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SOME DILATOMETRIC OBSERVATIONS
OF SEVERAL COMMERCIAL STEELS

THESIS FOR THE DEGREE OF M. S.

Carlos M. Heath

1931

THESIS

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SOME DILATOMETRIC OBSERVATIONS

OF

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Approved
J.E. 1931
Pulley

Some Dilatometric Observations of Several
Commercial Steels

Thesis
Submitted to the Faculty
of
Michigan State College

In Partial Fulfillment
of the
Requirements for a Degree
of
Master of Science

Carlos M. Heath

June, 1931

THESE

ACKNOWLEDGEMENTS

The writer wishes to express his appreciation and to thank Professor H. E. Publow for the valuable direction and assistance that he has generously given on this problem.

He is also grateful to Gordon Stumpf, of the Reo Motor Car Company, for the machined and carburized samples in this work.

ABSTRACT

In order to understand some of the reasons why there is shrinkage in case carburized stock upon heating through the Ac_1 critical range, a general study has been made here of the dilatation of iron, S.A.E. 1020, 1015, and 4615 steels, both carburized and non-carburized. A study of some quenched samples was also made. All these dilatation curves were taken on the Chevenard Industrial Thermal Analyser.

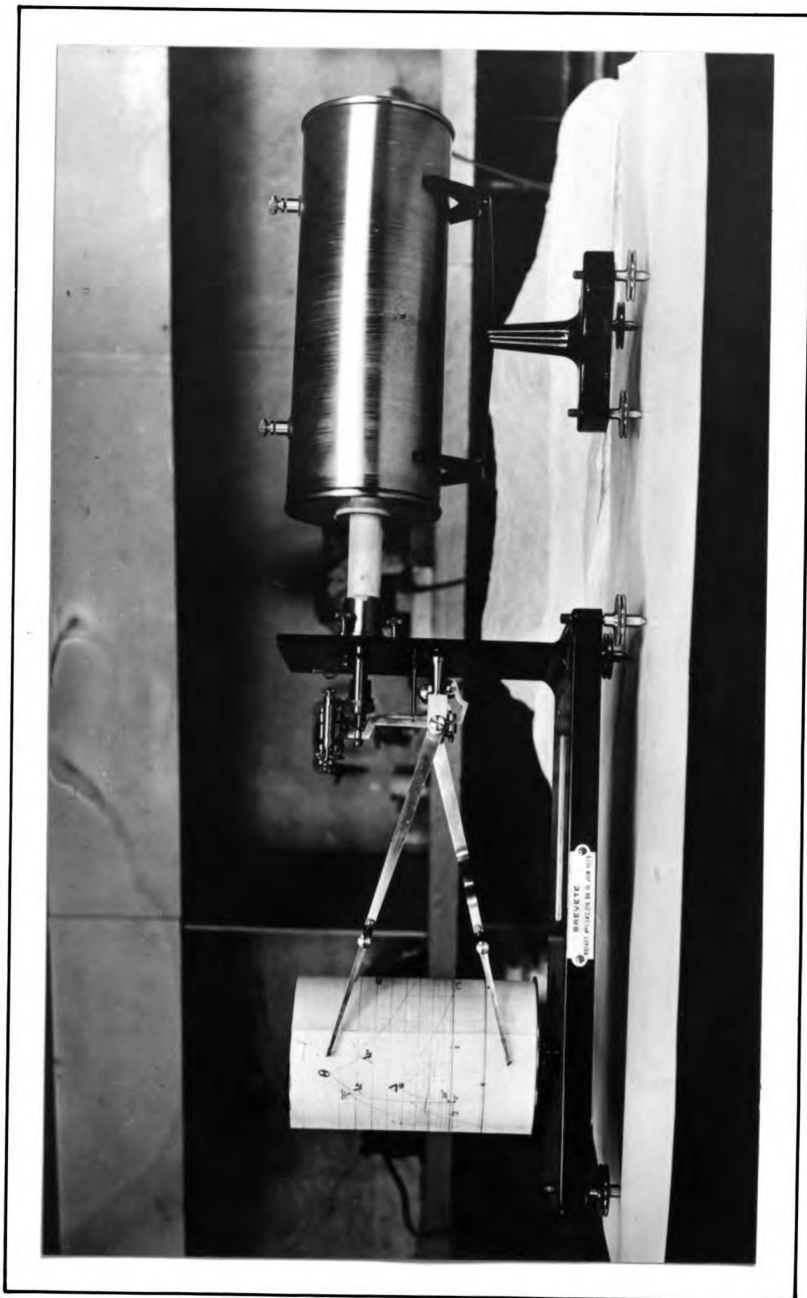
It has been found that all steels have a tendency to shrink upon returning to room temperature after having been heated above the Ac_1 range and slow cooled. On any of the above steels except iron and carburized stock, if all strains are removed by a slow heat, a soak, and a slow cool through the critical ranges, then the piece may be heated again, up and down, slowly through the critical range and return to its original size. Any piece that is under a mechanical strain or one due to heat treating strains will invariably decrease in size after heating through the critical range. A piece of steel that has been quenched will reduce in size, if it is only heated to 100°C . Any steel, if it is in the pearlitic state seems to return to its original size, if it is not heated over the Ac_1 range.

Case carburized stock and low carbon iron will shrink no matter what condition it is in, if it is heated above the Ac_1 range and slow cooled. Quenching of course will expand steel. If these two stocks are perfectly annealed from above the critical also as to remove work strains and any other strains present, the piece will not shrink nearly so much upon reheating through the critical ranges.

INTRODUCTION

It is known in practice that case carburized stock tends to shrink and warp upon heating through the critical ranges. The dilatation of two carburized stocks, S.A.E. 1015 and 4615 were investigated to find out what caused the shrinkage and how to prevent it, if possible. A Chevenard Industrial Thermal Analyser was used for the purpose. The preliminary results showed that the carburized stock always shrank upon heating through the critical range and slow cooling, or at least cooled slowly enough to keep it in the pearlitic state. We not only found out that the carburized stock shrank but also the non-carburized stock had tendency to shrink although not so marked and very erratic. This led to a general study of non-carburized stock together with a very low carbon, in order to explain the more complicated dilatation of the case carburized stock.

Figure 1.
Chevenard Dilatometer

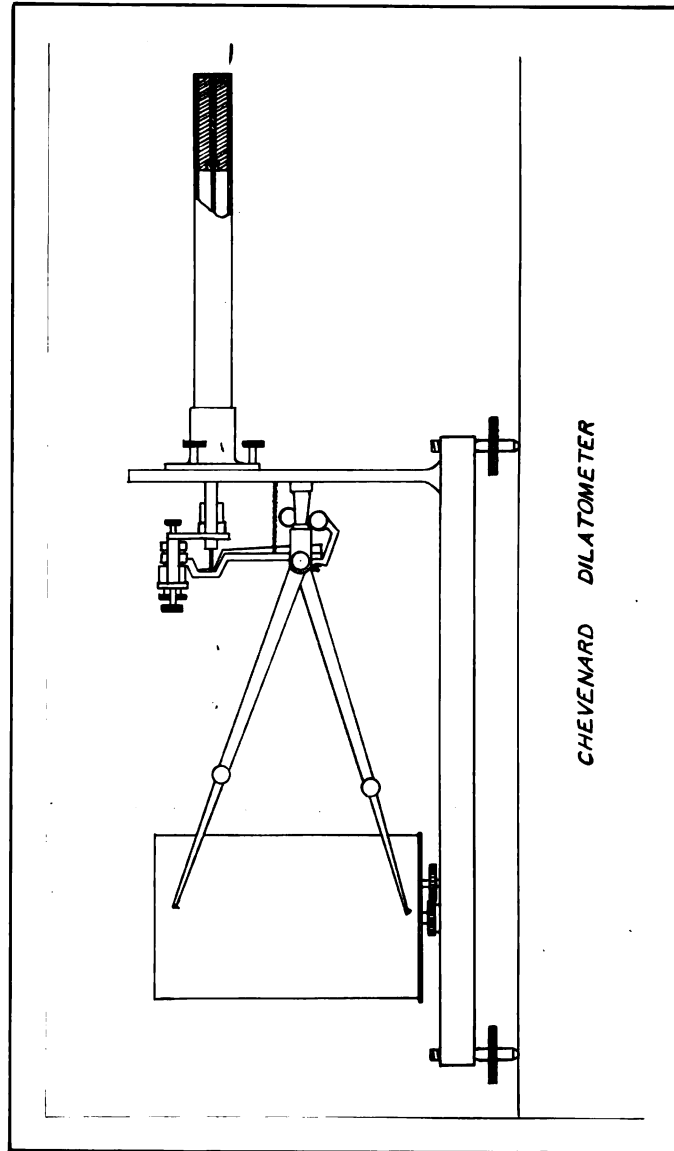


APPARATUS

The Chevenard Industrial Thermal Analyser used is a simple but delicate form of dilatometer. It consists of a silica tube holding a sample. Its expansion upon heating is transmitted by a small silica rod to the machine proper where it is enlarged approximately seventy times by a system of levers. This is recorded on a drum moved by clock work around a vertical axis. The temperature is automatically recorded at the same time and on the same drum by the expansion of the standard piece of non-corrosive alloy known as pyros. Pyros has a fairly constant rate of expansion, at least to 1000°C. the limit of the machine.

A photograph of the instrument is shown in figure 1 with a furnace covering the silica tube. Figure 2 shows a side elevation of the apparatus without the furnace and with a cut in the silica tube to show the piece inside. The steel sample is a cylinder, 55 millimeters in length and sixteen millimeters in diameter. The standard pyros is fifty millimeters in length and four millimeters in diameter. This is placed within the cylindrical hole which runs through the steel sample horizontally or rather longitudinally. Both pieces rest firmly against the end of the tube. The expansion is transmitted from the two pieces by

Figure 2



silica rods to two brass rods running through a bushing in the cast iron framework of the machine. These rods transmit the motion to two arms, which are suspended on points, by means of a moving contact. This motion is in turn transmitted to the pen arms also by sliding contacts. The mechanism is kept taut by two springs, as shown in the drawing. The movement of these arms is measured by a circular scale which is calibrated in 100°C. units on one side and expansion in 2 millimeters per millimeter times 10^{-3} on the other. These units of expansion are so large that $\frac{1}{20}$ of each space may also be measured down to 5 degree units. This apparatus has checked results very well measurements taken on an interferometer.

An auxillary silica tube was used with the apparatus in this experiment which was much easier to handle and was just as accurate in most respects. This other tube was made for unmachined specimens. The tube in the far end was cut away a little longer than the length of the specimen and to the axis of the tube. A silica platform supported the specimen and the pyros which are now placed side by side. The pyros used here is 50 millimeters long and has an 8 millimeter square cross section. The sample is made somewhere near the same cross section and is also 55 millimeters in length. A half circular cut out of the tube

is used to cover the piece. The specimens may be cooled more rapidly and may be removed without first removing the silica tube.

In either case pure powdered charcoal is inserted in the tube to keep away oxidation, which would naturally introduce errors into the results, if the silica is eaten away by the iron oxide. The pieces are heated with a horizontal type, tube muffle, electric furnace. The current is controlled with a finely graded rheostat, so that the rate of heat may be controlled very well. A small amount of heat may be used while cooling, so that most any cooling rate may be obtained down to an air quench. A very fast heating rate may be obtained by heating the furnace up before putting the furnace over the silica tube.

