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A RAPID MAGNETOMETRIC TECHNIQUE TO PLOT ISOTHERMAL TRANSFORMATION DIAGRAMS

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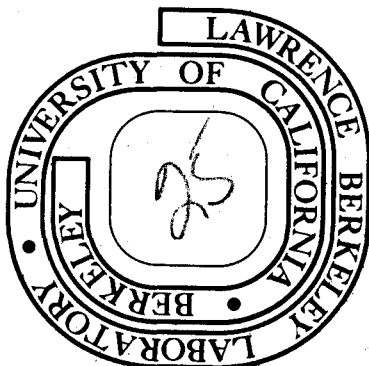
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A RAPID MAGNETOMETRIC TECHNIQUE TO PLOT  
ISOTHERMAL TRANSFORMATION DIAGRAMS

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ABSTRACT

A sensitive magnetic permeability method for rapid determination of isothermal transformations diagrams in steels and iron base alloys is described. The method consists of quenching the sample from an austenitizing temperature to a subcritical temperature in an isothermal bath, and holding it within the magnetic field of an inductor coil. The increase in permeability accompanying austenite decomposition increases the inductance of the coil, and this changes the resonant frequency of the circuit. An automatic continuous recording of the corresponding period provides a convenient and accurate method for following the austenite decomposition of AISI 4340 in the bainite and martensite temperature ranges. This method provides quantitative information on austenite decomposition kinetics within two seconds after the start of quenching.

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## INTRODUCTION

Since the pioneering work of Davenport and Bain in 1930,<sup>1</sup> a voluminous quantity of information on the isothermal transformation behaviour of steel has been obtained. The original metallographic methods developed by Davenport and Bain to study pearlitic and bainitic transformations are time consuming and laborious, and require considerable skill in the metallographic examination of a series of samples reacted isothermally for various periods of time. In some instances, these methods lack accuracy, and consistency is difficult to ensure from sample to sample.<sup>2</sup> The microscopic method, commonly referred to as the quench-temper technique described by Greninger and Troiano<sup>3</sup> for the determination of the martensite transformation range, is also subject to limitations. This technique is restricted to steels of high bainitic hardenability because it is virtually impossible to distinguish by optical metallography between tempered martensite and lower bainite when these constituents are present together.<sup>4</sup>

A number of investigators have followed the course of austenite transformation by measuring changes in properties such as specimen dimensions,<sup>1</sup> electrical resistance,<sup>5</sup> magnetic characteristic,<sup>6,7</sup> and hardness.<sup>8</sup> Most TTT diagrams have been determined by the combined application of metallographic and dilatometric methods, the former to determine the initial stages of transformation, and the latter to follow the subsequent progress during transformation. Measuring the dilation of long, thin specimens has been criticized<sup>9</sup> as not truly being a measure of the volume fraction transformed, particularly in the

case of formation of acicular products. Electrical resistivity methods have also been criticized<sup>2</sup> as not being a simple function of the volume fraction transformed because other factors also affect resistivity.

Magnetic permeability methods have been used by relatively few investigators. Previous investigations have utilized either an electrical transformer<sup>7</sup> or an electromagnet having a secondary search coil.<sup>10</sup> The specimens in the earlier work were held isothermally in an air gap in the magnetic core or the transformer, or within the air gap of an electromagnet. The induced emf in the secondary winding due to the magnetic change in the sample was recorded visually or photographically. The magnetic method described herein is simpler and more sensitive, and can be used to study austenite transformations at all temperatures below the Curie temperature of the alloy and in time periods ranging from two seconds to many hours. Details of this method are given in the following sections.

#### EXPERIMENTAL APPARATUS

The apparatus consisted of a vertical tube furnace for austenitizing the sample, an isothermal quenching system for holding the sample at the desired subcritical temperature, a sensing coil, and an electronic system to measure and record the extent of transformation in the sample. A sketch of the furnace, the quenching system, and the sensing coil is shown in Fig. 1. A block diagram of the electronic system is shown in Fig. 2.

### Tube Furnace

The mullite tube furnace, 1 in. (25.4 mm) in internal diameter and 33 in. (838.2 mm) long, was mounted vertically and had a uniform hot zone over a 2 1/2 in. (63.5 mm) length. This uniform zone was 7 in. (177.8 mm) from the bottom of the tube. The sample was attached with a tantalum wire to the end of a long Hastelloy rod (both non-magnetic). The rod passed through a water cooled vacuum seal at the top of the furnace. The vertical movement of the rod inside the tube was guided by a sliding heat shield. Both ends of the tube were water cooled. The bottom end could be closed and sealed with a lid for evacuation. During evacuation the specimen was held in the bottom cold zone. The long cold zone at the top of the furnace was needed to contain the part of the rod that heated up when the sample was attached to the rod outside of the bottom opening of the furnace. The hot rod could not be moved through the Wilson seal.

### Isothermal Quenching System

This part of the system contained an inconel tube, 1 in (25.4 mm) in internal diameter and 18 in. (457.2 mm) long, mounted coaxially with the tube furnace. The quenching medium (quenching oil for temperatures up to 200°C, and molten salt for higher temperature) was circulated through the tube by an impeller pump located at the base of the reservoir. The quenching medium was gravity fed to the pump through a central hole in the reservoir. The impeller shaft passed through this hole and was driven by a variable speed motor mounted at the top of the reservoir. The level of the quenching medium in the reservoir was maintained low enough to facilitate its gravity return

from the inconel tube. The direction of flow of the quenching medium during circulation is shown by the arrows in Fig. 1.

The system was insulated to minimize heat losses during circulation of the quenching medium. The temperature of the medium was controlled during circulation by controlling the power input to the heating coil that surrounded the reservoir.

#### Sensing Coil and Electronic System

The sensing coil, 5 in. (127 mm) long and 2 in. (50.8 mm) in internal diameter, surrounded the inconel tube. It had an inductance of 90 mH; the sensing coil circuit had a resonant frequency of 1.428 kHz. After austenitizing, the Hastelloy rod was thrust downward, placing the sample simultaneously into the circulating quench medium and within the field of the inductor coil. The increase in permeability of the sample, which occurred with increasing amounts of non-magnetic austenite transforming to low temperature phases which are magnetic, caused an increase in the inductance (L) of the coil. The inductance of the coil determined the resonant period of oscillation (P) of the electrical circuit. A variable frequency oscillator (VFO) was used to measure the period. The output of the VFO was fed into a divider counter, which divided the frequency, or in effect multiplied the period of oscillation by 1600. The control timing decoder monitored the count of the divider counter and provided start, stop, and reset signals to a time interval counter and a punch command to a paper tape punch, as indicated in Fig. 2. Considering the resonant frequency of the circuit at some instant to be 1.428 kHz, the resonant period as measured by the VFO was 0.0007003 sec/cycle. The resonant period as measured



by the divider counter, which multiplied the output of the VFO by 1600, was equal to 1.12045 sec/cycle. In other words, it took 1.12045 second for the divider counter to count 1600. Thus, between start and stop signals, i.e., between counts 0000 and 1000 of the divider counter (or in a time interval of 0.70028 sec.) the time interval counter with a 1 MHz time base had averaged the period of oscillation of the VFO over 998 cycles. When the count reached 1100, i.e, after 0.77 sec from the start of the count, a punch command was sent to the tape punch, and the output from the time interval counter was recorded by the punch. Thus, the fastest recording time interval was every 0.77 sec.

