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PROLONGED TEMPERING AT 100 C AND AGING AT ROOM TEMPERATURE OF 0.8 PERCENT CARBON STEEL

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ABSTRACT

Thermomagnetic analysis has been applied to the study of quenched 0.8 percent carbon steel to show the effect of tempering for prolonged periods of time at 100 C and also the effect of aging at room temperature for considerably longer periods. Some specimens were also cooled in liquid air, tempered, and aged. The changes taking place in magnetization are interpreted as constitutional changes taking place in the steel.

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I. INTRODUCTION

In a previous paper [1] there were pointed out some of the advantages and possibilities of thermomagnetic analysis as a method of studying constitutional changes occurring during the tempering of quenched steel. It was possible by this method to follow the progress of the transformations while they were occurring, and some new evidence as to their nature was obtained. The method has been used in the present investigation to study the aging at room temperature of quenched high carbon steel and the effect of prolonged heating at 100 C.

II. MATERIAL AND METHODS

Cylindrical specimens 10 centimeters in length and 5 millimeters in diameter were prepared from basic open-hearth steel of the following composition:

	Percent
Carbon	0.78
Manganese	.56
Phosphorus	.020
Sulphur	.031
Silicon	.11
Chromium	.06
Nickel	.05
Copper	.06

[1] G. A. Ellinger, Thermomagnetic investigation of tempering of quenched 0.75 percent carbon steel, B.S.J. Research, 7, 441(1931); R.P.350; also R. L. Sanford and G. A. Ellinger, Thermomagnetic phenomena in steel, Trans., A.S.S.T., 20, 263(1932).

After normalizing, the specimens were heated in a lead bath maintained at a temperature of 825 C and then quenched in water at 20 C. In order to obtain uniform action during quenching, four specimens were heated and quenched together in a small jig, so designed as to allow free circulation of water around each specimen. After quenching, some of the specimens were immersed in liquid air for a period of 2½ hours. Details of the treatment of each specimen are given in table 1.

TABLE 1.—*Treatment of specimens after quenching from 825 C into water at 20 C*

Specimen	Treatment	Specimen	Treatment
1	As quenched.	8	Tempered 1,500 hours at 100 C, and aged 2 years at room temperature (approximate range 0 to 35 C).
2	Tempered 168 hours at 100 C.	9	Aged 2 years at room temperature.
3	Tempered 360 hours at 100 C.	10	Cooled 2½ hours in liquid air (−190 C).
4	Tempered 750 hours at 100 C.	11	Cooled 2½ hours in liquid air, tempered 1,500 hours at 100 C, and aged 2 years at room temperature.
5	Tempered 1,500 hours at 100 C.		
6	As quenched.		
7	Tempered 1,500 hours at 100 C.		

As an aid in conciseness and brevity specimens held 1,500 hours at 100 C are designated as "tempered" and those kept at room temperature (an approximate extreme range of 0 to 35 C) for 2 years as "aged."

In addition to the thermomagnetic analysis which was carried out with a slightly modified form of the apparatus previously described,[2] the microstructure was examined and the hardness was determined on auxiliary specimens, notched before heat treatment so that small pieces could be broken off when desired during the course of the treatment. The hardness was determined with a Vickers hardness tester.

III. OBSERVATIONS AND RESULTS

In carrying out the thermomagnetic analysis the specimens were subjected to a constant applied magnetizing force of 277 oersteds. The dimensional ratio of the specimen was such as to bring the magnetization always into the third stage. The uncertainties usually encountered when the magnetization is in the unstable second stage, represented by the steep part of the B–H curve were thus avoided.

Two methods of procedure were used. In the first method the specimen was heated at a uniform rate of approximately 1.5° C per minute and the degree of magnetization noted at suitable intervals. In the second method, the procedure consisted in heating a specimen to a definite temperature and holding it at that temperature until the magnetization reached a constant value. This was done at temperatures of 200, 235, and 300 C, with several runs at 400 C.