As stated before the apparatus is fairly accurate but errors are introduced, if the proper care is not used in placing the wedges behind the rods to adjust the height of the pen arms. Beside the deterioration of the silica tube there may also be a slight amount of slippage, if the piece is not placed properly. This results in one or both of the hands coming back below normal on cooling.

As stated before there are two curves on the drum graph. The upper is ruled off in 50°C. lines and the lower curve is the dilatation of the piece. The

dilatation readings are taken for every 100°C. temperature and points where there was an change in either curve. These points were plotted, dilatation on the ordinate and temperature on the abscissa. The heating curve is solid and cooling curve is dashed.

All 1020 pieces are hot rolled, flat, bar stock. The iron is hot rolled iron thermocouple wire with a carbon content of about .026 %. The S.A.E. 4615 and 1015 steel are hot and cold rolled rod stock, respectively.

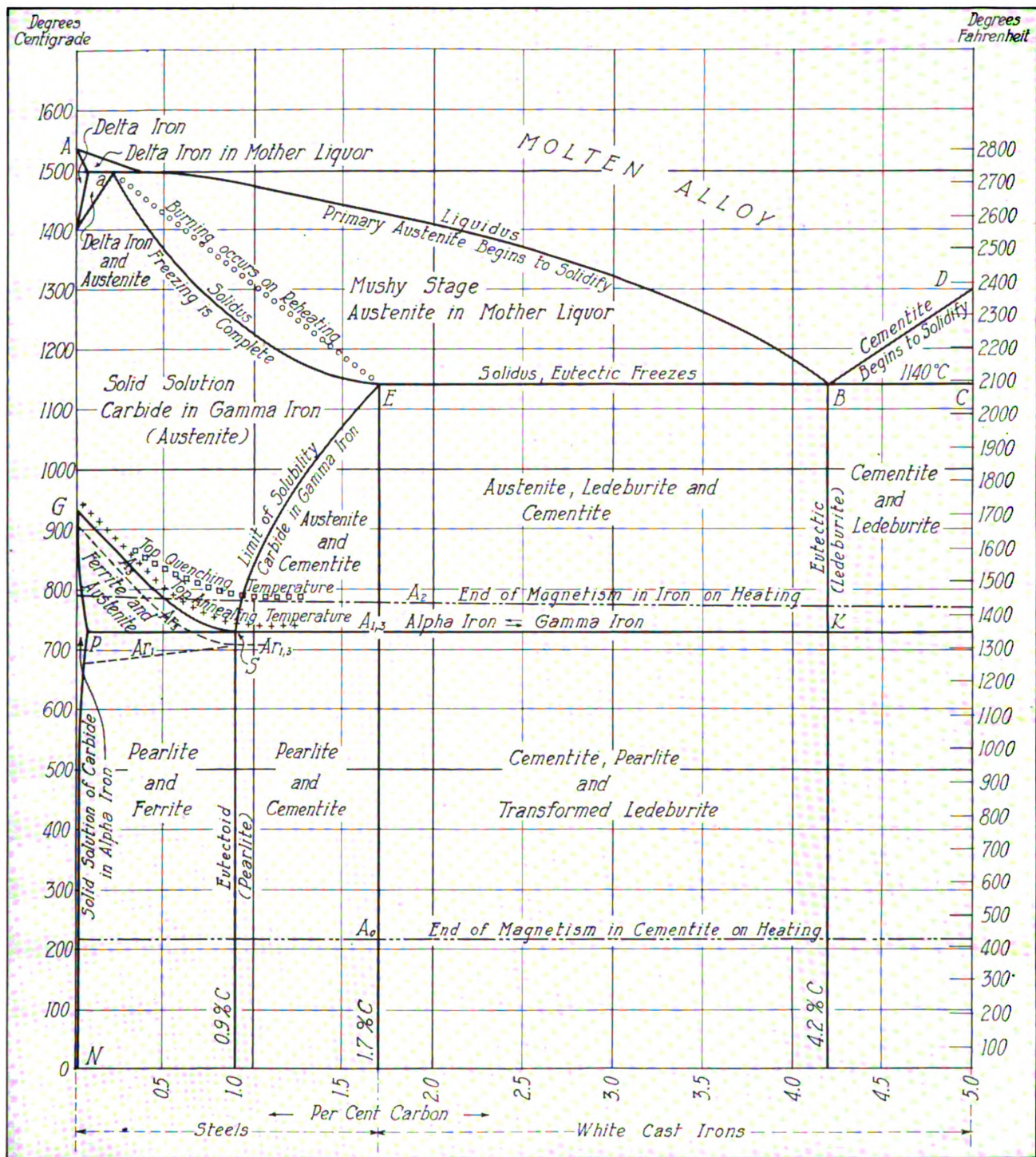
DISCUSSION

S.A.E. 1090 Steel

Plain eutectoid carbon steel is used as a standard for calibrating this machine. This is used as it has only one critical range to go thru or rather the majority of the piece has only one critical to go through, as may be seen in figure 3, of the equilibrium diagram of Iron and Iron Carbide. This steel may be heated up to the lower critical and will expand to a limit, as shown by the solid line of curve 3 in figure 4. At the lower critical the steel will change completely over to gamma iron, and the cementite going into solution without a change in temperature. This is not true with other steels as may be seen by the other curves on the same sheet. Upon cooling the eutectoid steel shrinks until it hits the A_1 critical again. The only change here until the iron is changed to alpha is the rise of about 5 or 10 degrees that takes place due to the giving off of heat of transformation. The pyros being inside the steel and very small, is very quick to record this evolution of heat. This is one advantage of the closed tube over the open one. It will be noticed that when the piece returns to its original size, as this does, that the change in expansion at the criticals is about the same for the change from alpha to gamma as it is from gamma to alpha. On cooling the curve returns practically back to the heating curve

Figure 3.

The Equilibrium Diagram of Iron and Iron Carbide



Delta Iron Regions according to Ruer, *Ferrum*, Vol. 11, 1914.

Liquidus according to Ellis, *Metals & Alloys*, April, 1930.

Solidus according to Kaya, *Science Papers*, Vol. 2, No. 4, Japanese Institute of Physical & Chemical Research.

Top Quenching and Annealing Temperatures from recommended practices, *National Metals Handbook*, 1930 Edition.

Burning line according to Jominy, 1929 *Transactions*, American Society for Steel Treating.

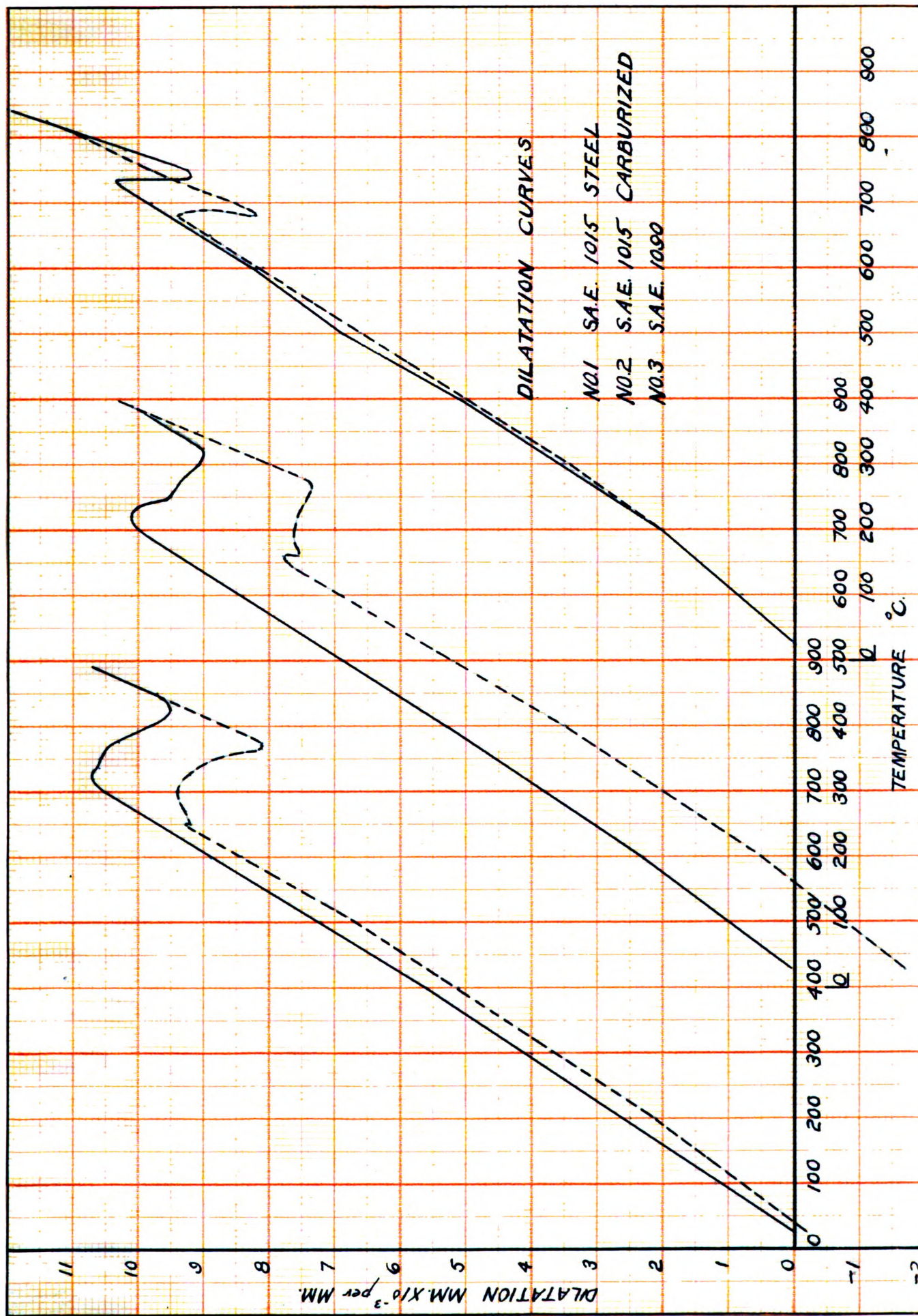
A₂, A₁, and A₀ according to Hoyt and Dowdell, "Metals and Common Alloys," 1921.

Line GPN according to Yensen, 1929 *Transactions*, American Institute of Mining & Metallurgical Engineers.

Line SE according to Honda and Endo, 147th Report, Imperial Japanese Research Institute.

Magnetic Lines according to Honda, 60th Report, Imperial Japanese Research Institute.

Figure 4.