It should be noted from Fig. 2 that the timing decoder obtains its time signal from the resonant period as measured by the divider counter. Thus, the recording interval was determined by the resonant period. When the resonant period changed because of the changes in inductance of the sensing coil, the recording interval also changed. For the size of the sample used, the resonant period was found to change by a maximum of 0.00002 sec/cycle for the complete transformation of the sample. Accordingly, the recording interval would change from 0.77 sec to 0.79 sec. This was ignored especially since the error was considered to be negligible, but a correction could be made for the above error by using a multiple period averaging counter or an independent time signal which could be fed into the circuit.

To ensure the thermal stability of the system, the VFO was stored in a constant temperature container. The sensing coil was also maintained at a constant temperature by surrounding it with a water cooled copper jacket. The system was stable enough to measure reproducibly 1/10 of 1% transformation.

### EXPERIMENTAL PROCEDURE

To determine the validity of the transformation data derived by the magnetic permeability method, it was necessary to use this technique to plot the TTT diagram of a steel, whose isothermal transformation characteristics are well documented in the literature. A commercial low alloy steel, AISI 4340 steel was chosen for this purpose, and it was decided to determine the bainite and martensite regions of the TTT diagram using the magnetic method. The steel used in this study was of commercial aircraft quality, and its chemical composition in wt% was: 0.39 carbon, 0.70 manganese, 0.28 silicon, 0.76 chromium, 1.70 nickel, 0.20 molybdenum and 0.22 copper. The steel was received in the form of 5/8 in. (15.9 mm) thick bar stock and in a fully annealed condition. Specimens for isothermal treatments were prepared by cutting 0.06 in. (1.5 mm) thick strips from the bar stock and then cold rolling the strips to 0.03 in. (0.76 mm) thickness. From the rolled lengths, samples with dimensions of 2 in (50.8 mm) by 0.56 in. (14.2 mm) by 0.03 in (0.76 mm) were prepared.

To prevent oxidation and decarburization, the samples were austenitized in a vacuum of  $8 \times 10^{-5}$  Torr ( $10.7 \times 10^{-3}$  N/m<sup>2</sup>) or better. Carbon analysis on samples, prior to and after austenitization showed no evidence of decarburization. Also, microhardness tests of a reacted sample indicated no surface decarburization.

It was important to know the cooling rate of the sample upon quenching into the isothermal bath. Temperature measurements were made by using a thermocouple consisting of 0.002 in. (0.05 mm) chromel and alumel wires, spotwelded to the opposite sides of a sample at its center.

The temperature as sensed by the thermocouple, spotwelded in this manner, was an average of the temperature gradient across the thickness of the specimen. When a laminar flow of the quenching medium was used, a 0.03 in. (0.76 mm) thick sample required 6 sec, and a 0.01 in. (0.25 mm) thick sample required 4 sec to cool and stabilize at the temperature of the quenching medium. By using counter current turbulent flow, the times were reduced to 2 sec and 1 sec, respectively.

The experimental procedure consisted of the following steps:

(1) The sample was held in the cold zone at the bottom of the furnace, the lid was closed and the tube was evacuated.

(2) The quenching medium was circulated and allowed to stabilize at the desired temperature.

(3) The electronic system was switched on and allowed to warm up.

(4) After a good vacuum in the tube was obtained, the sample was raised into the hot zone and was austenitized at 900°C for 15 minutes.

(5) At the end of 15 minutes the ceramic tube was isolated from the evacuating system.

(6) The latch holding the bottom lid was released.

(7) The tape punch was turned on.

(8) An inert gas was introduced into the tube to force the bottom lid open.

(9) The sample was then quenched and held in the inconel tube within the field of the coil.

Initially, the tape punch recorded the period at intervals of 0.77 sec. In the later stages of transformation, recording was done at intervals of 7.77 sec or 77 sec. In the six digit printout, the last

five digits gave the measured period, while the first digit was coded to indicate the recording interval.

#### ANALYSIS OF DATA

For a given alloy the magnetic permeability is a function of temperature, strain and structure of the phases present.<sup>11</sup> To interpret the data obtained at different temperatures, a calibration was needed for each alloy to account for the temperature and structure dependence of the magnetic permeability. To obtain quantitative values of the kinetics of the austenite decomposition, it was necessary to know for the alloy being studied the value of  $\Delta P$  for 100% transformation at each reaction temperature.

In alloy steels, in the upper part of the bainite range, it is known that the bainite reaction stabilizes short of complete austenitic transformation.<sup>12</sup> In some alloy steels the bainite reaction is very slow and it can take several hours before the reaction is completed or stabilized short of completion. Thus it is time consuming and sometimes impossible to follow the reaction to completion in order to determine the value of  $\Delta P$  for 100% transformation. In 4340 steel, virtually complete transformation to martensite could be achieved by quenching a sample in room temperature water and then refrigerating it in liquid nitrogen. Fully martensitic samples were tempered at various temperatures corresponding to the isothermal holding temperatures. These tempered martensitic structures were assumed to have magnetic properties similar to those of bainitic structures and they were used as comparative standards for the isothermally transformed samples.

To test this assumption, fully bainitic structures were obtained by transforming in the lower part of the bainite range for sufficient periods of time. The completion of the transformation was determined

by the tapering off of the steady increase in  $\Delta P$ , and was verified by optical metallography. The steady values of  $\Delta P$  were obtained for fully martensitic samples that were tempered at the same temperature and for the same time that was required for the fully bainitic samples to transform isothermally. The values agreed for the two types of samples within 5 percent.

The standard sample was heated to successively higher temperatures and the corresponding  $\Delta P$  values were noted. The  $\Delta P$  vs temperature relation so derived is presented in Fig. 3. The value of  $\Delta P$  for 100% transformation for a particular reaction temperature was read from the calibration curve.