Figure 1 shows the results obtained by the first method, that is by uninterrupted heating to 300 C of specimens quenched and tempered at 100 C for various periods up to 1,500 hours. The magnetization increased with temperature to a maximum which occurred at 280 C and then decreased with increasing temperature. The maximum was lowered progressively by tempering at 100 C, the change being

[2] R. L. Sanford, Apparatus for thermomagnetic analysis, BS J. Research, 2, 659 (1929); RP 50.

most marked in the early part of the tempering period. The abrupt increase in magnetization took place at higher and higher temperatures as the tempering period was increased.

Figure 2 illustrates the change in hardness as tempering progressed. The part of the curves corresponding to relatively short tempering periods is indicated by dotted lines, as the trend in this region is somewhat uncertain. Several investigators have reported an increase in hardness for quenched specimens tempered for short periods of time at 100 C. In this investigation, no hardness determinations

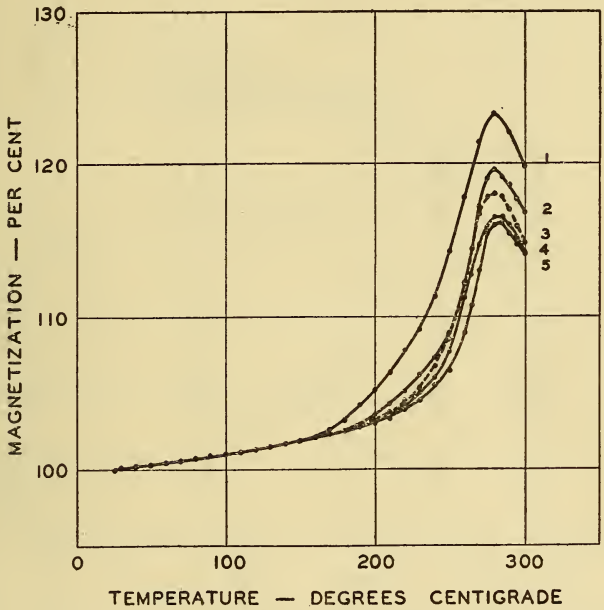

FIGURE 1.—*Effect of prolonged tempering at 100 C upon magnetization*

Details of treatment of each specimen (numbers 1, 2, 3, 4, and 5) are given in table 1.

were made on specimens aged for less than 1 week, at which time the hardness was appreciably lower than that of the quenched material.

Figure 3 is typical of the results of thermomagnetic analysis when the second procedure given above was followed. Here are shown the changes in magnetization which took place upon heating to, holding at, and cooling from a predetermined temperature. The magnitude of the changes observed upon holding at a constant temperature and the time required for the changes to be completed depended upon the treatment to which the various specimens had previously been subjected. All of the other curves were substantially similar in form to those shown.

In this procedure a progressive increase in magnetization took place immediately upon heating, at the rate of increase depending upon the rate of heating. Upon holding at 200 C the magnetization continued to increase over a period of several hours, after which no further increase was noted. On cooling to room temperature little, if any, change in magnetization was observed.

Upon reheating, no appreciable change in magnetization was noted until the temperature was approximately 230 C. On holding at 235 C an increase in magnetization was observed, which continued for approximately 40 minutes. No further change took place upon holding for an hour but in cooling to room temperature, a progressive increase in magnetization was again observed.