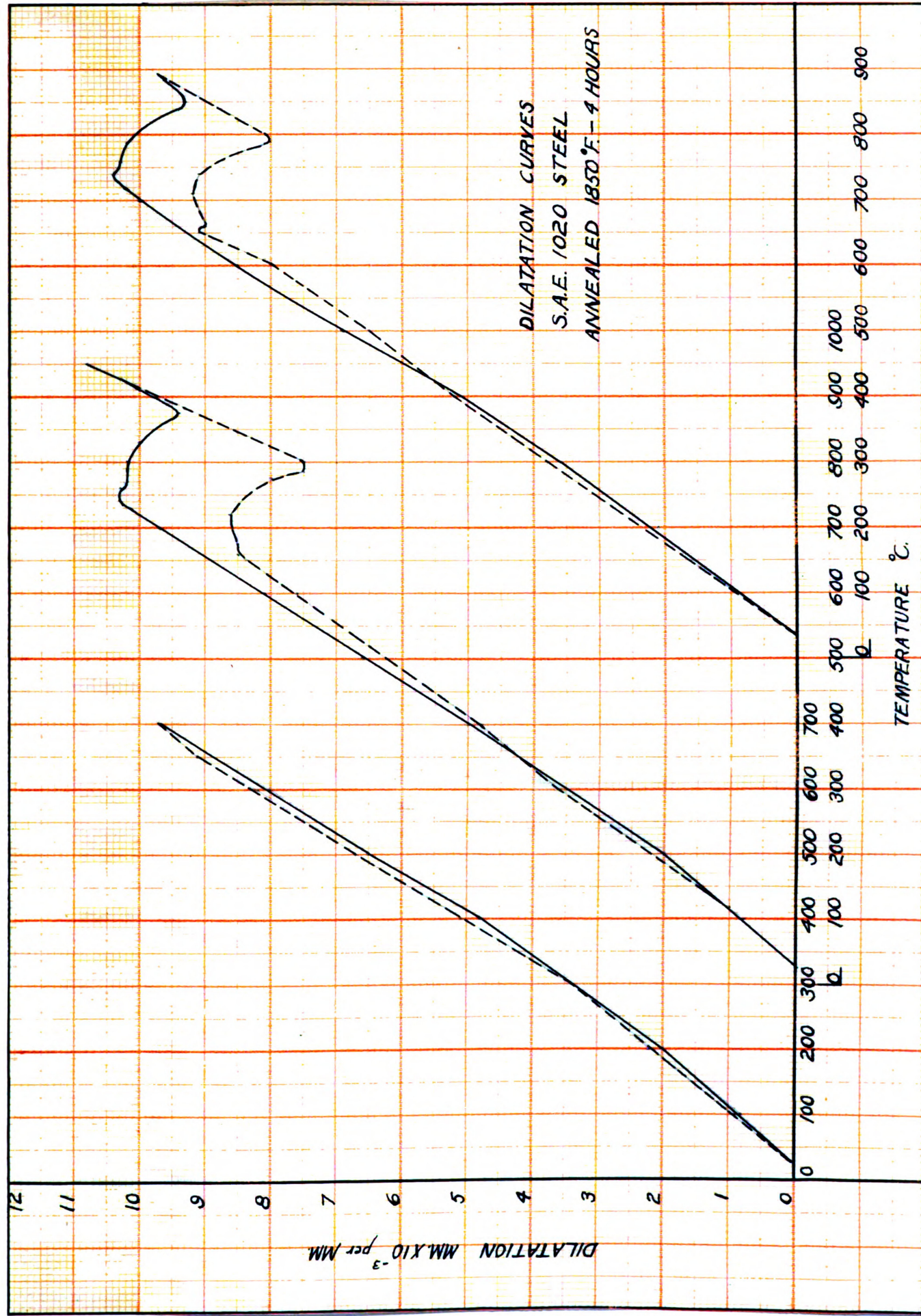


by the time the expansion of gamma to alpha is complete. This must be true, if the coefficient of expansion is the same on heating and cooling, to have the piece return to its original size.

S.A.E. 1015 & 1020

None of the annealed steels shrink below their original size when heated anywhere below the Ac_1 critical. Curve 1, figure 5, shows the heating curve (solid line) and the cooling curve (dashed line) come within plotting errors of each other. Curve 2 on the same sheet is the dilatation of a piece of S.A.E. 1020 brake drum stock run on the platform silica tube. After cold pressing a preliminary anneal of 1350°F . for four hours. This was heated to 950°C . and slow cooled. This piece being only one fourth pearlite does not show much reaction at the lower critical range. This may be followed also in the equilibrium diagram, in figure 3. As the heat reaches this range nothing happens for awhile. The temperature continues to rise until there is enough energy built up to throw the iron in the pearlite into solution with the cementite, forming islands of austenite in the steel and causing the slight amount of shrinkage. This accounts for the small nob on the heating curve at 740°C . From there the temperature continues to rise, the austenite continuing to absorb more ferrite to form more gamma iron, consequently more

Figure 5.



shrinkage. This takes place until all the iron or ferrite is in solution in the austenite. Then the austenite on further heating expands as shown by the dilatation curve after 370°C . is reached.

The reverse takes place on cooling. Nothing happens when the temperature lowers to the theoretical, upper critical. The temperature lowers 70°C . below the Ac_3 , in this particular instance, before enough energy is built up to throw ferrite out of solution. It then throws out quite a quantity of ferrite, accounting for the decided rise in the curve. As the temperatures continue to lower the less ferrite can be held in solution according to the equilibrium diagram and collaborated by the dilatation curve, which shows a continual expansion to alpha-iron as the temperature lowers. When the lower critical range is again reached nothing happens. The mass, being all changed to alpha except the potential pearlite, begins to shrink, and all at once there is the final change accounting for the austenite changing to pearlite. When the metal is heating through the criticals all parts not changing from alpha to gamma or that have changed are expanding even though the total mass is contracting. The curve reproduced is the resultant of all the reactions. The reverse is true when cooling through the criticals.

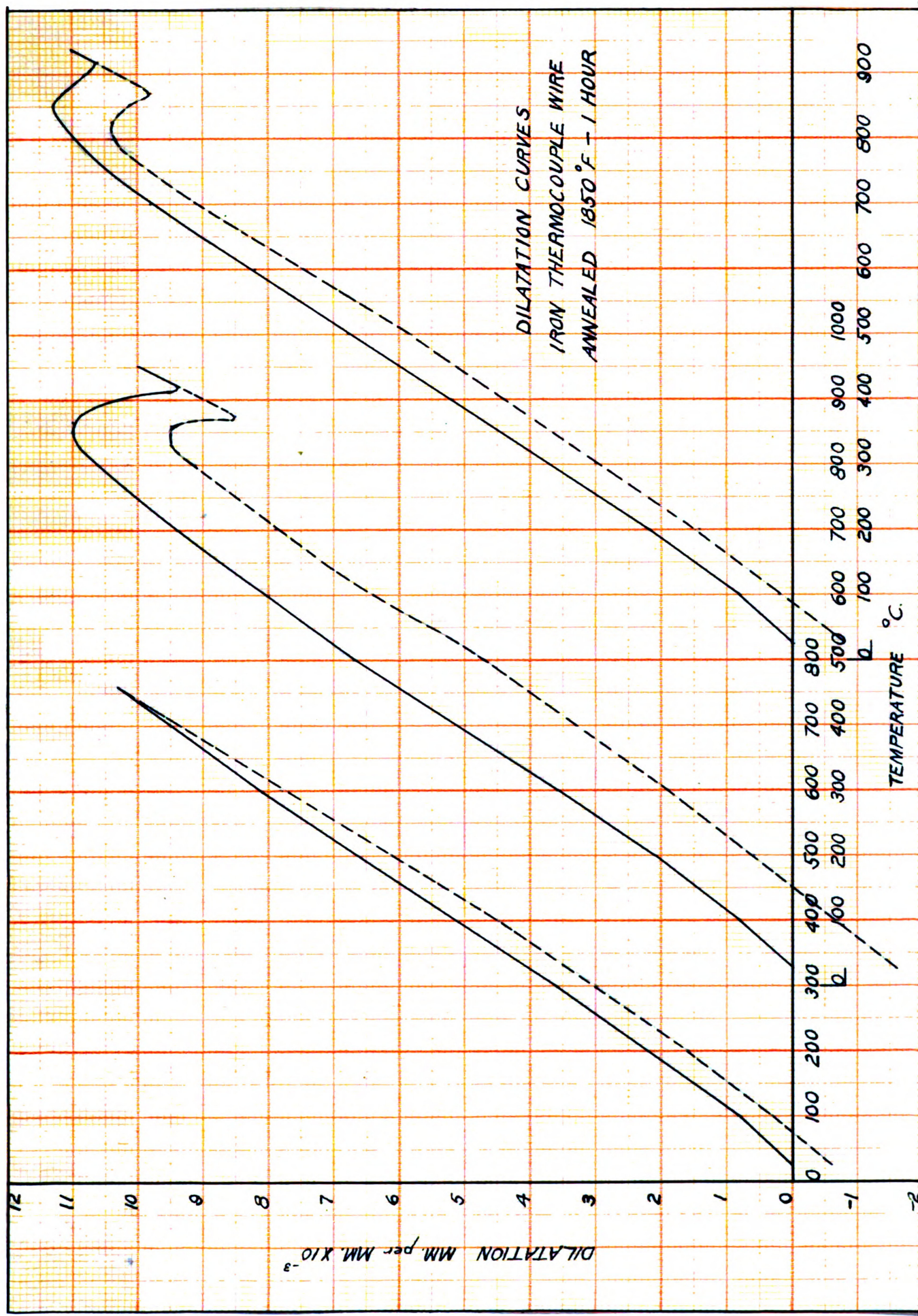
The third curve shows a similar piece of metal heated up in the same manner. Curve 1, in figure 4, shows an S.A.E. 1015 steel heated up in the same manner in a closed silica tube. This piece shows slight shrinkage, however.

In any plain carbon steel the ferrite thrown out of solution is not pure iron but at least a solid solution of iron and carbon in some form to about .03%. Upon lowering to room temperature the carbon content lowers to .02%. In most steels this effect is not noticeable but it has a more appreciable effect on iron containing in the neighborhood of .03% C, as will be shown.

Iron

Pure iron seems to shrink more than plain carbon steel for some reason or other. It isn't due to oxidation either, as plenty of charcoal was used although the open silica tube had to be used due to the fact that the low carbon iron was in the form of no. 8, thermocouple wire. Figure 6, curve 1, shows this iron heated in the region of the lower critical. Due to the fact that solid solution range in ferrite is so small and the critical is so vertical, there is no sudden change in volume before before the gamma range is reached. Besides there is no change from alpha to gamma unless the iron in the cementite is gamma.

Figure 6.



When iron is heated up through the critical ranges as shown by curve 2, on the same sheet, there is a different story. As the temperature runs over $300^{\circ}\text{C}.$, the expansion slacks off gradually, as the ferrite gradually becomes leaner in dissolved carbon according to the steel diagram. This means that there are small particles of austenite formed which are gamma and are shrinking as the temperature is increased and the residual ferrite is suddenly changed to gamma iron forming a weak austenite. This is shown by the steep decrease after $370^{\circ}\text{C}.$ is reached.

The reverse is true in cooling of iron also. The majority of the weak austenite is changed to ferrite instantly and then there is a slow decrease for a while until all the remaining austenite is changed back to ferrite. Then the decrease is more rapid. This sluggish period is represented in temperature on the equilibrium diagram by the distance between the intersections of a vertical line representing an .026% Carbon and the A_3 and the line PG.