For small changes in inductance  $L$ , the relation between the resonant period of oscillation,  $P$ , and  $L$  in the equation  $P = 2\sqrt{LC}$  (where  $C$  is the capacitance) is substantially linear, as shown in Fig. 4. The change in  $L$  with the volume fraction transformed is not necessarily linear. To determine this relation, samples of different thicknesses, but with the other dimensions the same, were transformed to 100% martensite by quenching to room temperature, and in each case the resulting change in period,  $\Delta P$  corresponding to the change in inductance  $L$  was noted. Because the other dimensions were the same, the change in thickness of the specimens could be correlated to their change in weight and in turn, the volume fraction transformed. For example, the strength of the signal of a 2 gm specimen that is 100% transformed should be equivalent to that of a 4 gm specimen that is 50% transformed. The experimentally derived dependence of  $\Delta P$  with weight is shown in Fig. 5. This relationship was not linear, but could be described quite well

by the equation,  $\Delta P = 34430(1 - e^{-0.13K})$ . A 100% transformation "value" of  $\Delta P$  established a value for  $K$  to represent the 100% volume fraction. Based on this, other values of  $\Delta P$  could be converted into their corresponding volume fractions of transformed austenite.

A computer program written by C. E. Ericsson<sup>13</sup> was used to decode the paper tape output, to correct the non-linearity of the change in  $\Delta P$  vs volume fraction transformed, to convert the corrected change in  $\Delta P$  to absolute volume fractions, to plot the kinetics of transformation of the isothermal curves, and finally, to plot the initial TTT diagram. The isothermal reaction data were plotted in both linear and log forms. Some typical isothermal transformation curves obtained for 4340 steel are given in Fig. 6. The TTT diagram derived from these data is given in Fig. 7. The diagram reported in the literature<sup>14,15</sup> for 4340 steel is reproduced in Fig. 8. A comparison of the two diagrams will be made in the next section.

## RESULTS AND DISCUSSION

### Magnetometric Technique

The magnetic permeability method permitted a continuous measurement of the fraction of austenite decomposed in the sample as it was isothermally transformed. The initiation (incubation) and finish times of the isothermal reactions and their associated kinetics could thus be determined. The decomposition products could be identified by observing the kinetics of the transformation and by correlating the kinetics with microstructure.

The martensitic reaction was recognized by its athermal behavior and by the rapid rate at which it formed. Below the  $M_s$  temperature,

the rapid martensite reaction was followed by the time dependent bainite reaction, the beginning of which was signified by an inflection point in the isothermal reaction curve (Fig. 6). Above the  $M_s$  temperature, the isothermal reaction was characterized by an incubation period during which there was no detectable transformation, followed by a period during which there was a time dependent decomposition of austenite. With proper calibration, the amount of untransformed austenite remaining after the bainite reaction had stabilized, or when the bainite reaction was interrupted by quenching, could be determined. Also, the amount of austenite that transformed while cooling to room temperature could be measured. By measuring the permeability of the sample before and after refrigeration in liquid nitrogen, the amount of austenite transformed during refrigeration could also be measured.

The magnetic technique provided a direct experimental method for determining the  $M_s$  temperature and the temperatures required for different amounts of martensite to form. The  $M_s$  temperature was defined to be that temperature at which only 1% of the austenite transformed rapidly to martensite, and the rest transformed slowly to bainite. From isothermal reaction curves, the various percentages of martensite obtained at the different reaction temperatures could be easily determined.

#### Published Diagram vs Presently Derived Diagram

Comparing the diagram derived in the present study (Fig. 7) with the well known diagram reproduced in Fig. 8 for AISI 4340 steel, it can be noted that the two are very similar except for a few differences in the lower bainite and martensite ranges. In the published diagram, the bainite range is shown as a smooth C-shaped curve. The present

investigation showed that the incubation period for the formation of lower bainite decreased at temperatures just above  $M_s$ , and gave an S-shaped curve for the bainite reaction. The acceleration of austenite decomposition just above  $M_s$  has been observed previously<sup>16-18</sup> in several steels. The bainite reaction that follows the martensite reaction below the  $M_s$  is not clearly established in the published diagram. In the diagram derived in the present study, the bainite curve extending below the  $M_s$  and overlapping the martensitic range was C-shaped, as shown in Fig. 7. Below the  $M_s$ , the bainite reaction began almost immediately at the end of the martensitic reaction. This rapid onset of the bainite reaction at temperatures below  $M_s$  is well established in the literature, and is associated with the accelerating effect of the presence of strained martensite - untransformed austenite interfaces on the nucleation of bainite. Decreasing the temperature below the  $M_s$  produced more martensite but less bainite. The decreased rate of bainite formation at temperatures well below  $M_s$  (below about 250°C) was presumably due to the lower diffusion rates of carbon.

The  $M_s$  and  $M_f$  temperatures as determined by the magnetic method were 298°C and 105°C respectively. The values agree quite well with those determined by using the formula of Steven and Haynes.<sup>4</sup> This  $M_s$  temperature is within 5°C of that given in the published diagram, and this difference can be attributed to the small differences in the chemical compositions of two different steels used to determine the two diagrams. The  $M_{50}$  values also agree fairly well. However, the  $M_{90}$  temperature given in the published diagram is higher than the  $M_{70}$  temperature determined in the present study. The volume fractions of



martensite obtained at different temperatures below the  $M_s$  in the present study agree with the values computed by using the empirical equation derived by Harris and Cohen.<sup>19</sup>

The existence of isothermal martensite without the presence of the athermal form is well established in certain alloys and steels, but in most of the commonly used steels, the martensite reaction is believed to be athermal. The present investigation showed that the martensite reaction did not stop when the cooling was arrested within 2 seconds of quenching (Fig. 6), and that some retained austenite did transform isothermally in the next few seconds, before the onset of the bainite reaction. Such behavior was previously observed by Averbach and Cohen.<sup>20</sup>

The reaction curves for the formation of bainite are similar to those of a nucleation and growth process. However, other kinetic features such as the  $B_s$  (the temperature above which austenite will not transform to bainite) and  $B_f$  (the temperature below which fully bainitic structures are obtained by isothermal transformation) temperatures are analogous to those of the martensitic transformation. The  $B_s$  and  $B_f$  temperatures for 4340 steel were found to be around 530°C and 430°C respectively in this study. These values agree fairly well with those shown in the published diagram. Also, the general shape and position of the upper part of the bainite curve in the two diagrams are very similar. A similar range of temperature dependent incomplete transformation to bainite (between the  $B_s$  and  $B_f$  temperatures) can be seen in both the diagrams.