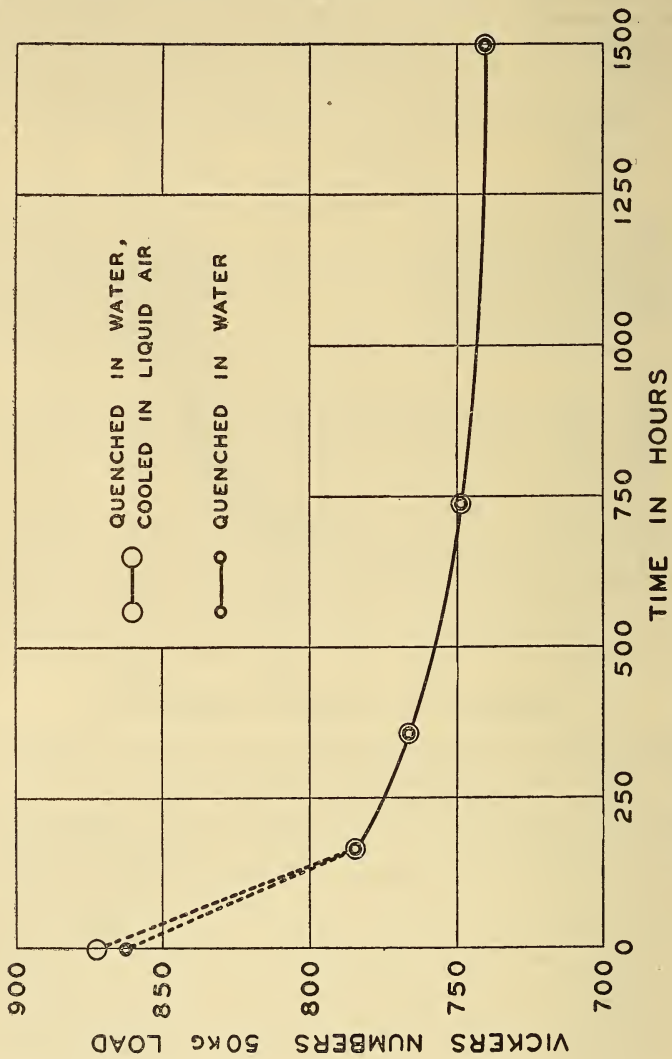

FIGURE 2.—*Effect of prolonged tempering at 100 C upon hardness*

On reheating to 300 C a gradual progressive decrease in magnetization took place, which continued for some time as that temperature was maintained constant. Upon cooling, a pronounced A_0 transformation was observed which indicated the presence of cementite. Specimens reheated to 400 C revealed, in no case, any further change in magnetization as the specimen was held at that temperature.

From this type of curve were derived the data shown graphically in figure 4, in which the total changes in magnetization at each of these

temperatures are plotted on a percentage basis. The value for 200 C was the total change upon heating to and holding at that temperature. The values given for the other temperatures were the changes that took place only during the time the temperature was held constant.

The increases in magnetization occurring at 200 C show several features. The maximum increase occurred in freshly quenched specimens (no. 6) while the effect was somewhat less in specimens aged for 2 years (no. 9). Tempered specimens (no. 7) and those tempered and aged (no. 8) showed the least increase, both having practically

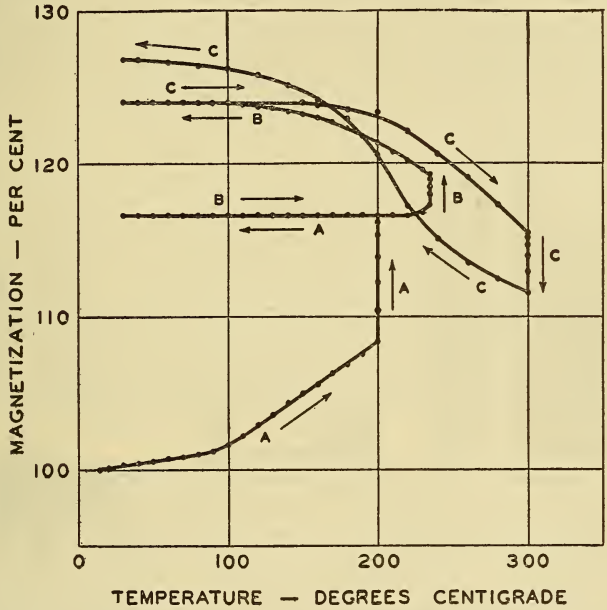

FIGURE 3.—*Typical thermomagnetic curve showing changes in magnetization in quenched steel*

A—Maximum temperature 200 C.
B—Maximum temperature 235 C.
C—Maximum temperature 300 C.

the same value. After treatment with liquid air the increase in magnetization after tempering and aging (no. 11) was the same as that observed immediately after the liquid air treatment (no. 10).

The increase in magnetization observed upon holding the specimens at 235 C was affected by neither the simple liquid air treatment (no. 10) nor tempering (no. 7). Aging for 2 years (nos. 8, 9, and 11), on the other hand, very greatly reduced the change.

The decrease in magnetization occurring when a specimen was maintained at 300 C was apparently affected very little by tempering (no. 7) but aging (nos. 8, 9, and 11) influenced this change materially. The maximum change was noted in the aged specimens regardless of previous treatment. Figure 5 shows the differences in time required for the changes occurring at 300 C, to be completed. Specimens aged 2 years at room temperature (nos. 8 and 9) required more than twice as long for completion of the change as did the freshly quenched

specimens (no. 6). In both cases, the liquid air treatment (nos. 10 and 11) increased the time required.

