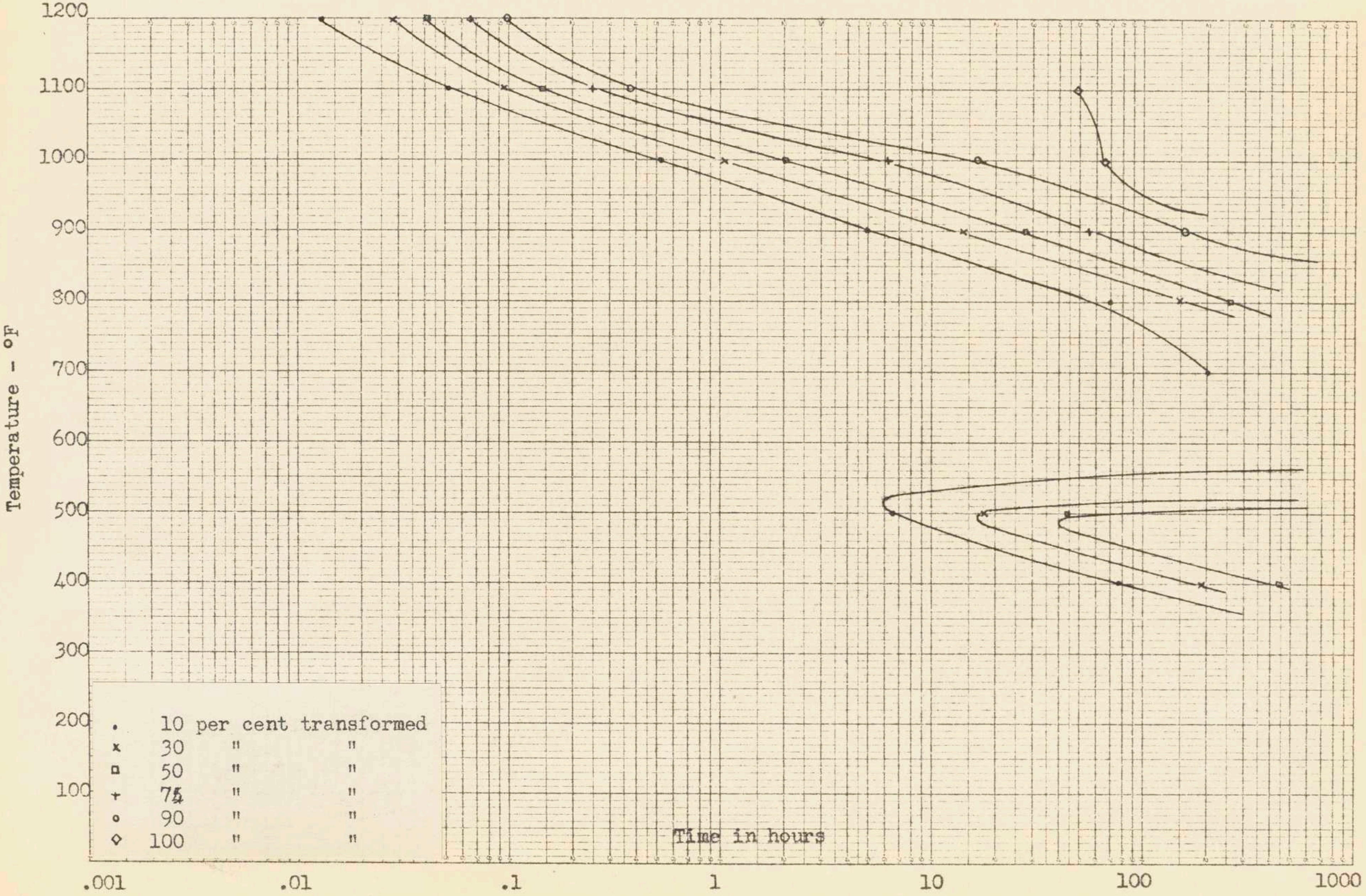


FIGURE 50 Transformation curves of retained austenite for 1% C 5% Cr steel air cooled from 2000° F.

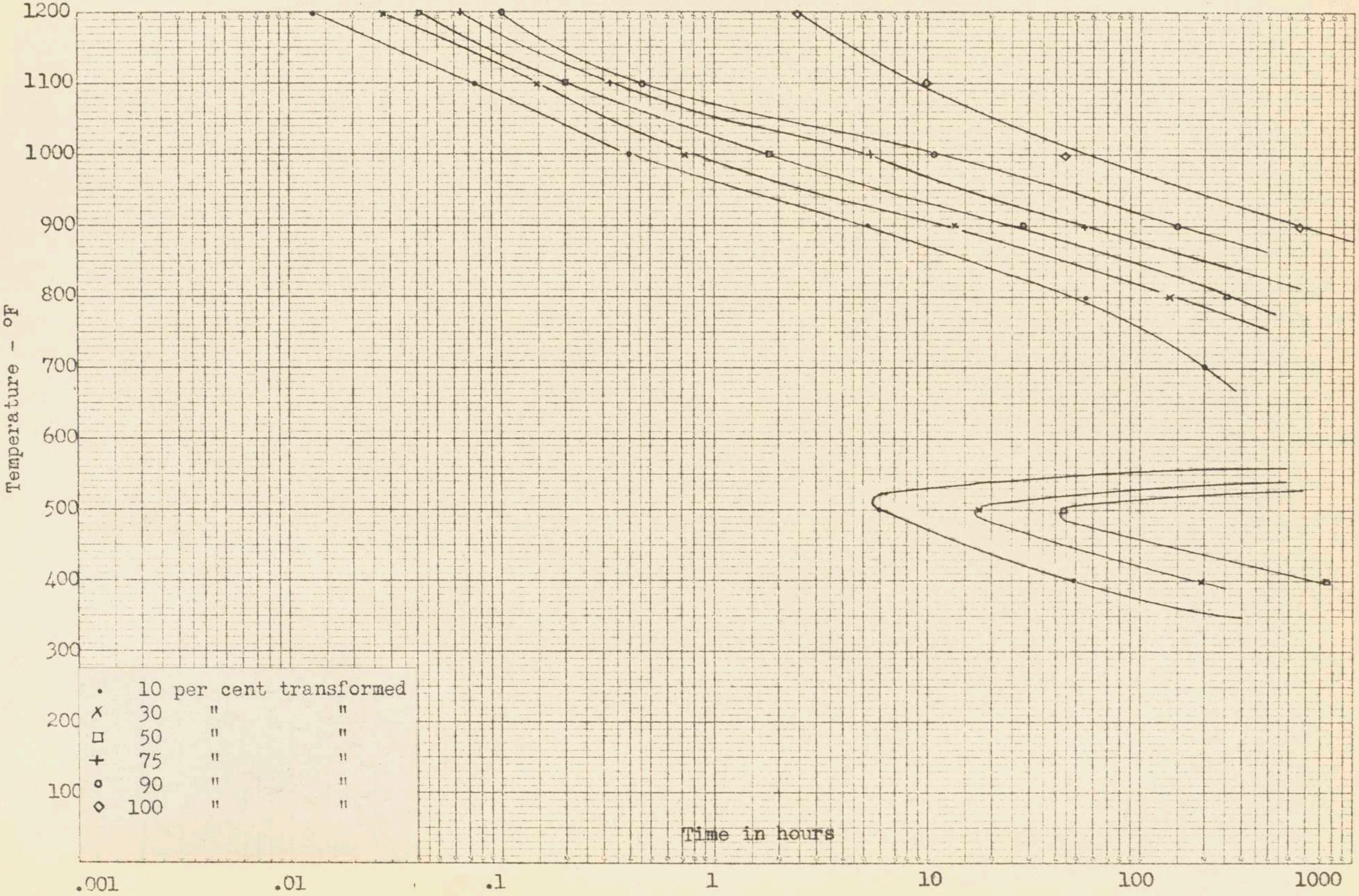


FIGURE 5/ Transformation curves of retained austenite for 1% C 5% Cr steel lime cooled from 2000° F.

In Figures 52, 53, and 54 are shown the transformation curves for specimens cooled from 1700° F. in oil, air and lime, respectively. The times for 10 per cent transformation were quite low, but there was a considerable spread to the 30 per cent transformation curve. The austenite was more reactive the slower the specimen was cooled from the hardening temperature. Austenite in the oil-hardened specimen transformed in the low temperature range only up to 30 per cent. The austenite in the air-hardened specimen transformed up to 50 per cent at 400° F. at a minimum time of 60 hours. The comparable value for the austenite in the lime-hardened specimen was 18 hours at a temperature of 500° F.

In Figures 55, 56, and 57 are shown the transformation curves for specimens cooled, from 1800° F. in oil, air, and lime, respectively. The times for 10 per cent transformation were considerably longer than for the 1700° F. heat-treated specimens. The 10 per cent transformation curves were the only ones which exhibited high reactivity at the lower temperature ranges. The minimum transformation times of the retained austenite at 500° F. were 0.4, 0.3, and 0.2 hours for the oil-, and air-, and lime-hardened specimens, respectively. The room temperature transformation was greater than that at either 212 or 300° F.

In Figures 58, 59, and 60 are shown the transformation curves for specimens cooled from 2000° F. in oil, air, and lime, respectively. No highly reactive range at lower temperatures was noted and the 10 per cent transformation curves closely followed the curves for greater transformations.

The curves in the aforementioned figures do not give any indication as to the absolute amounts of retained austenite, but show only the times to transform given fractions of the amount present, whatever it be.

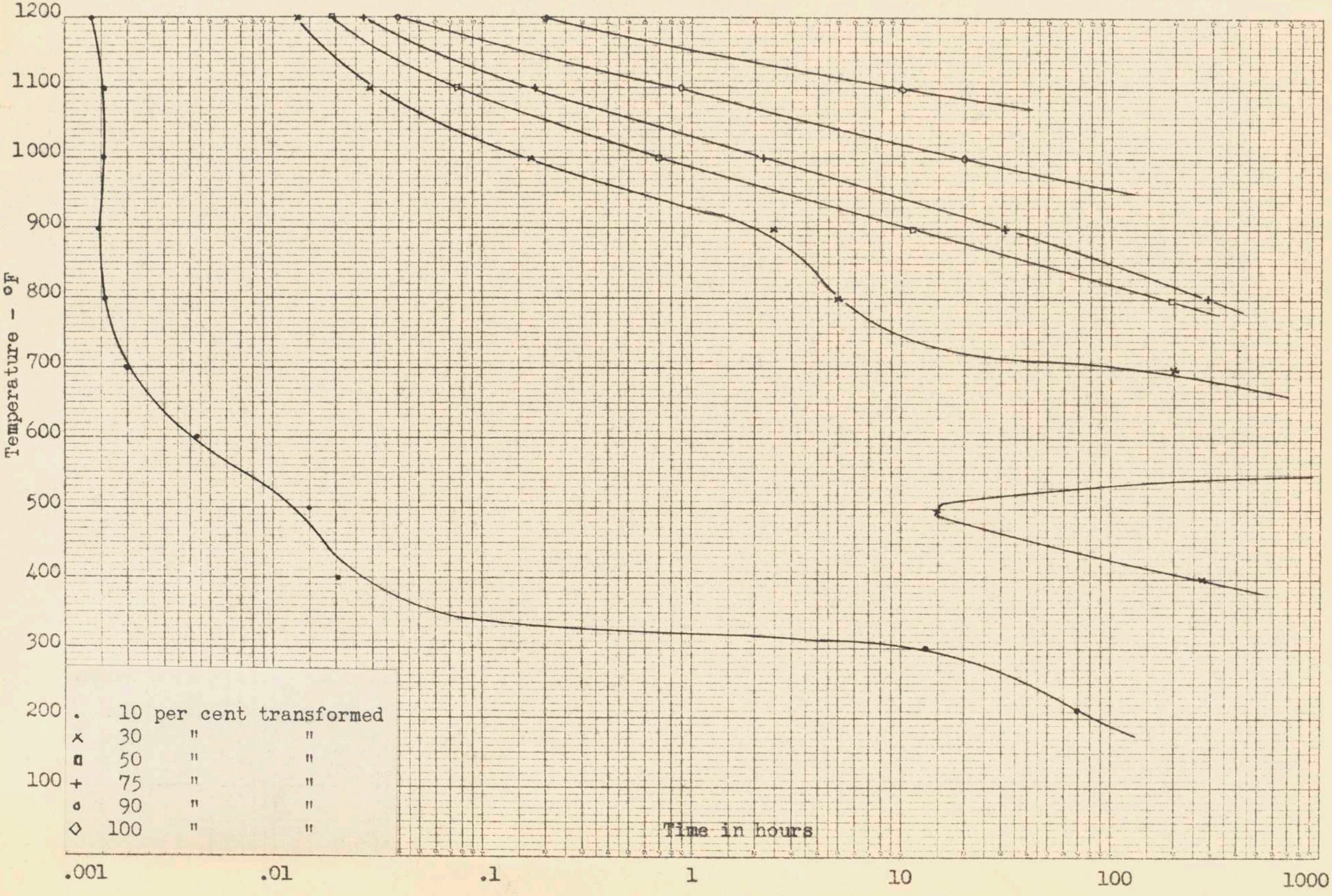


FIGURE 52 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel oil cooled from 1700° F.

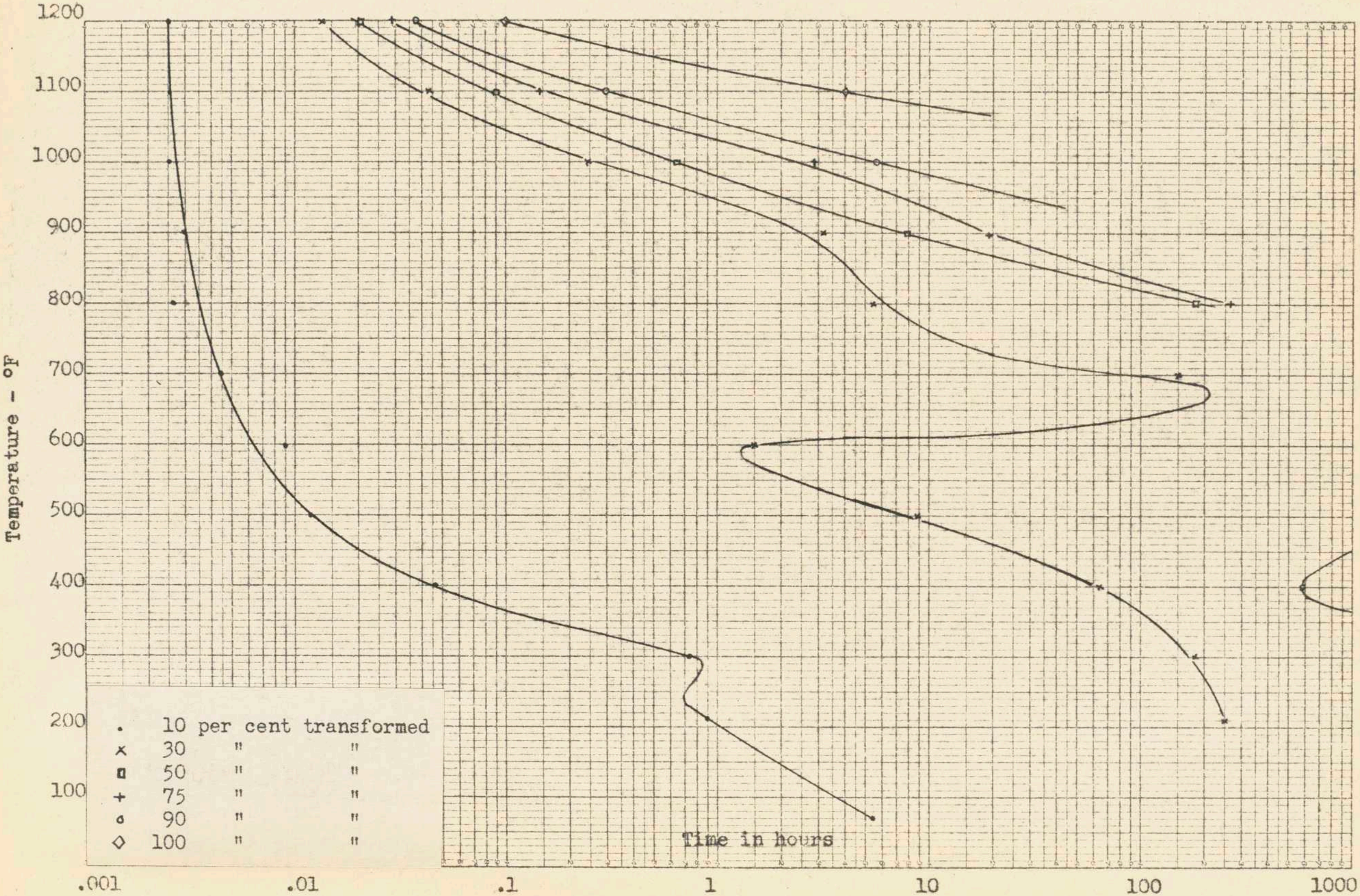


FIGURE 53 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel air cooled from 1700° F.

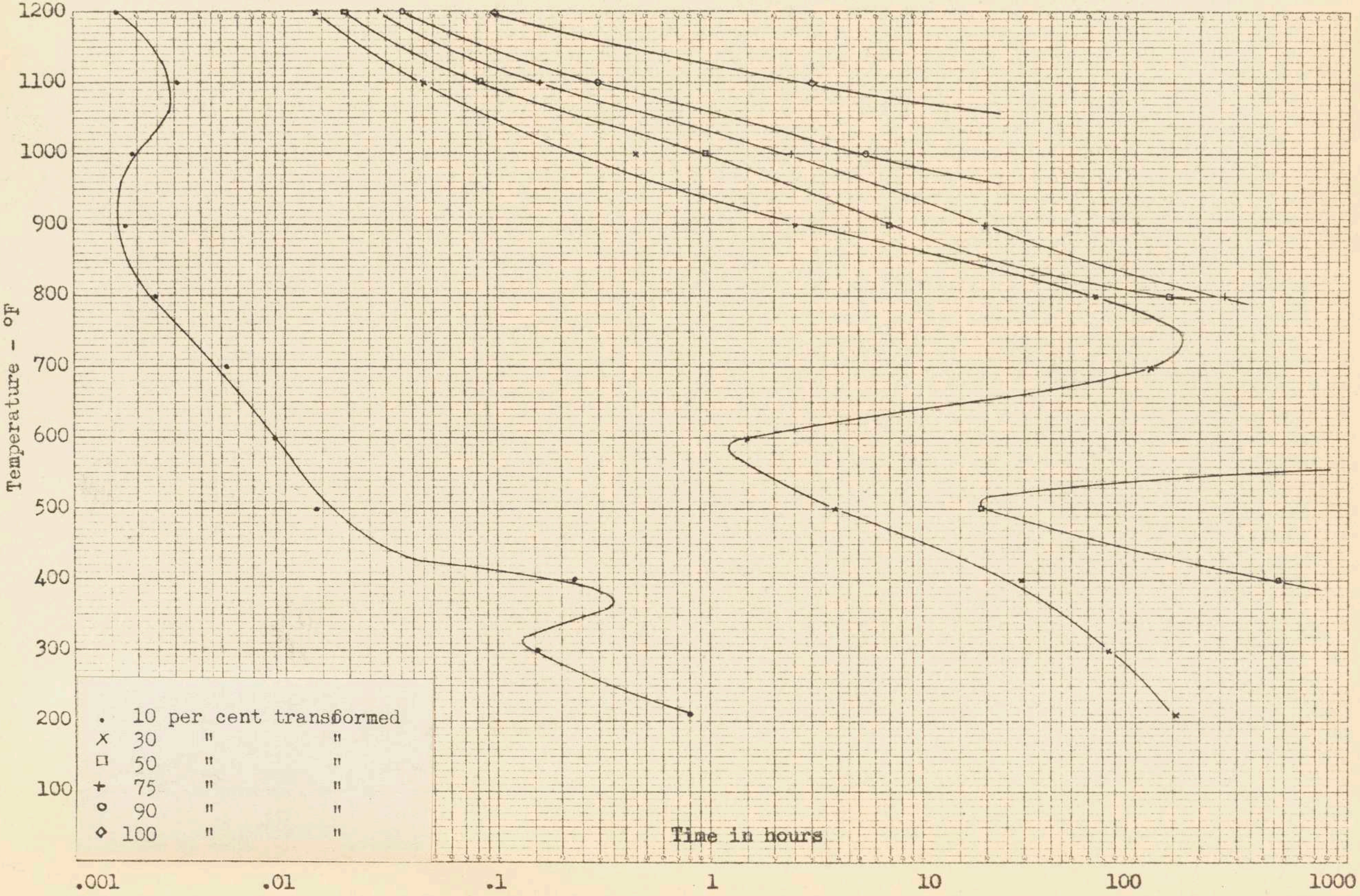


FIGURE 54 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel lime cooled from 1700° F.

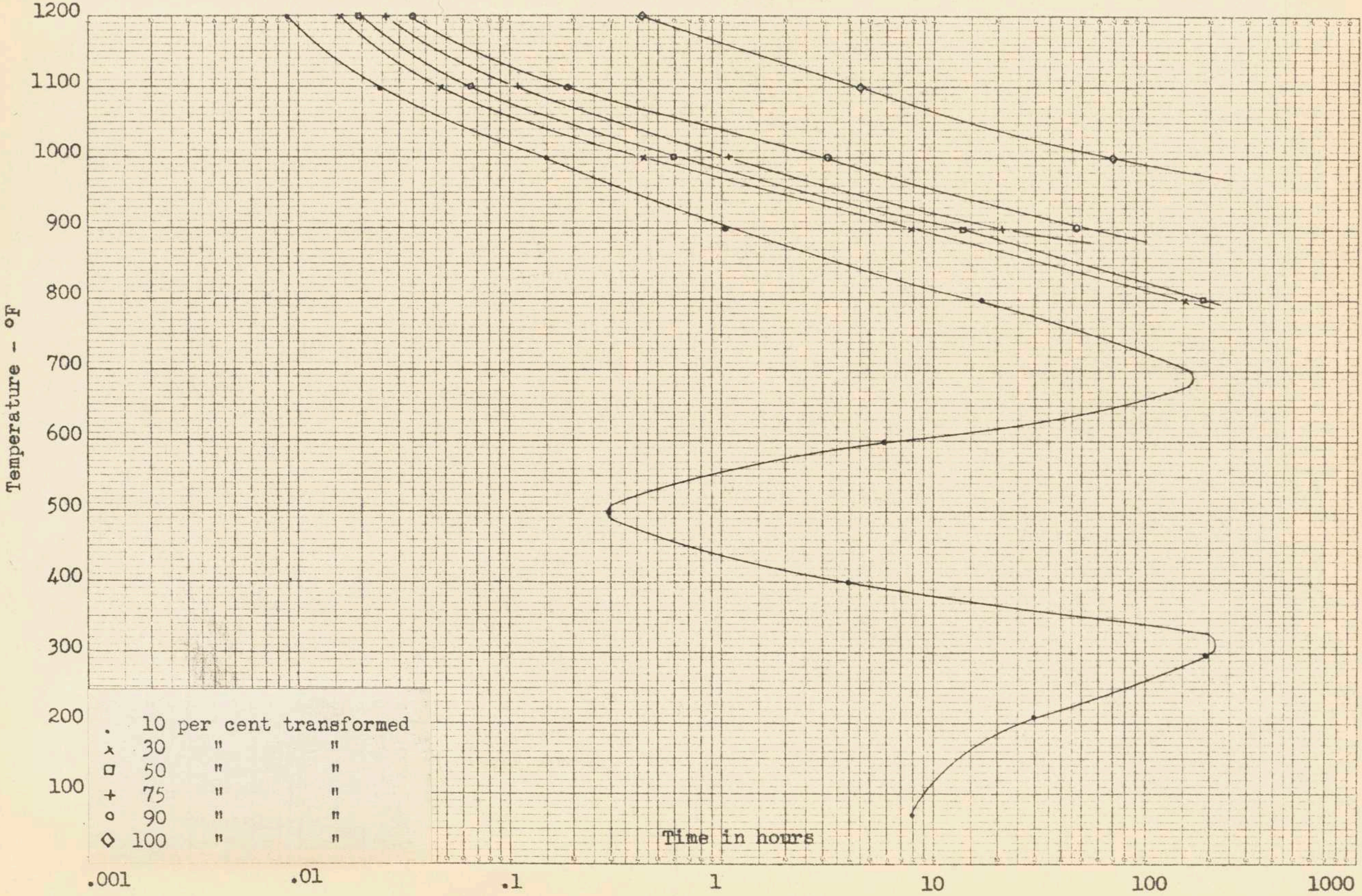


FIGURE 55 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel air cooled from 1800° F.

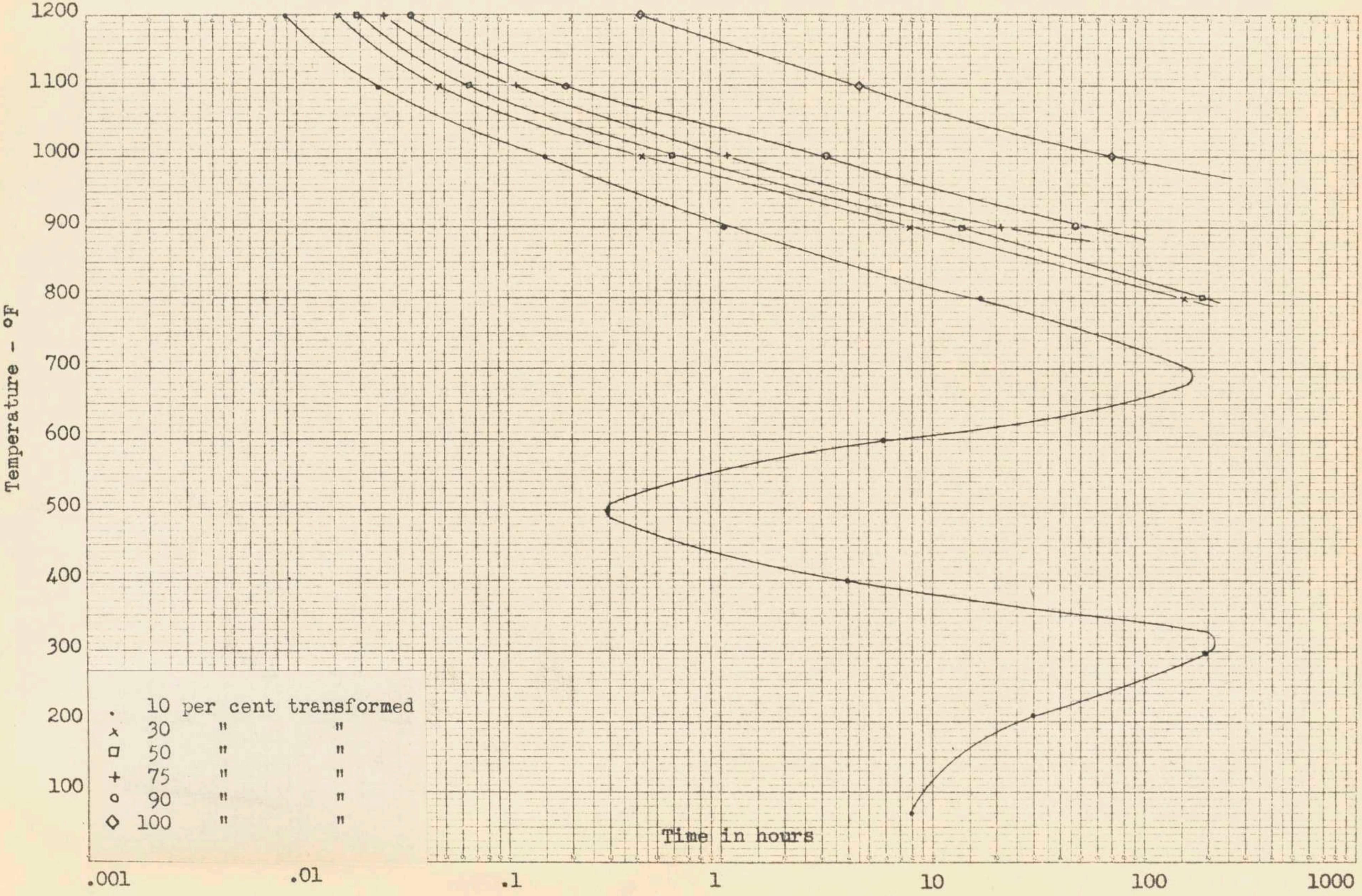


FIGURE 56 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel air cooled from 1800° F.