### Limitations of the Magnetic Technique

Like other indirect methods used to follow austenitic decomposition, the magnetic method required that a proper calibration be made to determine the quantitative values of kinetics. Some errors do arise in such estimations. The main source of error was in correlating  $\Delta P$  with phase change. In order to establish a 100% transformation value of  $\Delta P$ , a reasonable assumption was made that tempered martensitic structure has magnetic properties similar to those of bainitic structures formed at the corresponding tempering temperatures, and that the amount of retained austenite did not exceed a few percent. Also, the tempering occurring during the transformation would probably cause a slightly higher  $\Delta P$  than what would be caused by transformation alone. These errors were, however minimal and considered to be negligible in estimating the start and early stages of the transformation, because, at the beginning of the transformation, the value of  $\Delta P$  changed from zero to a positive value. The present system was stable enough to measure less than 0.5% transformation. Although the magnetic method was used in the present study to follow austenite decomposition at temperatures below 550°C, the method is potentially useful at all temperatures up to the Curie temperature of the steel (which is about 770°C).

### SUMMARY

A sensitive magnetic permeability method has been described as a rapid and accurate means of obtaining isothermal transformation data. It has been demonstrated that the method can be successfully used to study austenite transformation in the bainite temperature range when

austenite decomposition is substantially complete. A computer aided analysis was employed to provide accurate quantitative values of the kinetics of transformation.

#### ACKNOWLEDGEMENTS

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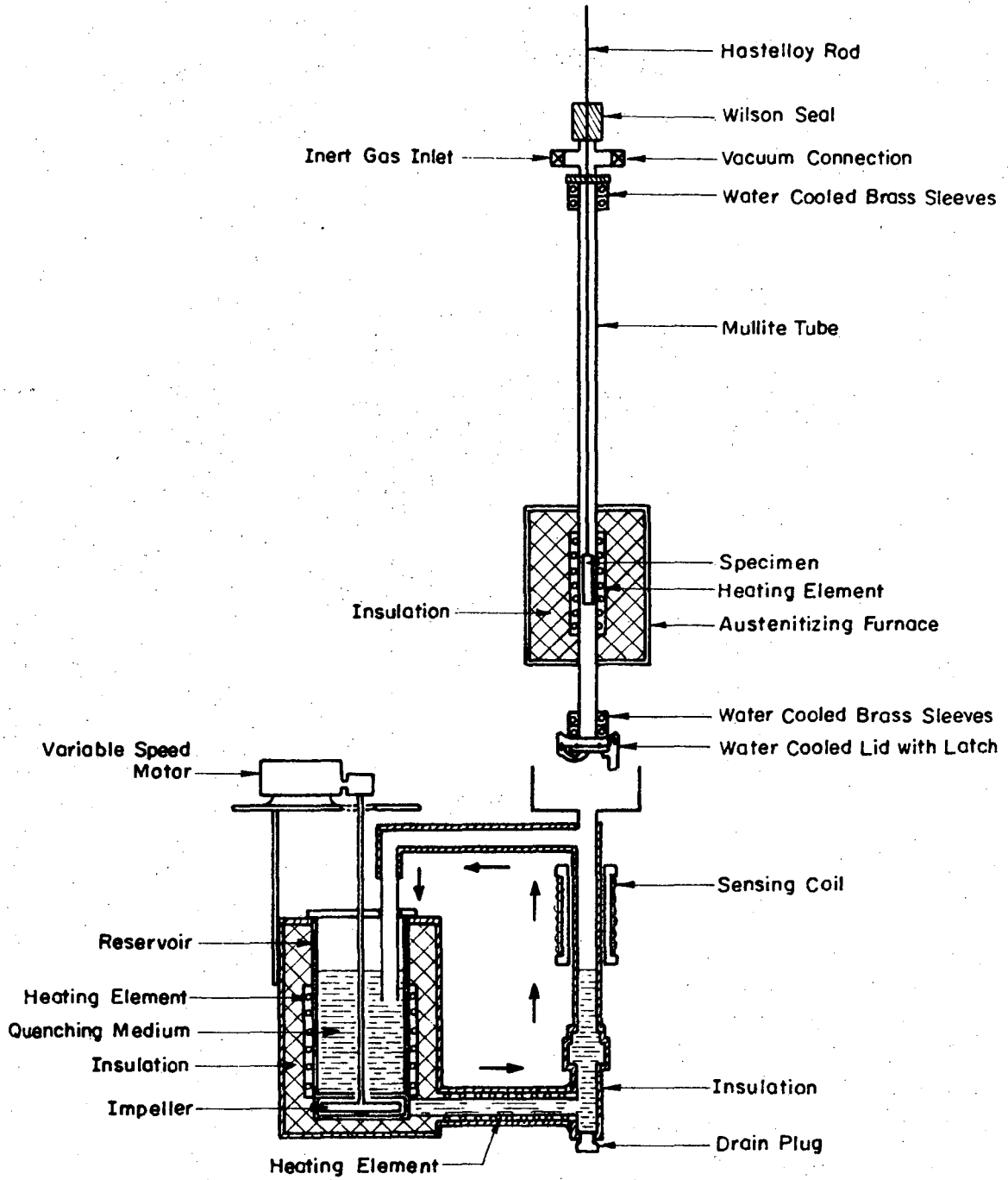
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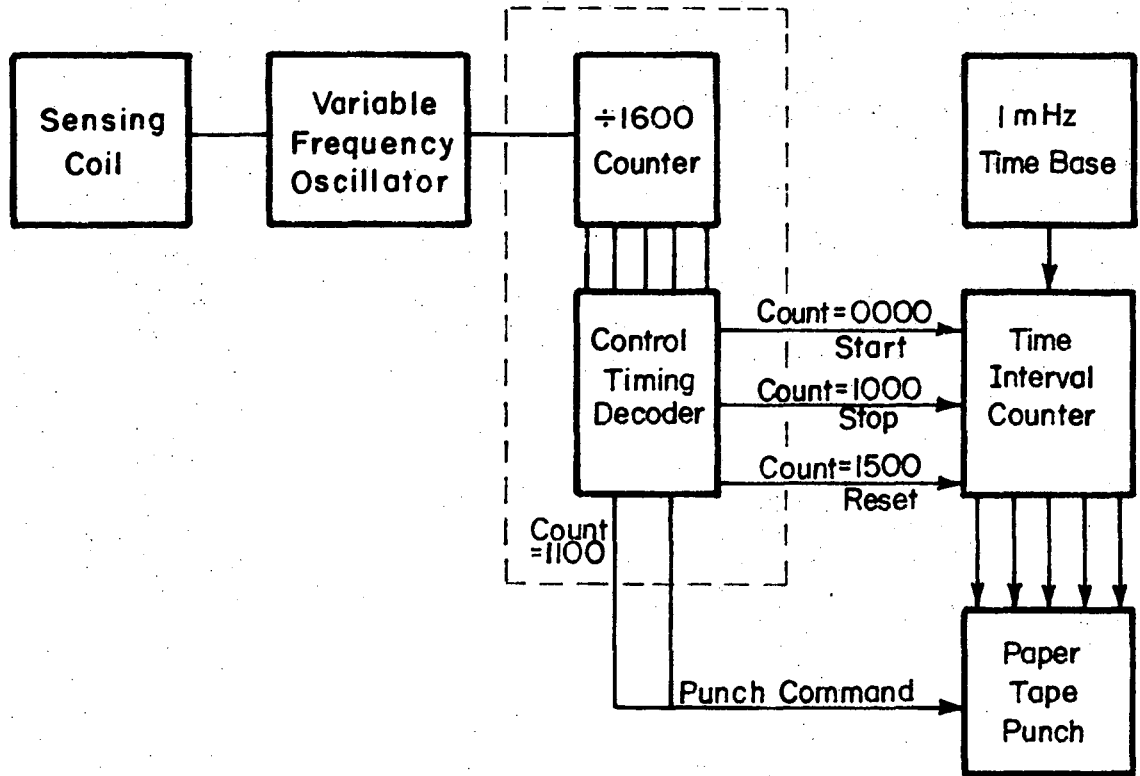
FIGURE CAPTIONS