A study of the microstructure of the quenched and tempered specimens was made. Typical structures are shown in figures 6 to 8, inclusive. The martensite needles showed a progressive darkening as time of tempering increased and a gradual breaking down, particularly in the latter stages, until after 1,500 hours it was difficult to reveal structural details with any degree of sharpness.

IV. DISCUSSION

From the results it is evident that, even at room temperature, quenched steel undergoes progressive changes, the magnitude of which

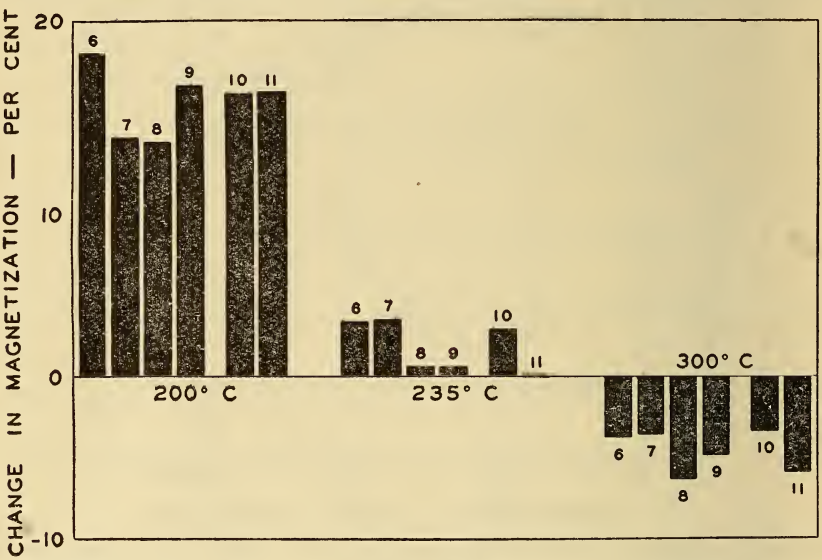

FIGURE 4.—*Changes in magnetization taking place at 200, 235, and 300 C*

Details of treatment of each specimen are given in table 1.

is determined by preliminary treatment such as cooling in liquid air or heating for prolonged periods at 100 C. It is apparent further, that the effect of these changes differs from the "age hardening" phenomena in other metals, but rather is a softening effect.

In considering the changes at 200 C, it was observed that the maximum increase in magnetization occurred in freshly quenched specimens and was least in tempered specimens. These changes have previously [3] been attributed to the gradual release of stresses induced by quenching.

It is known from the results of other investigators that martensite is in a highly stressed state, the stresses being so high as to convert the normal cubic lattice of alpha iron into a pseudo-tetragonal form. Furthermore, martensite is in the state of a super-saturated solid solution, such solution being forced by the extremely rapid cooling with no chance for the precipitation of carbon and the formation of cementite.

Low temperature tempering or sufficiently long aging periods are believed to relieve these stresses materially through the gradual

[3] See footnote 1.