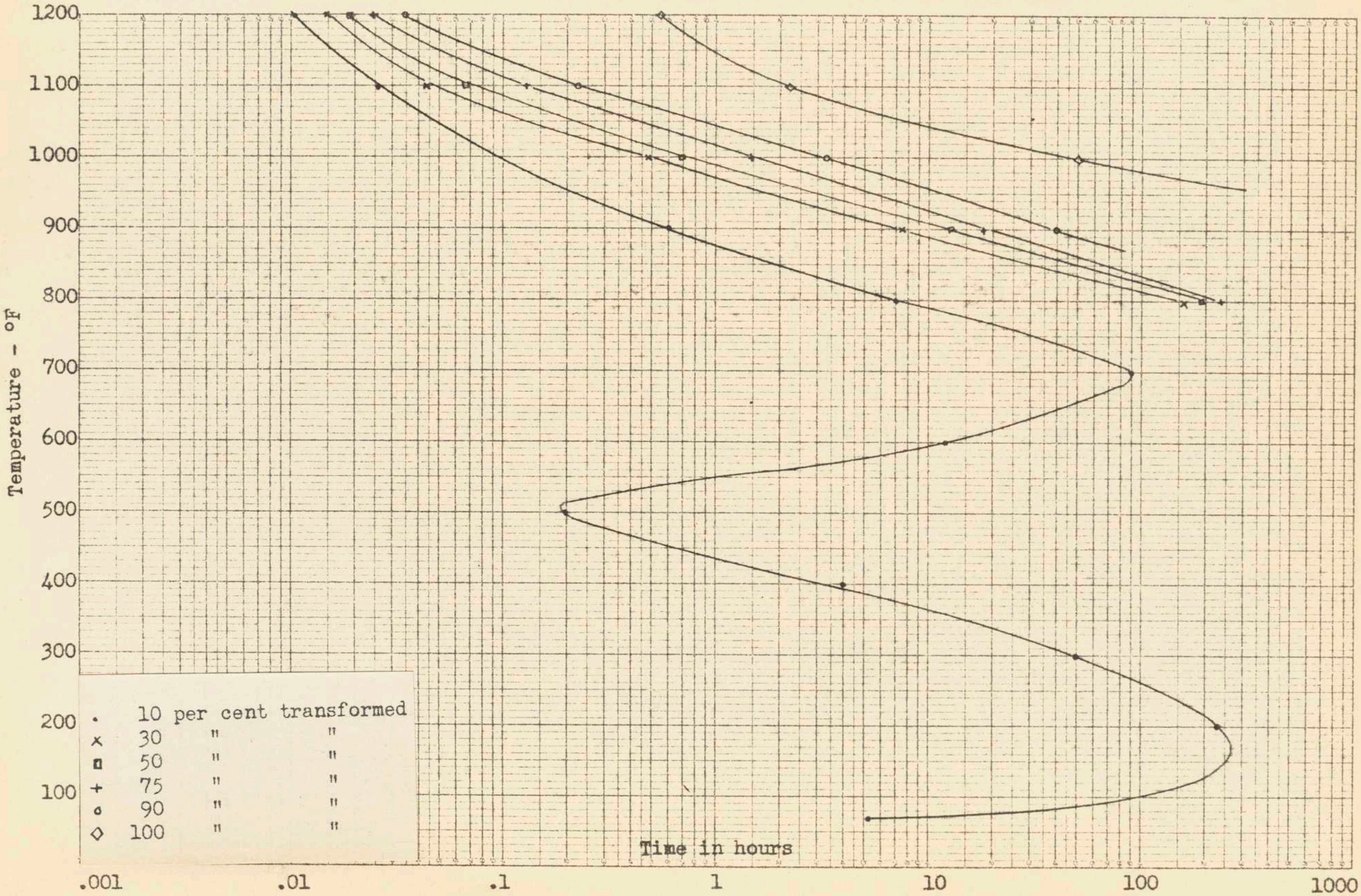


FIGURE 57 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel lime cooled from 1800° F.

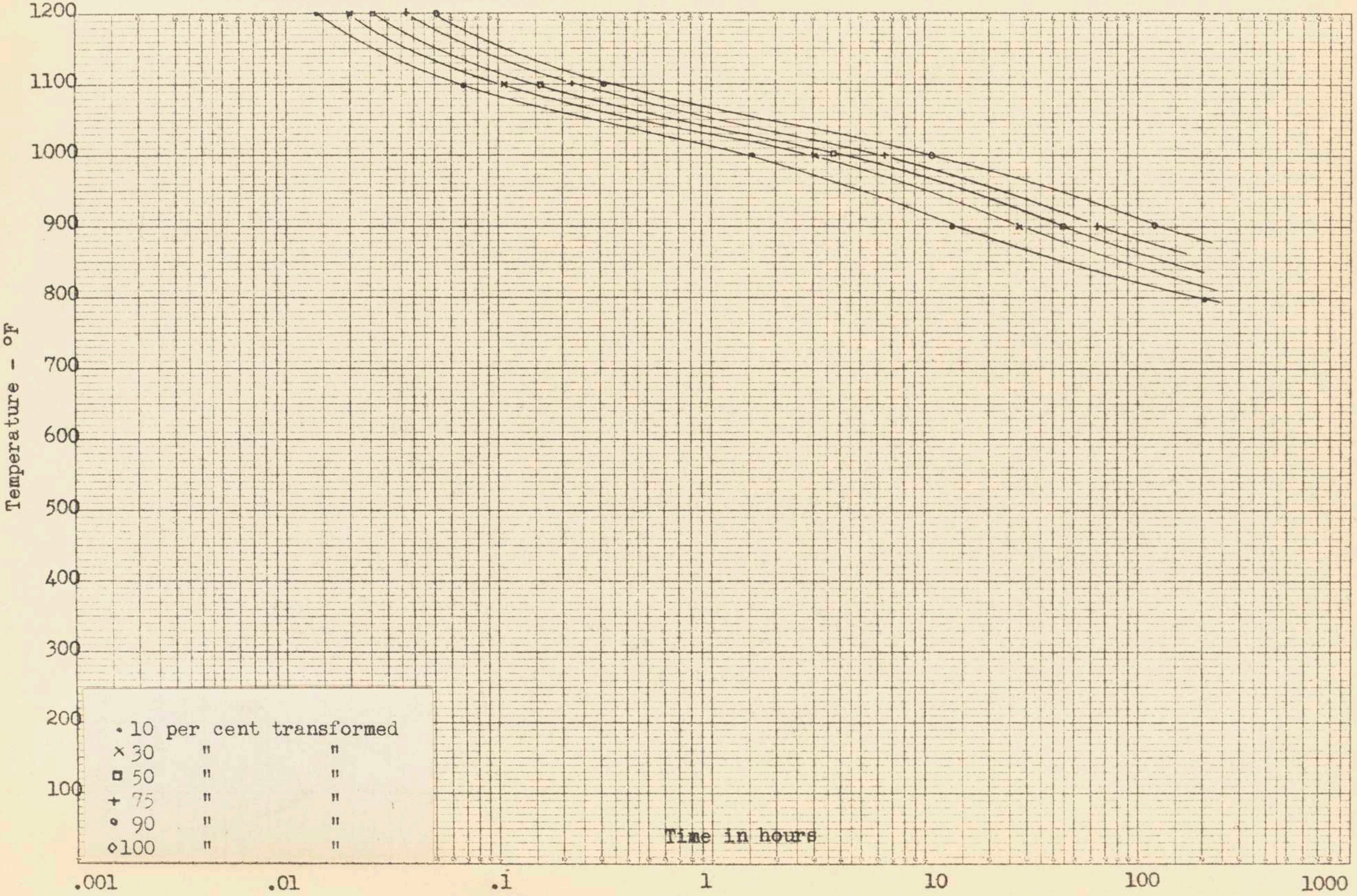


FIGURE 58 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel oil cooled from 2000° F.

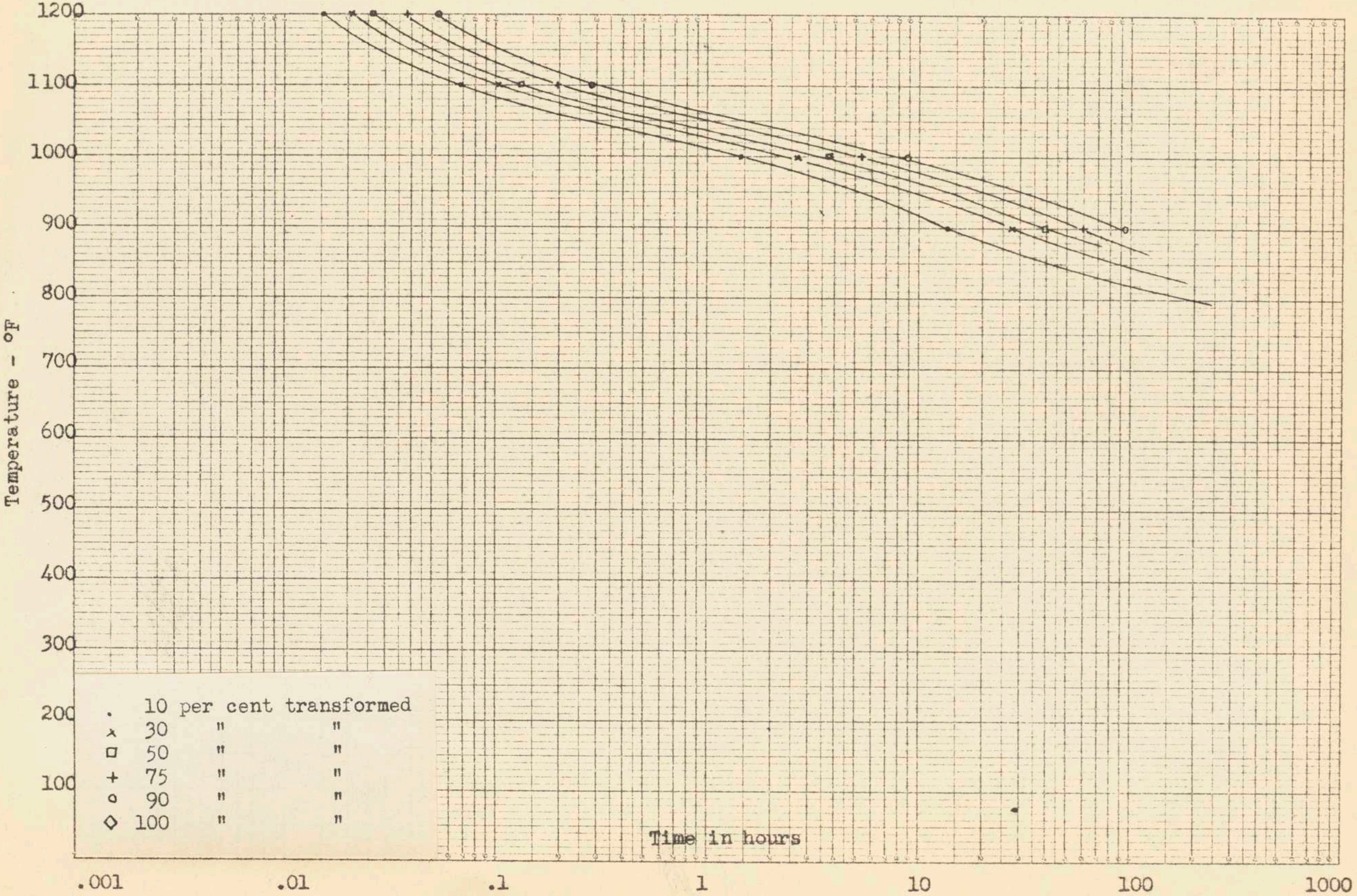


FIGURE 59 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel air cooled from 2000° F.

FIGURE 60 Transformation curves of retained austenite for 1.5% C 11.5% Cr steel lime cooled from 2000° F.

This missing information may be found in the accompanying graphs, shown in Figures 61 and 62. They show the relations between the retained-austenite content in the as-hardened state and the hardening temperature. Three rates of cooling are shown--oil, air, and lime.

It is clear that the amount of retained austenite increased sharply with the hardening temperature. In the usual range of hardening temperatures, the amount of retained austenite increased with the slowness of the cooling medium. This relation was changed at the higher temperatures.

The two steels were entirely similar in regards to the above-mentioned facts. The lower chromium steel, however, had more retained austenite at all the hardening temperatures used in this particular study. At higher hardening temperatures, however, from 2050° F. up, the 1% C - 5% Cr steel contained less austenite while the 1.5% C - 11.5% Cr steel became, except for the carbides, austenitic.

In order to allow a comparison of the minimum and maximum reaction rates for the two steels and the various heat treatments, these data have been assembled from Figures 43 to 60 inclusive and presented in Table IV and V. The minimum reaction rates will be expressed by the times and temperatures corresponding to the tips of the "bays" of the curves. The maximum reaction rates will be expressed by the times and temperatures corresponding to the tips of the "noses" of the curves.

The following facts relative to the transformation characteristics of the retained austenite in the 1% C - 5% Cr steel were obtained from Tables IV and V.

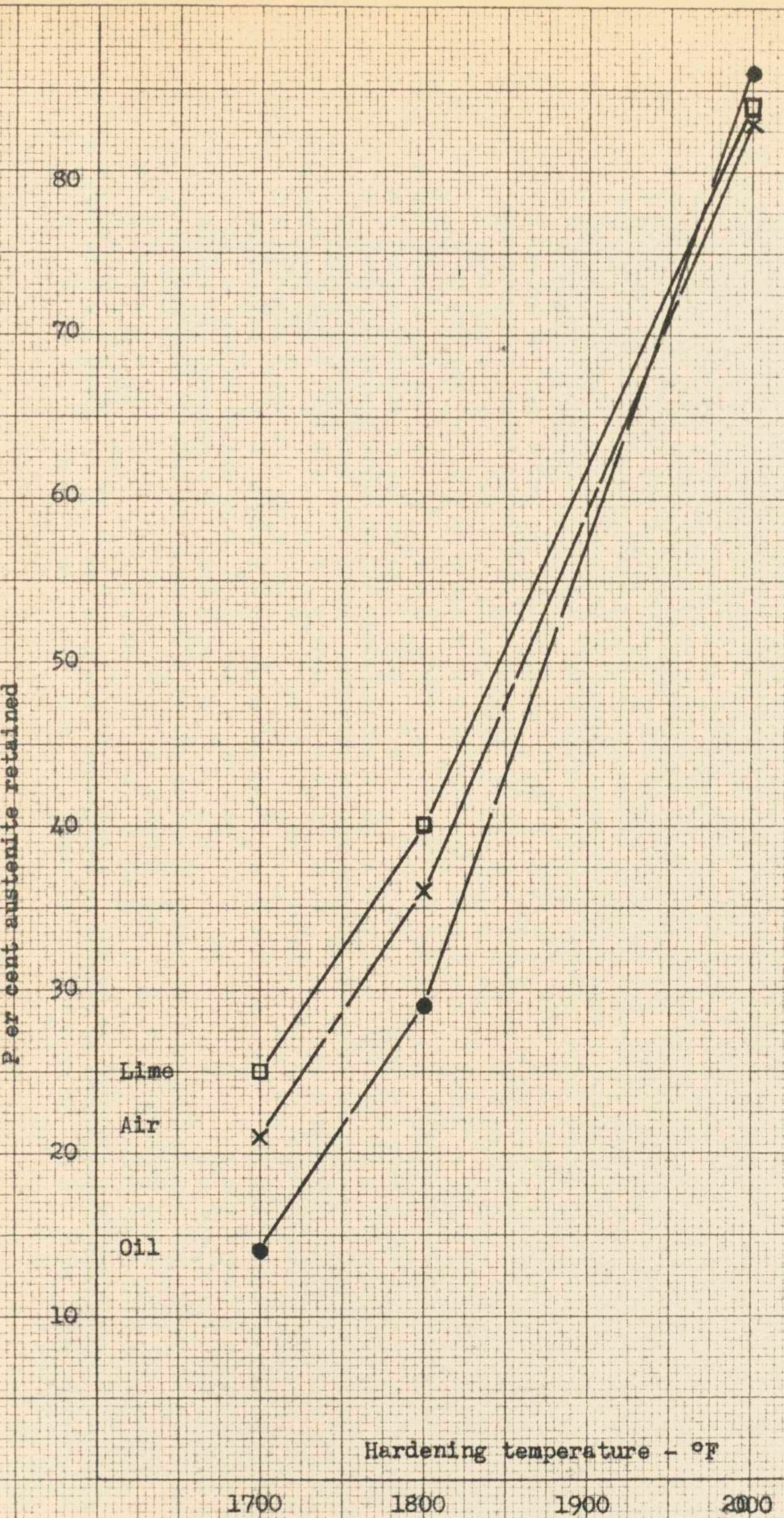


FIGURE 61 Relation between the amount of retained austenite and the hardening temperature for a 1% C 5% Cr steel cooled as shown.

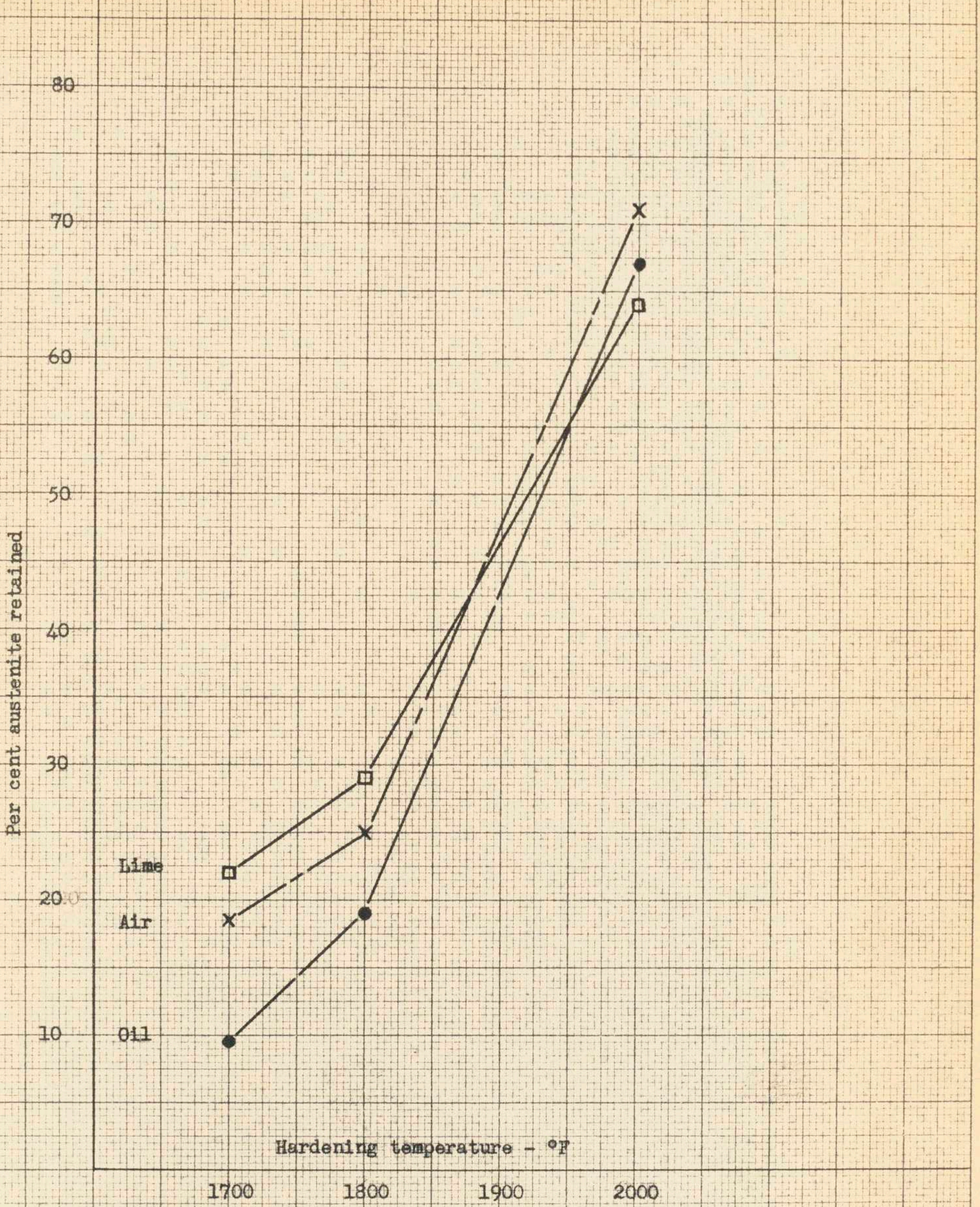


FIGURE 62 Relation between the amount of retained austenite and the hardening temperature for a 1.5% C 11.5% Cr steel cooled as shown.

TABLE IV

NOSE TIMES AND TEMPERATURES FOR LISTED EXTENTS OF TRANSFORMATIONS

Hardening Temperature °F.	Steel	Cool Medium	10		30		50		75		90	
			Hours	°F.	Hours	°F.	Hours	°F.	Hours	°F.	Hours	°F.
1700	1% C-5% Cr	Oil			.0035	990						
			.0035	500	.01	800	.07	700	.26	610	1	600
		Air	.012	590	.0045	1000						
			2.	210	.006	820	.06	690	.22	610	.45	600
		Lime	.0015	880								
			.01	580	.007	1000						
			.3	300	.01	800	.05	710	.2	620	.75	600
1800		Oil	.014	1000								
			.04	650	.25	610	1.2	570	5.8	520	14	510
		Air	.013	800								
			.045	600	.17	610	.6	590	4.7	510	13	500
		Lime	.014	1100								
			.35	700	.18	620	.6	590	3.3	530	9.5	520
2000		Oil	8	510	24	500	320	480				
		Air	6	510	17	500	40	480				
		Lime	5.5	510	17	500	42	490				
1700	1.5% C-11.5% Cr	Oil	.03	370	1.5	500	600	400				
		Air	.7	240	1.3	590	19	510				
		Lime	.0016	900	1.3	580						
			.13	310								
1800		Oil	.4	500								
		Air	.3	500								
		Lime	.2	500								
2000		Oil										
		Air										
		Lime										

TABLE V

BAY TIMES AND TEMPERATURES FOR LISTED EXTENTS OF TRANSFORMATION

Hardening Temperature °F.	Steel	Cool Medium	10		30		50		75		90	
			Hours	°F.	Hours	°F.	Hours	°F.	Hours	°F.	Hours	°F.
1700	1% C- 5% Cr	Oil	.02	650	.015	1100						
			>1000	200	4	900	45	800	700	750	>1000	720
		Air	.015	650	.025	1100						
			6	300	1.5	900	10	870	700	740	>1000	720
		Lime	.015	650	.05	1100						
			.45	400	1.5	900	20	790	700	730	>1000	720
1800		Oil	.025	1100								
			2.5	900	>1000	750	>1000	720	>1000	700	>1000	600
		Air	1.5	900								
			>1000	200	>1000	720	>1000	700				
		Lime	.75	910								
			>1000	200	>1000	700						
2000		Oil	670	610	>1000	650						
		Air	>1000	600								
		Lime	>1000	600								
1700	1.5% C- 11.5% Cr	Oil			>1000	620						
		Air	.8	300	200	680						
		Lime	.003	1080								
			.35	370	160	730						
1800		Oil	100	690								
			450	150								
		Air	160	690								
			210	320								
		Lime	90	700								
			260	170								
2000		Oil										
		Air										
		Lime										

1. The most outstanding difference in behavior of the retained austenite in the two steels was the region of high reactivity at low temperatures in the 1% C - 5% Cr steel. The following facts are relative to this steel:

a. The higher were the retained-austenite contents in the specimens hardened from a given temperature due to more-slow cooling from this temperature, the greater were the reaction rates for transformations from 50 to 90 per cent for the 1700° F. hardening, from 30 to 90 per cent for the 1800° F. hardening; but, the lower were the reaction rates for 100 per cent transformation.

b. The greater the extents of retained-austenite transformation considered the lower were the temperatures and the greater were the time values at which the maximum reaction rates existed.

c. The greater the extents of retained-austenite transformation considered the lower were the temperatures and the greater were the time values at which the minimum reaction rates existed.

d. The lower the hardening temperature the less the time required to effect any given percentage transformation.

The curves for the low percentage transformations as obtained from the 1700 and 1800° F. heat treatments for both steels are unusual in appearance. The abnormality is most apparent in the 1700° F. treated specimens. This is very likely due to the considerable instability of the retained austenite resulting from this treatment. The curves were made to follow the points because of the definite trends thus indicated.

2. The retained austenite in the 1.5% C - 11.5% Cr steel could not be completely transformed at temperatures below 900° F. The range highly reactive for the retained austenite in the 1% C - 5% Cr steel was also slightly effective here for the specimens hardened from 1700° F.

The lack of stability in the austenite retained in either steel after a 1700° F. heat treatment was further confirmed by the fact that limited room-temperature transformation would take place. The 2000° F. treatment did not produce an austenite which experienced any transformation at room temperature. It would prevail to some extent in the specimens heat treated from 1800° F.

B. Kinetics of the Transformation

1. Heating-to, Holding-at, and Cooling-from the Tempering Temperature

The kinetics of the phenomena attending the tempering of the steels used were determined in the following way. The hardened specimen was introduced into the magneto-dilatometer and the furnace power was turned on. The deflections of the galvanometer and the changes in length of the specimen were recorded at intervals of 25° F. during heating-to and cooling-from the tempering temperature.

The controls were set to give a heating and a cooling rate of 5° F. per minute for all the runs discussed in this chapter. All of the hardening was done in oil. The magnetic studies were all carried out under a field of 770 gauss. The time at the tempering temperature of 1000° F. was 2 hours.

Figures 63, 64, and 65 are typical of the plots made during a run. The changes in length and the galvanometer deflections were measured during the heating-to and the cooling-from the tempering temperature of 1000° F. A second tempering treatment was likewise studied for each specimen of the series. The length changes are plotted in inches $\times 10^{-4}$.

Figures 66 to 69, inclusive, were made by superimposing the first tempering curves of the abovementioned runs.

Since the absolute values of magnetization were not important here and the relative values only were of major interest, the values plotted were the observed galvanometer deflections, to which the magnetization values are directly proportional. Furthermore, the constant value of the coil-deflection was included with the various specimen deflections in the values titled galvanometer deflections.

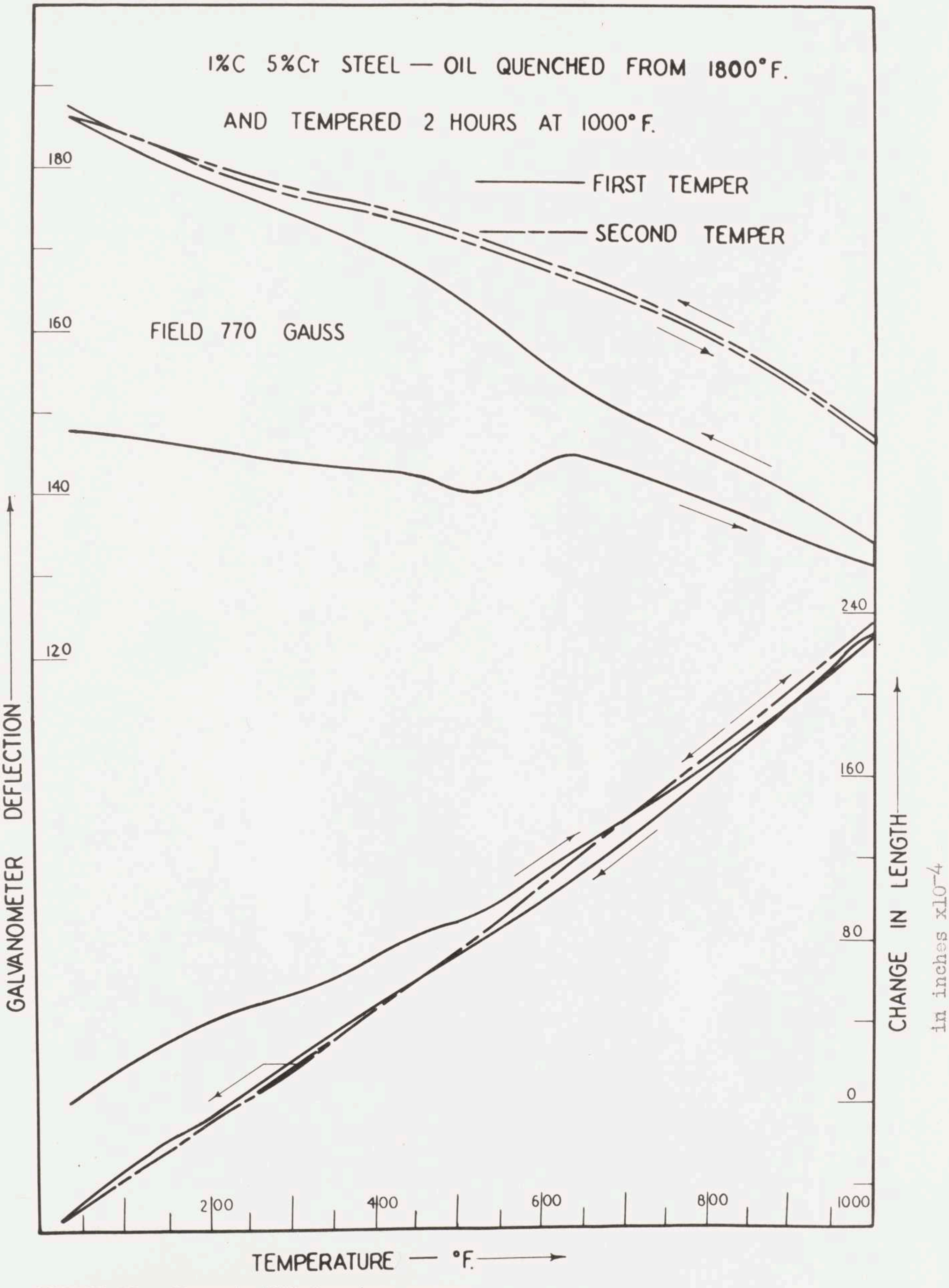


FIGURE 63. Magneto-Dilatometer curves.