- Fig. 1. Schematic of the tube furnace, the quenching system and the sensing coil.
- Fig. 2. Block diagram of the electronic system.
- Fig. 3. Calibration curve for change in period ( $\Delta P$ ) with temperature for AISI 4340 steel.
- Fig. 4. Plot of resonant period of oscillation (P) vs inductance (L) of the coil.
- Fig. 5. Calibration curve for the change in period with the amount of transformation.
- Fig. 6. Isothermal transformation curves for AISI 4340 steel austenitized at 900°C. Plot (a) is on a log-log scale and (b) is on a linear scale.
- Fig. 7. Time-temperature-transformation diagram for AISI 4340 steel austenitized at 900°C. Only the bainite and martensite ranges were determined.
- Fig. 8. Time-temperature-transformation diagram for AISI 4340 steel austenitized at 870°C as found in the literature.<sup>14,15</sup>  
Only bainite and martensite ranges are shown.



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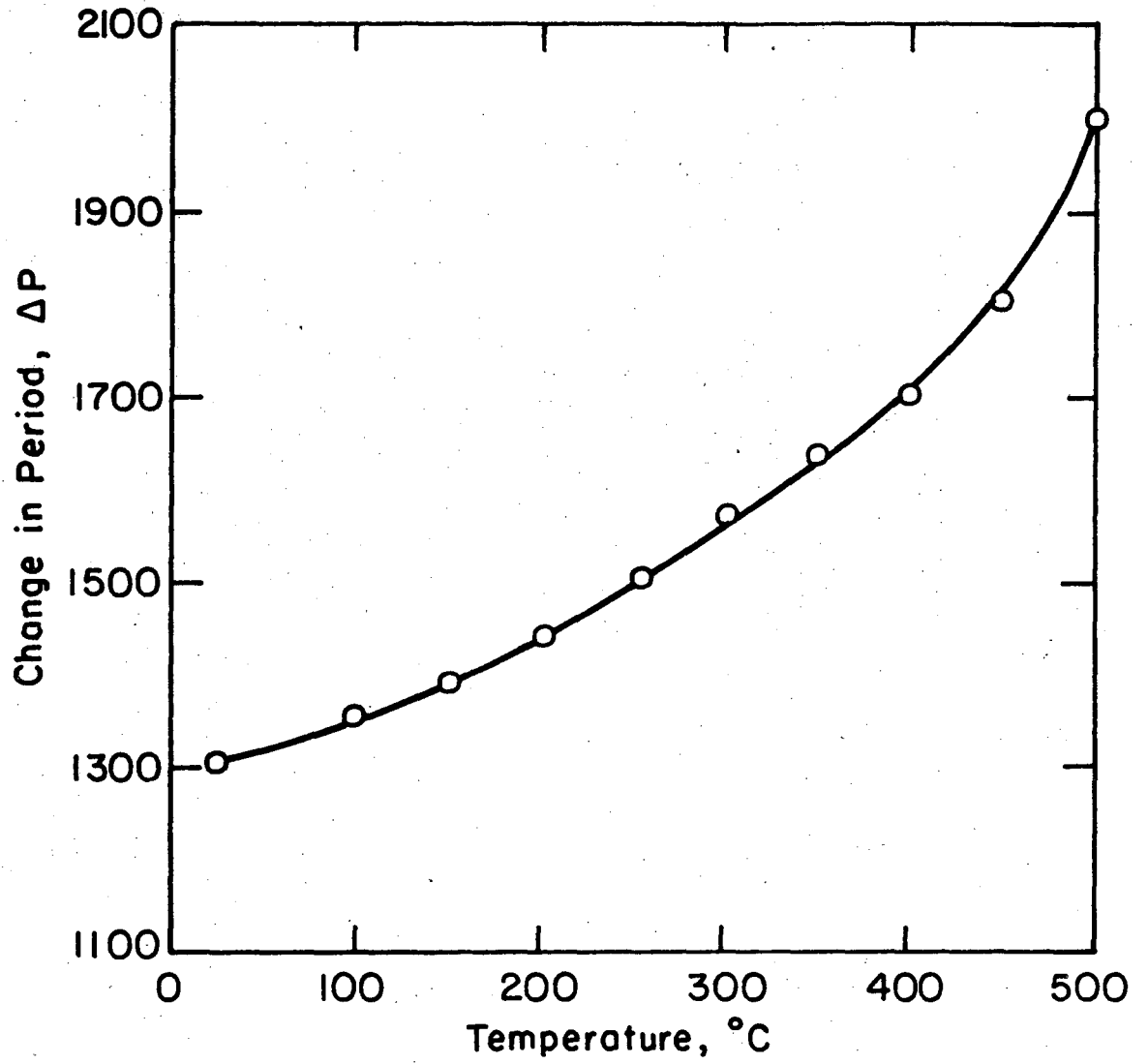
Fig. 1.