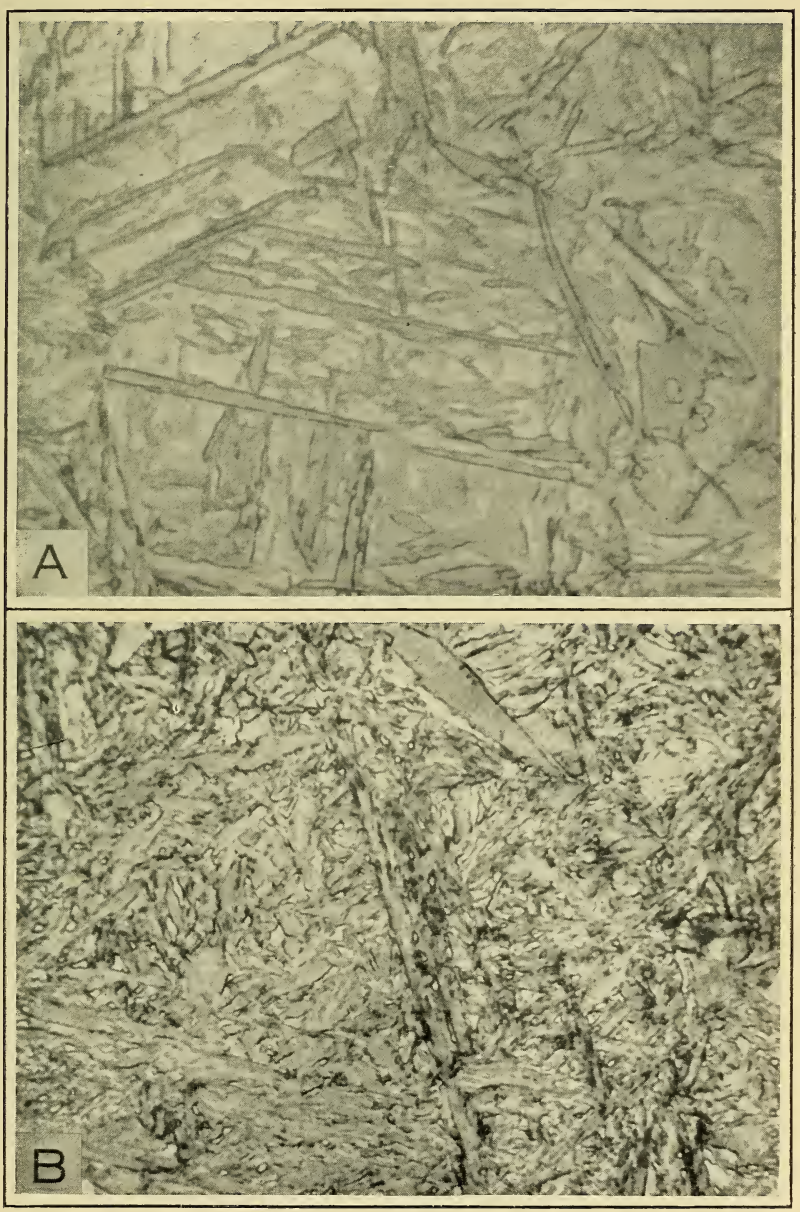

FIGURE 6.—A, *Structure of specimen freshly quenched in water,* × *1500;* B, *structure of specimen after tempering 168 hours at 100° C,* × *1500.*