Figures 7 and 8 show the iron, that has been annealed at $1000^{\circ}\text{C}.$ for one hour and slow cooled, at 100 and 500 diameters, respectively. The latter shows the small particles of cementite that are thrown out of solution, as the temperature passes through the range PN, on the equilibrium diagram. This happened to be one of the richer areas in carbon discovered. Microscopically

Figure 7.
Iron Thermocouple Wire
100 X

Figure 8.
Iron Thermocouple Wire
500 X

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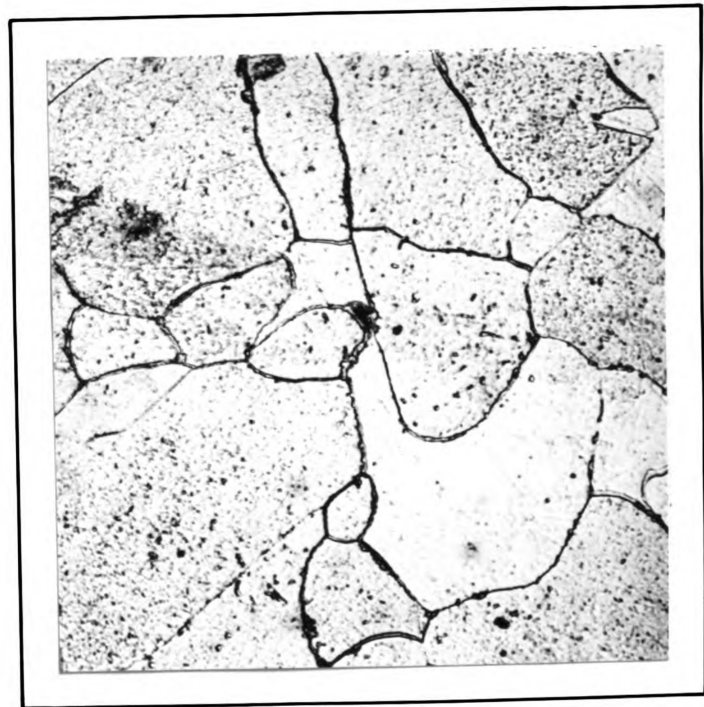
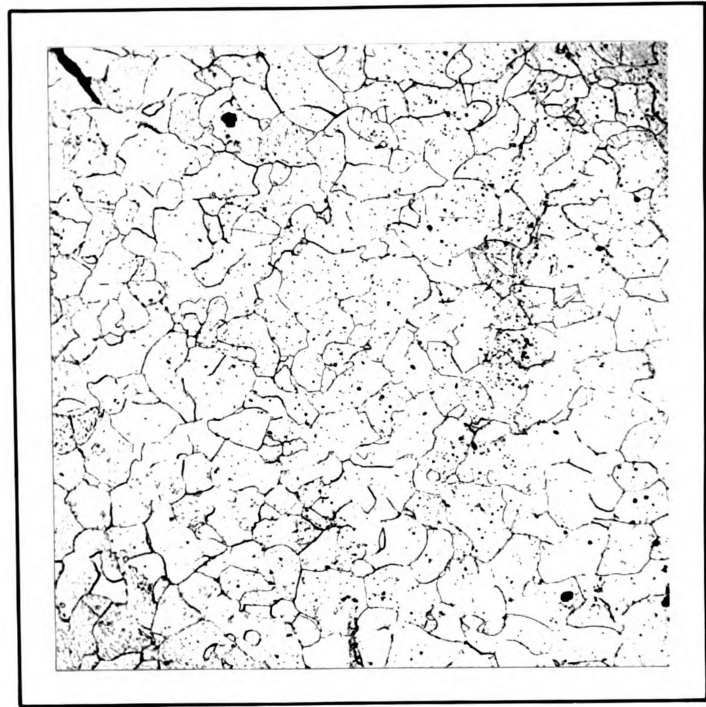
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X 111

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X 111



the carbon content varied quite a bit.

S. A. E. 4615

The alloy steels are not quite true to the iron-carbon equilibrium diagram of course. The heating curve is very similar to that of the 1015 steel except for the spreading out of the Ac_3 range, shown by the flatness of the curve between 750° and $820^{\circ}C$. on curve 2, figure 9. The Ac_1 is also lowered at least $20^{\circ}C$. On cooling the action is very much the same as may be seen by the two curves in figure 9. The cooling is even more sluggish, as the Ar_3 is at least $40^{\circ}C$. lower. The Ar_1 is lower or at least spread out so that it still has some effect at room temperature. This spreading out of the Ar_1 , probably due to the sluggishness of nickel, changes the coefficient of expansion, as also may be seen in the diagram.

Figure 10, shows the curves of five consecutive rapid heats and coolings. It may be seen how heating below the critical has very little effect on the shrinkage and as the temperature is elevated into the critical ranges, the greater becomes the shrinkage.

Other Peculiarities of Heat Treating.

There are a great many variables to be taken into account when studying the dilatation of steel. Unless these are taken into account results obtained will seem very unreliable. That is why we studied the plain carbon steels along with the carburized stock, thus

Figure 9.

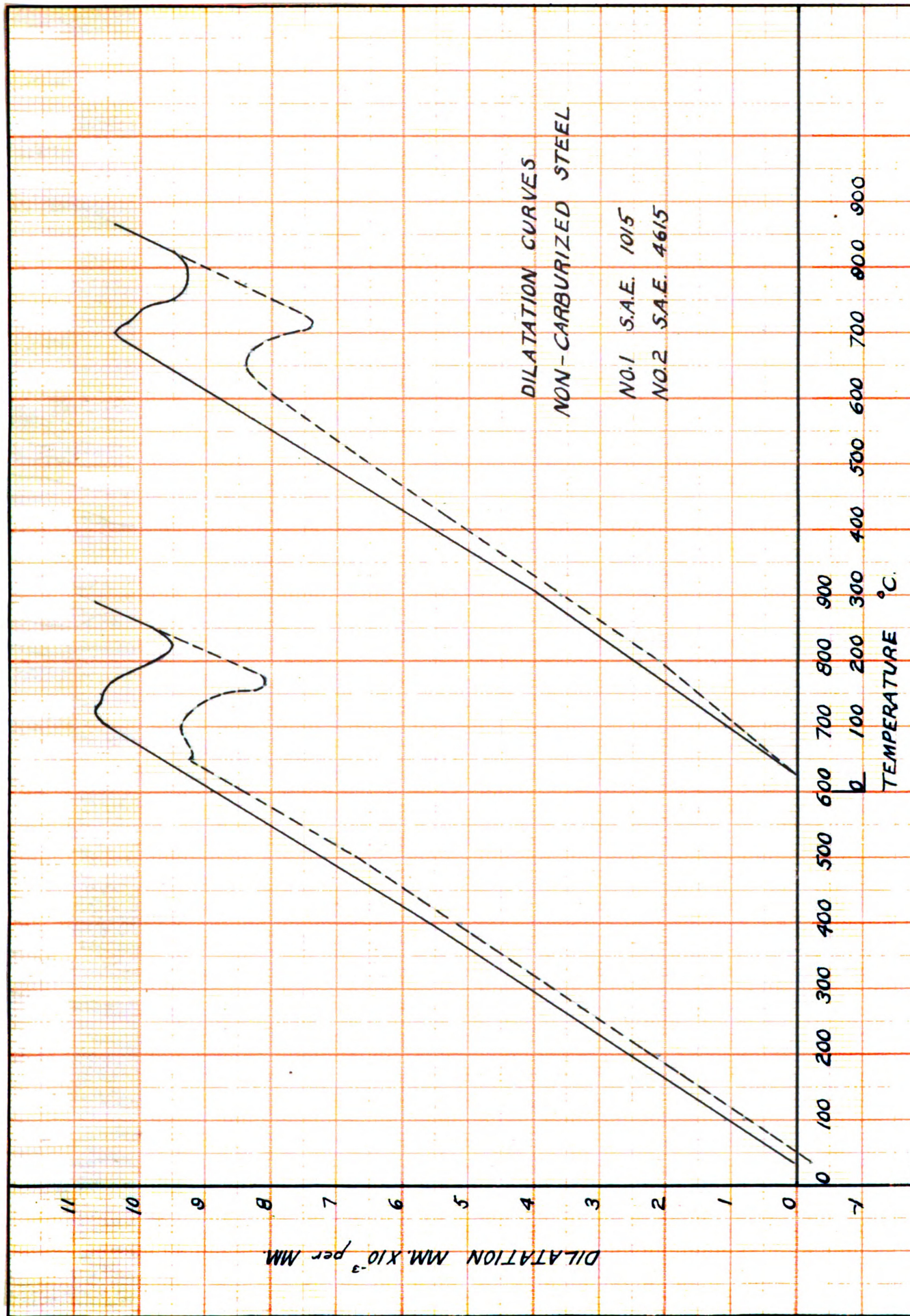
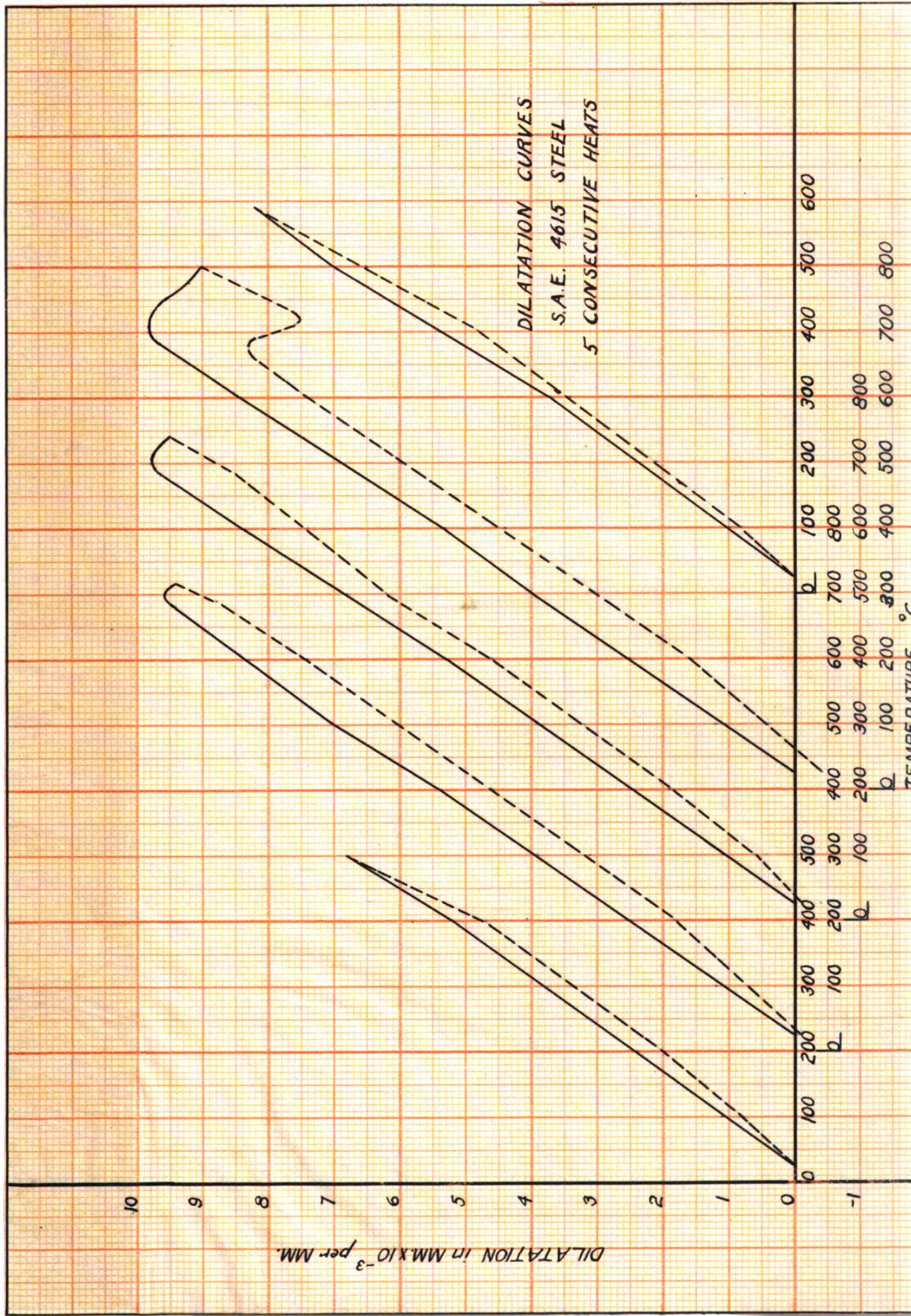


Figure 10.



eliminating some of the variables. Two great effects are, rates of heating and cooling, and other internal strains produced by mechanical work.