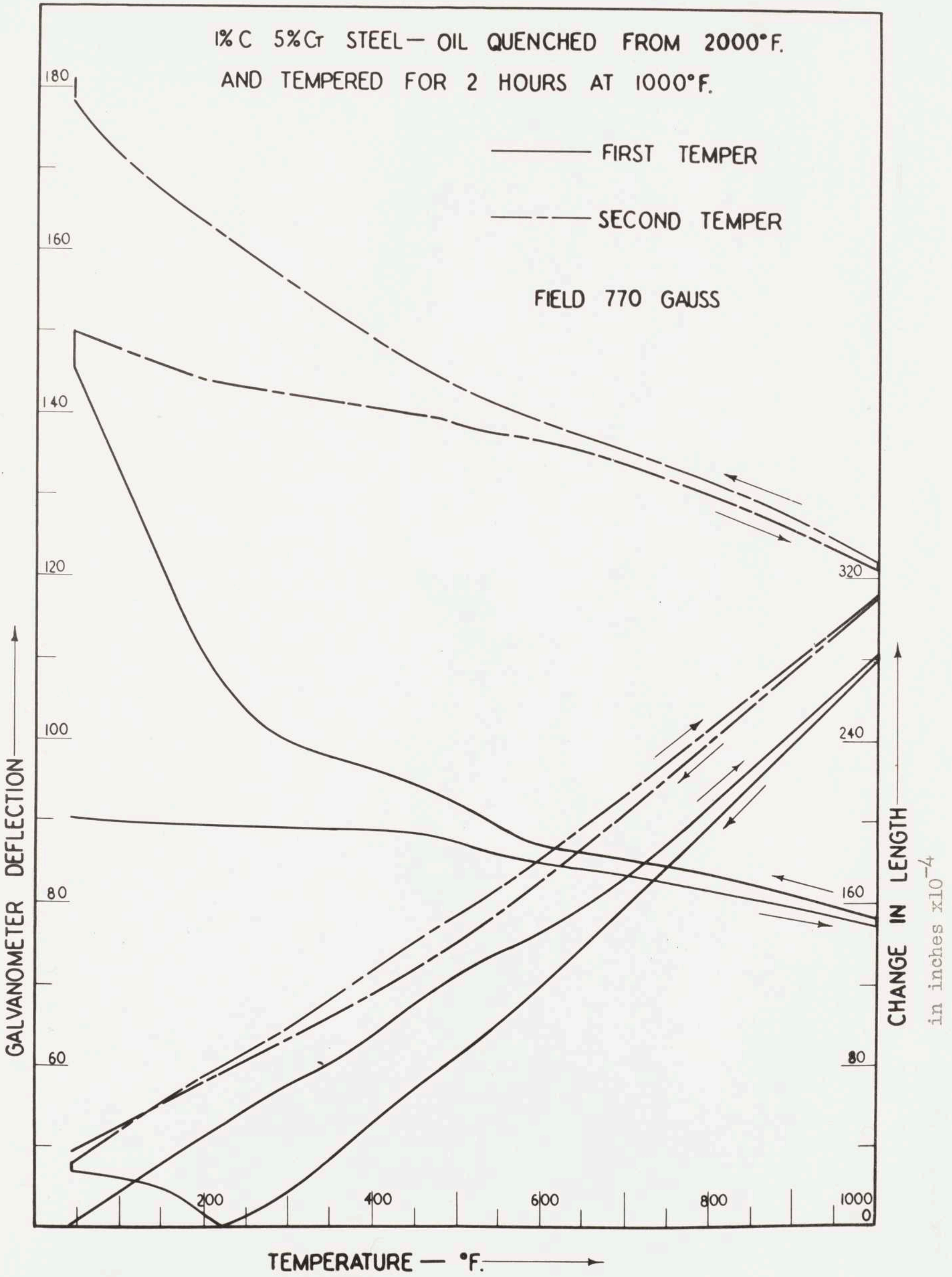


FIGURE 64. Magneto-Dilatometer curves.

In Figure 63 are shown the first and second tempering curves of a 1% C - 5% Cr steel specimen oil-quenched from 1800° F. The solid lines designate the course of the first tempering curves and the broken lines designate the course of the second tempering curves. An applied field of 770 gauss was used and the total deflections for the coil and the specimen were recorded. The changes in length were read to $\pm 1 \times 10^{-5}$ in. but were recorded only to $\pm 3 \times 10^{-5}$ in. The dimensions of the specimens were 4-1/4 in. in length and 1/4 in. in diameter.

The magnetization curves are shown in the upper portion and the dilatometer curves are shown in the lower portion of the figure.

In Figure 64 are shown the first and second tempering curves of a 1% C - 5% Cr specimen oil-quenched from 2000° F. The curves of Figures 63 and 64 show that the retained austenite in the specimen hardened from 1800° F. was completely transformed by the first temper and that retained austenite in the specimen hardened from 2000° F. was not completely transformed by even two tempers at 1000° F. The completion of the transformation is demonstrated by the coincidence of the heating and cooling curves (both magnetization and dilation) for the second tempering of the 1800° F.-hardened specimen.

The expansion due to the retained-austenite transformation in the 1800° F.-hardened specimen was considerably less than the two contractions experienced during the heating period. When the much greater amount of retained austenite in the 2000° F. hardened specimen transformed, however, the coincident expansion resulted in an over-all increase in the room-temperature-length of the specimen.

During the two-hour-hold of the specimens at 1000° F. an increase in magnetization was always produced. However, in the 1800° F.-hardened specimen the change in length was one of expansion while in the 2000°F.-hardened specimen it was one of contraction. This can be explained by the fact that the amount of retained-austenite transformed at 1000° F. was greater for the 1800° F.-hardened specimen.

It should be noted that the courses of the second-tempering heating (magnetization) curves bear no relation to the first-tempering cooling (magnetization) curves. This is proof that the sharp rises in the cooling curves were results of austenite transformation and not results of curie transformations.

The second-tempering heating (dilation) curve of the 2000° F.-hardened specimen showed the same contractions as the first-tempering heating (dilation) curve but to a lesser degree. This indicated that the transformation of retained austenite during the cooling from 1000° F. produced a state similar to the transformation pf primary-austenite during the cooling from the hardening temperature.

Only one of these double-tempering runs is given for the 1.5% C - 11.5% Cr steel. Figure 65 shows the first and second tempering curves of a 1.5% C - 11.5% Cr steel specimen oil-quenched from 2000° F. The close similarity of these curves to the corresponding ones for the 1% C - 5% Cr specimen should be noted.

Another interesting fact comes from the vertical portions at room temperature of the cooling curves, both magnetization and dilation. These lines represent the increases in magnetization and in dilation that occurred over a period of 12 hours (the time interval between the

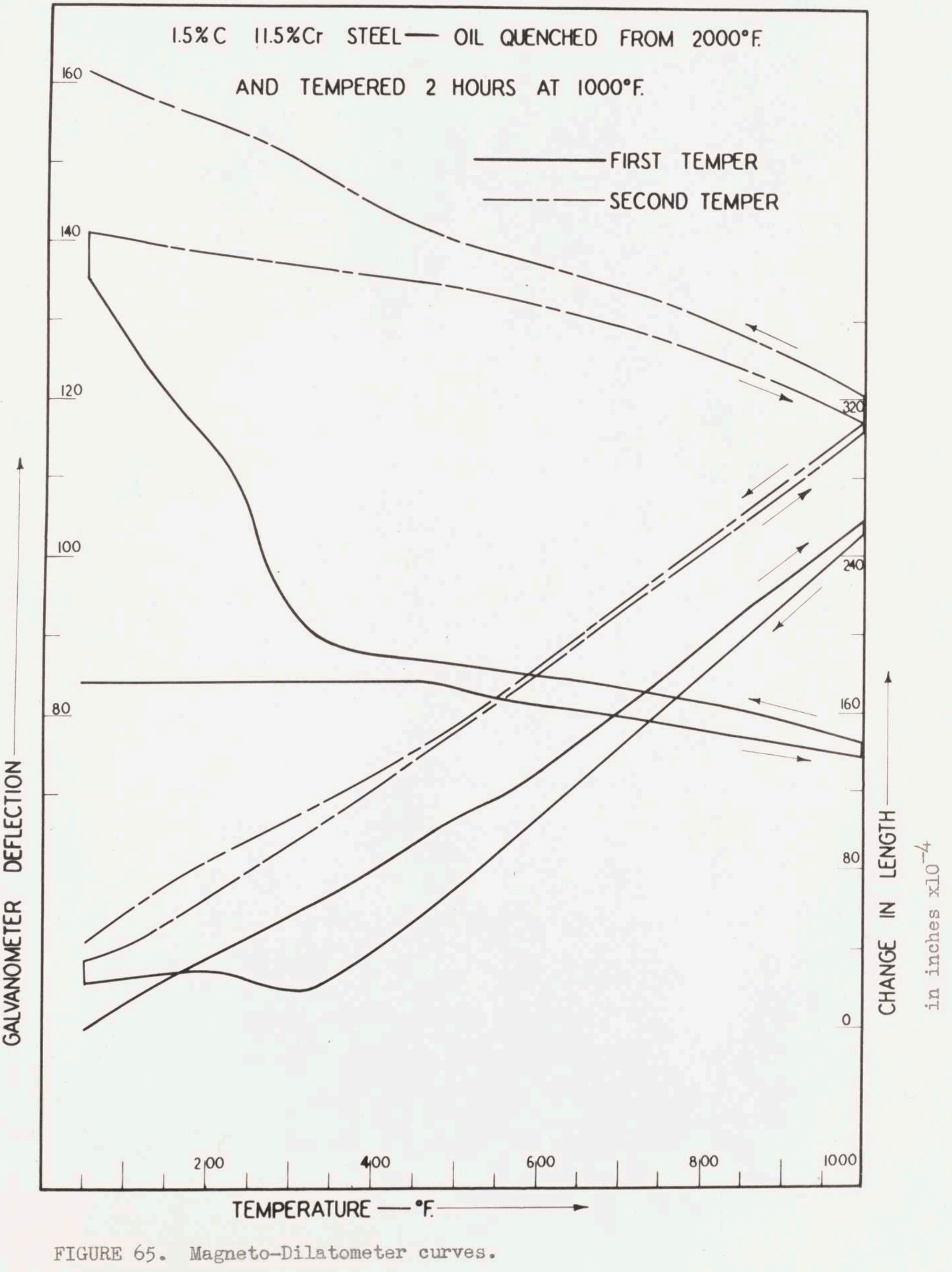


FIGURE 65. Magneto-Dilatometer curves.

completion of the first, and the start of the second tempering run). They can only be explained as being attendant to room temperature transformation of the retained austenite.

Fourteen of these runs were made, seven for each steel. The heating-magnetization, cooling magnetization, heating-dilation, and cooling-dilation curves for the first tempering operations of the seven runs for each steel were each assembled on separate plots by super-imposing them. Thus, the displacements of the curves are not relatively significant.

In Figure 66 are shown the heating (magnetization) curves of 1% C - 5% Cr specimens oil-quenched from 1700, 1800, 1850, 1900, 2000, and 2200° F. The cooling (magnetization) curves are shown in Figure 67. The heating (dilation) curves are shown in Figure 68, and the cooling (dilation) curves are shown in Figure 69.

From these curves the following facts may be obtained:

a. Transformation of the retained austenite occurred during heating in the specimens quenched from 1700, 1800, 1850, and 1900° F.

b. This transformation upon heating was less in extent the higher the quenching temperature.

c. In the range 450 - 550° F. each of the seven heating (magnetization) curves displayed a drop of greater slope than in the preceding portion of each curve.

d. The phenomenon whose magnetic effect was mentioned in (c) caused a contraction over the same range in the heating (dilation) curves.

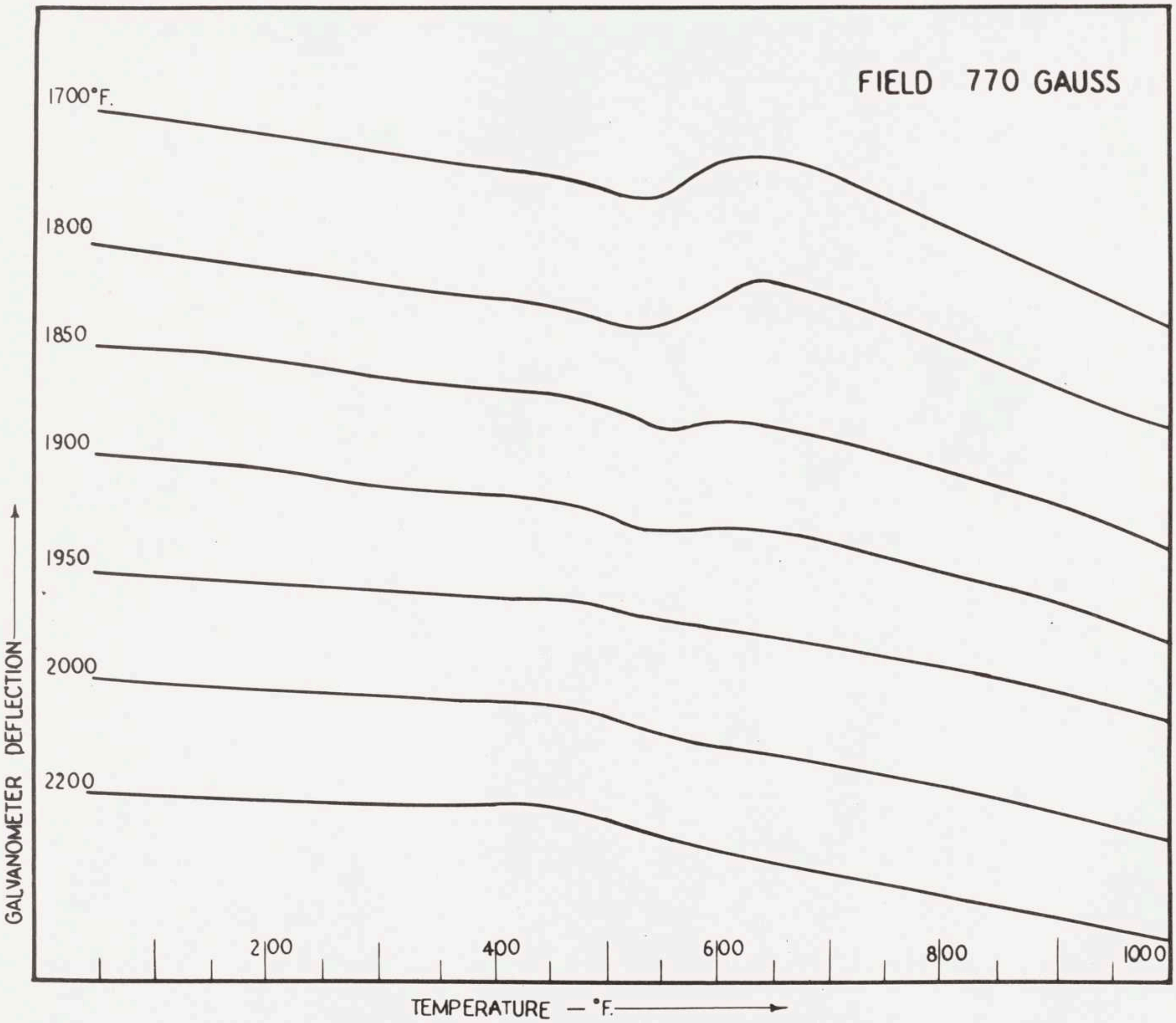


FIGURE 66. HEATING CURVES OF 1%C 5%Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN.

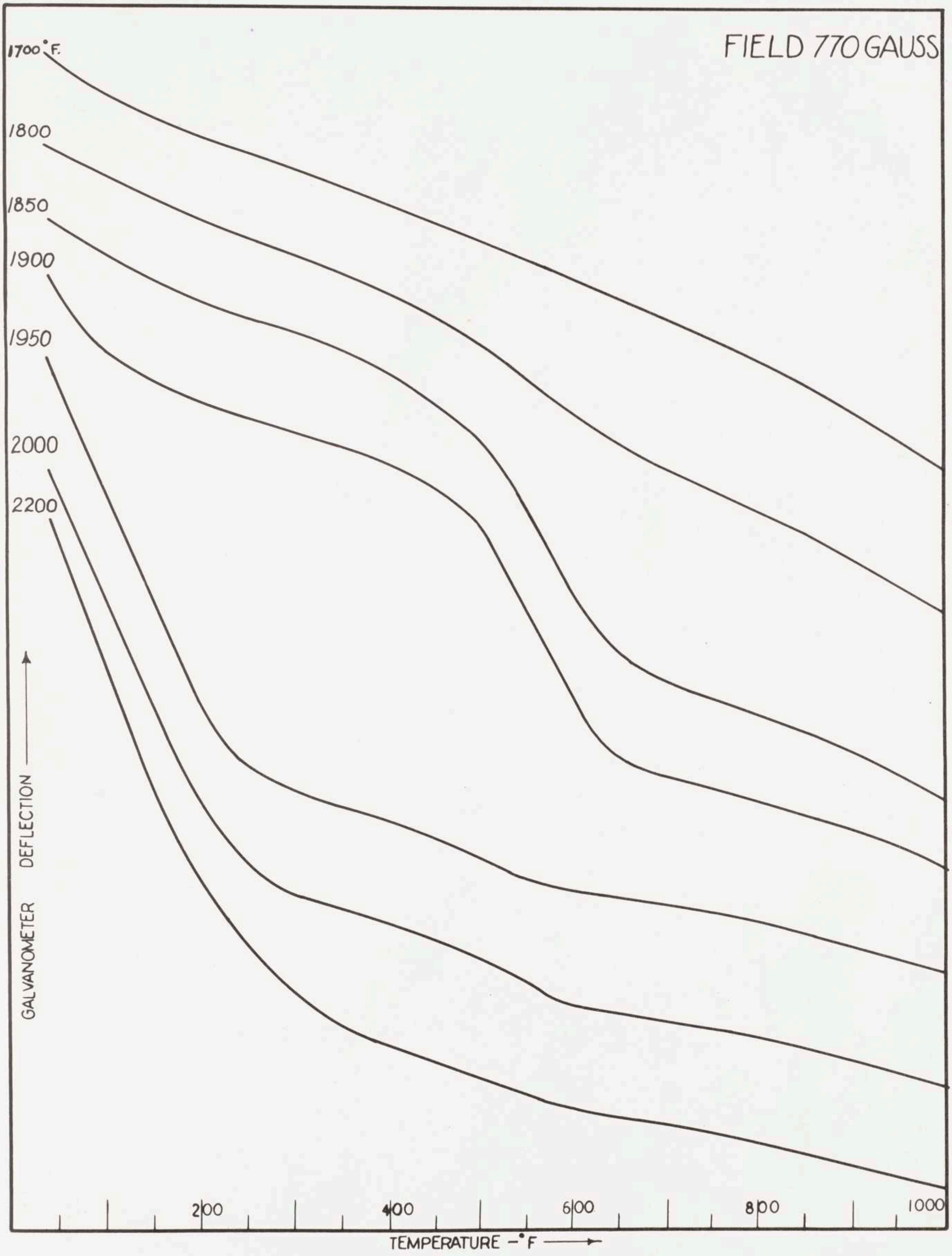


FIGURE 67. COOLING CURVES OF 1% C 5%Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN AND TEMPERED 2 HOURS AT 1000° F.

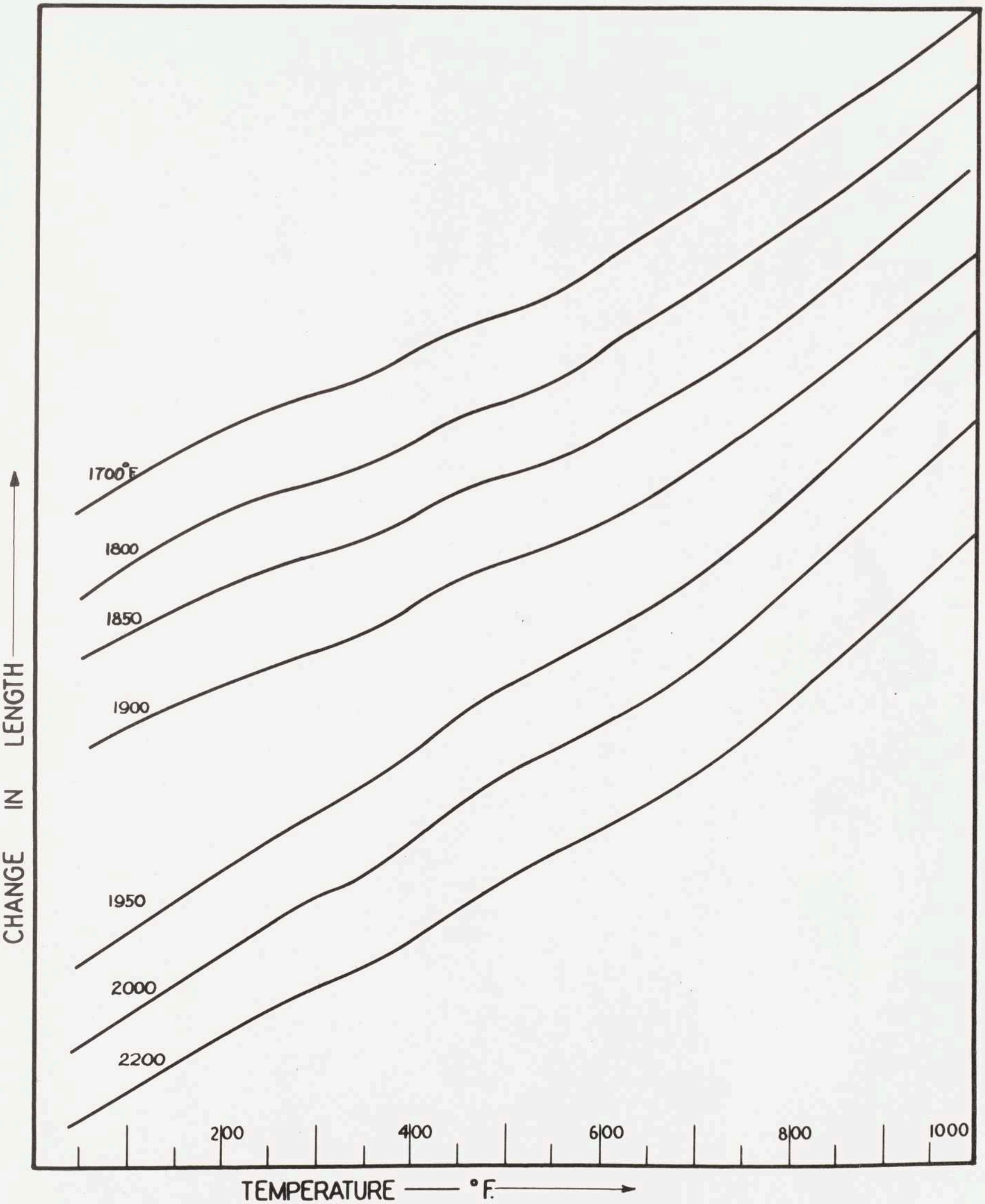


FIGURE 68. HEATING CURVES OF 1% C 5% Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN

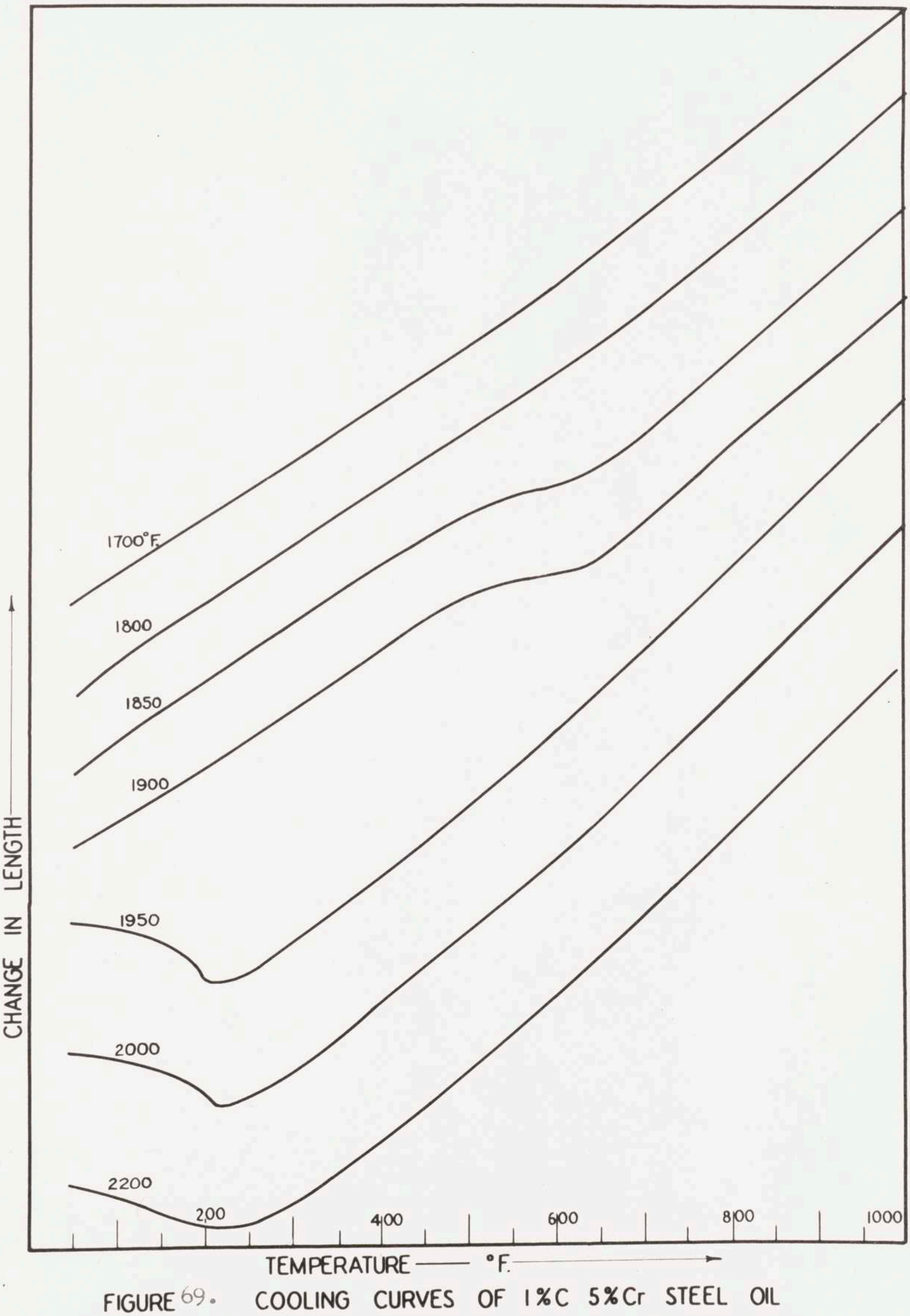


FIGURE 69. COOLING CURVES OF 1%C 5%Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN AND TEMPERED 2 HOURS AT 1000°F.

e. The contraction observed in the range 275 - 350° F. of the heating (dilation) curves was not attended by a magnetic effect.

f. The transformation of the retained austenite that took place upon cooling from the tempering temperature was greater in extent (1) the lower the extent of the austenite transformation that took place during heating, and (2) the greater amount of austenite retained by the quench.

g. The retained austenite in the 1700° F.-quenched specimen was all transformed during the heating-to and holding-at the tempering temperature (1000° F.).

h. Progressively smaller amounts of the retained austenite in the 1800° F.-, 1850° F.- and 1900° F.-quenched specimens were transformed during the heating-to and holding-at periods and progressively greater amounts of their retained austenite transformed during cooling from 1000° F.

i. The austenite transformation that existed during the cooling-from 1000° F. of the 1800° F.-, 1850° F.-, and 1900° F.-quenched specimens started close to 600° F. and finished close to 500° F. (An exception was the 1900° F.-quenched specimen, in which a small second stage of the transformation started below 150° F.).

j. The transformations of the retained austenite in the 1950° F.- 2000° F.- and 2200° F.-quenched specimens were definitely two-stage, with the second stage the more marked one.

k. In two-stage transformation, the first stage started close to 600° F. and the second close to 200° F.