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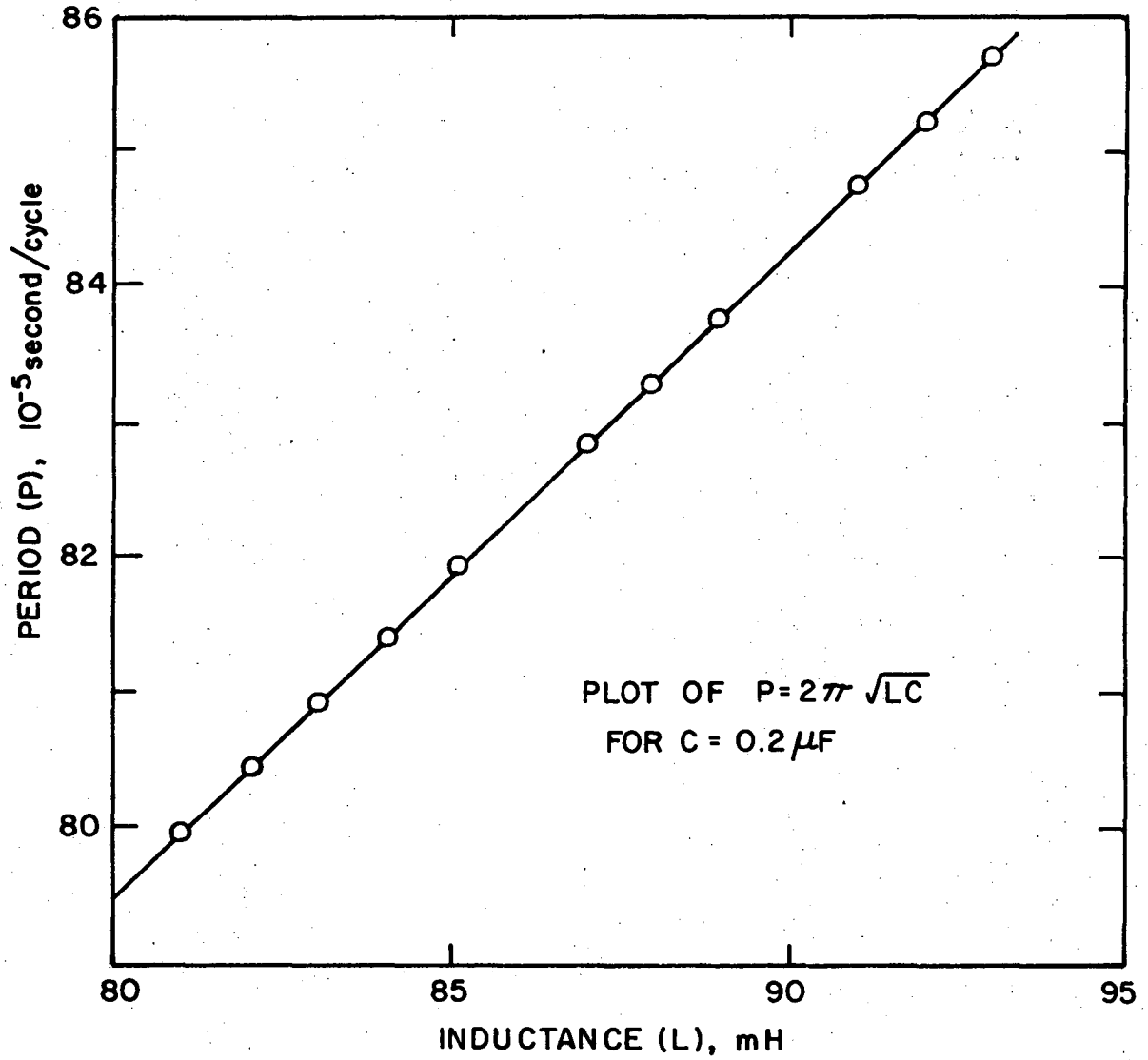
Fig. 2.





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Fig. 3.



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Fig. 4.