Everyone knows that quenching in water or oil has a marked effect on the shape and strains of a piece of steel. Figure 11, curve 1, is the dilatation curve of a piece of S.A.E. 4615 steel that has been quenched from $760^{\circ}\text{C}.$, in brine. These are both carburized pieces shown here. The second curve is a piece that has been furnace cooled on carburizing. Evidently the quenching expands the piece because when the quenching strains are relieved it should return somewhere near the normal size minus one normal annealing shrinkage. The start and finish of curve 1 must represent the quenched and normal size of the piece plus the shrinkage of an ordinary anneal, respectively. The shrink in curve 1 minus the shrink in curve 2 should give somewhere near the expansion on quenching.

Figure 12 shows the curve of a piece that had been quenched from the box. The first heat was to $190^{\circ}\text{C}.$ The second curve is a subsequent heat to $830^{\circ}\text{C}.$ and slow cool. On curves 1 of figure 11, it will be noticed that there are two changes in the coefficient of expansion on heating. These same jogs would be in the curve in sheet eleven except not so marked as the quench was not so drastic. The upper jog is present but the lower

Figure 11.

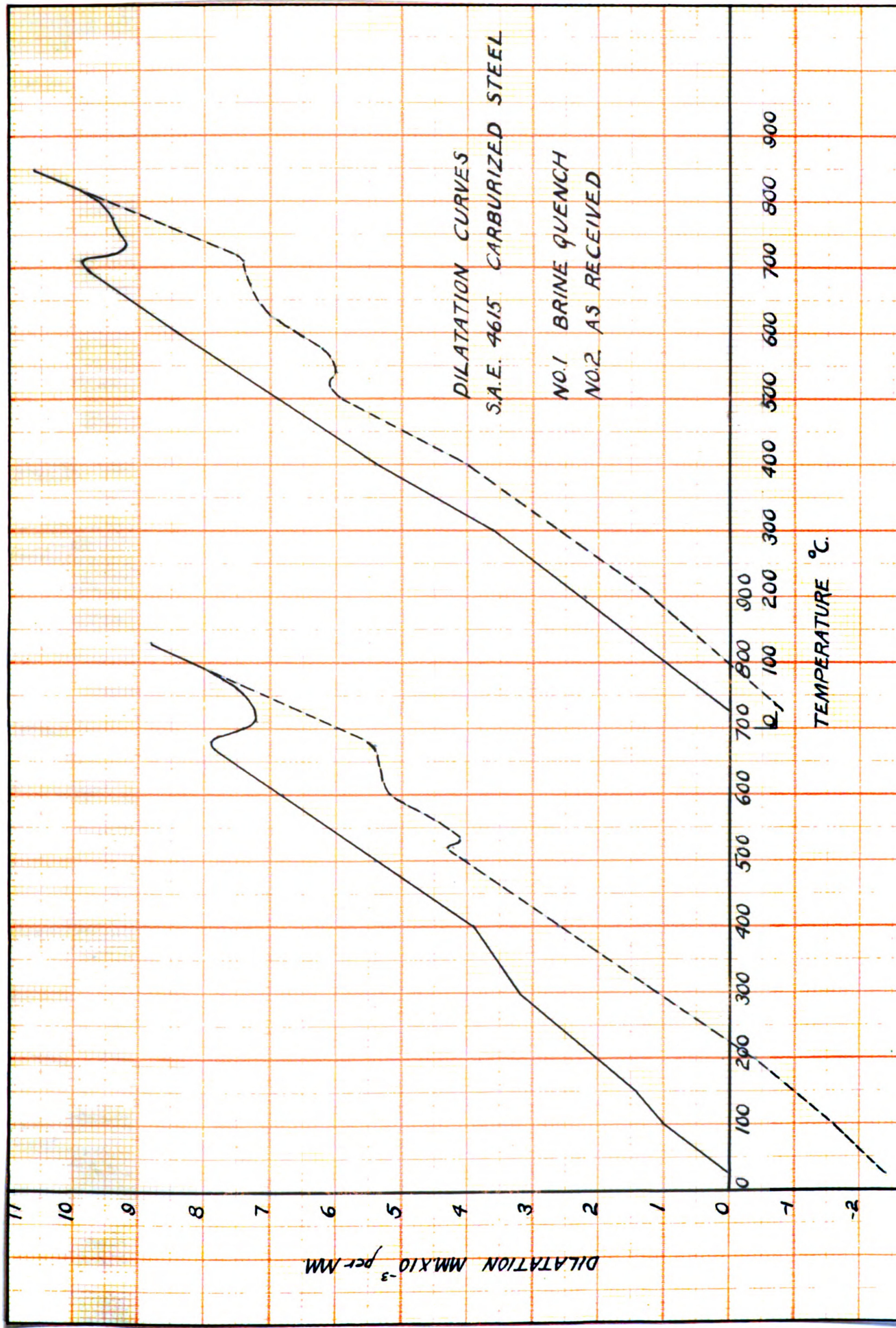
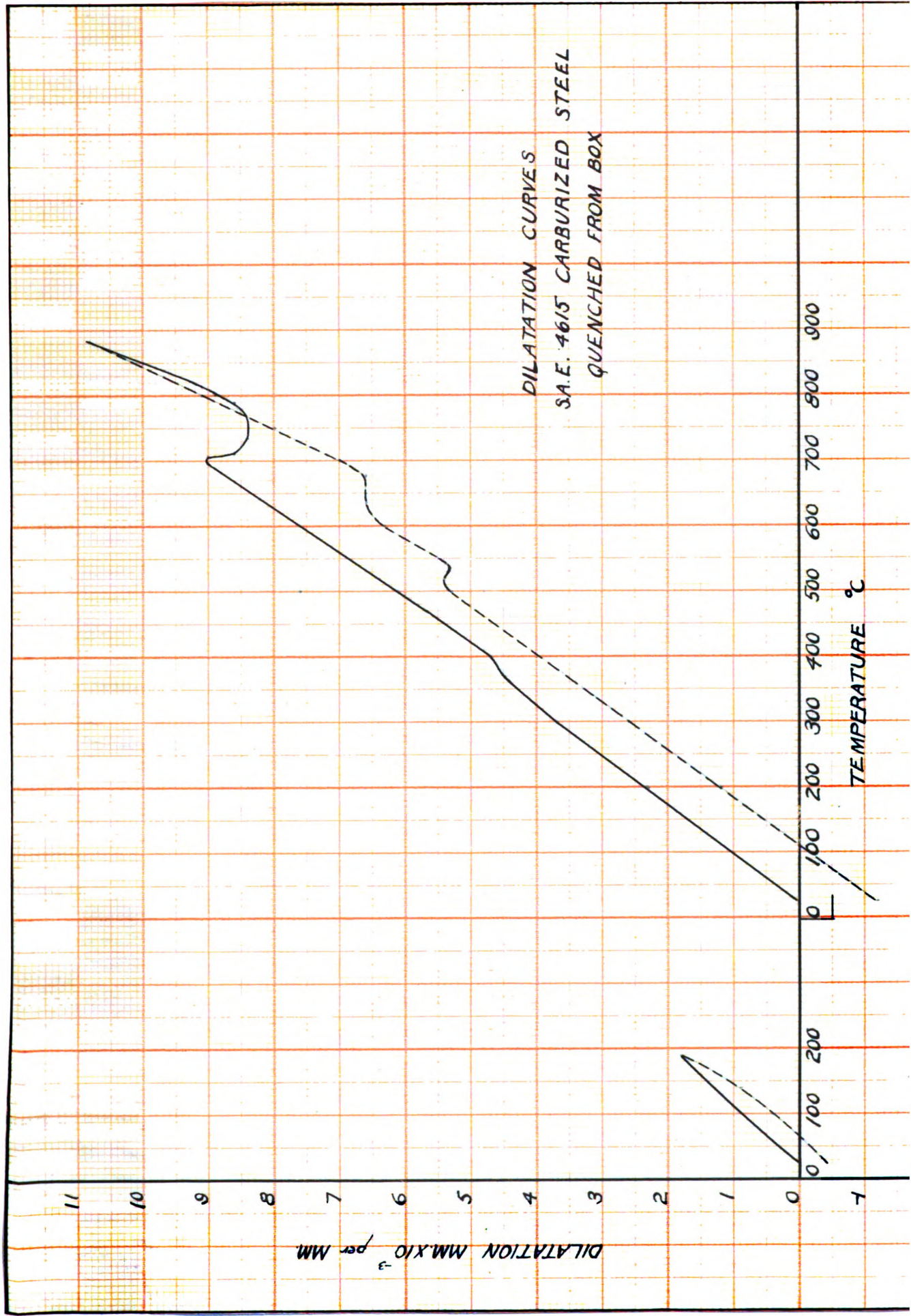


Figure 12.

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DILATATION CURVES
S.A.E. 4615 CARBURIZED STEEL
QUENCHED FROM BOX

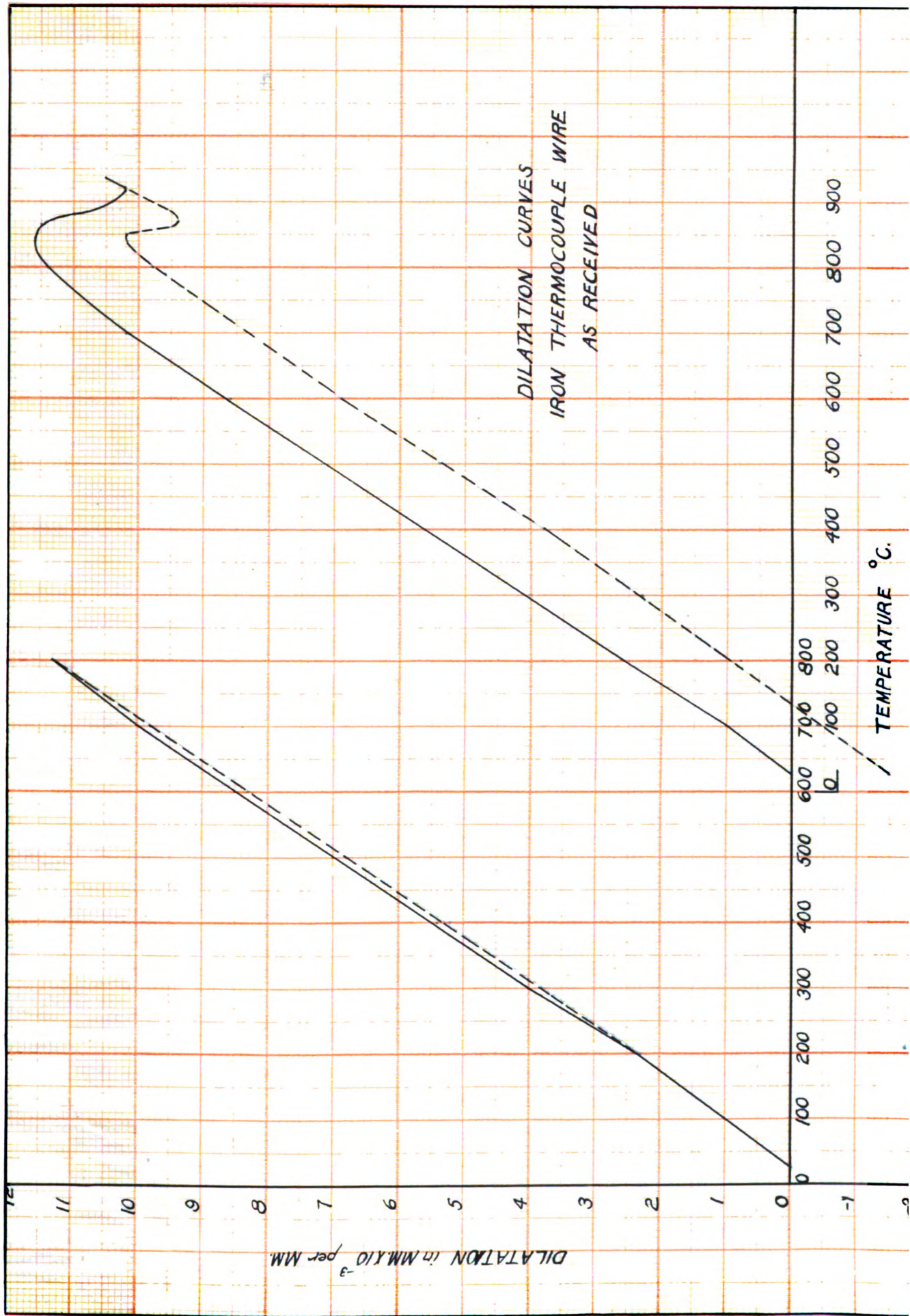


jog was removed by the 190°C . draw.