In Figure 70 are shown the heating (magnetization) curves of 1.5% C - 11.5% Cr specimens oil-quenched from 1700, 1800, 1900, 2000, 2050, 2100, and 2200° F. The cooling (magnetization) curves are shown in Figure 71. The heating (dilation) curves are shown in Figure 72, and the cooling (dilation) curves are shown in Figure 73.

From these curves the following facts may be obtained:

a. No transformation of the retained austenite occurred during heating.

b. The slopes of the heating (magnetization) curves for the steels quenched from 1700, 1800, 1900, and 2000° F. were of two kinds: (1) practically level before 500° F. and (2) fairly steep beyond 500° F.

c. The heating (magnetization) curves of the specimens quenched from 2050, 2100, and 2200° F. were straight lines parallel to the abscissa.

d. The heating (dilation) curves for the specimens quenched from 1700, 1800, 1900, and 2000° F. showed two contractions--one over the range 275 - 350° F. and the other over the range 450 - 550° F.

e. The heating (dilation) curves for the specimens quenched from 2050, 2100, and 2200° F. were straight lines of much greater slopes than the over-all slopes of the curves mentioned in (d).

f. No retained austenite transformed during cooling of the 1700° F.-quenched specimen. Some transformed to a slight extent in the 1800° F.-quenched specimen. The transformation proceeded to a considerable extent in the 1900° F.- and the 2000° F.-quenched specimens. No transformation was noticed in the 2050° F.-, 2100° F.-, and 2200° F.-quenched specimens.

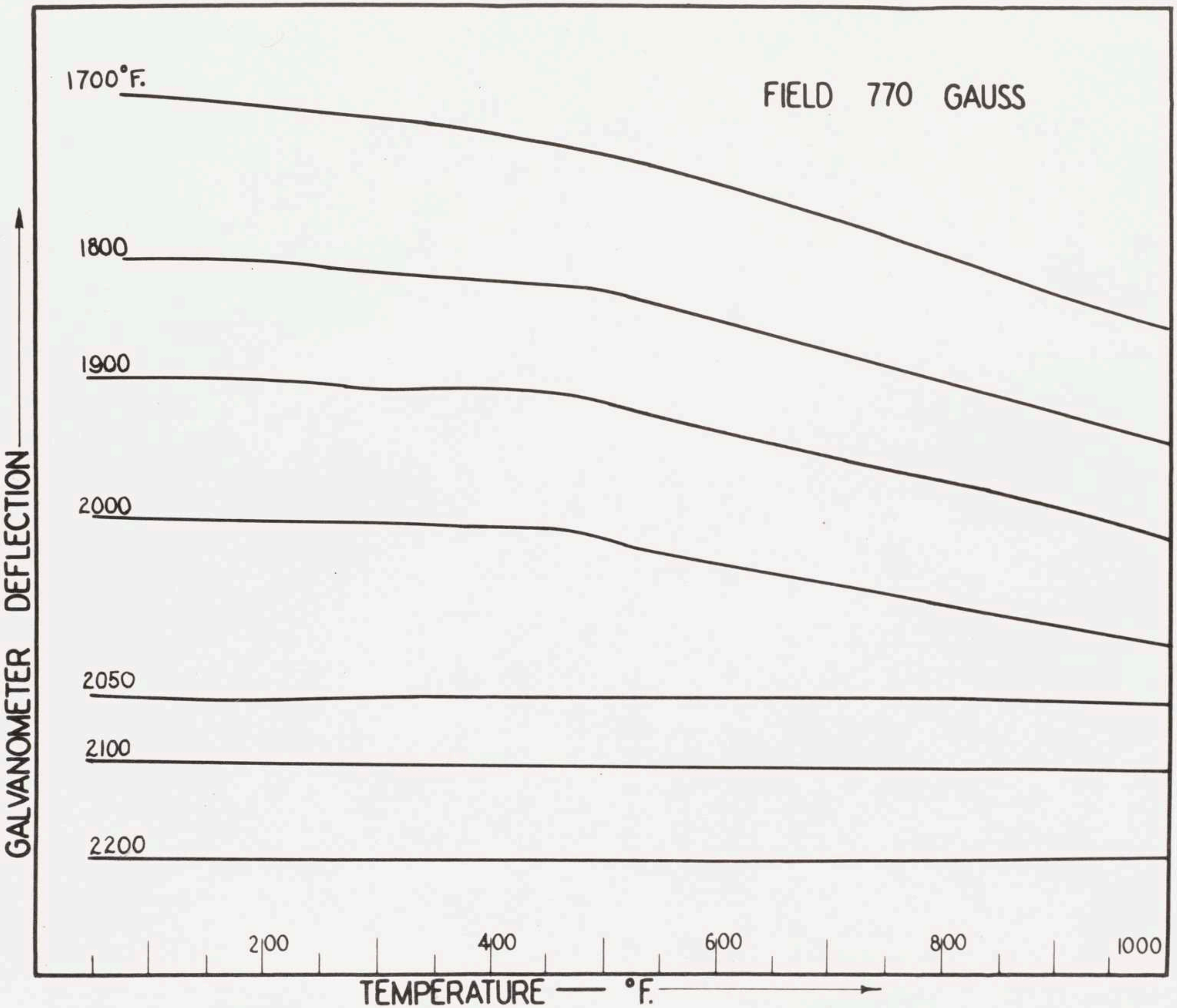


FIGURE 70. HEATING CURVES OF 1.5% C 11.5% Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN.

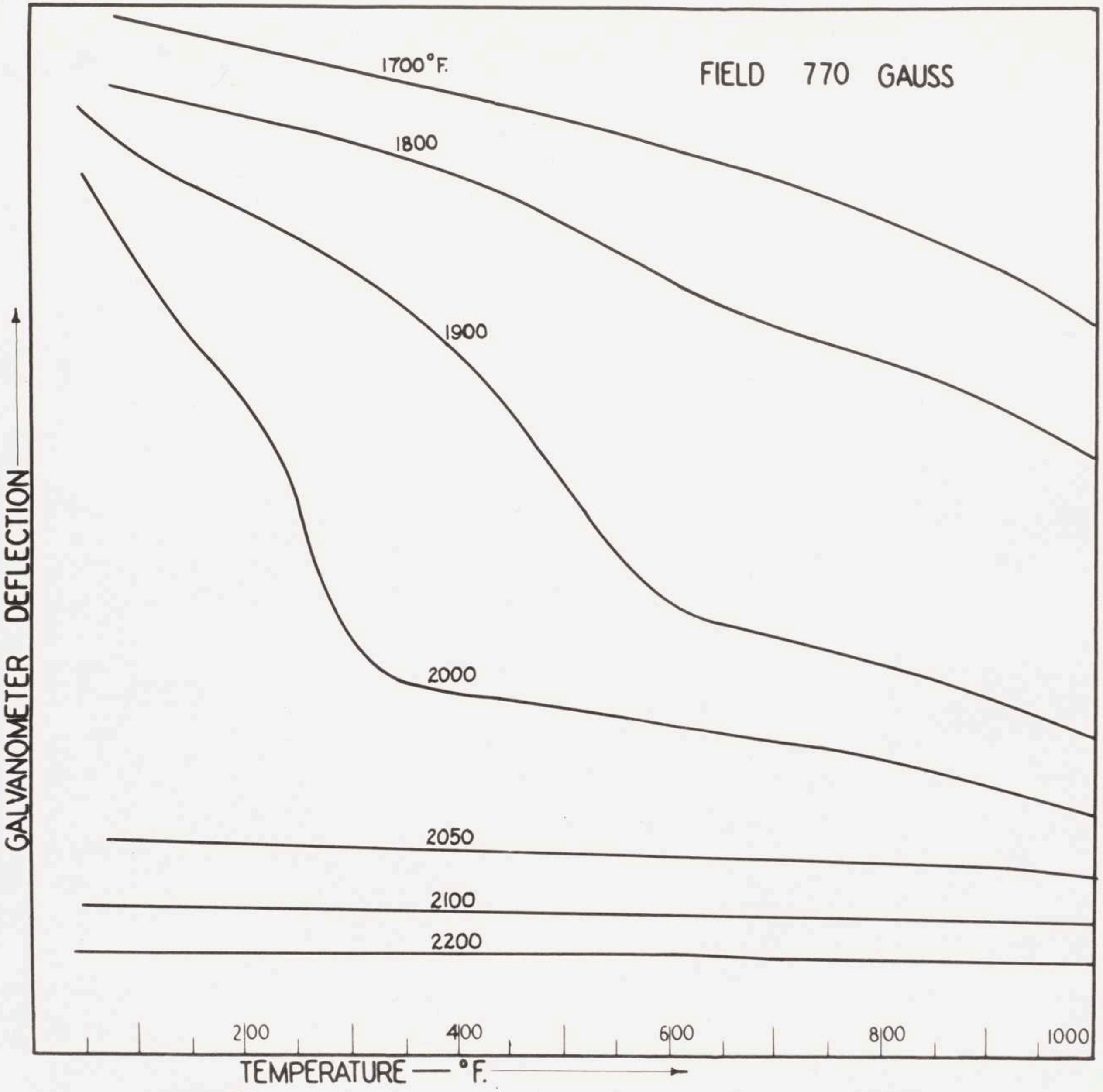


FIGURE 71. COOLING CURVES OF 1.5%C 11.5% Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN AND TEMPERED 2 HOURS AT 1000° F.

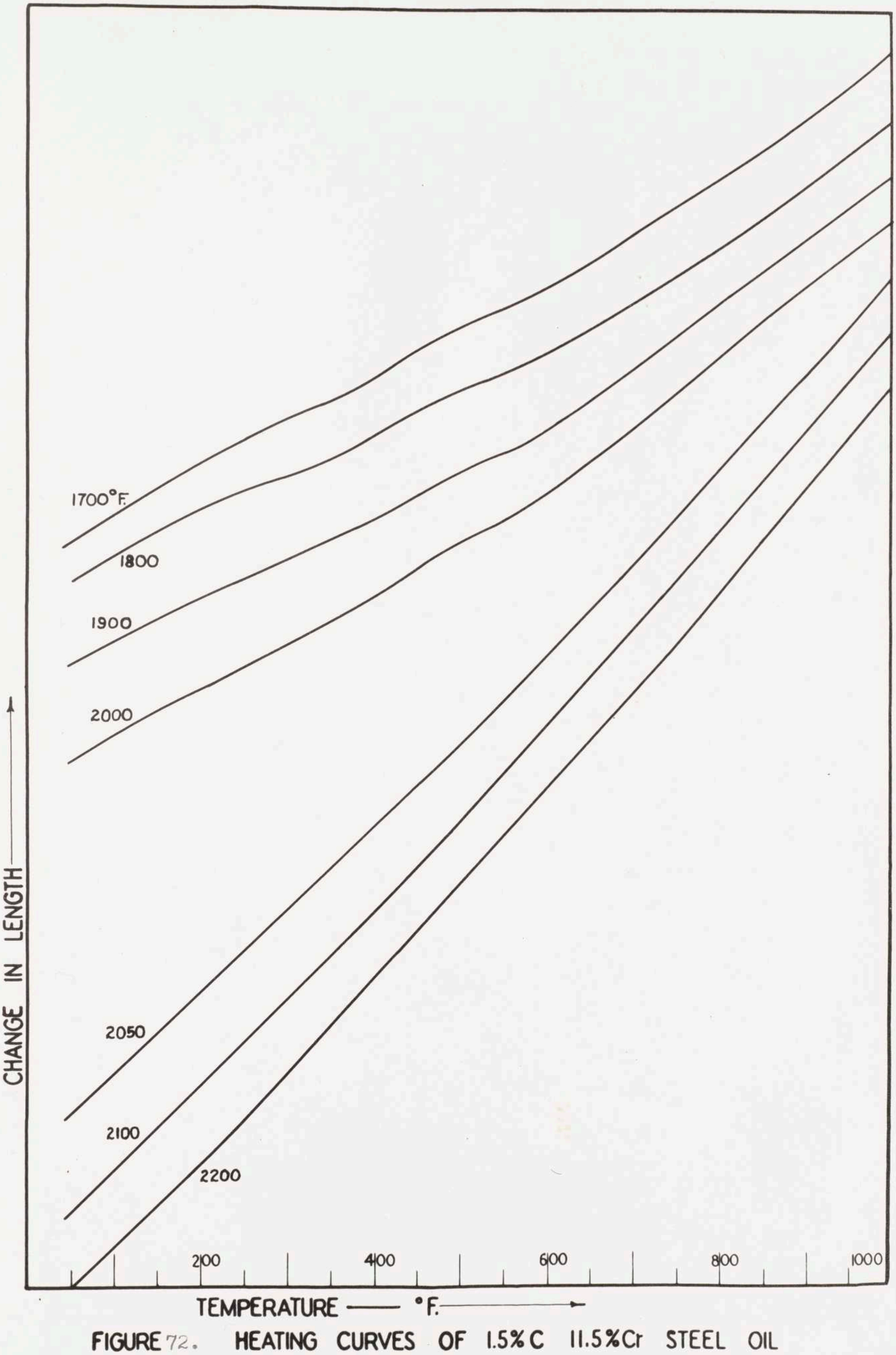


FIGURE 72. HEATING CURVES OF 1.5% C 11.5% Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN.

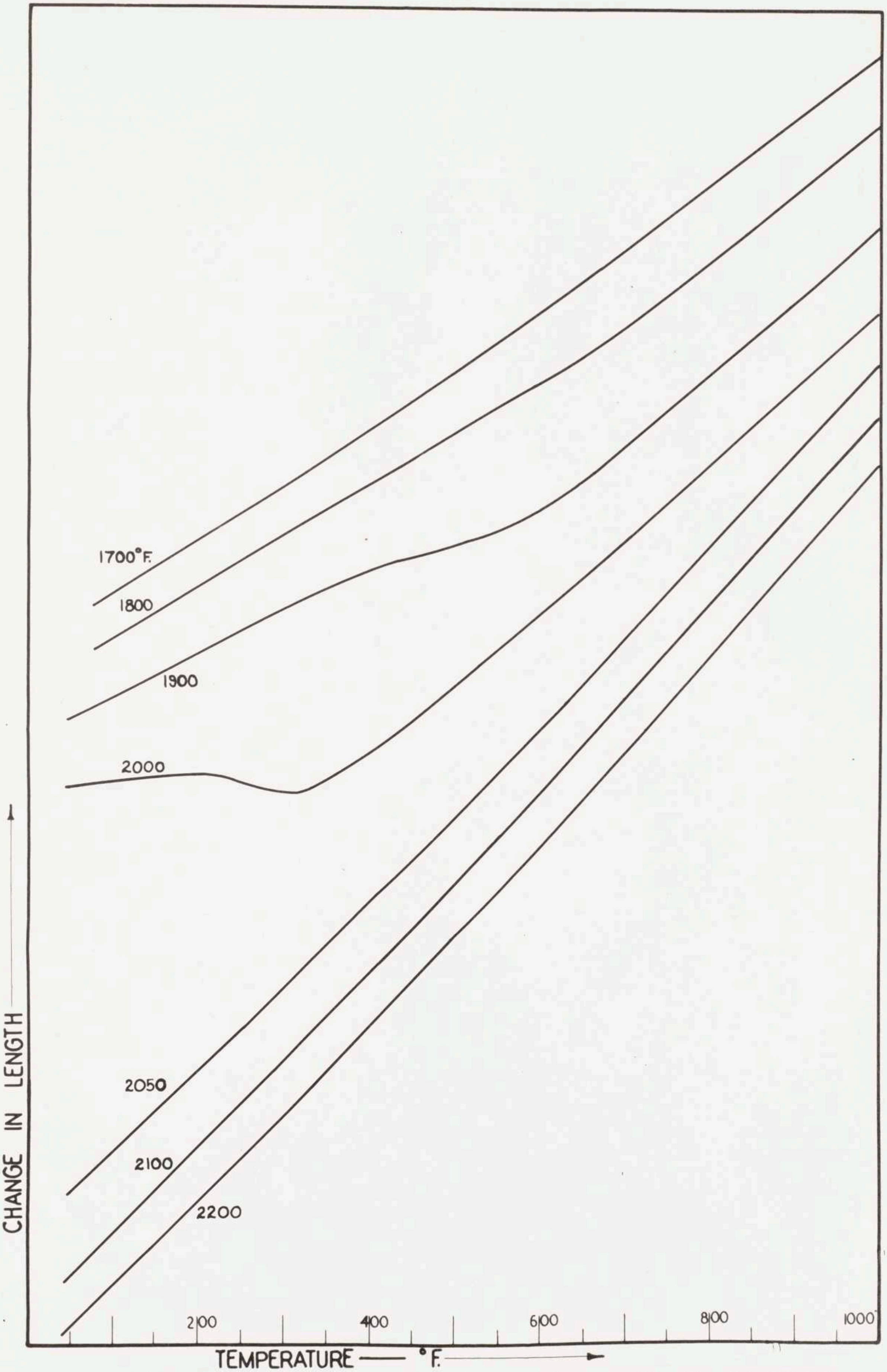


FIGURE 73. COOLING CURVES OF 1.5%C 11.5%Cr STEEL OIL QUENCHED FROM TEMPERATURES SHOWN AND TEMPERED 2 HOURS AT 1000°F.

g. The transformations during cooling started at 600° F. for the 1800° F.-and the 1900° F.-quenched specimens and at 325° F. for the 2000° F.-quenched specimen.

2. The Effect of Temperature and Time at Temperature

The curves in Figure 74 show the effects of tempering temperature and time at temperature upon the transformation of retained austenite in specimens of 1% C - 5% Cr steel oil-quenched from 2000° F. In all, the rates of heating and of cooling were identical (5° F. per minute). Curve 7 shows the transformation that occurred as a result of a 48-hour temper at 1200° F.; curve 6, 48 hours at 1100° F.; curve 5, no holding time at 1200° F.; curve 4, 48 hours at 1000° F.; curve 3, no holding time at 1100° F.; and curve 2, no holding time at 1000° F. Curve 1 shows the magnetization changes, common to all of the specimens, experienced during heating to the tempering temperatures.

It is to be noted that the retained austenite could be made to transform on heating if the temperature were high enough. The 2000° F.-quench eliminated the transformation on heating that would begin at 525° F. for a lower temperature quench; however, transformation resulted on heating to a temperature of 1140° F. The other treatments, with the exception of the temper with no hold at 1000° F. and 48-hour hold at 1200° F., resulted in a two-stage transformation of the austenite during cooling. One stage started below and near 700° F. and the other below and near 300° F. The austenite could be transformed in one stage during cooling either by

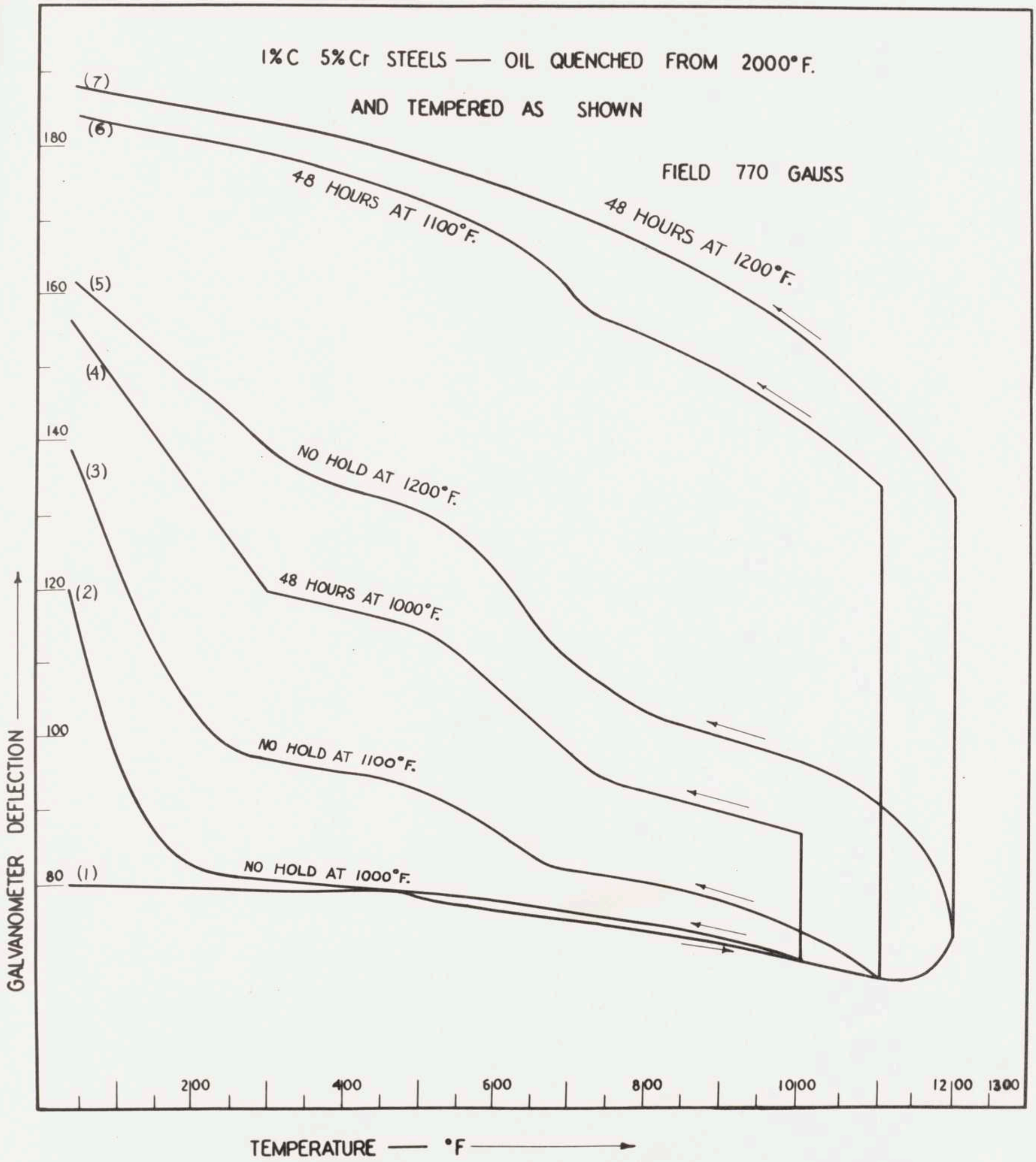


FIGURE 74. Effects of temperature and time at temperature.

using an elevated-temperature temper for a long time, or by using a lower-temperature temper for a very short time. In the former case, the transformation started at a temperature near 700° F., while in the latter, it started at a temperature near 300° F.

3. Effect of Rates of Heating-to and Cooling-from the Tempering Temperature

It is to be expected that the time of heating to any particular tempering temperature is of important concern to tempering reactions. It is, however, not usually expected that the time of cooling from the tempering temperature is of significance to these reactions.

The following curves point out the need of adopting a standard practice of heating and cooling for any series of experiments. In Figure 75 is shown the effect of varying the heating time upon the tempering reactions of specimens of 1% C - 5% Cr steel air-cooled from 1800° F. All measurements were made under a field of 1000 gauss. Curves 1' and 1 represent the changes in magnetization which resulted from a 5° F. per minute heating period, a two-hour hold at 1000° F. and a subsequent 5° F. per minute cooling period. Curve 2 shows the effect of a rapid heat (67° F. per minute) to the tempering temperature with the following treatments identical to those for the first specimen. The austenite transformation during heating was suppressed and transformation did not occur until the specimen was cooled below 700° F. Curve 3 shows the effect of a rapid heat (67° F. per minute), no holding time at the tempering temperature, and a rapid cool (17° F. per minute). This treatment suppressed the transformation so that it took place to a limited extent only upon cooling below 250° F.

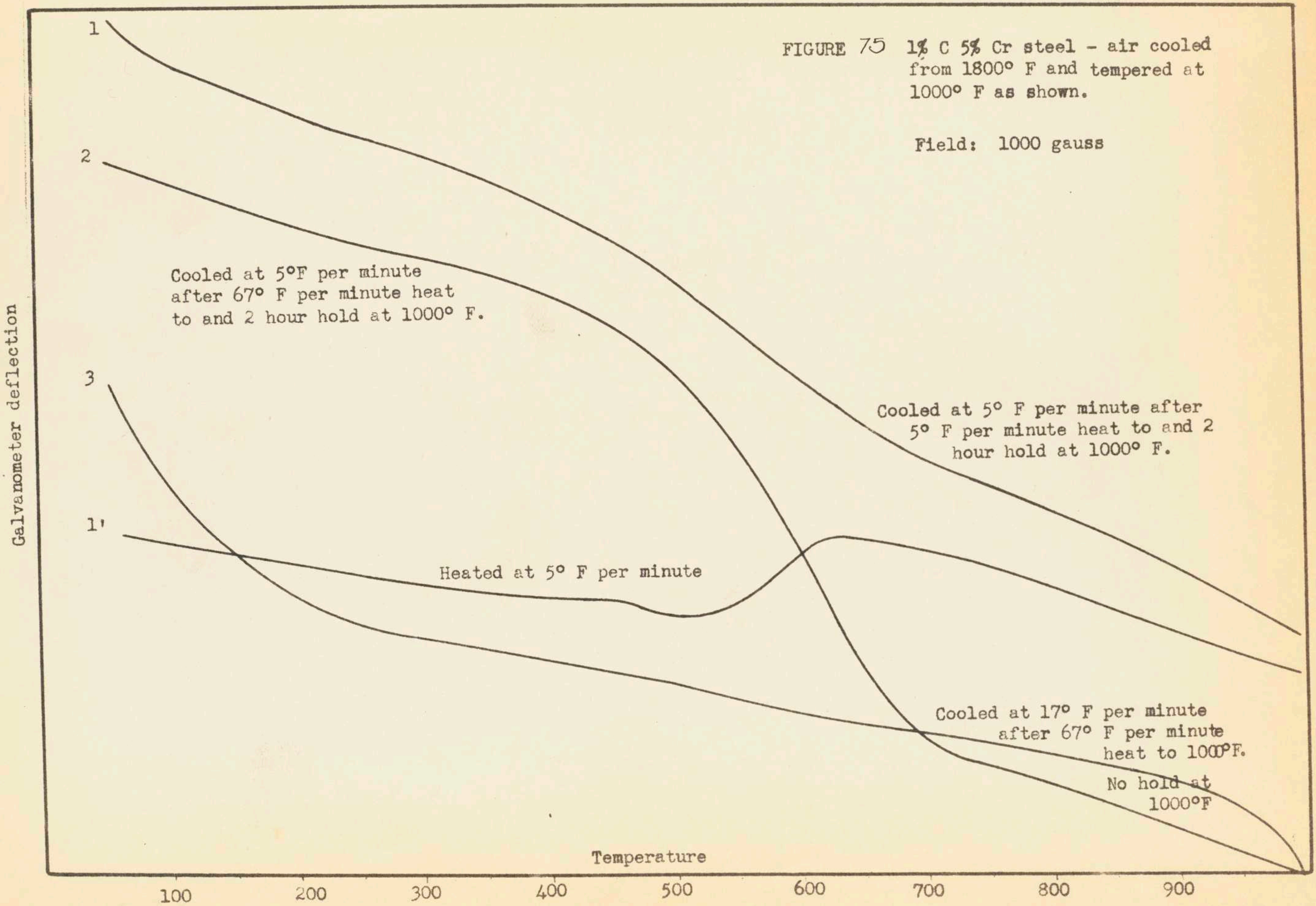


FIGURE 75 1% C 5% Cr steel - air cooled from 1800° F and tempered at 1000° F as shown.

Field: 1000 gauss

Figure 76 gives similar information for another treatment on specimens of the same steel--oil quenched from 2000° F. These measurements were made under a field of 770 gauss. Curve 1 shows the cooling of a specimen heated to 1000° F. at the rate of 5° F. per minute and held there for 48 hours. Curve 2 shows the same for a specimen treated similarly with the exception that the heating rate was 67° F. per minute. Although the 48 hours had equalized the reactivities of the two steels so that they underwent the transformations upon cooling at the same temperatures the total transformation was less in that specimen heated rapidly to the tempering temperature.