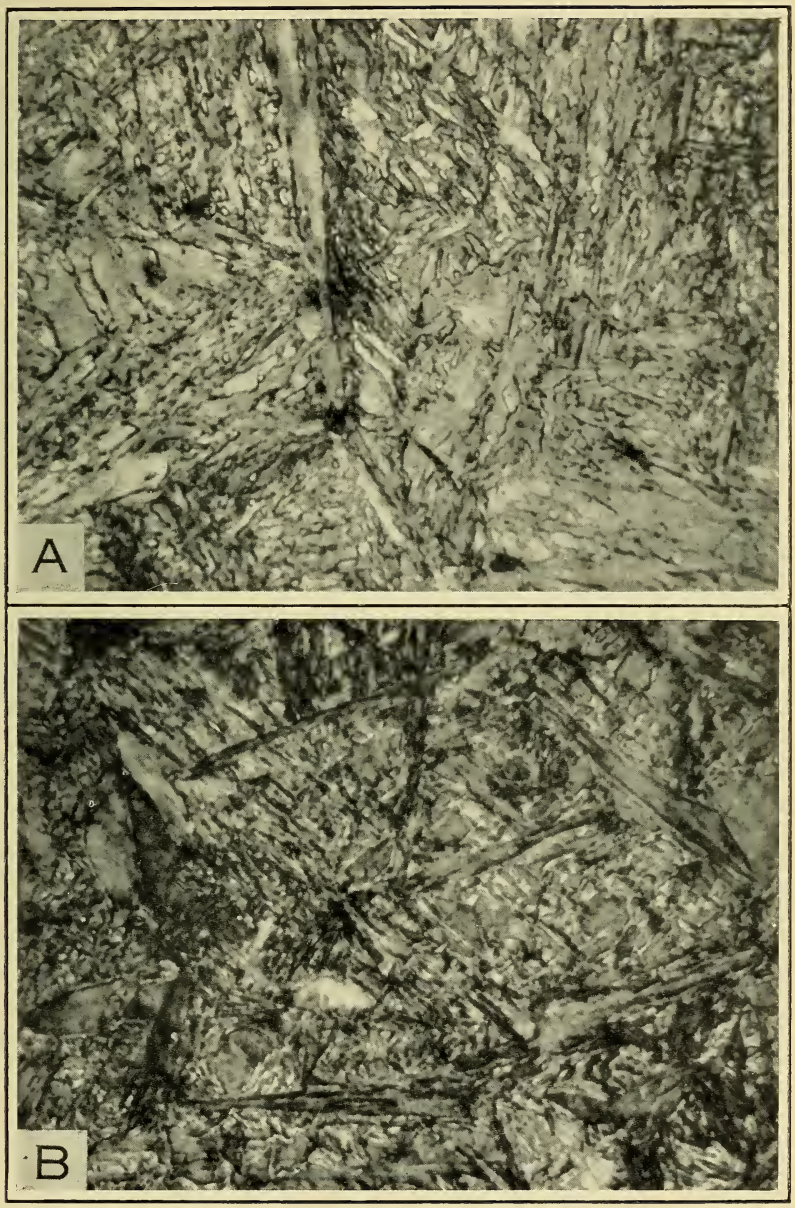

FIGURE 7.—A, *Structure of specimen after tempering 360 hours at 100° C., × 1500;* B, *structure of specimen after tempering 750 hours at 100° C, × 1500.*

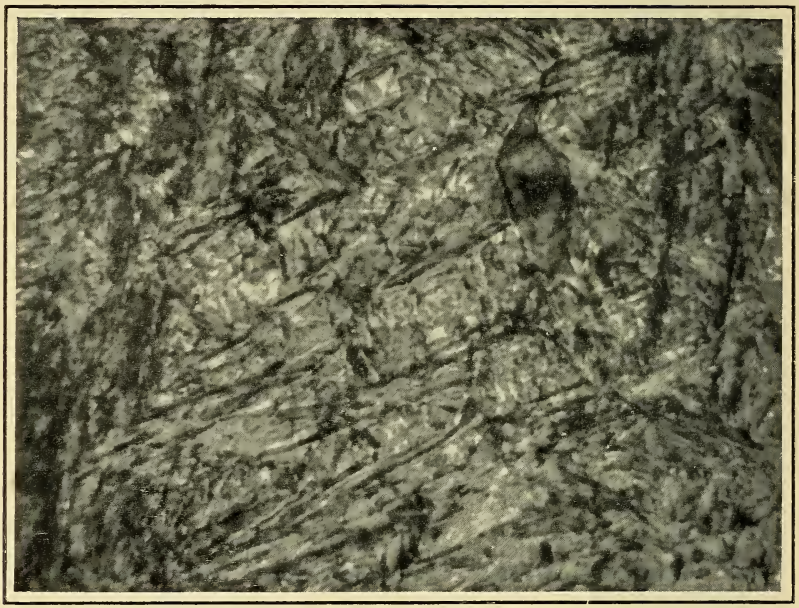

FIGURE 8.—*Structure of specimen after tempering 1500 hours at 100 C, × 1500.*

precipitation of carbon, either as atomic carbon or some constituent other than cementite. The lattice then assumes more nearly its cubic dimensions with a resultant decrease in hardness. Likewise, the stability of the tempered or aged martensite is increased.

The effect of cooling specimens in liquid air is quite pronounced. The magnitude of the change at 200 C was affected by neither tempering nor aging, which indicates that little significant change took place in the martensite. Greater stresses must have been induced by cooling in liquid air than in water quenching and certain of these stresses must have been released upon subsequently warming to room temperature. This release must have been of considerable magnitude as evidenced by the diminished increase of magnetization at 200 C. The effect of liquid air treatment, not unlike that of tempering, appears to be one of stabilization of martensite. This is further indicated by the fact that tempering and aging apparently have no effect upon the magnitude of the magnetic change at this temperature.