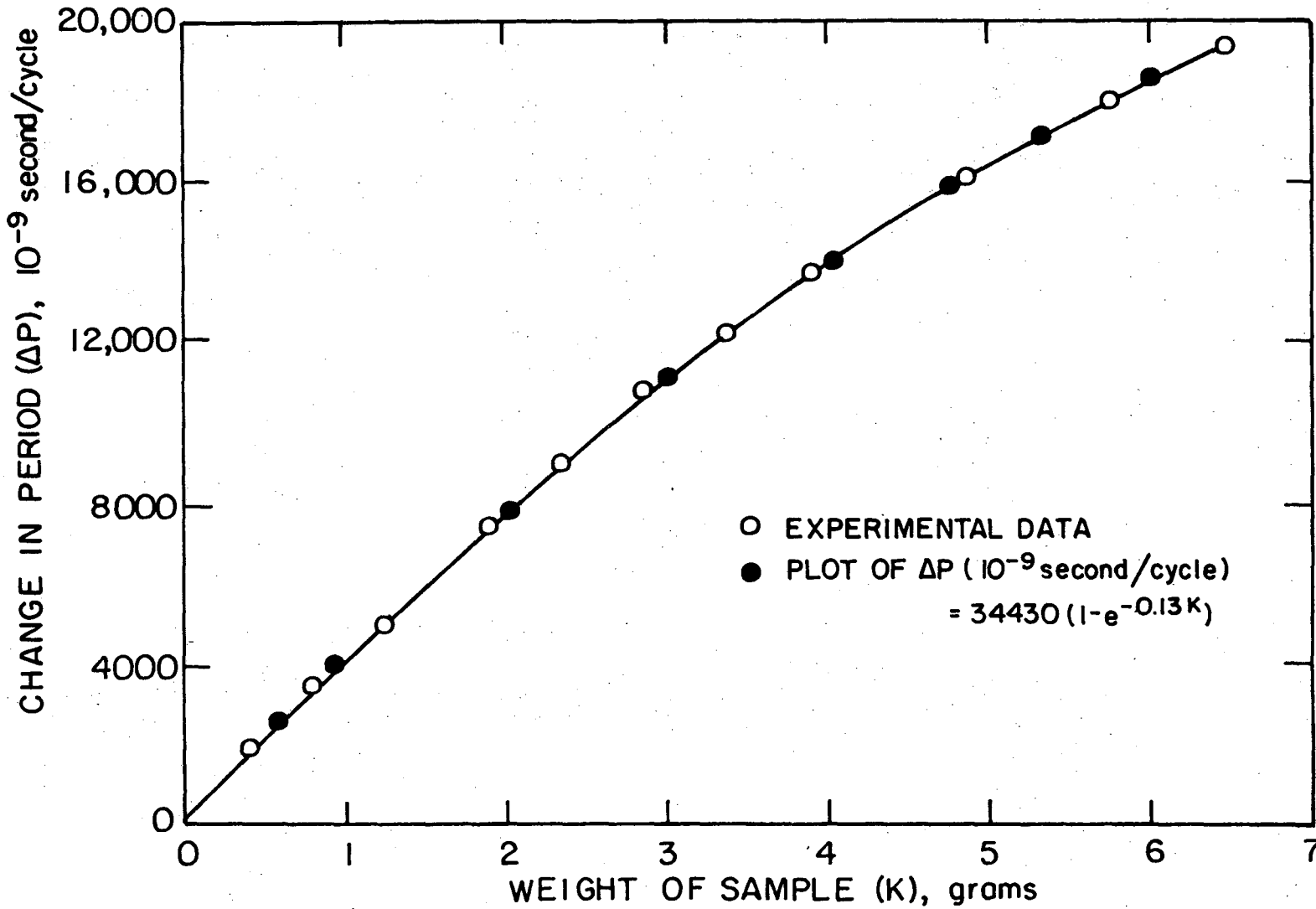
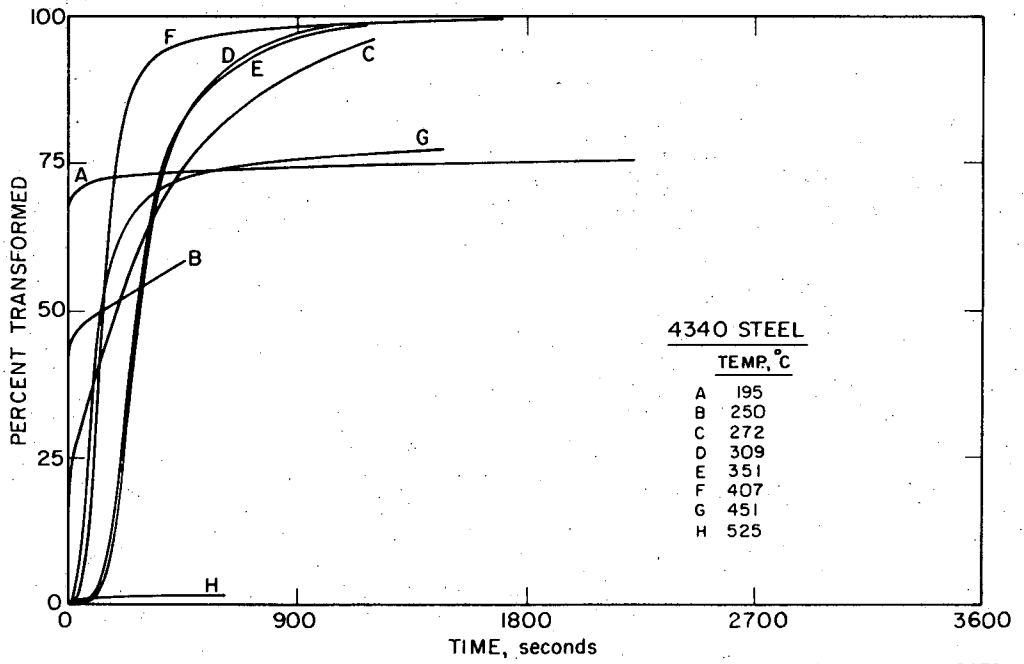
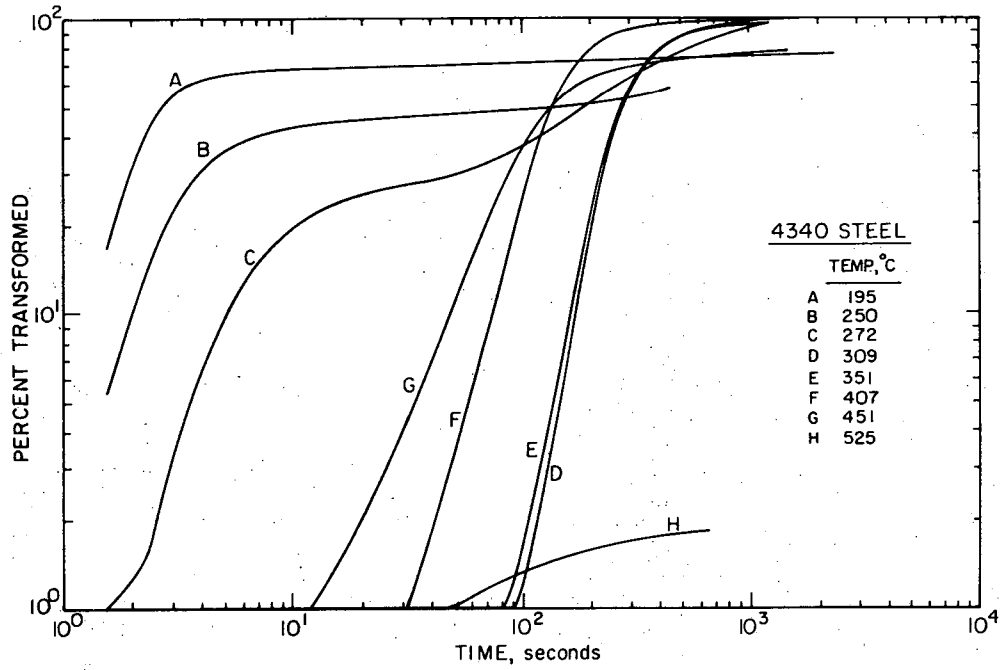


Fig. 5.

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Fig. 6.