Curves 3, 4, and 5 show the effect of rate of cooling from the tempering temperature upon the transformation temperature. All three of the specimens (curves 3, 4, and 5) were heated rapidly (67° F. per minute) to the tempering temperature (1000° F.) and then cooled immediately to 400° F. at rates of 5, 15, and 30° F. per minute. The transformation temperatures were continuously lowered.

It is evident that for a series of tempering experiments from which relative values are desired the heating and cooling rates must be maintained at a constant value.

4. Effect of the Rate of Cooling from the Hardening Temperature

It is now a generally accepted principle that cooling rates exceeding the critical result in less retained austenite because of the effect of stresses. This is well born out by the Table VI showing the relation

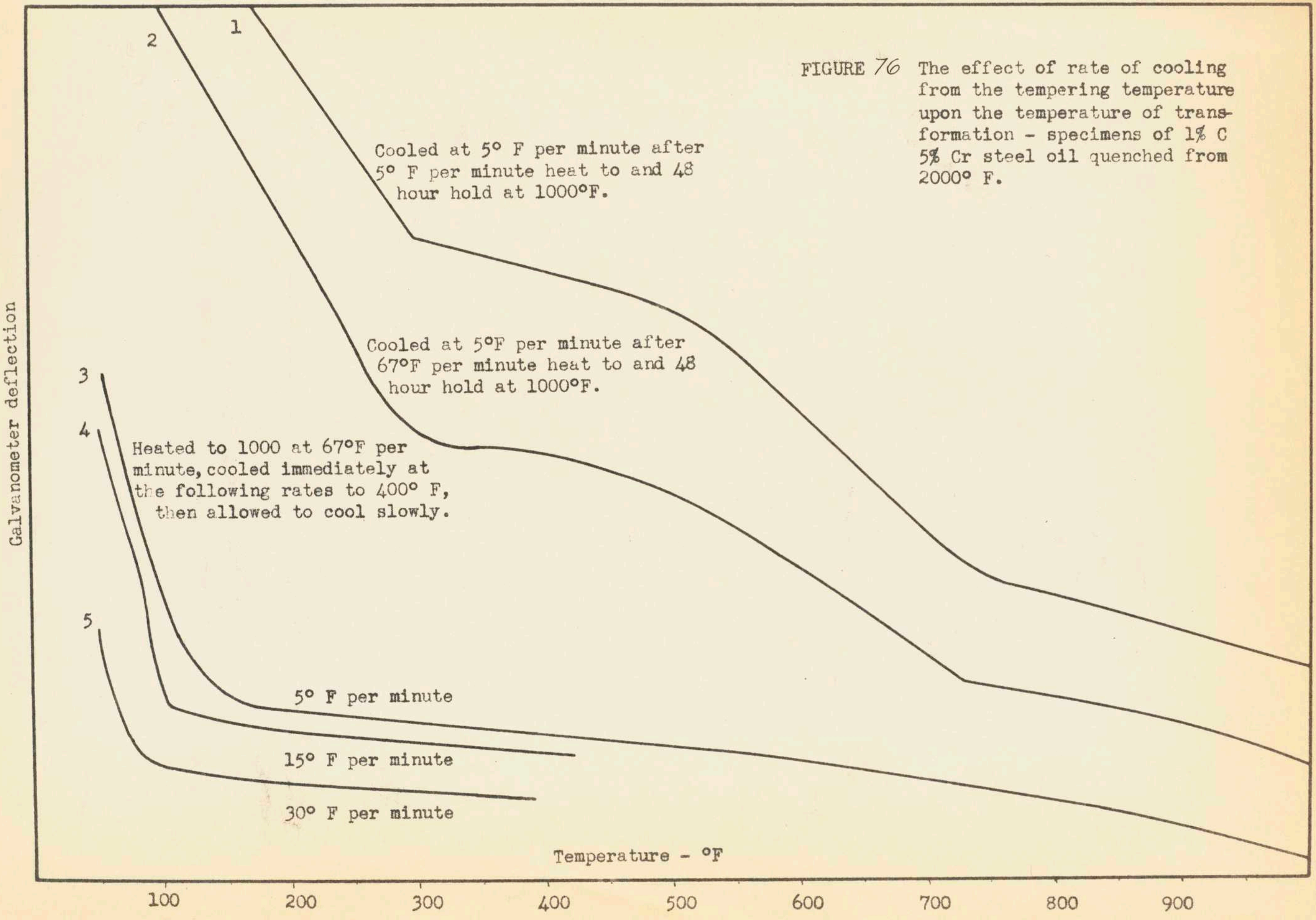


FIGURE 76 The effect of rate of cooling from the tempering temperature upon the temperature of transformation - specimens of 1% C 5% Cr steel oil quenched from 2000° F.

TABLE VI

EFFECT OF THE RATE OF COOLING FROM THE HARDENING TEMPERATURE ON THE AMOUNT OF RETAINED AUSTENITE

Steel - 1% C - 5% Cr

Quenching Temperature - 1800° F.

Time at Temperature - 1/2 hour

Applied Field - 1000 gauss

Cooling Medium	Per Cent Retained Austenite
Furnace cool	0
1800° - 1300° Furnace - 950° Furnace - Lime	5
Water	14
Oil	18
Air Blast	23
Still Air	24
Lime	26.9
1800° - 950° Furnace - Lime	27

between the amount of retained austenite and rate of cooling from 1800° F. The amount of retained austenite increased with decrease in the cooling velocity, up to a limit slightly less than the rate of lime cooling.

The curves in Figures 77, 78, and 79 also illustrate this principle. Figure 77 shows the changes in magnetization (field of 265 gauss) with temperature, up to 775° F., for specimens heat treated from 1800° F. Curves 1 and 2 are heating (magnetization) curves for specimens of 1% C - 5% Cr steel cooled in lime and in brine. Curves 3 and 4 (shown as broken lines) are cooling curves corresponding to curves 1 and 2. The specimens were cooled immediately after they had reached 775° F.

Figure 78 also shows the changes in magnetization (fields of 265 and 1000 gauss) with temperature, up to 1000° F., for specimens heat treated from 1800° F. Curves 1 and 1', 2 and 2', and 3 and 3' are heating (magnetization) curves of specimens cooled in lime, in air, and in water, respectively (the primes designate curves measured under a field of 1000 gauss). Curves 4 and 4', 5 and 5', and 6 and 6' are cooling (magnetization) curves corresponding to curves 1 and 1', 2 and 2', and 3 and 3'.

Figure 79 shows the changes in magnetization (fields of 265 and 1000 gauss) with temperature up to 1000° F. for specimens heat treated from 1700° F. Curves 1 and 1', 2 and 2', and 3 and 3' represent air-, water-, and lime-cooled specimens, respectively. Curves 3 and 3', 4 and 4' and 5 and 5' are cooling curves corresponding to curves 1 and 1', 2 and 2' and 3 and 3'.

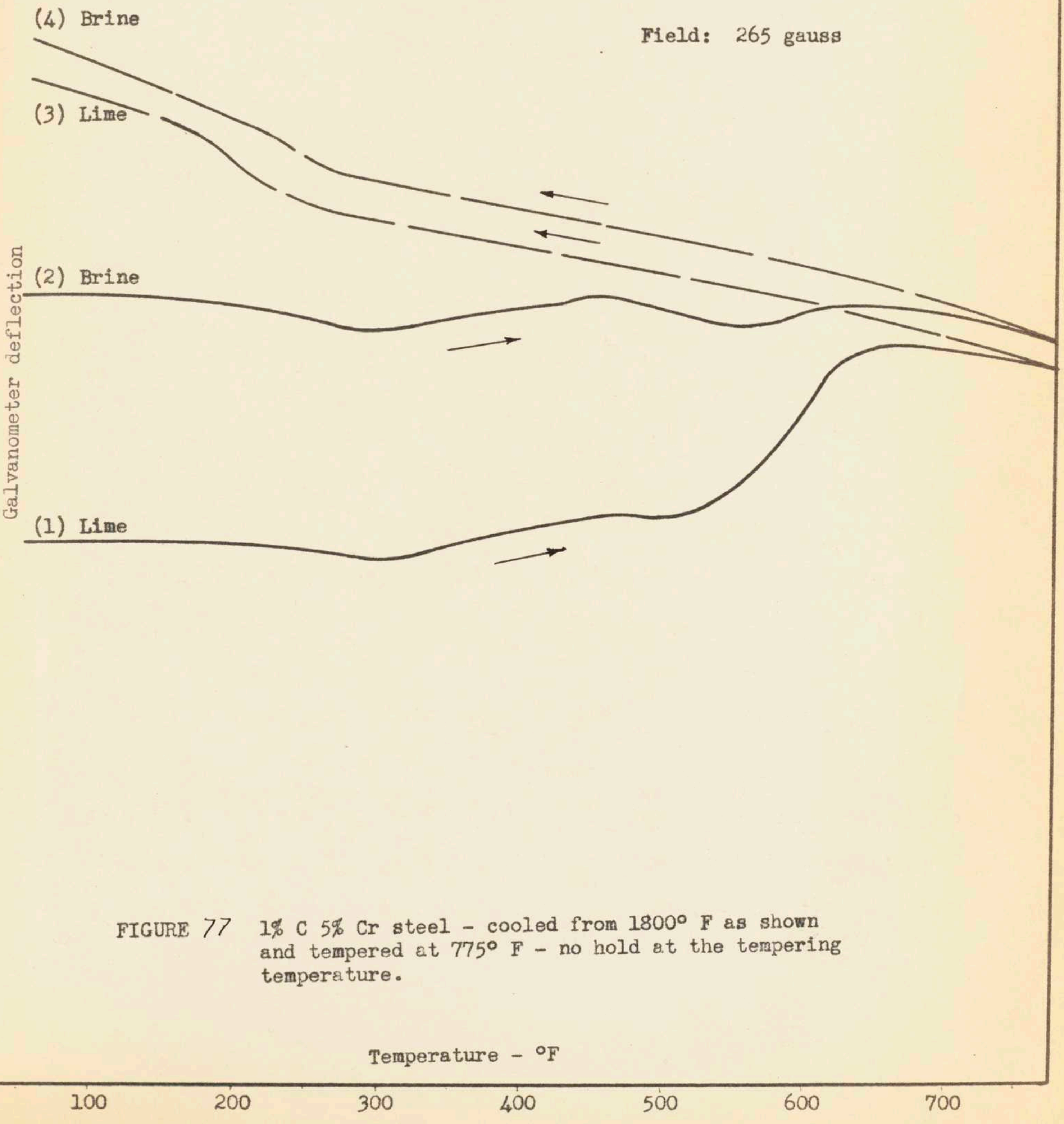


FIGURE 77 1% C 5% Cr steel - cooled from 1800° F as shown and tempered at 775° F - no hold at the tempering temperature.

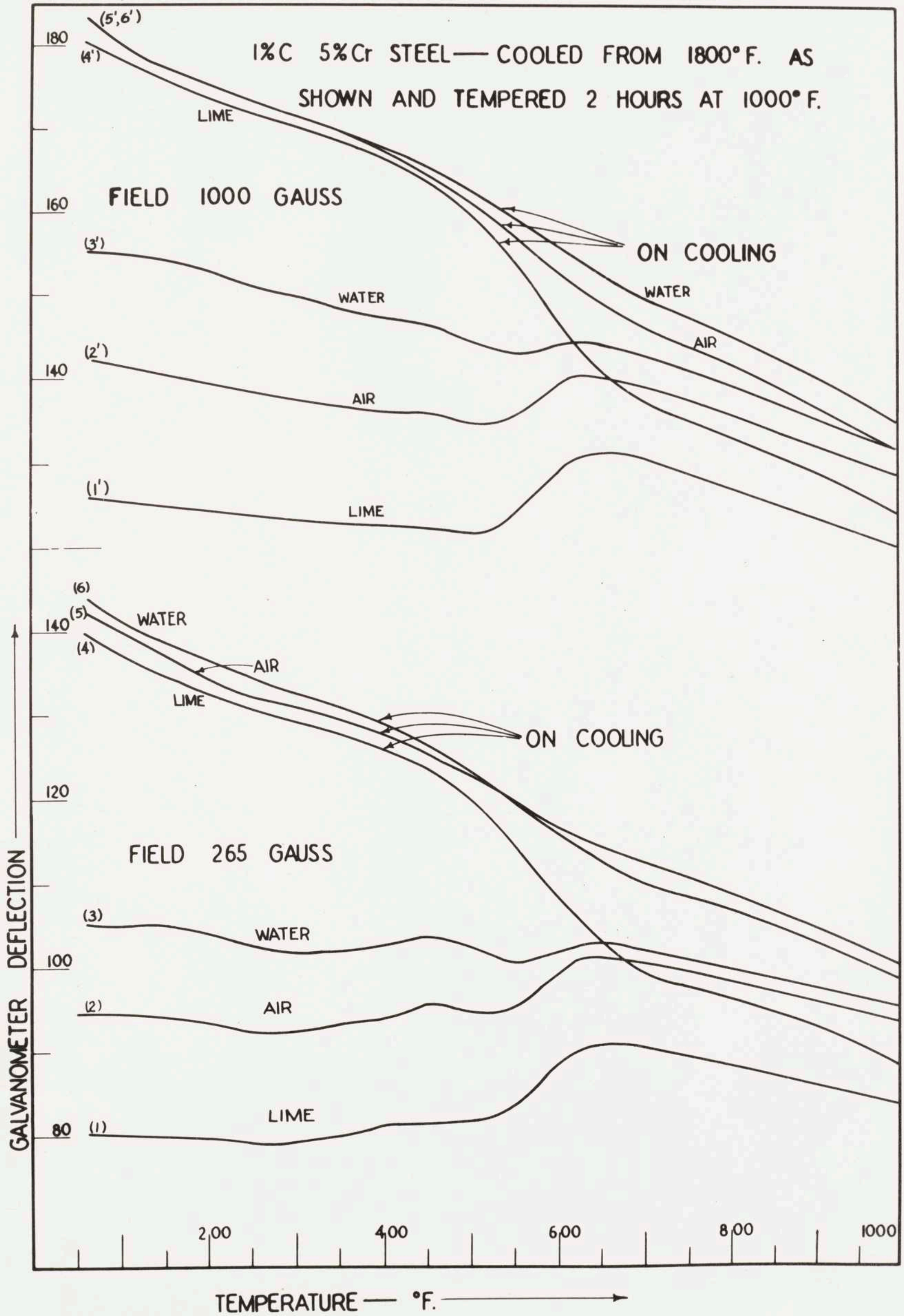


FIGURE 78. Heating and cooling (magnetization) curves.

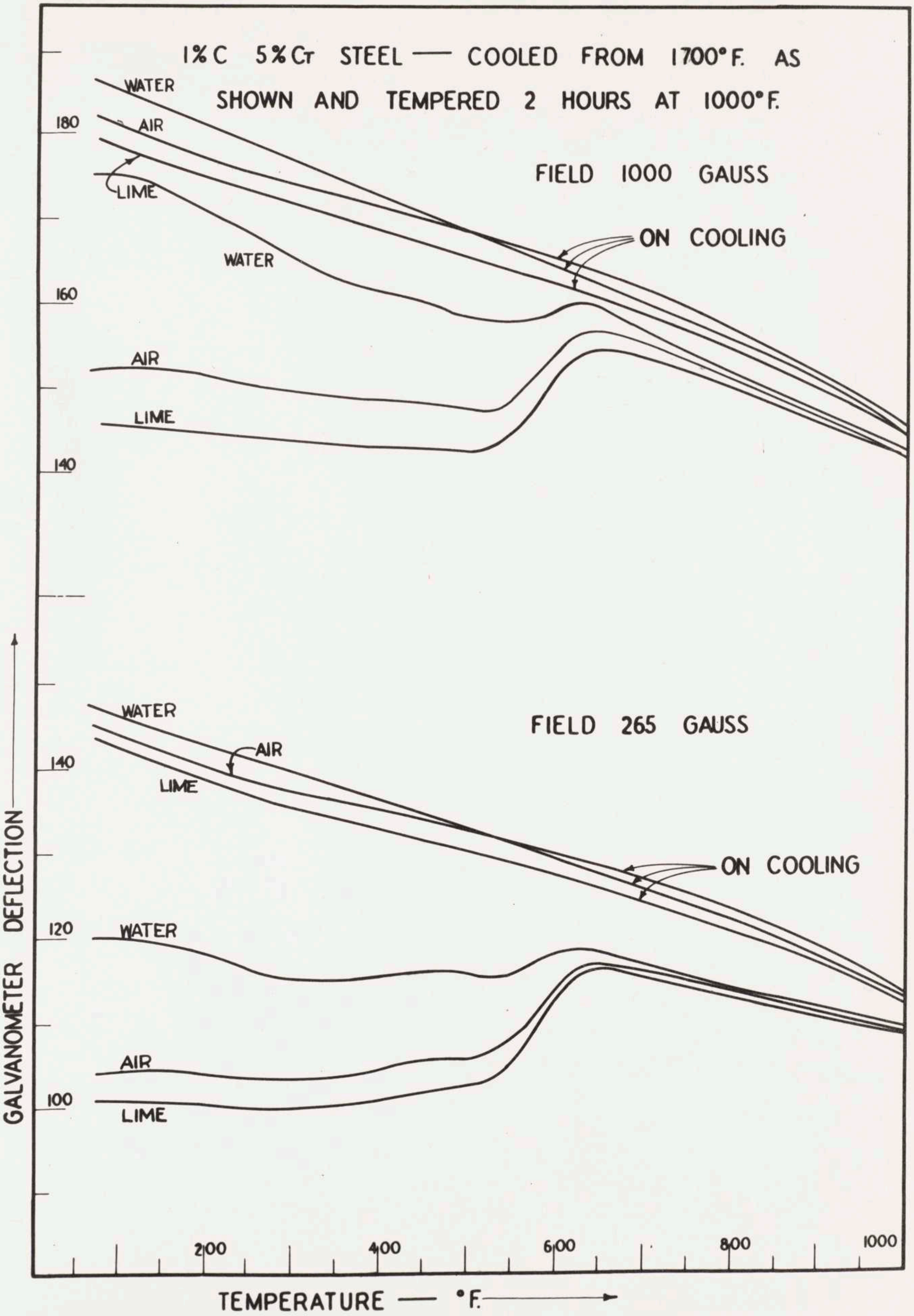


FIGURE 79 Heating and cooling (magnetization) curves.

In Figure 80 are shown the heating (dilation) curves of specimens of 1% C - 5% Cr steel cooled from 1800° F. in water, in air, and in lime.

From the figures given in this section the following facts may be obtained:

a. The amount of austenite retained in specimens hardened from 1700° F. and from 1800° F. was increased with a decrease in the rate of cooling from the hardening temperature.

b. The more slowly cooled the specimen the greater the amount of austenite that transformed during heating-to and cooling-from the tempering temperature.

c. The retained austenite in the specimens tempered to 775° F. without any hold at temperature transformed only slightly during cooling below 250° F. The heating period of 2 hours at 1000° F. resulted in transformation of considerably greater extent during cooling below 700° F.

d. The heating (dilation) curves of the specimens cooled in water, in air, and in lime showed two contractions and one expansion.

e. The contractions in the first range, 275 - 350° F. were more pronounced the more vigorous was the method of cooling.

f. The contractions in the second range 450 - 550° F., were practically equal in extent.

g. The expansions, in the range 550 - 650° F., were more pronounced the slower the method of cooling.

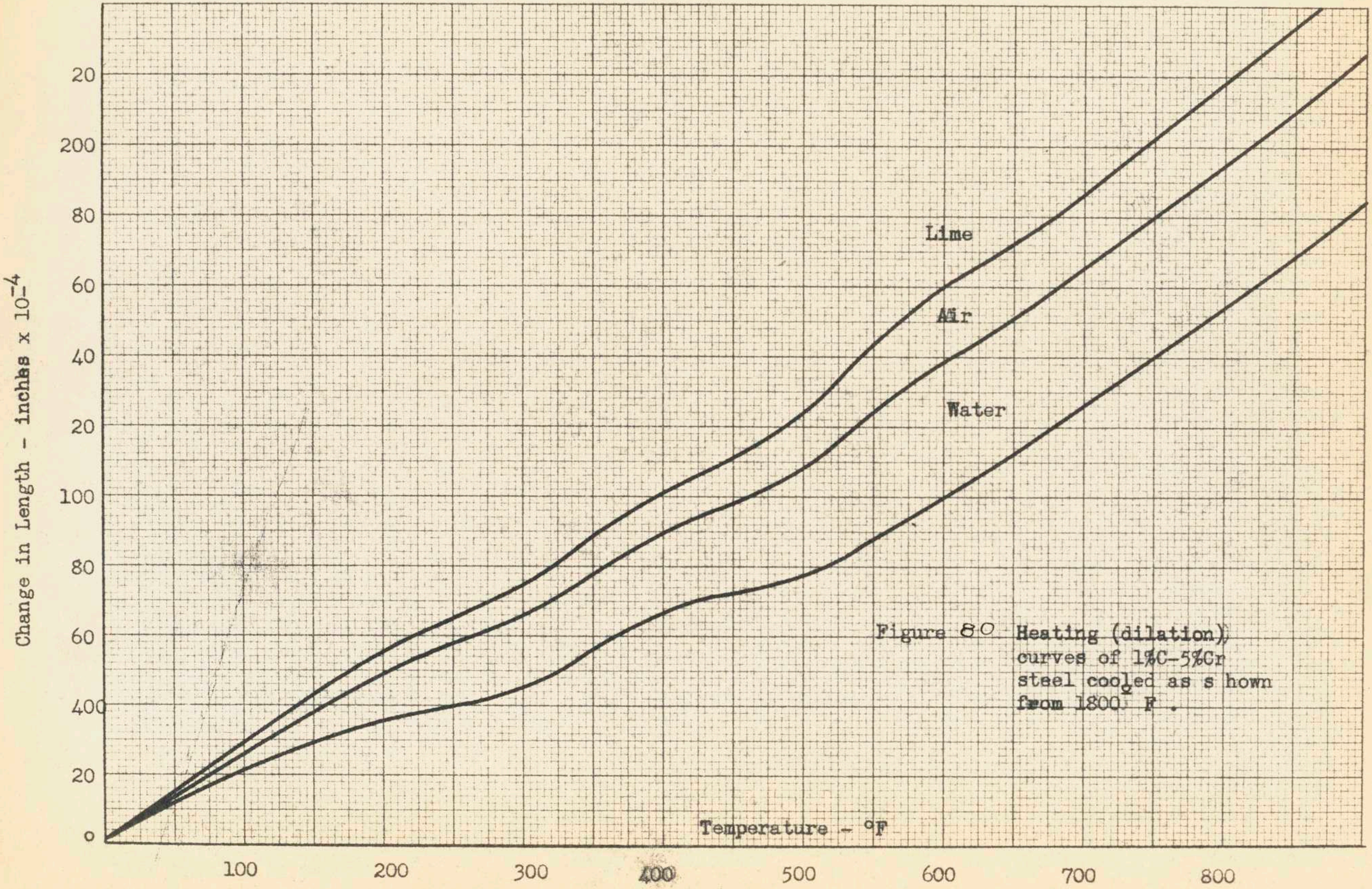


Figure 80 Heating (dilation) curves of 1%C-5%Cr steel cooled as shown from 1800° F .

5. The Effect of Stress on Magnetism

Virtually all hardening treatments of metals will produce residual stresses. The quench-hardening of steels produces residual stresses of two kinds: macro and micro. The macro stresses come under the category of stresses produced by inequality in cooling rate across the cross section or length of a specimen. The micro stresses are produced by the formation of martensite from austenite; thus the interfaces of the austenite and martensite regions are localities of high-stress content. The integrated stress in a quenched steel has been resolved into components in the longitudinal, tangential and radial directions by Bühler, Buchlotz, and Schulz(34).

Accordingly, any study of tempering must necessarily be concerned with the phenomenon of stress relief, especially so when that study relates to the kinetics of tempering.

It is well known that the magnetic properties of a metal are affected by the state of stress existant in that metal. This effect is most noticeable at the lower field strengths.

At saturation the effect of stress is lost. This has been shown to be true for stresses produced by cold working by Messkin(26) who measured the saturation induction of cold-worked 0.78 per cent carbon steel, and by Buehl, Hollomon, and Wulff(88), whose measurements will be discussed below. From the literature surveyed, it can be said that no proof of the stress independency of magnetization at high-field strengths for steels hardened by quenching has been given.

According to Buehl, Hollomon and Wulff(88) the presence of strain can be ascertained by a plot of magnetization against $\frac{1}{H^2}$. The extrapolation to infinite field should give the same magnetization for specimens which differ only in their strain content. They applied this method in their investigation of cold-worked stainless steel. The increase in magnetization produced by a holding period near 600° F. was found to be due to relief of internal strain.

An investigation was made of the relief of stress in hardened specimens of the 1% C - 5% Cr steel (Figure 81). The specimens were oil-quenched from 1800° F. The magnetic study of the preceding section, Figures 78 and 79, revealed two distinct trends of interest to this particular discussion. At both high and low fields (1000 and 265 gauss, respectively) there was a general drop in magnetization with temperature up to 300° F. From 300° F. the magnetization remained constant at high field and increased at low field. This behavior suggested that stress relief occurred in the range, 300 - 450° F.