Even less drastic heats and cools have some effect on the critical temperature and the amount of expansion. This is especially true on larger pieces. If a piece is heated too rapidly the outside will heat up much more rapidly than the inside. This will cause a lowering of the total expansion of the piece at the lower critical. As the piece approaches the lower critical the outside will begin to change to gamma iron while the inside is still expanding. This will have a tendency to keep the inside of the piece from expanding. Hence the total expansion may be lower at the Ac_1 . Also at the Ac_3 the dip may not be so great due to the action of case again. On cooling the outside will again reach the Ar_3 first and the inside will be held from shrinking its full amount, as the outside will be expanding again to alpha. The same damping effect will take place at the Ar_1 . The net effect of heating and cooling is to flatten out the expansion changes in the critical range on fast heating. Of course this effect would probably not take effect on these small samples used but there is a possibility.

Probably the greatest factor causing shrinkage in non-carburized pieces besides quenching is strains set up by mechanical work. Figure 13 has two curves of iron thermocouple wire heat treated as received. And figure 6 has three curves of the same wire that has been

Figure 13.



heat treated at 1000⁰C. for one hour. The grain of the two irons are shown in the next two figures. Figure 14 is, as received, and figure 15 is the annealed piece. The first curve in figure 13 shows no shrinkage when heated below the Ac_1 and the second curve shows quite a bit of shrinkage. The fact that the shrinkage to gamma iron is so much greater than the return to alpha, made us wonder, if some of the shrinkage was not due to hot rolling or some other mechanical work. We annealed some of this iron at 1000⁰C., as shown. This showed a shrinkage when heated both below and above the criticals. We blame this on the fact that the first two curves on sheet 6 were obtained from pieces that were straightened slightly before using. The third curve was run from iron that was not strained in any manner after annealing and the shrinkage as it will be noticed, is a lot less. If time permitted this would have been checked.

Figures 16, 5, 17, and 18 show the same thing for S.A.E. 1020 non-carburized stock. Figure 17 shows the hot rolled bar stock, as received, and Figure 18 shows the same after it has been annealed for 4 hours at 1000⁰C. In comparing sheets 16 and 5, it will be noticed that the shrinkage of the unannealed stock is much greater. Also the maximum expansions at the Ac_1 are inclined to be greater.

In looking over all these curves it will be seen

Figure 14.
Iron Thermocouple Wire
As Received
100 X

Figure 15.
Iron Thermocouple Wire
Annealed
100 X

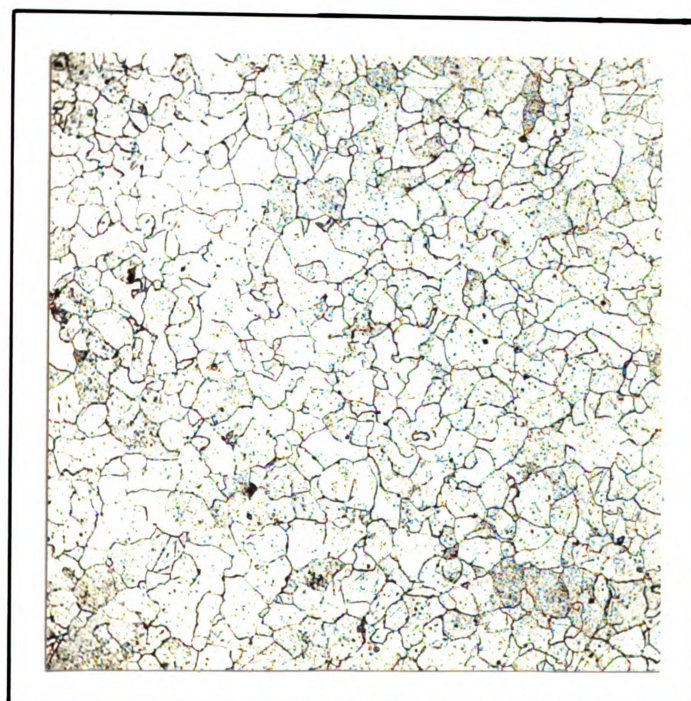
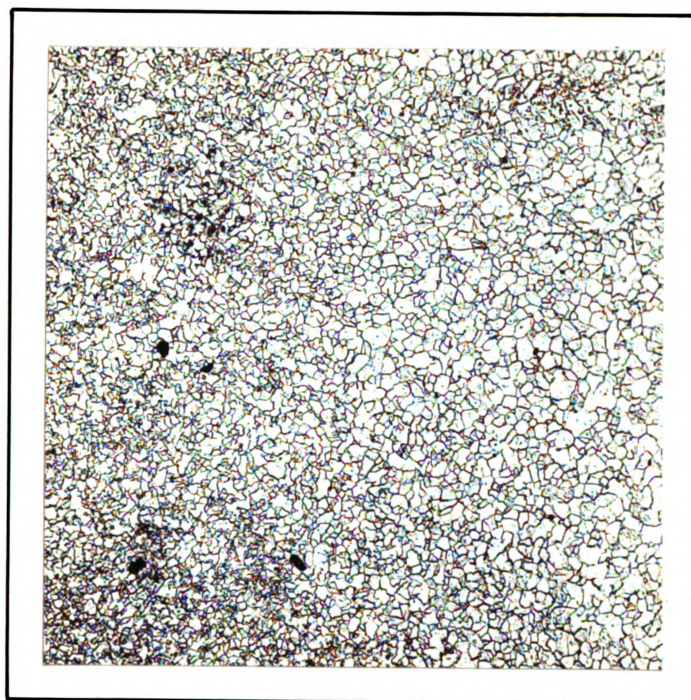


Figure 16.

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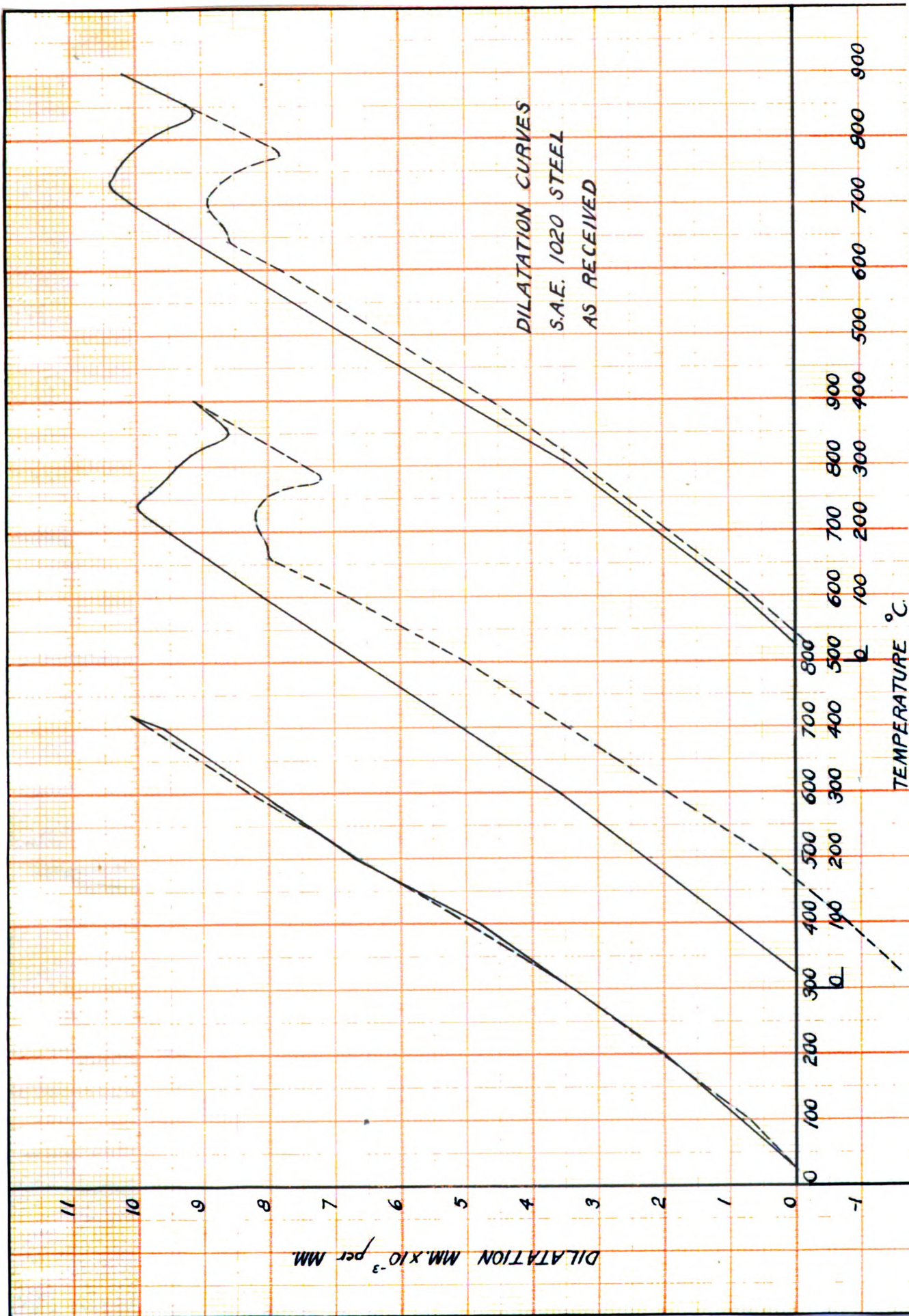