Small amounts of austenite usually exist in freshly quenched steel. This constituent is unstable, being forcibly detained in the undercooled state and will transform to stable phases as soon as permitted. That such was the case is supported by the data. The freshly quenched steel evidently contained an appreciable amount of austenite which decomposed upon heating to 235 C. Even after tempering there was little or no change in the amount of austenite present, but after aging for 2 years this amount was reduced materially. Those specimens which, in the freshly quenched condition, showed the presence of austenite increased somewhat in magnetization upon cooling from 235 C. The nature of the curves, figure 3, suggests that a small amount of cementite was formed at this point, most of which probably came from the decomposition of austenite.

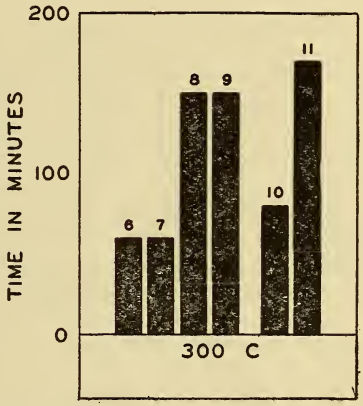

FIGURE 5.—*Time required for decrease in magnetization to be completed at 300 C*

The decrease in magnetization taking place at 300 C is attributed to the formation of cementite by the combination of carbon precipitated from martensite with alpha iron. As this took place above the A_0 transformation temperature of cementite (normally considered as 215 C), that portion of alpha iron which combines with carbon is rendered ineffective magnetically with a resultant net reduction in the total magnetization. A hypothesis was set up previously [4] indicating that in the range from 235 to 300 C atoms of carbon precipitated from the supersaturated martensite solution would unite with atoms of iron to form cementite.

Tempering or cooling in liquid air apparently had little effect upon the magnitude of the changes taking place at 300 C. On the other hand, aging for a considerable period of time produced a noticeable effect. The magnitude of the change and its duration were materially increased.

[4] See footnote 1.

The evidence points to a more rapid formation of cementite from the decomposition products of freshly quenched martensite than from those of aged martensite. The amount of carbide formed depends upon the amount of carbon in the steel and should be the same for a given percentage of carbon regardless of its form. A certain amount was formed while heating to 300 C and the remainder while holding at that temperature. The intensity and duration of the change at 300 C was greater in aged than in freshly quenched specimens. This leads to the belief that in aged specimens less carbide was formed in the range between 235 and 300 C than while holding at 300 C, while in unaged specimens most of the carbide is formed between 235 and 300 C. The sluggishness indicates the possibility that carbon atoms are in less favorable position to unite with iron atoms after aging and require considerably more time for the reaction to take place.

V. SUMMARY

Thermomagnetic analysis has been applied to the study of 0.8 percent carbon steel quenched in water and subsequently tempered 1,500 hours at 100 C and aged at room temperature (0 to 35 C) over longer periods of time. Some quenched specimens were cooled in liquid air for 2½ hours and subsequently tempered and aged. Changes took place during heating of these specimens as follows:

1. An increase in magnetization occurred during the heating of the material and while it was held at 200 C, the magnitude of which, in quenched specimens was appreciably affected by previous tempering, aging, and cooling in liquid air. Tempering and aging had no effect on specimens previously cooled in liquid air.
2. An increase in magnetization occurred in specimens held at 235 C which was not appreciably changed by previous tempering or liquid air treatment but was materially changed by aging.
3. A decrease in magnetization was observed in specimens held at 300 C which was changed appreciably in both magnitude and duration by previous aging.

The explanations offered for the behavior of this material after tempering and aging, based upon observations of changes taking place at various temperatures are as follows:

The carbon of martensite is gradually precipitated both after prolonged tempering at 100 C and after aging, so that the more stable cubic form results. Martensite is also changed by cooling in liquid air but is not further changed by either tempering or aging.

The small amount of austenite retained in 0.8 percent carbon steel after quenching is not affected by tempering but is decreased materially by aging. Liquid air treatment has no apparent effect upon this retained austenite.

Between 235 and 300 C, in what is believed to be the cementite formation range, previous tempering causes no observable difference in magnitude or time. Aging, on the other hand, increases both magnitude and time of the reaction.

The authors wish to express their appreciation and gratitude for the conscientious assistance and cooperation of E. G. Bennett of the National Bureau of Standards, in making many of the determinations in this investigation.

WASHINGTON, June 19, 1934.