One specimen was then tempered to 300° F. and one was tempered to 450° F.; both were not held at the draw, and were heated and cooled at a rate of 5° F. per minute. The field-magnetization relation was determined for each specimen, and then from this data, plots were made of magnetization against $\frac{1}{H^2}$ (Figure 82). Although the divergence of the two curves at low fields was rectified at the higher fields, the curvature made extrapolation to infinite field difficult. A repetition of the plot against $\frac{1}{H}$ (Figure 82) gave better results. The main point, however, is that the difference in magnetization of the two specimens which was marked at low fields disappeared at high fields, thus proving that the difference in the specimens was their stress content. In other words,

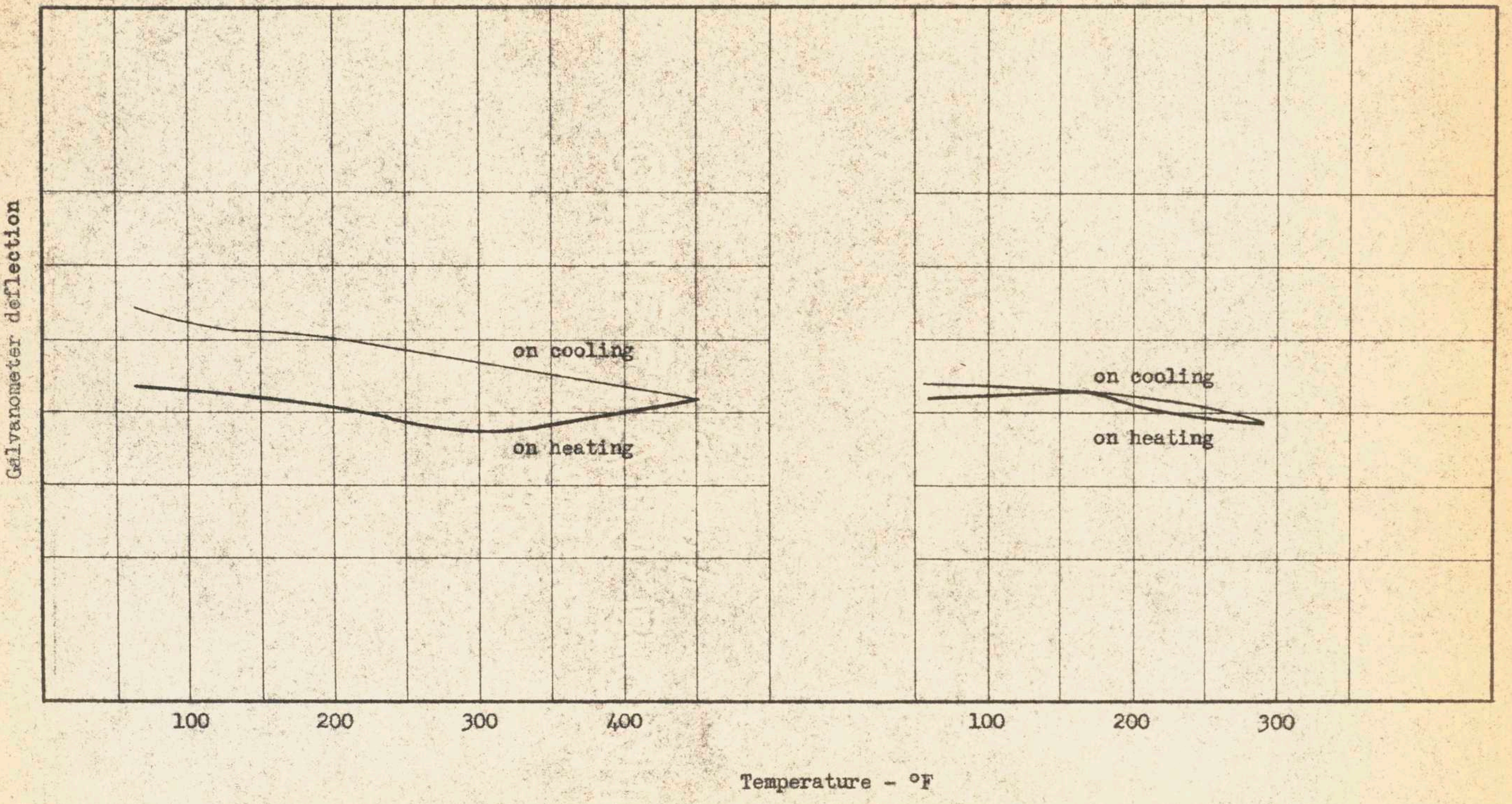
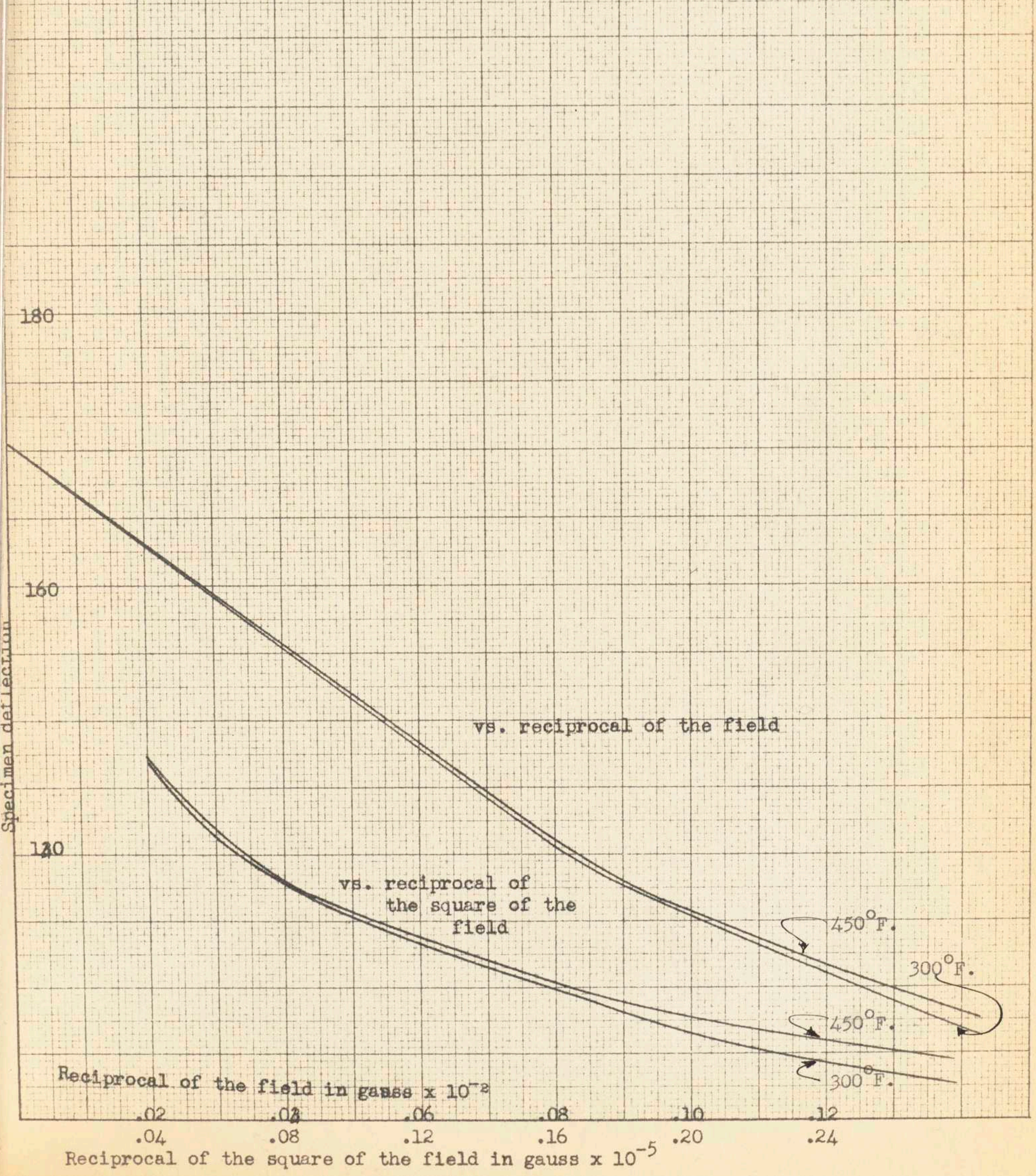


FIGURE 8/ 1% C 5% Cr steel oil quenched from 1800° F and tempered as shown.

Field- 265 gauss.

FIGURE 82 Magnetization extrapolation curves for 1%C 5%Cr steel oil quenched from 1800 °F and tempered as shown.



the increase in magnetization noticed at low fields upon tempering a hardened specimen of 1% C - 5% Cr in the range, 300 - 450° F., was due to stress relief. This was later shown to be true for the 1.5% C - 11.5% Cr steel.

The coercivity changes in these two specimens were also of bearing to this question. In the as-quenched state the coercivity was 60.5 gauss. The temper at 300° F. elevated this to 64; but, the specimen tempered at 450° F. only had a coercivity of 48.5. These data coincided with the proposition that stress relief occurred between 300 and 450° F. The property of coercivity is, among other things, a measure of the strain in a metal. It should be expected then that the stress-relieved specimen would have the lower coercive force; the above data verifyed this completely.

The question of the interpretation of coercivity is, indeed, a moot one. Many attempts have been made to relate it to mechanical hardness, but the discrepancies are numerous.(72)(58)(41) Dean and Clayton(74) have claimed that there is a complete lack of parallelism between the two; they proposed that coercive force was a function of the internal surface. The increase of coercivity with grain size has been reported for electrolytic iron sheet by Dahl, Pawlek and Pfaffenberger(59). Köster(23) proposed a relation between the coercivity of tempered steels and the carbide particle size. Regardless of the relation between mechanical hardness and coercivity, the introduction of strain which produces increases in mechanical hardness also produces increases in coercivity. Liwshitz(42) found an increase in the coercivity of steel wire with an increase in the extent of cold-drawing. This relation was also found by Messkin(26) when the strain was introduced by cold rolling. One point that is above contention, however, is that for any given steel, its coercivity is a direct function of the stress content, other factors being eliminated.

Two objections may be raised, however, to the conclusion that the rise in magnetization intensity of a quenched specimen with temperature in the range from 300° F. to 450° F. was a result of stress relief. One possible objection that could be raised is that this rise is similar to the phenomenon of the rise in magnetization immediately preceding a curie point observed at low field strengths by Honda, Masumoto, and Kaya(16), and Dussler(14). This objection may be considered untenable when the fact is recognized that these changes were irreversible; that is, the steel has undergone a permanent change upon the experience of passing through the range 300 - 400° F. subsequent to a quenching treatment (See Figure 80).

The other possible objection is that this rise in magnetization in the range 300° F. to 450° F. is due to agglomeration of carbides previously precipitated from the martensite. This process should, however, result in a softening of the steel. This was not observed. The hardness was found to be constant at 58 to 59 Rockwell C on tempering in this range.

The above evidence seems quite convincing that the phenomenon of a rise in magnetization intensity with temperature at low field strength in the range 300 - 400° F. is due to stress relief.

An experiment was conducted on a hardened plain-carbon steel. The results are shown in Figure 83. The steel contained 1.25 per cent carbon

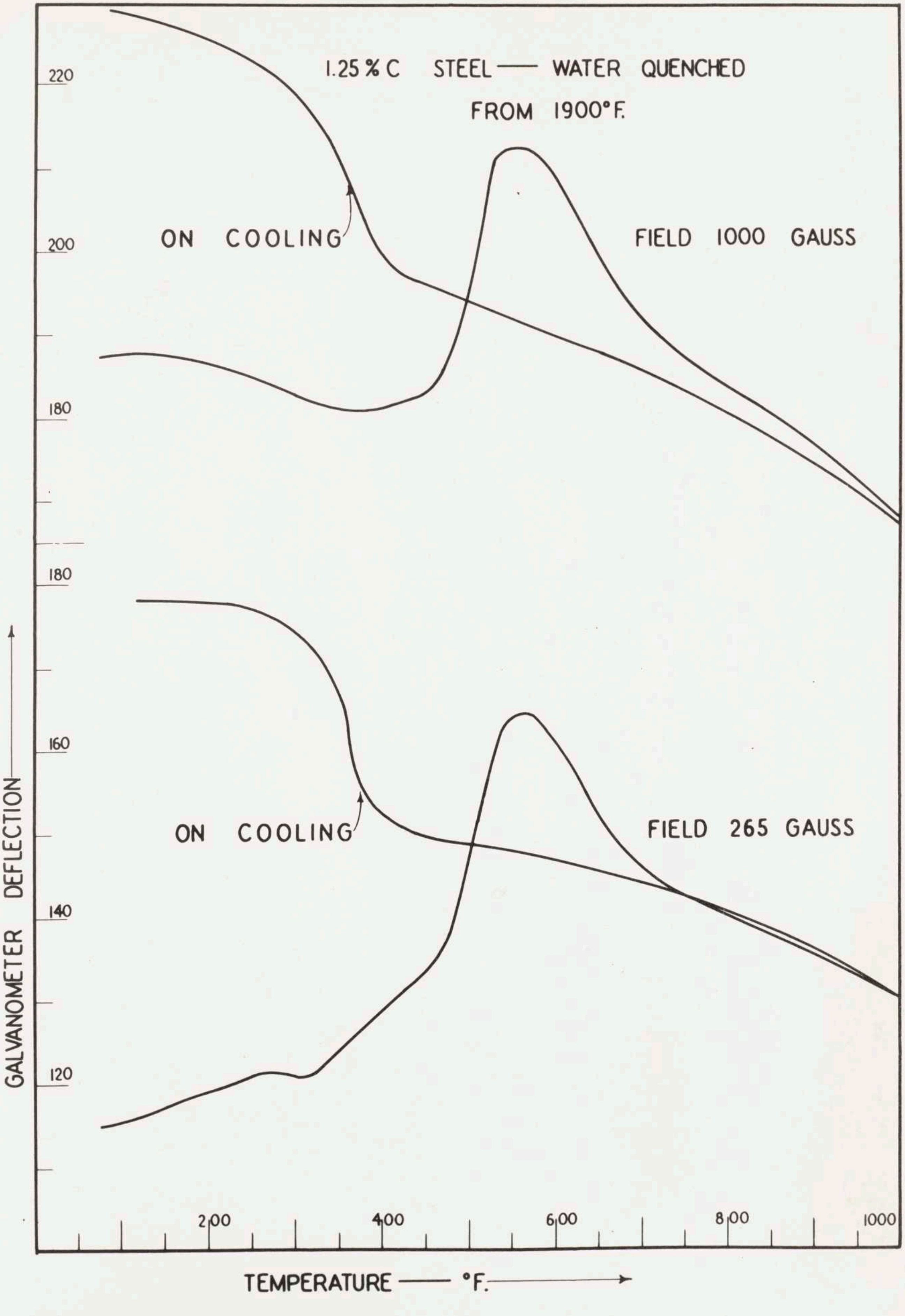


FIGURE 83. Magnetization curves.

and was water-quenched from 1900° F. Measurements were made of magnetization versus temperature during the heating-to and cooling-from 1000° F. at the rate of 5° F. per minute). One series of measurements were made under an applied field of 265 gauss. The lower field curves should be compared with those of Mikami given in Figure 5, Chapter III - C.

The portion of these curves that is believed produced by stress relief is the second rise in the low-field curve. This view has also been adopted by Ellinger(31).

In order to find out if the relief of stress produced by cold-working occurred in the same temperature range as did the relief of stress produced by martensite-hardening, the following experiment was run. A specimen of Wemco iron was cold-swaged to a reduction of 56 per cent in area. Magnetization versus temperature curves were made at fields of 265 and of 1000 gauss. The low-field curve was a straight line and the high-field curve showed a gradually increasing drop. These results showed that the relief of stress produced by cold-working does not occur in the same temperature range as does the relief of stress produced by martensite-hardening.

6. Effect of Carbide Precipitation

In all the heating (magnetization) curves, except those that represented specimens containing no martensite, a change in slope started at 450° F. and was completed at 550° F. In this same range the heating (dilation) curves showed a contraction. Henceforth, both the change in slope of the magnetization curve and the contraction of the dilatometer curve shall be referred to as breaks.

The question has arisen as to whether or not these phenomena are attendant to a curie point. The factors in favor of a curie point are:

a. The (magnetic) break occurred in both steels and for all quenches, which did not give 100 per cent austenite, in the same temperature range.

b. The phenomenon in question was followed by austenite transformation in the specimens of 1% C - 5% Cr steel hardened from 1700 and 1800° F., but was not followed by austenite transformation in specimens of the same steel quenched from higher temperatures nor in any specimen of the 1.5% C - 11.5% Cr steel.

The factors which are not in favor of a curie point are:

a. Coincident with breaks in the magnetization curves were breaks in the dilatometer curves which represented considerable contractions.

b. The breaks in the dilatometer curves of plain carbon steels at the curie point of the carbide showed slight expansions.

c. No breaks occurred in the two annealed steels of this investigation.

An experiment was conducted to settle this question by find out if the process was reversible. The results are shown in Figure 84.

A specimen was quenched in oil from 2000° F. and was then tempered at 600° F. Starting at 500° F., the magnetization curve showed a drop, the intensity of which increased to 550° F. and then attained a constant value. The dilatometer curve showed that a contraction existed simultaneously.

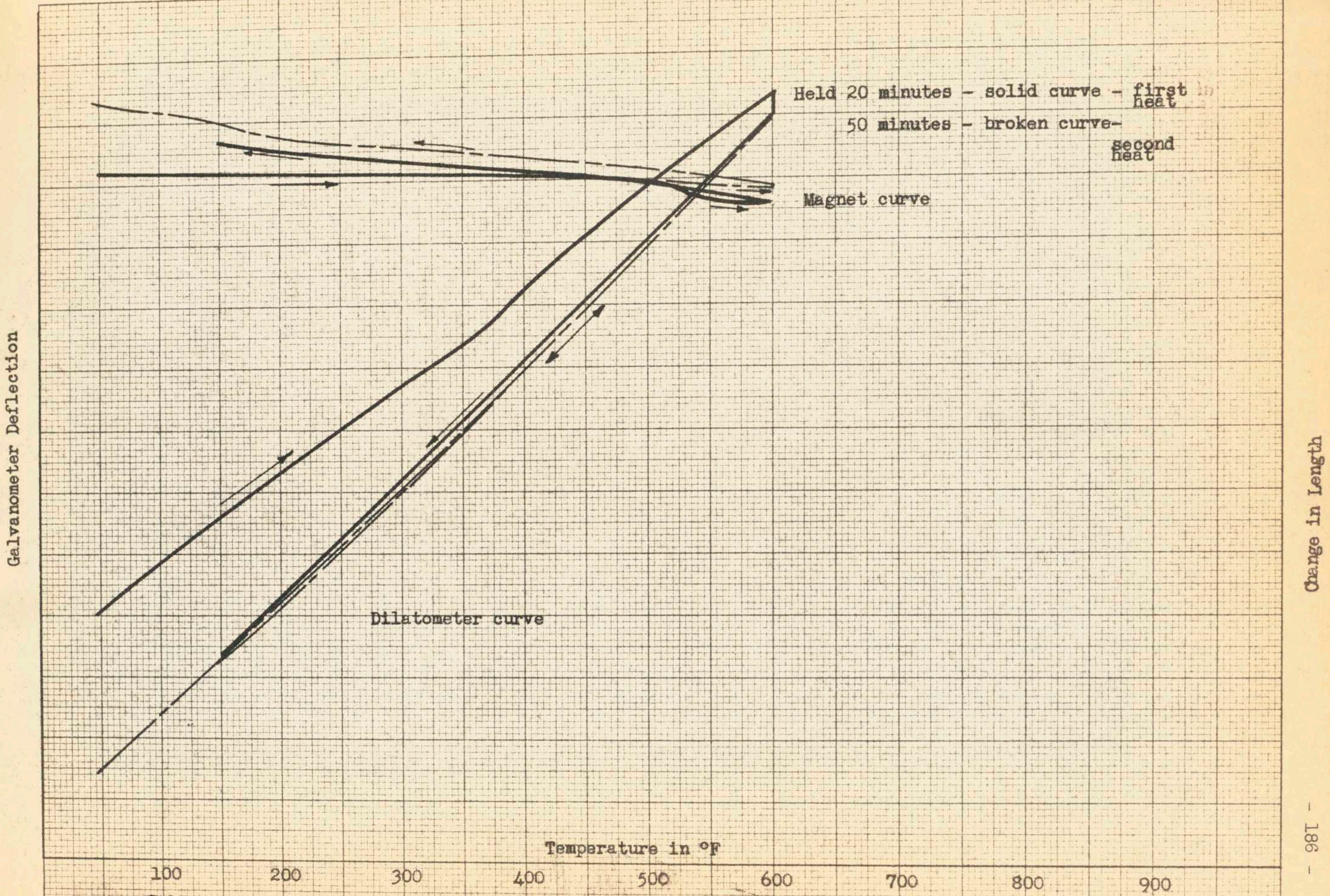


FIGURE 84 1%C 5%Cr steel oil quenched from 2000 °F and tempered as shown at 600 °F.

Holding at 600° F. for 20 minutes resulted in continued contraction and a slight increase in the magnetization. Upon cooling, no sharp increase was noticed in the magnetization in the temperature range wherein a sharp drop had been noticed during the first heating. The second heating and cooling curves showed no change in this range.

This means that the break noticed during the first heating was either due to a curie transformation or that if it was a curie point the carbide exhibiting it was decomposed by the heating period at 600° F. Honda and Murakami(2) have shown that carbide decomposition may occur. However, the temperatures necessary to effect this decomposition were of the order of 1300° F., much higher than the temperatures considered here. The large contraction that attended the process, however, was the most forceful proof that a curie point was not present.

The process that will explain all these facts is carbide precipitation. The only disturbing fact is that the first cooling (magnetization) curve of Figure 84 comes back above, and not below the first heating curve. This may be explained, however, as being the result of a change in the temperature-coefficient of the steel. The curves in Figure 85 show that a change in the temperature-coefficient can be produced by merely heating to 425° F., which was below the carbide-precipitation range.

It is believed, then, that the process may be described as follows: Carbide containing some iron, as well as molybdenum and chromium, precipitated from the martensite. The drop in the magnetization was caused by the loss of iron. The temperature coefficient of the new state of the specimen was greater than that of the unprecipitated state. Thus, the curve on cooling comes back above the heating curve.

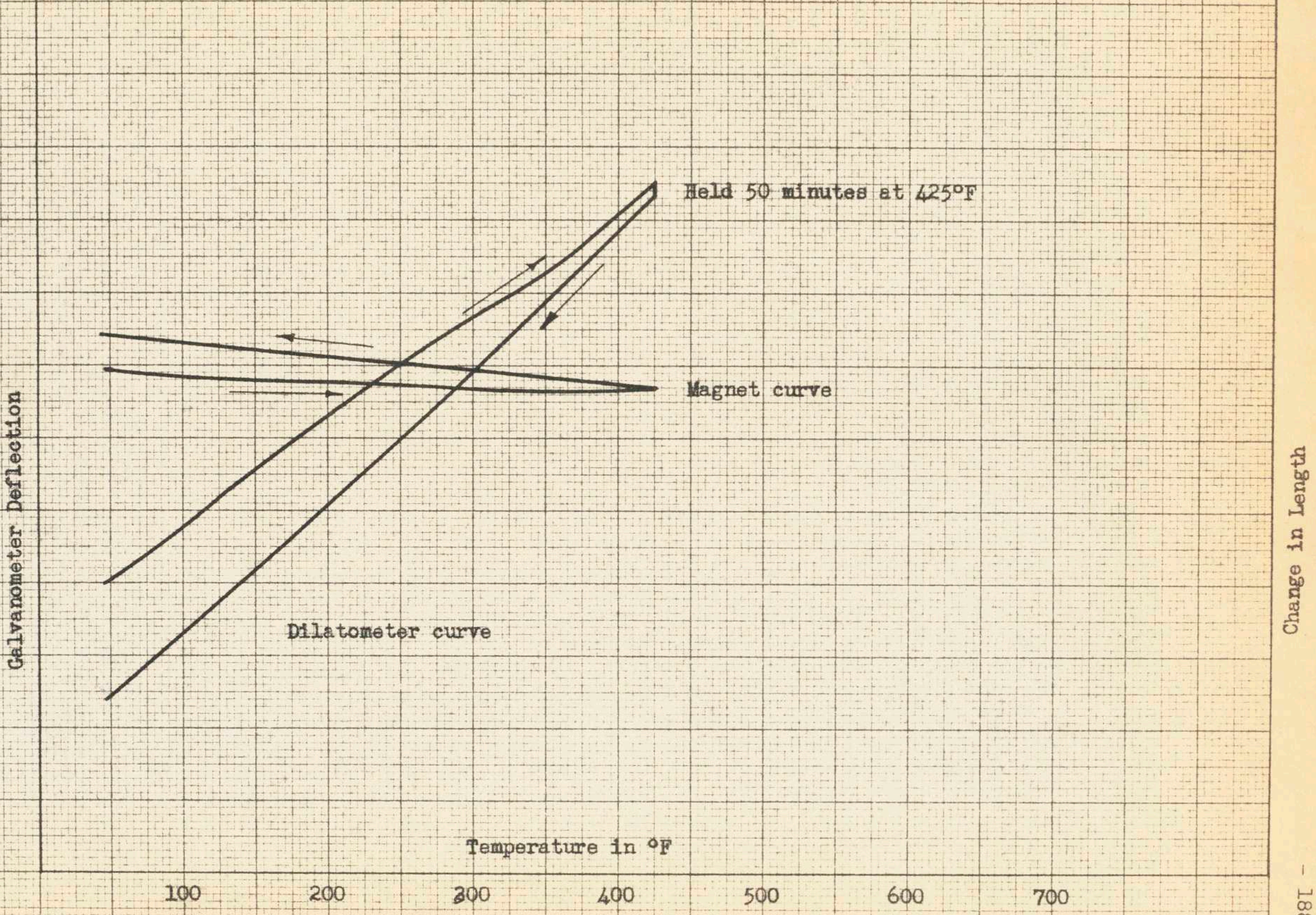


FIGURE 85 1% C 5% Cr steel oil quenched from 2000 °F and tempered at 425 °F.

7. Multiple Tempering

The heating (magnetization) curves of Figure 66 (Chapter VII) show the retained austenite in specimens of 1% C - 5% Cr steel oil-quenched from 2000° F. did not transform at 600° F. during heating at the rate of 5° F. per minute. Figure 74 (Chapter VII) shows that the retained austenite in these specimens will transform after being heated to 1000° F. and immediately cooled. The austenite will transform during the cooling into tetragonal martensite. The austenite left in the steel has been to some extent reactivated; for it will not start to transform at room temperature. Room temperature transformation could not be found in the untempered specimens.

It was thought that the active austenite that remained after the first temper would now transform, during heating, at 600° F., as did the austenite in specimens hardened from 1700° F. and 1800° F. The results of three temperings to 1000° F. with no hold at temperature on a specimen of 1% C - 5% Cr steel oil-quenched from 2000° F. are shown in Figure 86.

It can be seen that the activity of room-temperature-transformable austenite increased with the number of temperings. The expected transformation during heating, however, did not take place.

Several other important observations may be made from these curves. First, the three successive heating (dilation) curves all showed two contractions during heating, but were of diminished intensity in each heating. It has been proved by the X-ray study that the austenite transformed below 250° F. into tetragonal martensite. It can be further said that

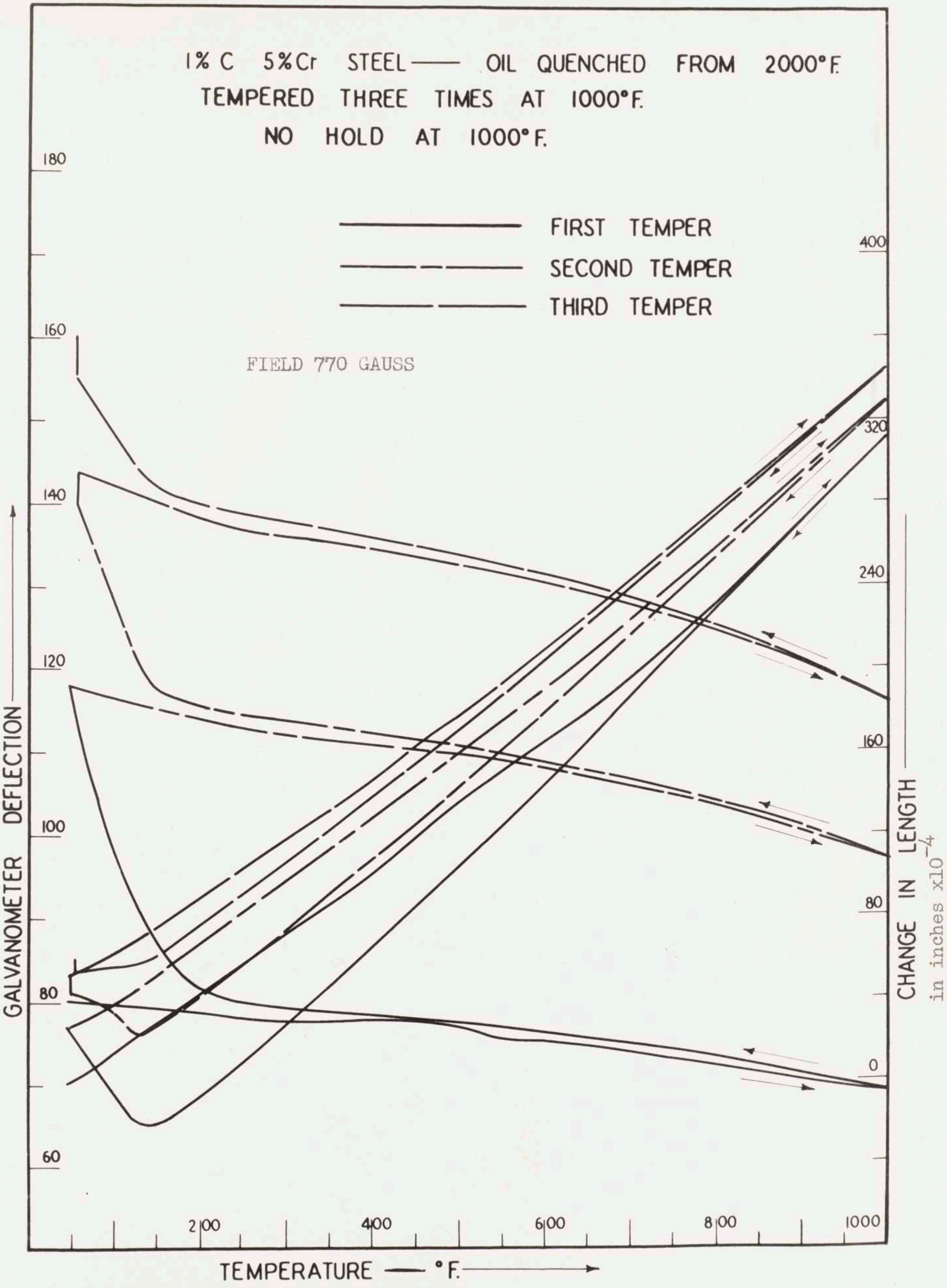


FIGURE 86. Magneto-Dilatometer curves.

martensite will form from the austenite that transforms on each tempering-cooling providing that the transformation temperature is below 250° F. This is proved by the existence of the two contractions mentioned above, the first for tetragonal-martensite tempering and the second for precipitation of carbide from the cubic martensite.

Second, the temperature of the austenite transformation decreased as the number of temperings increased.

An experiment conducted (notshown in thesis) on a specimen of the 1.5% C - 11.5% Cr steel quenched so as to be composed of only austenite and carbide gave results which substantiated the above conclusions. After some of the austenite was transformed, no increase in reactivity during heating was noted. The second heating (magnetization) curve showed the drop at 450° - 550° F. and the second heating (dilation) curve showed the contractions at 250° - 375° F. and at 450° - 550° F., whereas the first heating curves were straight lines. This all seems to prove that martensite was responsible for the phenomena of the heating curves.