Figure 17.
S.A.E. 1020
As Received
100 X

Figure 18,
S.A.E. 1020
Annealed at 1000°C.
100 X

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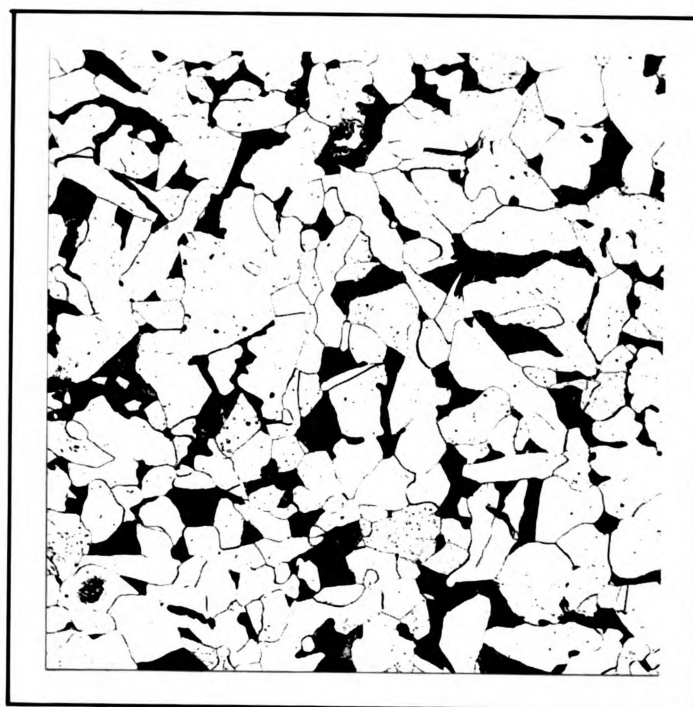
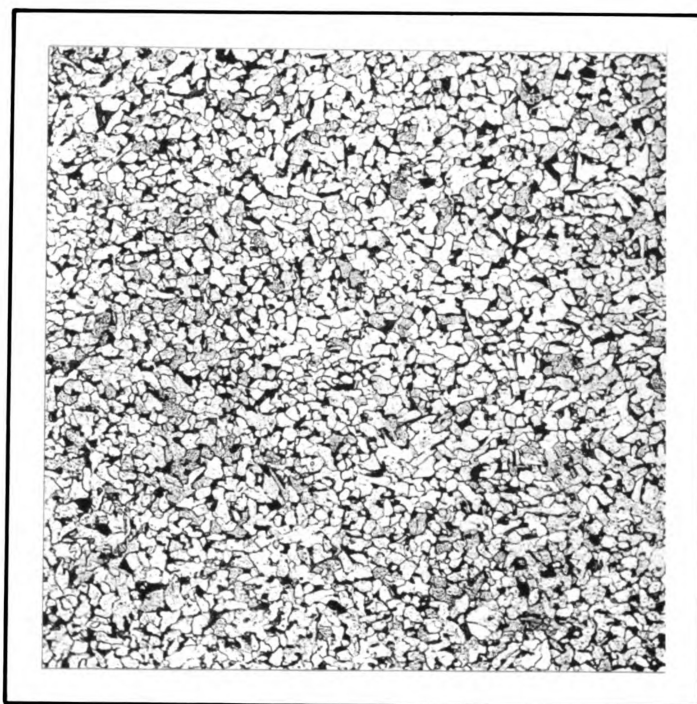
X 11. 11.

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X 11. 11.



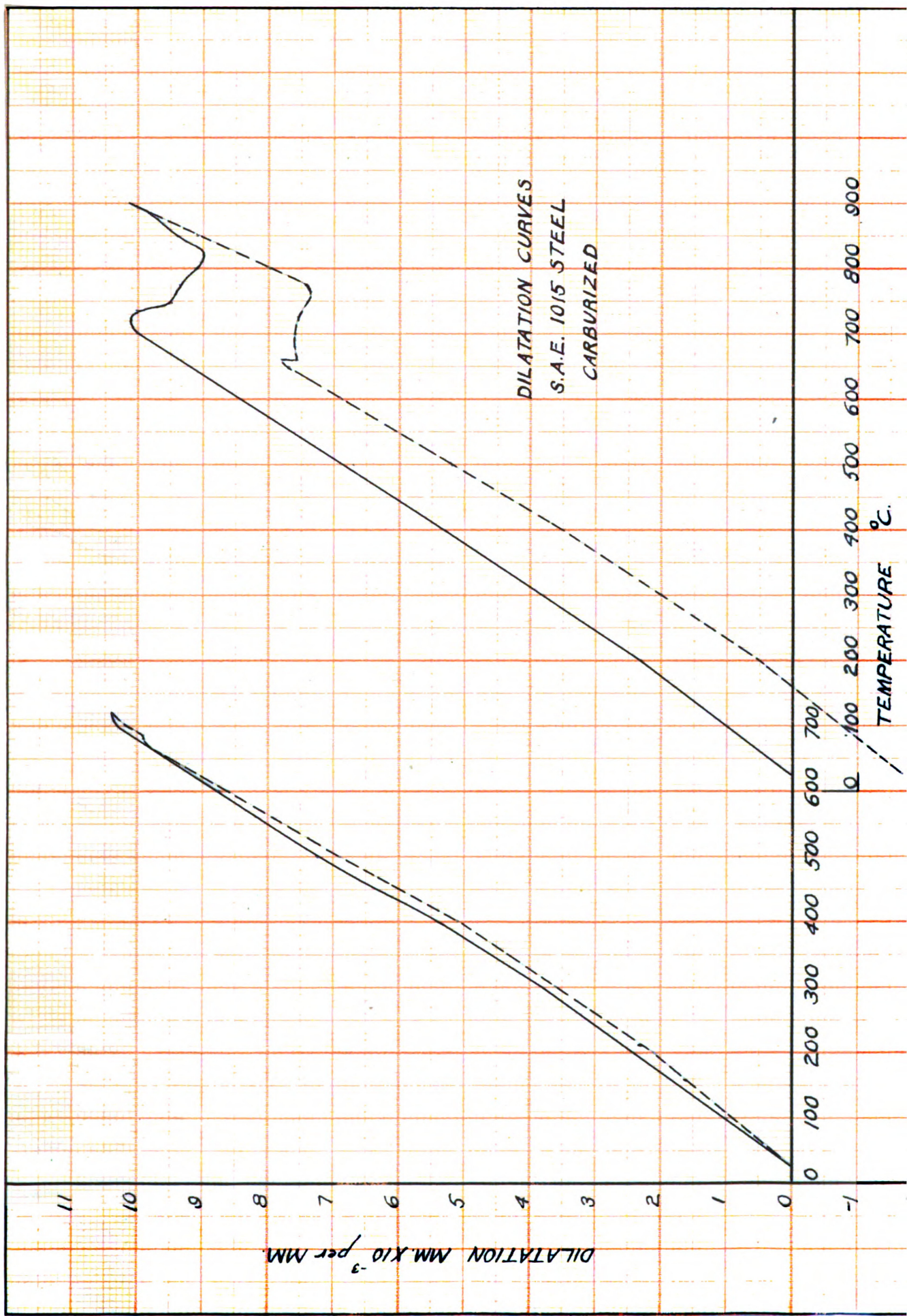
that there is a great deal of uncertainty about these curves, as to the amounts of dilatation in either direction. Part of this may be due, especially in iron, to the fact that the line GPN is so nearly vertical in figure 3. The composition may vary from nearly zero carbon content to over .026%, which is the average carbon content. Different pieces might act quite differently on annealing, as a consequence. This effect may be noticed in steel, the lower its carbon content. For once in a while S.A.E. 1015 stock will have a tendency, even though it has been annealed previously, to shrink.

It is difficult to say how much effect any of these variables will have on a piece, without a much more detailed study on each different phase, such as, rates of heating and cooling, temperatures gradients and mechanical strains.

Carburized Stock

All 1015 and 4615 carburized stock has been cooled in the box and in the furnace, except for one piece that was quenched from the box in oil, at 920°C. All pieces were originally cold and hot rolled. The 4615 were rolled hot and the 1015, cold. Figure 19 shows two curves from a piece of carburized, 1015 stock, one heated just to the critical and the other well through the criticals. As may be seen there is plenty of shrinkage in the annealed piece. The shrinkage of carburized

Figure 19.



pieces is consistent in all cases, also. In fact several pieces have been heated up and down through the criticals for a number of times and the total shrinkage is usually the exact multiple of the shrinkage of one single heat.

Going back to figure 4, we may see why this shrinkage takes place. On curve 1 you will see that the eutectoid constituent has little effect. As this carburized piece is heated up the steel begins to shrink at about 730°C . The core stops shrinking but the case keeps shrinking as much as the core will let it, as shown on curve 2. The core finally gets the upper hand and the piece stops shrinking markedly. The temperature increases, the case wishes to expand but the core is still changing to gamma iron. Evidently the shape of the curves is due to the strength of the phase change balanced against the strength of the dilatation of the piece. However, when the alpha is all changed to gamma the steel expands again.

Upon cooling, the case is still trying to shrink while the core is trying to expand after the A_{r3} is reached. The curve is flattened out quite a bit. At the lower critical the case wishes to expand and the core is then holding back and this expansion is also being suppressed. The total result of these suppressions being that the piece as a whole shrinks. Another thing very noticeable is the lowering of the maximum expansion at the A_{c1} , although the temperature of the A_{c1} is the same in either case. The coefficient of expansion is lower than the non-carburized 1015 stock from the lowest

temperatures. It seems to follow an average between the two curves until 700°C. is reached and then it falls off rapidly. This falling starts at the same temperature as the non-carburized stock but at a lower point of expansion due to the case. The case being the same composition as 1090 stock (eutectoid), acts as nearly like curve 3 in figure 4 as the core will let it. Consequently the the case will not reach its maximum height as the core is expanding hardly at all for about 20°C. before the Ac_1 is reached. This might be also be effected by a temperature gradient but it is doubtful. Possibly there is a better explanation.

Figure 20 brings out the same effects on 4615 carburized stock. It also has the same shrinkage and lowering of the Ac_1 dilatation. The carburized case brings out very well the submerging of the Ar_1 . This critical was not shown in the non-carburized stock due to the lack of eutectoid material.

Figure 21 shows the carburized 4615 stock heated to just below the Ac_1 and through the Ac_3 . Although the Ac_1 expansion is lowered the coefficient of expansion is the same on heating and cooling, when heated below the lower critical, as there is no shrinkage.

Figure 22 gives a comparison in the dilatation curves of carburized 1015 and 4615 steels, again showing that 4615 is a much better steel to use for carburizing as far as dilatation is concerned. Of course this curve

Figure 20.