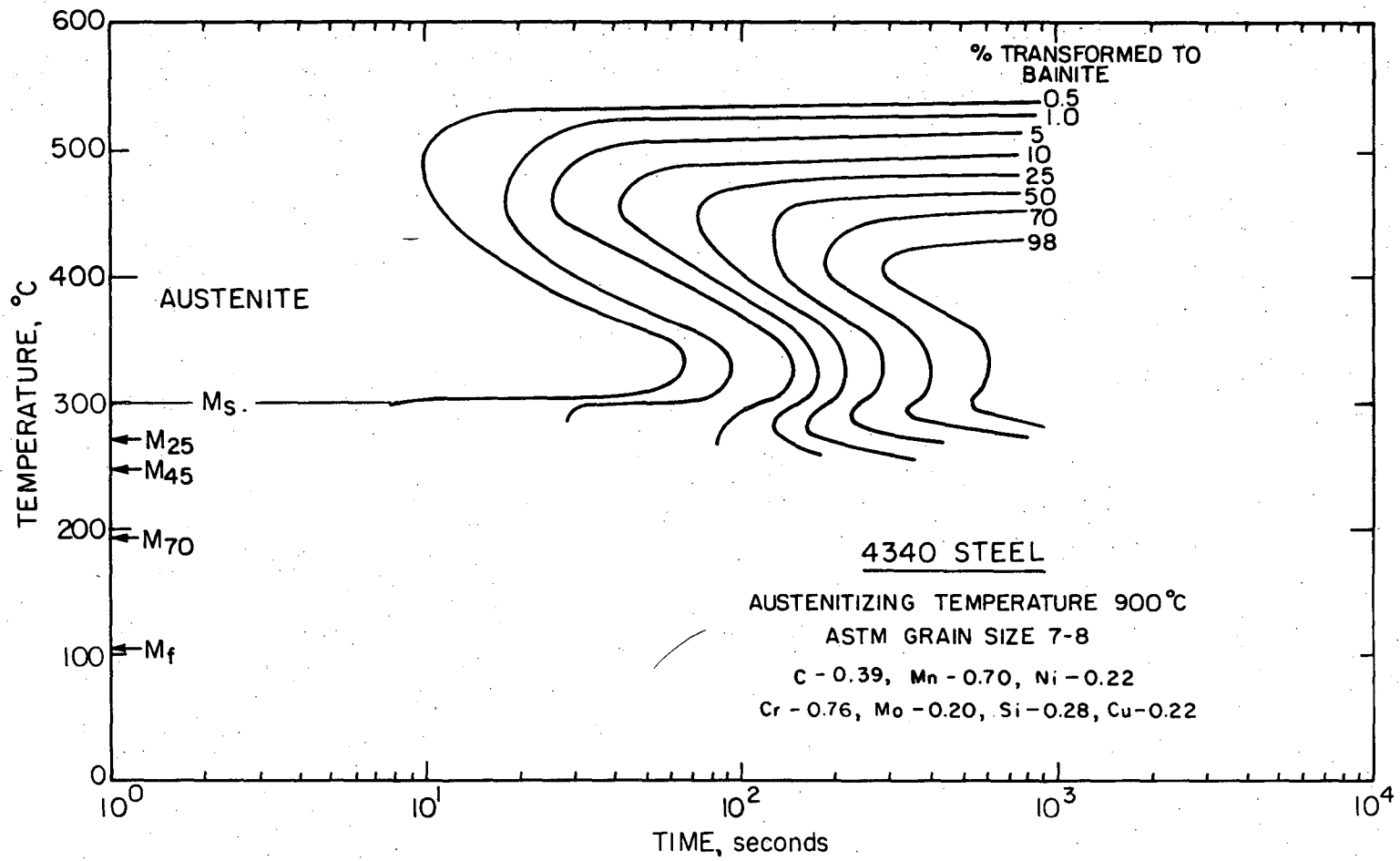
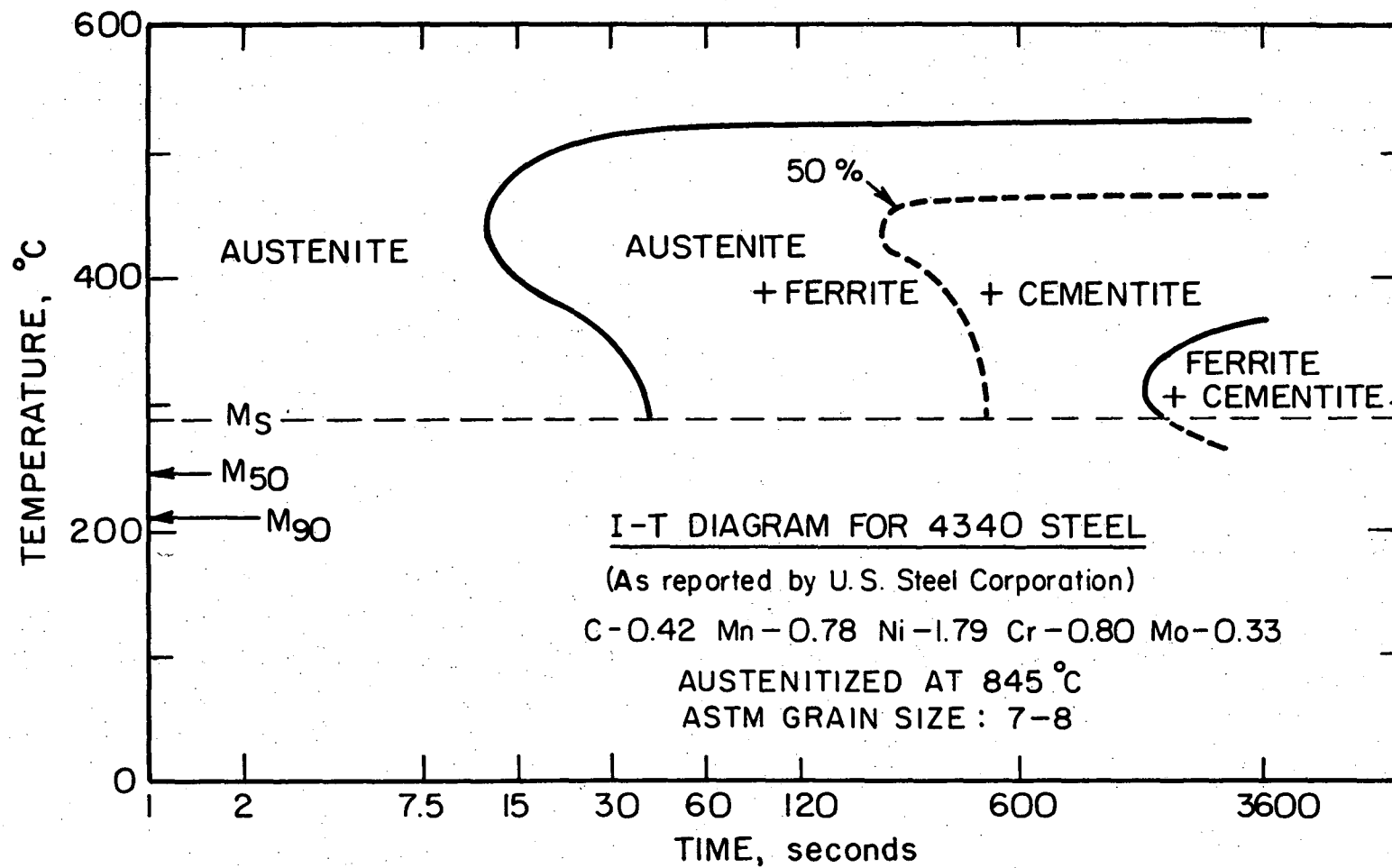


Fig. 7.



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Fig. 8.

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