While a certain extent of transformation could be produced by either a long hold or by numerous short holds at temperature, the resultant effects were different. That is, the time of holding at a temperature above 900° F. could be chosen so that almost all the austenite transformed during cooling into martensite. By numerous heatings and coolings the austenite could be transformed to the same extent as before, but all the martensite, except the last to form, would be tempered and stress relieved.

C. X-ray Results

1. Determination of the Amount of Retained Austenite

The Gardner method was applied to the steels of this investigation with the hope that through the data obtained, the field dependency of the magnetic method for the determination of the retained-austenite content could be evaluated.

Specimens of both steels were used. The X-ray specimens and the magnetometer specimens were hardened together at each temperature. All heat treating was done under vacuum.

The results obtainedfrom the X-ray and the magnetometer study agreed in general but not in detail. Most important of all, the ratio for the 100 per cent austenite state could not be obtained. The X-ray method gave different amounts of retained austenite for different portions of the specimen. The magnetic method is an integrating one and is affected by the austenite in all parts of the specimen. Typical results are shown in Figures 87 and 88.

Although exact values could not be obtained, the results of the X-ray study substantiated the results of the magnetic study. The amount of austenite was found to increase in both steels with increase in hardening temperature. Beyond 2000° F., however, the 1% C - 5% Cr steels were found to contain decreasing amounts of austenite; the 1.5% C - 11.5% Cr steels to increase in austenite content. Below 2000° F. the slower the cool the more austenite retained; above 2000° F. the relation did not hold.

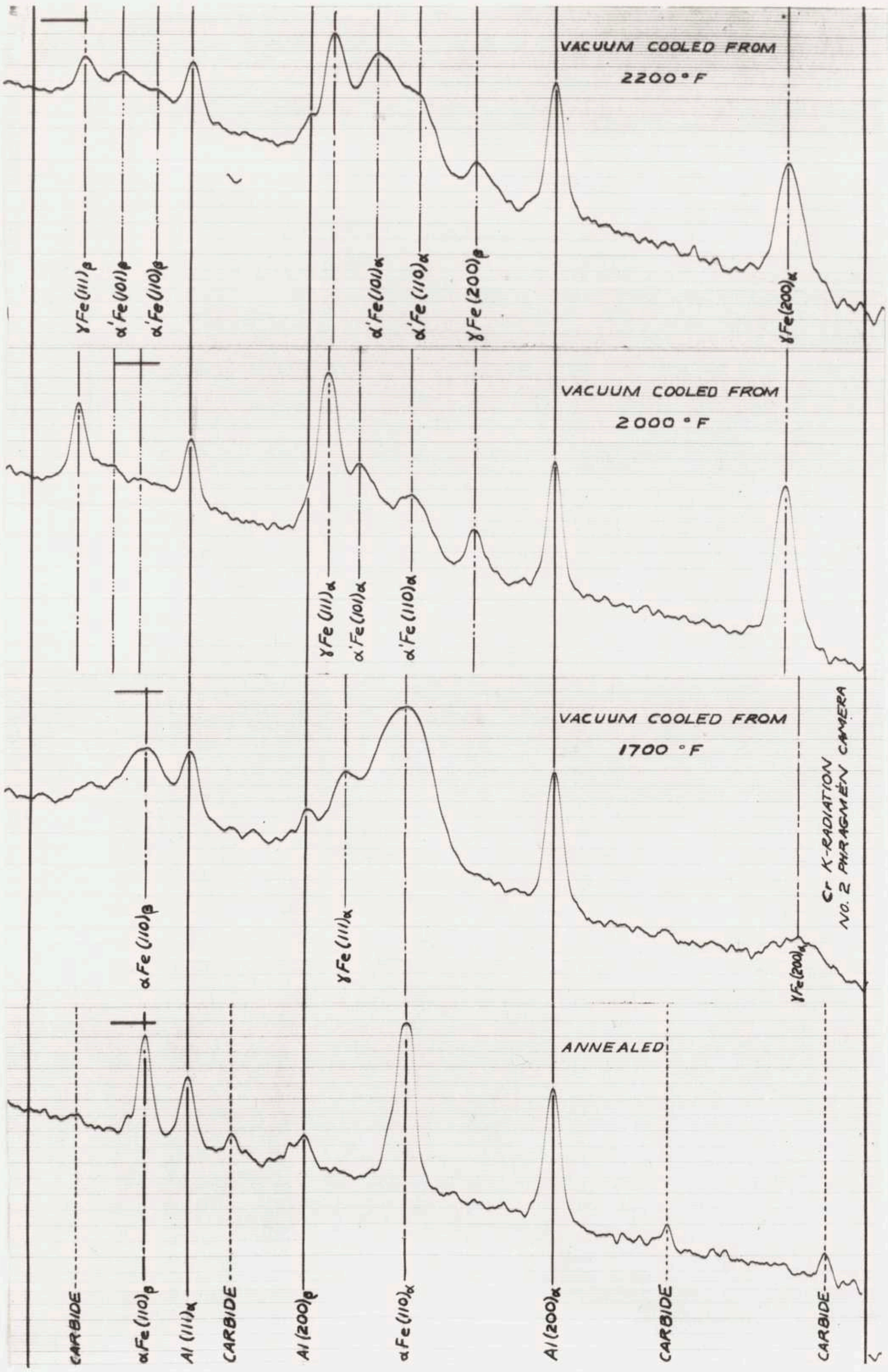


FIGURE 87. MICROPHOTOMETER TRACES OF 1% C 5% Cr STEEL HEAT TREATED AS SHOWN.

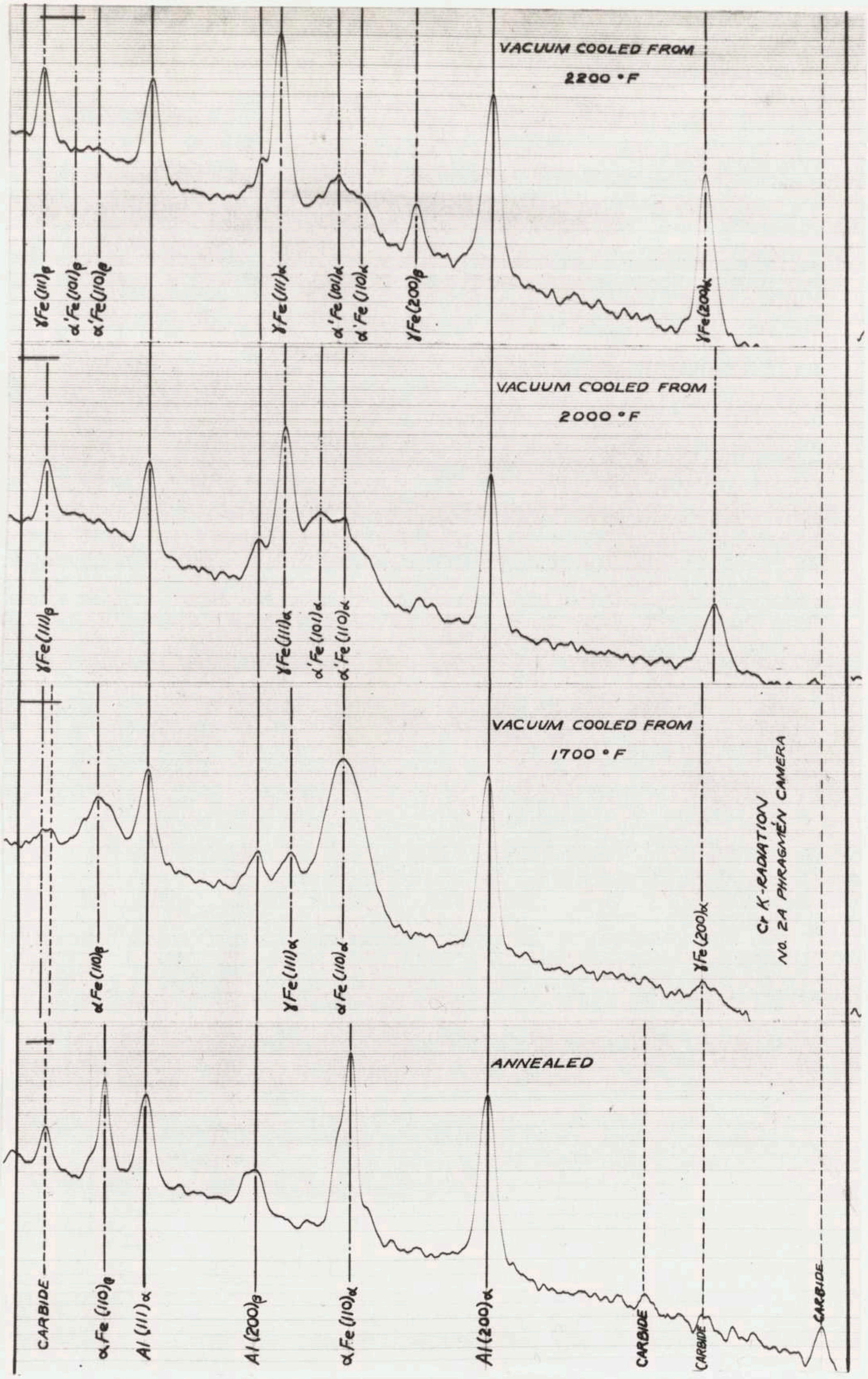


FIGURE 88. MICROPHOTOMETER TRACES OF 1.5 % C 11.5 % Cr STEEL HEAT TREATED AS SHOWN.

An interesting observation from these figures was that the tetragonal martensite doublet was only apparent in the hardened steels that contained considerable quantities of austenite.

The X-ray method requires a specimen of the steel in the completely austenite state for calibration purposes. Neither of the two steels studied could be made completely austenitic. The 1.5% C - 11.5% Cr steel could be made non-magnetic, but an examination of the micro-structure (Figure 95) showed the presence of carbides. The carbides present in the 1% C - 5% Cr steel could be dissolved but martensite was always present along with the austenite. In order to obtain the density ratios for the 100 per cent austenite states of the two steels, estimates had to be made of the amounts of the extra constituents.

Two other factors made the X-ray method unsuitable for determining the retained austenite contents in these steels. The first factor was the "skin effect." That is, the hardened specimens had more austenite in the core portions than in the surface portions (probably due to compression stresses exhibited by the surface portions on the core). The second factor was the segregations in the steels. The first factor could be eliminated by careful grinding followed by deep etching of the ground surface. The second factor, however, could not be eliminated.

2. Kinetics of Tempering

Although the X-ray study for the purposes of determining the retained-austenite content was not fruitful, some very fundamental information was obtained from the X-ray study for the purpose of clarifying the kinetics of tempering.

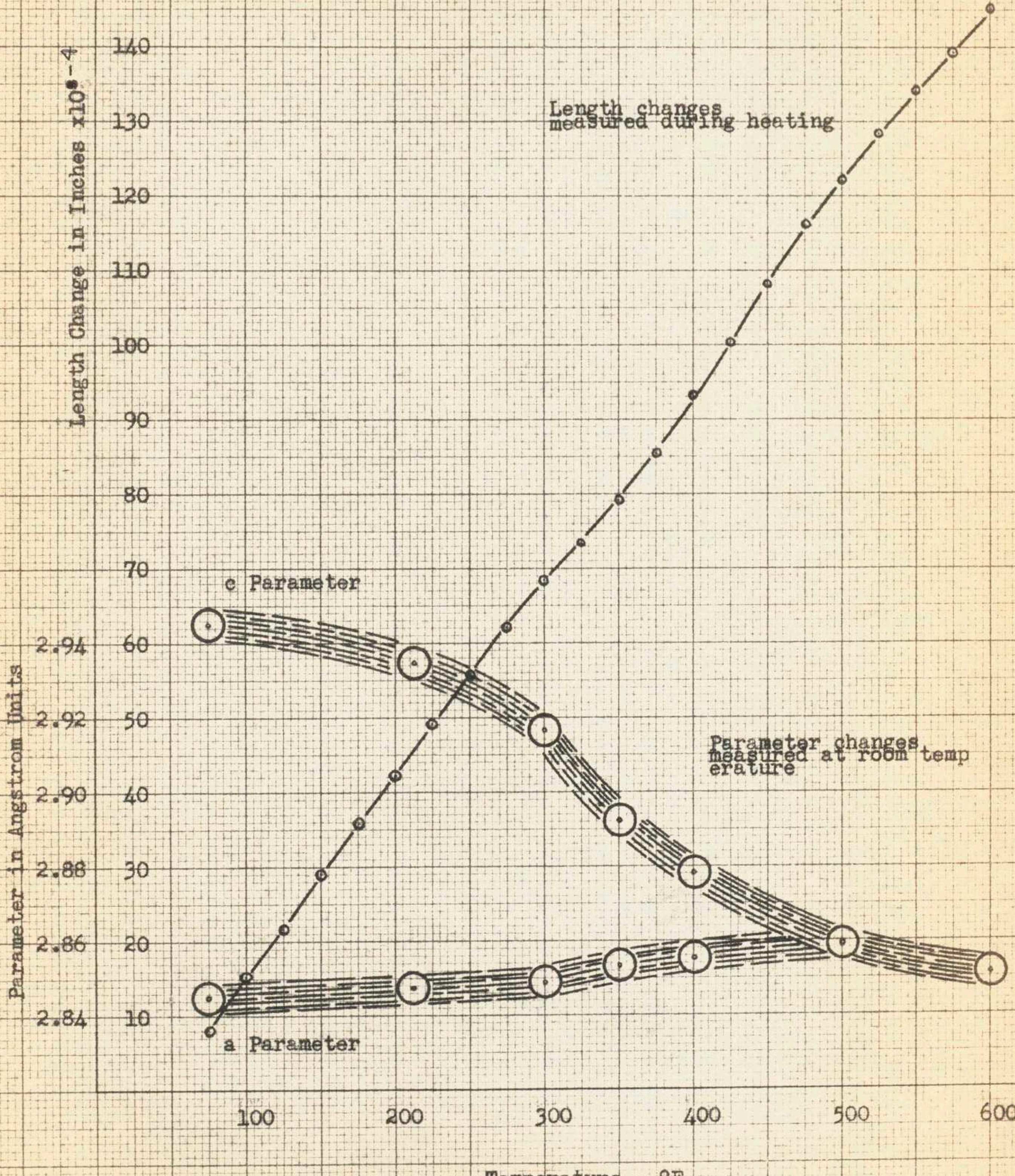
It was found in the microscopic study that no heat treatment used resulted in complete dissolution of the carbides in either of the two steels. These carbide lines may be seen in the traces of the annealed specimens (Figures 87 and 88). They were not, however, visible in the photograms of the heat-treated 1% C - 5% Cr steels. One carbide line only was visible in most of the photograms of the heat-treated 1.5% C - 11.5% Cr steels. The position of this carbide coincided with the carbide shown in the extreme right-hand side of the trace of the annealed-specimen's photogram, Figure 88. In both steels the carbide lines occurred in the same places as an austenite line and a martensite line. This superimposition distorted the tetragonal-martensite doublet in the 1.5% C - 11.5% Cr steels, as can be seen in the microphotometer traces of Figure 88, and prevented accurate measurements of parameter changes.

Both steels had the same ferrite parameter in the annealed state-- 2.854 Angstrom units. The visible carbide lines indicated that the carbides in the two steels were different in composition.

The axial ratio of the tetragonal martensite in the quenched steels could be determined accurately in the specimen of 1% C - 5% Cr steel hardened from 2000° F. This ratio was 1.035 in the quenched specimen; and was gradually reduced to unity by tempering as shown in Figure 89. The dilatometer studies showed that the contraction which accompanied the change from tetragonal to cubic martensite occurred in all the quenched steels (except those quenches which produced no martensite).

It was expected that the cause of a second contraction noted upon tempering through the range 450 - 525° F. was carbide precipitation. As can be seen from Figure 90 no carbide lines were visible in the

FIGURE 89 Parameter and length changes in a 1% C 5% Cr steel oil quenched from 2000°F and tempered as shown.



photograms of the tempered specimens. This can be taken to mean that either none were present, or, more probably, that they were too finely divided to give a line on the photogram. A study of the change in the ferrite parameter, however, disclosed that precipitation did occur. Allocation of this precipitation could also be made. The austenite parameters remained, with the precision of the measurement, at 3.596 Angstrom units while the ferrite parameter changed as shown in Figure 89. This meant that the precipitation occurred from the martensite.

A 1% C - 5% Cr steel specimen oil-quenched from 2000° F. was used for the study. (Because of interferring carbide lines, measurements on the 1.5% C - 11.5% Cr steel could not be made.) This specimen was tempered successively for one-half hour periods at temperatures of 212, 300, 400, 500, and 600° F. After each temper a photogram was made. The parameters of the ferrite and austenite lines were measured. The ferrite parameter changes are shown in Figure 89. Also plotted in the same figure are the results of a dilatometer run made on a similarly treated specimen. It is to be noted that the first contraction is accompanied by a closing of the tetragonal-martensite doublet and that the second contraction is accompanied by a decrease in the ferrite parameter.

A most important result of this investigation was obtained by increasing the tempering temperature further. It was found that during the cooling from a tempering treatment of 100 hours at 980° F. the austenite in a 1% C - 5% Cr steel vacuum-hardened from 2000° F. was transformed to tetragonal martensite. Microphotometer traces were made of the photograms of this study and are shown in Figure 90.

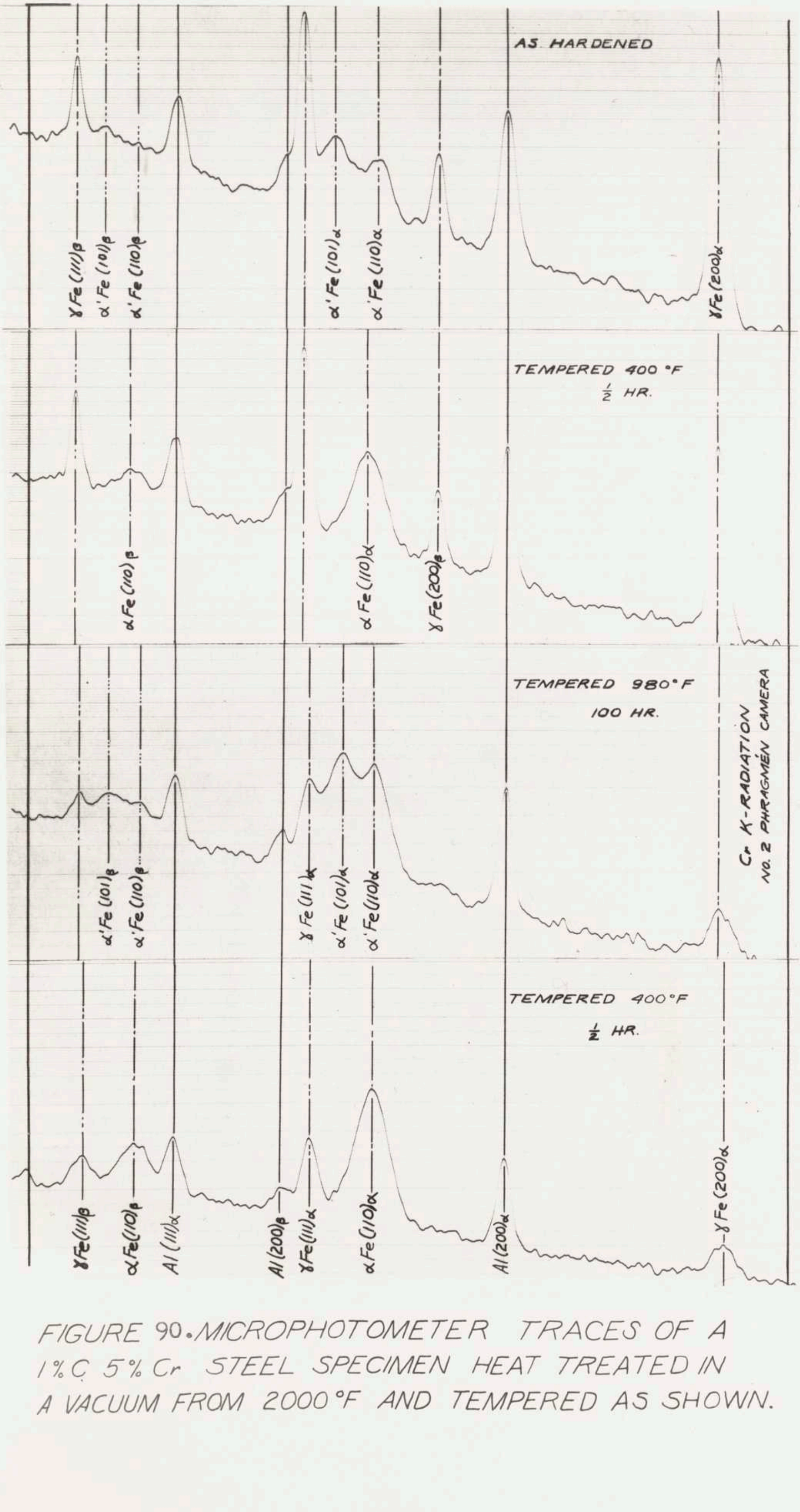


FIGURE 90. MICROPHOTOMETER TRACES OF A 1% C 5% Cr STEEL SPECIMEN HEAT TREATED IN A VACUUM FROM 2000 °F AND TEMPERED AS SHOWN.

The heating was all done in a vacuum, but the quenching was done by breaking the vacuum and quenching in oil. The top trace shows the lines of the as-hardened state. After tempering at 400° F. for 1/2 hour, the tetragonal doublet was closed. The reason for the lower intensity of the austenite line was not that some austenite transformed, but rather that the re-etching of the surface after the tempering treatment exposed a different portion of the steel to the X-radiation.

The third trace is the illuminating one, for it proves that the austenite transformed into tetragonal martensite. The axial ratio, 1.027, shows that the transformation occurred during cooling close to 300° F.

It should be noted that the traces given in Figure 90 were made from the photograms of one specimen. This specimen received successively the treatments indicated. Then, the last trace is highly significant. It shows that the two peaks were closed by the temper and thus were actually due to tetragonal martensite.

D. Microscopic Results

A systematic study of the microstructures was not undertaken because of the interference of the carbide structures.

All photomicrographs were made at 2000 magnifications unless otherwise noted. The specimens that contained martensite needles were tempered one-half hour at 400° F. (in some instances at 600° F. and 500° F.) to darken the martensite.

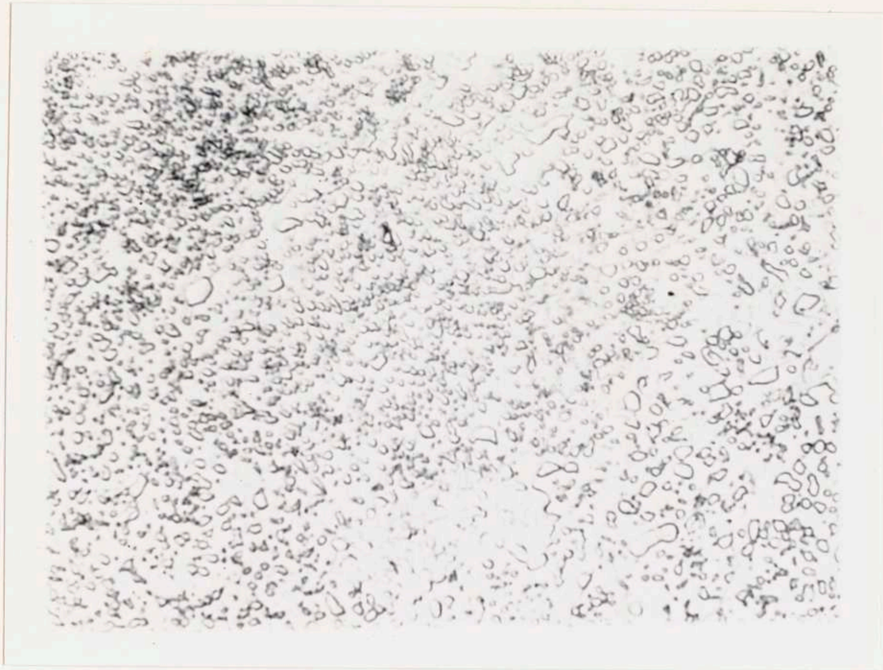
Figure 91 shows the microstructures of the annealed states. Carbides of various sizes may be seen. The heavier carbides were generally located in bands parallel to the rolling direction.

Figures 92 and 93 show the microstructures of 1700° F.- and the 1800° F.-vacuum-hardened states, respectively. The amounts of carbides are only slightly less than in the annealed states. A grain-boundary delineation may be seen in the 1800° F. structures.

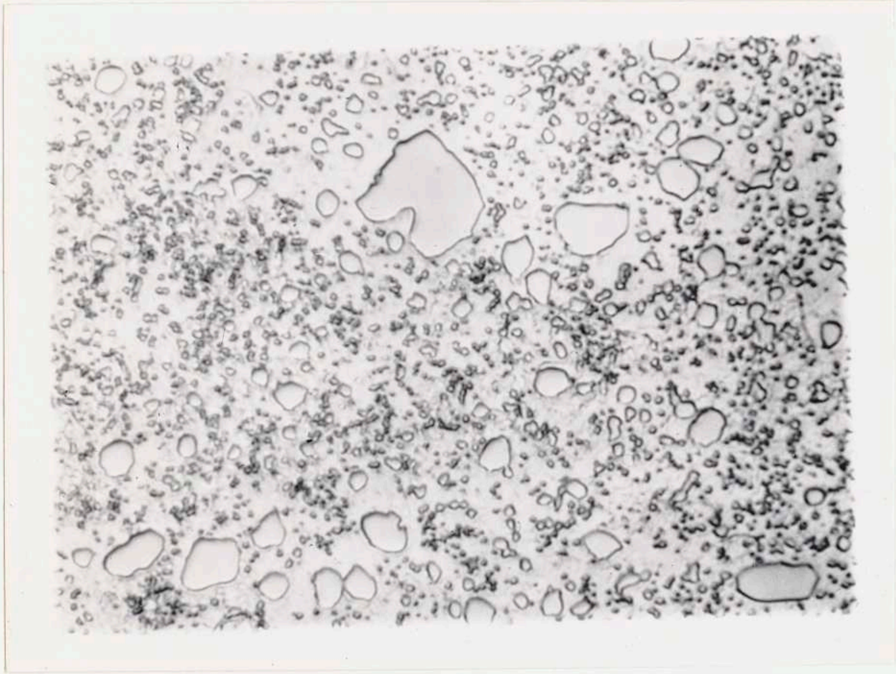
The grain-boundary effect is more clearly shown in the microstructures of Figure 97. The specimens were of 1% C - 5% Cr steel hardened from 2000° F. in air and in lime.

It can be seen that the grain-boundary delineation was more marked the more slowly-cooled was the specimen. This was found to be true for all hardening temperatures above 1700° F. It appears that this grain-boundary effect is due to transformation at the grain boundaries.

The 2000° F.- and the 2200° F.-hardened states are shown in Figures 94 and 95. Above 2000° F. the martensite in the 1% C - 5% Cr steel increased, and in the 1.5% C - 11.5% Cr steel it decreased.