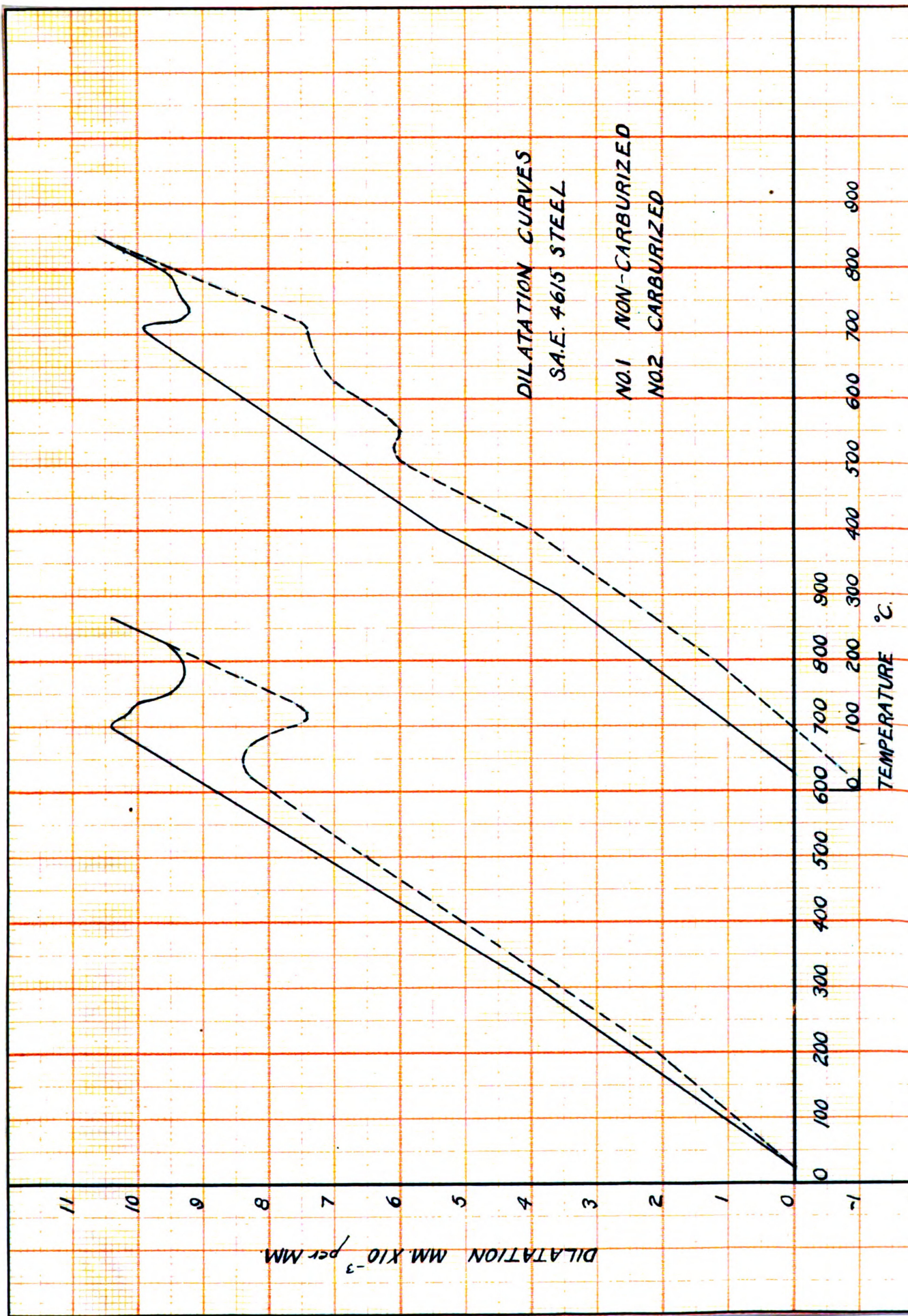


Figure 21.

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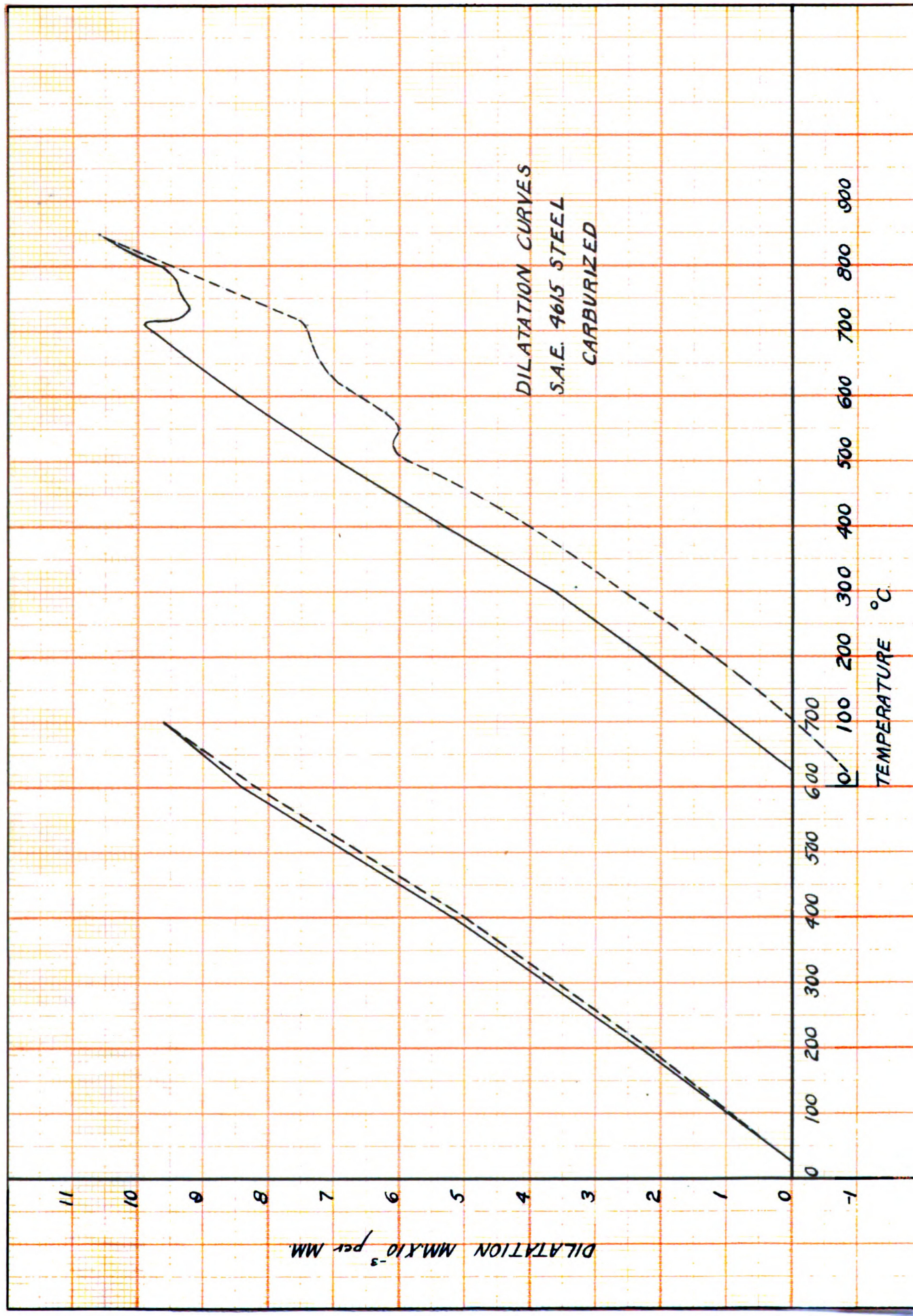
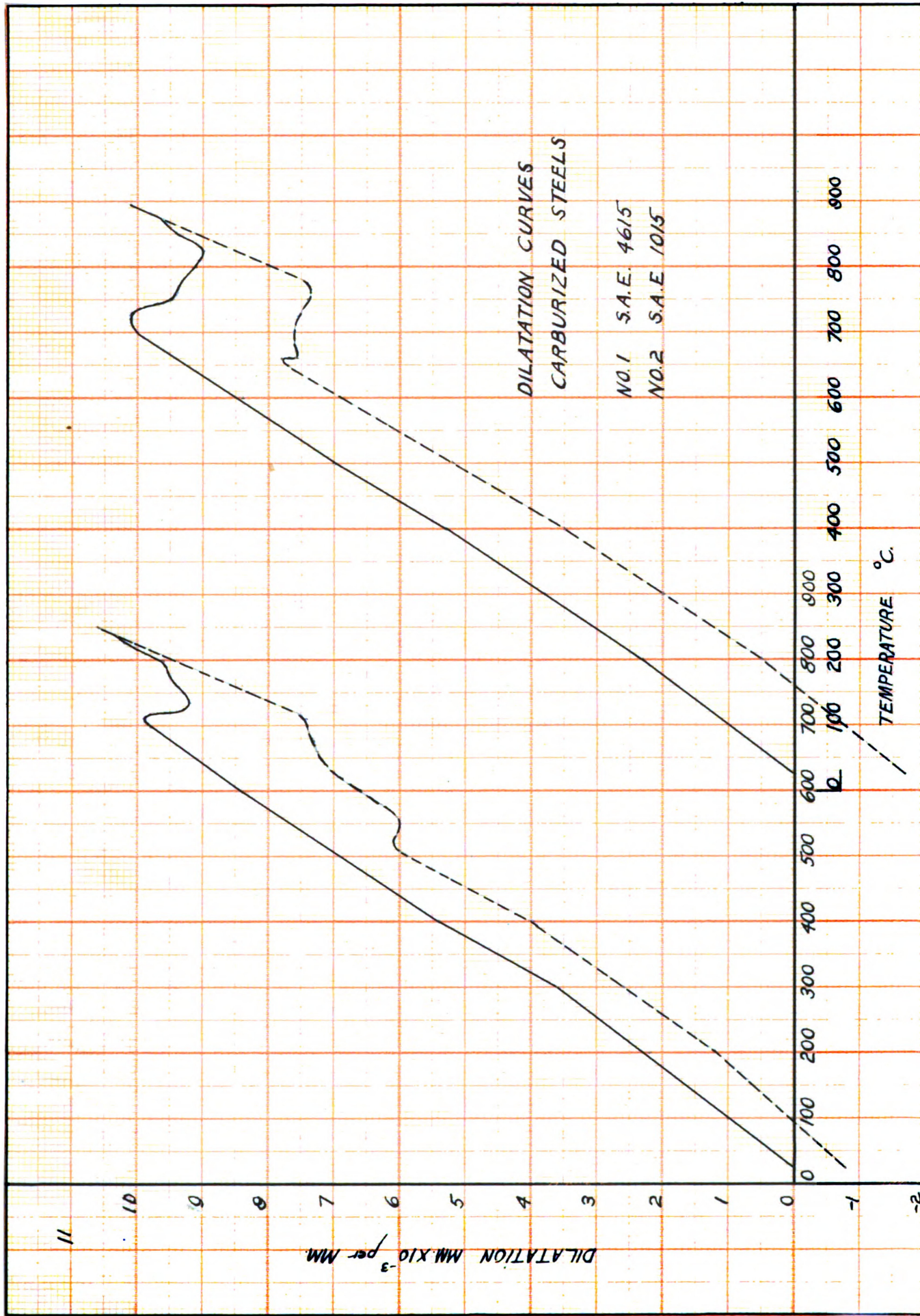


Figure 22.



of carburized 4615 stock is the resultant dilatation curve of the case and core. We were unable to obtain a any eutectoid molybdenum-nickel stock to find out how the case acted alone.

CONCLUSIONS

So far we have found nothing to prevent a case carburized steel from diminishing in size upon heating through the critical ranges and cooling slowly enough to keep the steel in the pearlitic state. All kinds of treatments were tried when heating above the critical to bring it back to its original size. The treatments tried were; a very slow heating and cooling, rapid heating and cooling, combinations of both and also changes in the rates of heating and cooling during the run. Of course the piece may be expanded by quenching but this sometimes gives the wrong properties, and anyway on the draw, if the draw is high enough, the piece will again shrink.

In order to reduce shrinkage as much as possible by heat treating, one should eliminate all the variables, such as cooling strains, working strains, strains set up due to too rapid heating and cooling during the run, and temperature gradients. This treatment also holds for pure iron and will eliminate shrinkage in non-carburized stock.

Alloying combinations may be also developed and studied, that would tend to reduce this shrinkage or eliminate it entirely. The 4615 steel has done this to a great extent. Of course to keep away from expense, it might be better to calculate for the shrinkage in the machining and forging operations. Of course this would not eliminate warpage and strains set up in the piece.

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