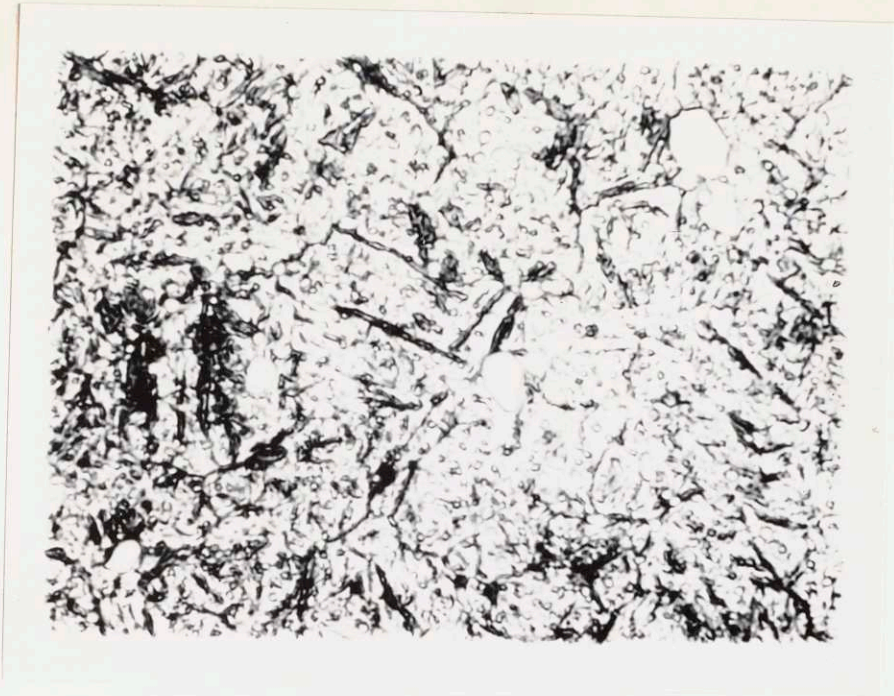


1% C - 5% Cr



1.5% C - 11.5% Cr

FIGURE 91. Microstructures of annealed states.
Mag. = 2000X Etch = 3% Nital

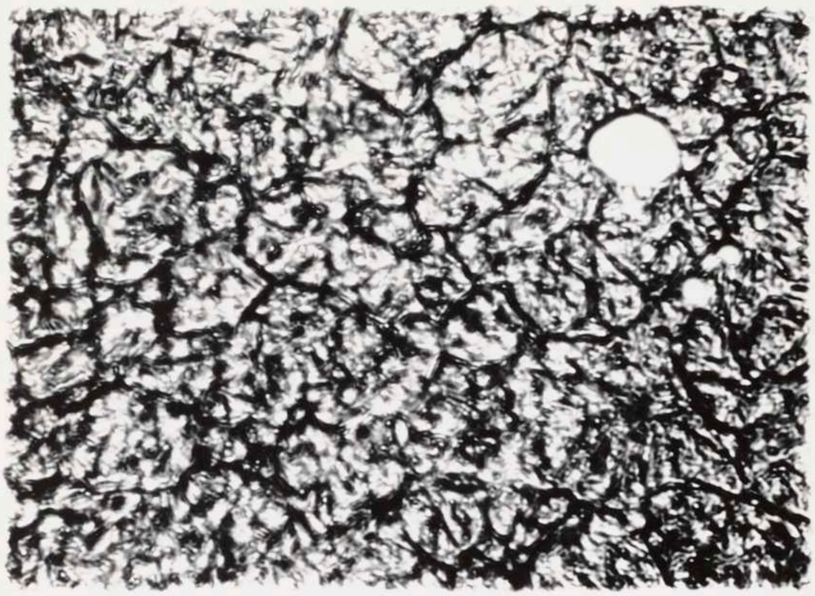


1700

1% C - 5% Cr

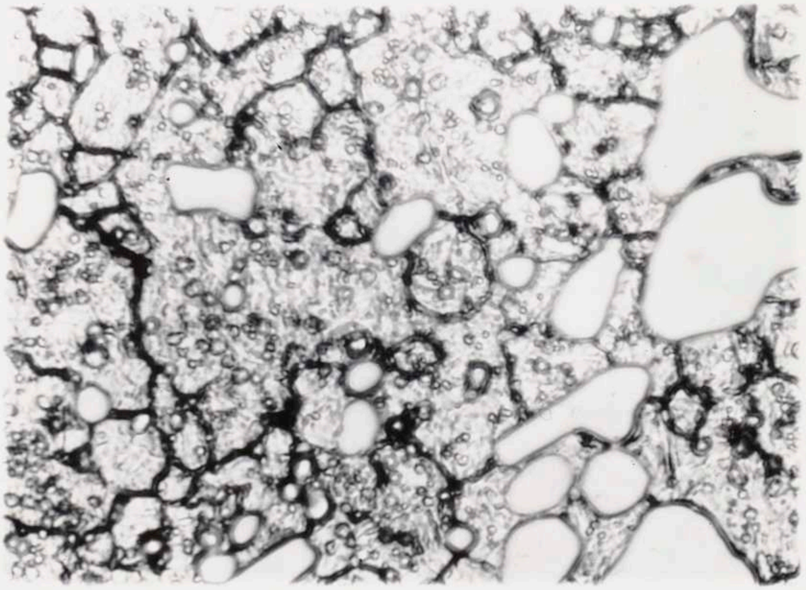
1.5% C - 11.5% Cr

FIGURE 92. Microstructures of vacuum-hardened (1700° F.) states. Mag. = 2000X Etch = 3% Nital.



1800

1% C - 5% Cr



1800

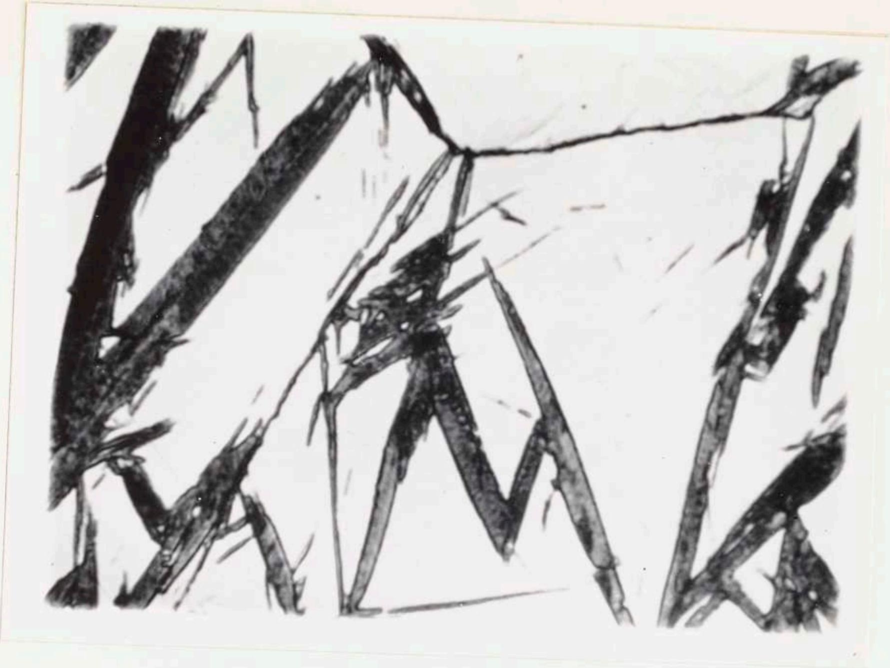
1.5% C - 11.5% Cr

FIGURE 93. Microstructures of vacuum-hardened (1800° F.) states. Mag. = 2000X. Etch = 3% Nital.

1% C - 5% Cr

1.5% C - 11.5% Cr

FIGURE 94. Microstructures of vacuum-hardened states (2000° F.). Martensite darkened by 600° F. temper. Mag. = 2200 X. Etch = 6% Nital.



2200° F.

1% C - 5% Cr

(Martensite darkened by 400° F. - temper)

2200° F.

1.5% C - 11.5% Cr

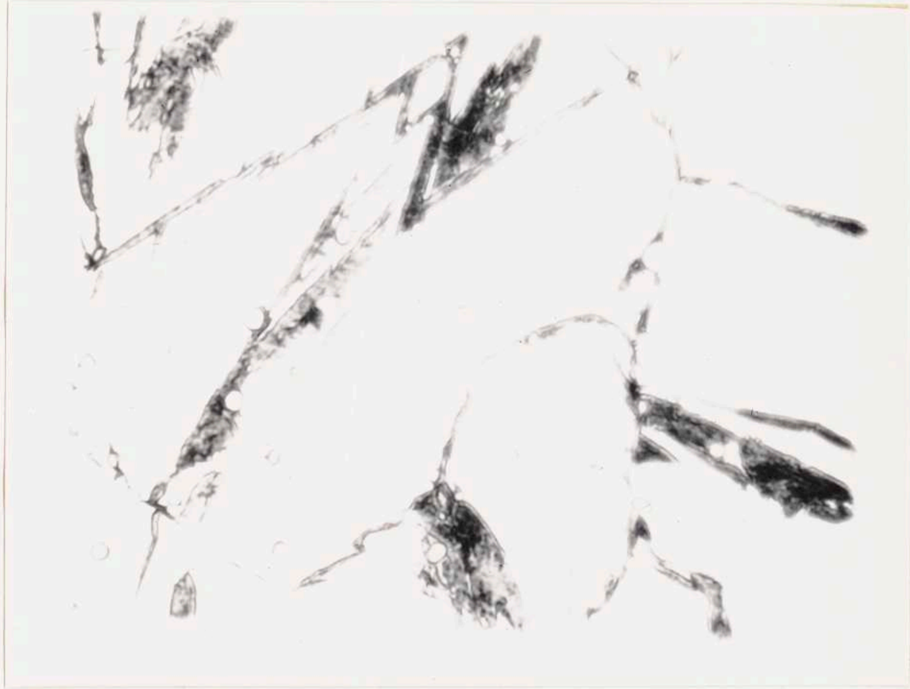
FIGURE 95. Microstructures of vacuum-hardened states (2200° F.). Mag. = 2200X. Etch = 6% Nital.

Water-hardened from 2000° F.

Oil-hardened from 2000° F.

FIGURE 96. Microstructures of water-and oil-hardened 1% C - 5% Cr steels (2000° F.). Martensite darkened by 500° F. temper
Mag. = 2000X Etch = 3% Nital

Air-hardened from 2000° F.



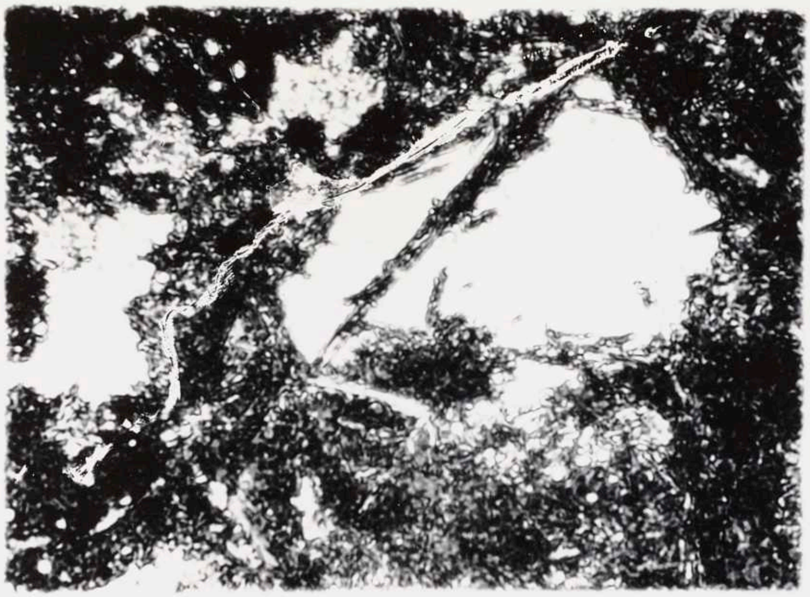
Lime-hardened from 2000° F.

FIGURE 97. Microstructures illustrating grain-boundary effect in 1% C - 5% Cr steel. All specimens tempered at 500° F. to darken the martensite.
Mag. = 2000X. Etch = 3% Nital.

Figure 98 gives representative microstructures of the transformed states. The lower-temperature-hardened steels gave complex structures similar to the 1% C - 5% Cr steel air-hardened from 1700° F. and transformed at 6000° F.

The partially-transformed state for the 1% C - 5% Cr steel oil-hardened from 2000° F. is included to show that the transformation at upper temperatures was a product of nucleation and growth.

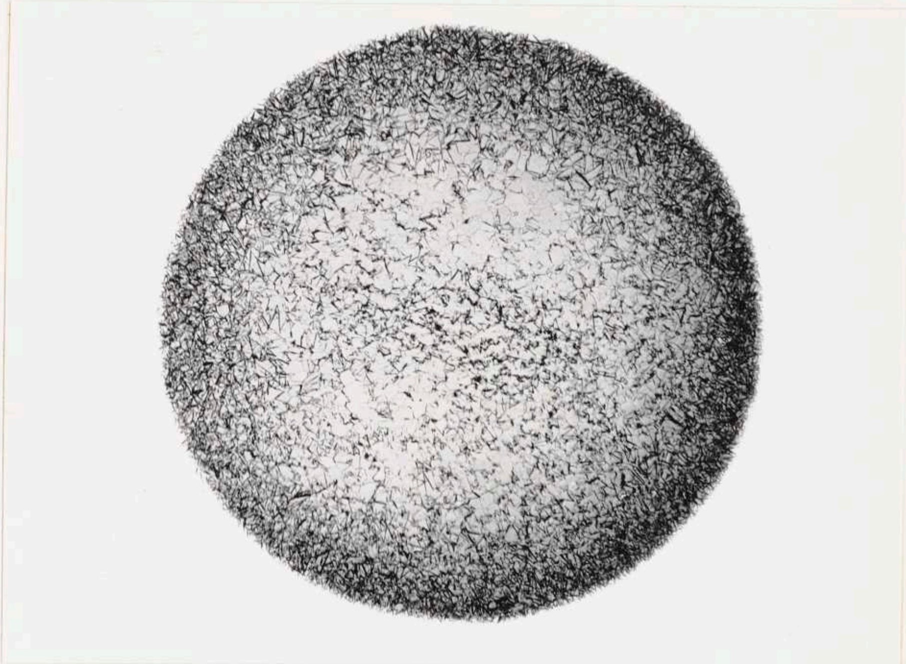
Figure 99 shows the case-effect in a 1% C - 5% Cr steel oil-hardened from 2000° F. This photomicrograph was made at a magnification of 15 diameters. This case amounted to 25 per cent of the diameter of the specimen.



1% C - 5% Cr steel oil-quenched from 2000° F., tempered 48 hours at 1100° F.

1% C - 5% Cr steel air-cooled from 1700° F., and completely transformed at 600° F.

FIGURE 98. Microstructures illustrating transformed states.
Mag. = 2000X Etch = 3% Nital.



1% C - 5% Cr Steel

Oil-hardened from 2000° F. and tempered at 400° F. to darken the martensite.

FIGURE 99. Microstructure illustrating case effect in 1% C - 5% Cr steel. Mag. = 15X Etch = 6% Nital

VIII

CORRELATION AND INTERPRETATION OF RESULTS

A. Transformation Characteristics

The heating magnetization and dilation curves showed that the austenite retained in the 1% C - 5% Cr steels by either oil-, air-, or lime-cooling from 1700° F. to 1900° F. transformed during heating at temperatures below 700° F. With the rate of cooling used, 5° F. per minute, these transformations started at 550° F. and ended at 650° F. The extent of the transformation depended on the stability of the retained austenite. The stability of the retained austenite was lower the lower the hardening temperature and the lower the rate of cooling.

The amount of the retained austenite was greater the higher the quenching temperature and the lower the hardening rate. The reason for the greater quantity of austenite in the progressively slower cools from the lower hardening temperatures must be the presence of stress. The more removed a given rate of cooling is from the critical cooling velocity the less austenite is produced. These results indicate that the critical cooling velocity at higher hardening temperatures is different from that at the lower temperatures.

The isothermal transformation results verified the facts on the stability of the austenite. The temperature of the heating transformation, however, was shown to depend upon the rate of heating. This temperature could be depressed to as low as 460° F. for complete transformation by heating slowly enough and it could be suppressed by heating rapidly.

The austenite retained in specimens of the 1.5% C - 11.5% Cr steel withstood transformation during heating. Only the austenite in the 1700° F.-hardened specimens transformed during very slow heating at temperatures below 700° F. The extent of the transformation was only 30 per cent.

The high rate of reaction of the retained austenite in 1700° F.- and 1800° F.-hardened specimens of the 1% C - 5% Cr steel in the range 590 - 600° F. would indicate that this range should be chosen for all tempering.

In the specimens hardened from the higher temperature, however, 600° F. tempering will result in a lower hardness value than tempering at 900° F. This is explained by the austenite's high reactivity at 600° F.; it all transforms at temperature into a bainite product. At 900° F. the austenite is less reactive and is only conditioned to transform during cooling, whereupon, the transformation temperature is lowered to 250° F. and below. Tetragonal martensite is formed.

The advantage of a multiple tempering process is seen to be great. A single temper at a temperature and time that will cause the austenite to be completely transformed during cooling into martensite will also produce a highly-stressed state. If a hardened specimen were tempered several times, a smaller amount of martensite would be produced each time. The hardness of a multiple-tempered specimen would be lower than the hardness of a single-tempered specimen.

Transformation of the retained austenite in the 1.5% C - 11.5% Cr steels, to over 50 per cent could not be had at temperatures below 900° F. The retained austenite in both steels could be transformed to a considerable extent by continuing the cooling from hardening down to the temperature of liquid air. The more stable austenite (in 2000° F.-quenched specimens) were only slightly transformed by the treatment. Furthermore, the extent of the transformation was lowered the longer the stay at room temperature before immersion in the liquid air.

Although a high temperature quench produced an austenite of high stability, once the austenite transformed, to any extent, some very unstable austenite was produced. The evidence of this unstable austenite was the extent of room-temperature transformation that occurred during subsequent tempering runs.

Room-temperature transformation of austenite was definite, but it did not occur to any extent of more than 20 per cent in the least-stable condition.

B. General Tempering Characteristics

The first phenomenon that occurred was the tempering of the tetragonal martensite. This was not accompanied by any distinct magnetic change other than a decrease. The slope of this initial part of the curve was greater the less austenite that was present. The nature of the change in the steel, however, was disclosed by the dilation curve, which showed a contraction. This process was complete at 300° F. A slight change in the slope of the magnetization curve at 1000 gauss then ensued and a flatter portion of the curve resulted. Most conclusive of the evidence supporting this concept was the X-ray data. A contraction of the axial ratio was displayed by tempering to the same temperature that produced a contraction.

The magnetization curve at low field strength displayed an entirely different appearance from the curve at high field. In the range from 300 to 450° F. the magnetization increased. The dilatometer curve showed an increasing slope after the contraction due to the tempering of the martensite, but no unusual change was noted. The coercivity was markedly lowered and the hardness was unchanged. This all indicated that the process which took place in this temperature range was that of stress relief.

A most important feature common to all specimens heat-treated from the varying temperatures was the contraction and the irreversible decrease in magnetization under high and low fields which existed in a temperature range from 450 - 525° F. Subsequent to this did or did not occur transformation during heating--depending upon the quenching temperature. This served to allocate the phenomena. For, if it represented a precipitation from the austenite it should have resulted in subsequent transformation at all temperatures.

Furthermore, if this were true, the process should have become increasingly manifest as the austenite content increased. Actually, there was little change in its intensity. The fundamental demonstration was secured from the X-ray data. These showed that the ferrite parameter decreased in this range. The conclusion derived from this is that the precipitate must form from the martensite. We are further led to the belief that martensite must exist in the cubic form between the temperatures of the restoration of the axial ratio to unity and the precipitation process.

The subsequent phenomena for usual heating rates depended upon the prior heat treatment of the steel and upon the analysis of the steel. If specimens of the 1% C - 5% Cr steel were quenched from the range 1700 - 1900° F., they gave magnetization curves which showed a rise at both fields, the rises reaching maxima at 600° F. The magnitude of the rise was greater the lower the hardening temperature. Confirmation that this rise in magnetization was due to austenite transformation came from the dilatometer curve which displayed an expansion. Further confirmation came from the X-ray data. The treatments that should transform the austenite as judged from magnetic measurements also caused the austenite lines to disappear from the X-ray photograms.

If the hardening temperature was above 1950° F. this rise in magnetization was not present. The magnetization curve at each field displayed a continuous drop the slope of which was fairly steep.

If the heating was continued to 1135° F., another highly reactive range of austenite transformation was entered. If not, the phenomena occurring during subsequent cooling from the tempering temperature would occur during cooling from the tempering temperature.

The transformation could be made to occur in one or in two stages, depending upon the hardening temperature and the tempering temperature as well as the time of tempering. The temperature of the first stage was generally 600° F. and the temperature of the second stage was usually below 250° F. Both stages when they occurred were manifest by sharp magnetization increases at both high and low fields and by expansions.

IX

CONCLUSIONS

It is believed that the present investigation on the tempering of high-carbon high-chromium steel justifies the following conclusions:

1. The transformation of the austenite retained in hardened specimens of these steels has an important effect on their dimensional-change properties.

2. The amount of retained austenite is greater the higher the hardening temperature (in the usual range of hardening temperatures) and the lower the cooling rate (15 to 25 per cent, oil to lime cool from 1700° F. and 30 to 40 per cent, oil to lime cool from 1800° F.).

3. For a given hardening treatment (in the usual range of hardening temperatures) more austenite is retained in the 1% C - 5% Cr steel than the 1.5% C - 11.5% Cr steel (10 to 20 per cent, lime to oil cool).

4. The stability of the retained austenite in the 1% C - 5% Cr steel is less than the stability of the austenite in the 1.5% C - 11.5% Cr steels.

5. A highly-reactive range at 500 - 600° F. for retained-austenite transformation exists in the specimens of 1% C - 5% Cr steel hardened from 1700 and from 1800° F.

6. The retained austenite in specimens of 1.5% C - 11.5% Cr steel hardened from all temperatures above and including 1700° F. could not be transformed to any extent above 30 per cent by a tempering temperature less than 900° F.

7. The first change to occur during the tempering of a usually-hardened specimen of either steel is the tempering of tetragonal martensite. The decrease in the axial ratio starts at 200° and is practically complete (to unity) at 400° F.

8. The second tempering process is micro stress relief. This starts at 300° F. and appears complete at 450° F.

9. The third tempering process is the precipitation of complex carbides out of the martensite.

10. These three processes will exist during the tempering of all specimens of these steels hardened so as to contain some martensite.

11. In specimens of the 1% C - 5% Cr steels hardened from 1700 and from 1800° F. retained austenite is present that will transform on heating in the range 500 to 600° F.

12. The transformation of this austenite can be suppressed by rapid heating through the range 500 to 600° F.

13. Tempering at temperatures above 800° F. of both steels, hardened from any temperature will produce austenite transformation. The major portion of this transformation will occur on cooling from the tempering temperature.

14. Depending upon the temperature and the time at temperature, austenite transformation may be made to proceed in one or two stages.

15. The temperature of single stage transformations is dependent on the tempering conditions.

16. If the transformation temperature of the austenite is below 250° F., tetragonal martensite will be formed.

X

SUGGESTIONS FOR FUTURE WORK

Retained austenite formed in the steels of this investigation by certain hardening treatments was able to transform at room temperature. This transformation was not extensive but has been shown to be attended by property changes in the steel. Retained austenite that was too stable to transform at room temperature did so when partially transformed by an elevated-temperature temper. This all suggests that a very illuminating research could be conducted on the general problem of room-temperature transformation.

Retained austenite in specimens of 1.5% C - 11.5% Cr steel could not be transformed to any amount greater than 30 per cent by tempering temperatures below 900° F. Immersion in liquid air, however, could produce 90 per cent transformation in the lower-stability austenites. This suggests that an adaption of the magnet to allow sub-room-temperature studies of austenite transformation would be very interesting and profitable to industry.

The ability of these steels to harden by slow cooling suggests further investigation of the limits to which this can be carried out.

The field dependency of the retained-asutenite content as determined by the magnetic method needs clarification. This can only be done with a steel whose retained-austenite content can be determined by the Gardner X-ray method.

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BIOGRAPHICAL NOTE

Otto Zmeskal was born in Chicago, Illinois, on July 16, 1915. After graduating from the public schools of that city, he entered the Armour Institute of Technology in 1932 and was graduated in 1936, receiving the degree of Bachelor of Science in Chemical Engineering.

He was appointed Instructor in Chemistry in 1936, and Instructor in Metallurgy in 1937 at the Armour Institute of Technology. In 1936 he matriculated in the Graduate School of the Armour Institute of Technology, and received the degree of Master of Science in Chemical Engineering in 1938.

Appointed Assistant in Metallography in the Department of Metallurgy at the Massachusetts Institute of Technology, 1938 - 1941, he entered the Graduate School of the Massachusetts Institute of Technology in 1938 to pursue a course of study leading to the degree of Doctor of Science in Physical Metallurgy.

He presented to the Faculty of the Massachusetts Institute of Technology in September, 1941 a thesis entitled, "The Tempering of High-Carbon High-Chromium Steels.

ABSTRACT

The high-carbon high-chromium steels containing molybdenum have become increasingly important in the last five years. The application of these steels is largely in die work and the reason for their popularity is their ability to be hardened with a minimum amount of distortion. The 1% C - 5% Cr steel has attracted more attention than the 1.5% C - 11.5% Cr steel because of its lower cost.

Present in both these steels in their hardened states are certain amounts of retained austenite. The 1% C - 5% Cr steel, which exhibits the minimum distortion, retains more austenite when hardened from the temperatures used in practice than does the 1.5% C - 11.5% Cr steel.

The tempering of these steels has a great effect upon their wear life and, again, upon their distortion. The transformation of the retained austenite during this tempering is intimately associated with these properties.

Thus, a study of the tempering characteristics of these steels with particular emphasis on the retained-austenite transformation should bring forth information of interest and importance to both the users of these steels and the theoretical student of metallurgy. Such a study was conducted in this present investigation.

There are several methods of studying austenite transformation: microscopic, dilatometric, mechanical-property, density, the newly-developed

X-ray, and magnetic methods. The nature of the steels, that is, their tendency to be non-homogeneous in austenite content, made it necessary to use a method that would allow an integrated or all-over measurement. The magnetic method is the only one which will do this quantitatively.

There have been recorded numerous devices, working on the magnetic principle, for the determination of the amount of retained austenite in a steel specimen. The apparatus built for this investigation has the advantage of being easily used and of allowing rapid measurement. An innovation was introduced that made the apparatus unique. This was the adaptation of a dilatometer to the apparatus, resulting in what has been christened the "magneto-dilatometer." From this apparatus simultaneous measurements of both magnetization and dilation changes could be obtained during the tempering of a specimen.

To aid in the interpretation of the results, X-ray and microscopic measurements were also conducted.

The study was carried out in two general divisions: (1) isothermal transformation of the retained austenite and (2) kinetics of the tempering process.

The isothermal investigations disclosed an uniquely-active range at 500 - 600° F. for retained-austenite transformation in the 1% C - 5% Cr steel. Transformation in this range took place at temperature. When the hardened specimens were tempered at 900° F. and above, transformation occurred mainly during cooling. The product of the 600° F.-temper was mainly a time-dependent structure, but the product of the 900° F.- and above temper was mainly martensite.

In specimens hardened from 2000° F., where the major constituent of the hardened structure was austenite, the martensite was definitely tetragonal. Furthermore, it was ascertained that the retained austenite as a result of the tempering operation also formed tetragonal martensite.

The retained austenite in specimens of 1.5% C - 11.5% Cr steel were adamant in their resistance to transformation of any extent over 30 per cent by tempering temperatures lower than 900° F. An immediate immersion of freshly-hardened (1700 - 1800° F.) specimens in liquid air, however, transformed 90 per cent of the austenite.

The following rates of hardening were used in this investigation: water, oil, air, and lime. Specimens of both steels hardened in this order from 1700° F. and 1800° F. contained increasing amounts of austenite. At 2000° F. the relation did not hold. The austenite present in increased amount in a specimen due a certain cool over that due to a faster cool is always more reactive. This austenite will start to transform before the austenite in the faster-cooled specimen has started to transform. The austenite in the more slowly-cooled specimen, however, requires a longer time to be completely transformed.

The investigation on the kinetics of the tempering process divulged some very illuminating facts. It was found that during the tempering of a hardened specimen of either steel (usual hardening temperatures) the following processes always occur. The first is the tempering of tetragonal martensite, the second is the process of stress relief and the third is the precipitation of complex carbides out of the cubic martensite.

Although these processes exist at successively higher temperatures, they do overlap somewhat. This is evident from the ranges listed below:

Tetragonal martensite to cubic martensite	275 - 400
Stress relief	350 - 450
Carbide precipitation from cubic martensite	450 - 550

These processes cannot be suppressed by rapid heating through the listed ranges, for they will be affected at higher temperatures.

A process sensitive to rate of heating, however, is that one which occurs in specimens of the 1% C - 5% Cr steel hardened from 1700° F. and 1800° F. This process is austenite transformation. At 550° F., a certain amount of austenite starts to transform. This transformation stops, whatever its extent, at 650° F. If the specimens are heated rapidly through this range the austenite will not be transformed. As mentioned before, austenite of such a degree of instability is not present in specimens of the 1% C - 5% Cr steel hardened from 2000° F. and in specimens of the 1.5% C - 11.5% Cr steel hardened from all temperatures above 1700° F.

The combined magnetic and dilatometric study was found to be very suitable for the above investigation. The changes of one kind could be immediately interpreted in terms of the changes of the other kind. The magnet is not very sensitive to the tempering of martensite, but the dilatometer is very sensitive. The magnet is very sensitive to the transformation of austenite (more so than the dilatometer). A unique application of the magnet is the determination of micro-stress-relief processes.

In conclusion the author wishes to state his wish that this investigation will lead to a wider application of these steels.