The Determination of the Ms Temperature by Dilatometric Method

By

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In 1939 Greninger and A. R. Troiano published a method to determine microscopically the Ms temperature of steels[1], and the method has generally been adopted for that purpose. The determination of the Ms temperature, however, is often met with difficulty as the distribution of tempered martensite is not always uniform in the specimens under examination.

The authors tried and succeeded in determining the exact Ms temperature by the application of sensitive delatometric analysis. The principle of this analytical method is to measure the amount of the primary shrinkage brought about by the change of martensite to so-called tempered martensite in the martempered steel on heating.

The present paper deals with the determination of the Ms temperature by the dilatometric method of analysis.

The chemical composition of the steel employed was as follows: 0.80% C, 0.32% Mn, 0.29% Si, 0.024% P, 0.019% S. The specimens, 5 mm in diameter and 50 mm in length, were taken from a long wire 5 mm in diameter. After austenitizing them for 30 minutes at 850°C. in an electrically heated quartz tube, they were quenched for a definite period of time in a salt-bath* kept at a fixed temperature, and subsequently were quenched in water. Their delatation was messured with a dilatometer, which was designed by one of the authors and is shown in Figure 1. As shown in the figure, it consists of a hollow silica tube 10 mm in diameter and 300 mm in length with one of its ends closed and fixed in a metallic cylinder (C). A specimen (S) is inserted in a silica tube (T_1).

The expansion of the specimen when heated, is transmitted to a piece (P) by a silica tube (T_2). As the piece (P) moves, a needle (N) rotates. In accordance with the rotation of the needle (N) a mirror rotates likewise and its movement is read by

* The chemical composition of the salt-bath was as follows:— $NaNO_3$ 25%, KNO_3 25%, $NaNO_2$ 50%. The weight of this mixture was about 2 kg.

a cathetometer placed 80 cm away from the mirror.

For more accurate measurement, differential method of analysis was adopted and the difference in the dilatation between the specimen and an annealed neutral body of the same kind, was measured by another dilatometer as shown in Figure 2. The mechanism of the apparatus is similar to that of the one shown in Figure 1.

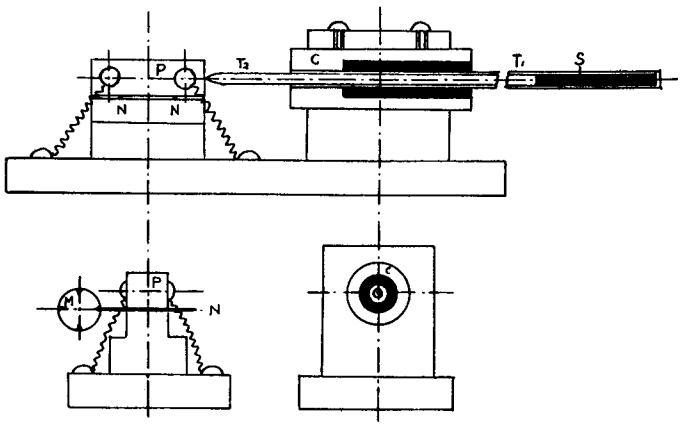

Fig. 1. The dilatometer

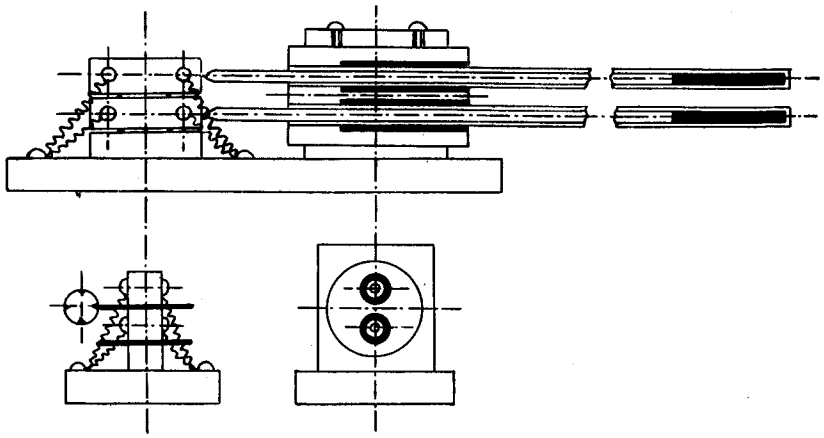

Fig. 2. The differential dilatometer

Preliminary Experiment.

The specimens described above were heated at 850°C. for 30 minutes, then quenched in the bath held at 250°, 210°, 180° and 150°C. respectively for one minute, and then quenched in water. The delatation change of these specimens on heating was measured with the dilatometer shown in Figure 1. The diameter of the rotating needle was 0.8 mm, which means that the magnification of dilation was about 2000.

Figure 3 shows the results of the experiment. As it is seen well in Figure 3 the water-quenched steel has two stages of shrinking on heating below the A_1 point.

The observation of the dilatometric curves of the specimens heat-treated as

described above, reveals that the extent of the primary shrinkage in the specimens is influenced greatly by the temperature of the quenching bath even if that of the secondary shrinkage may not.

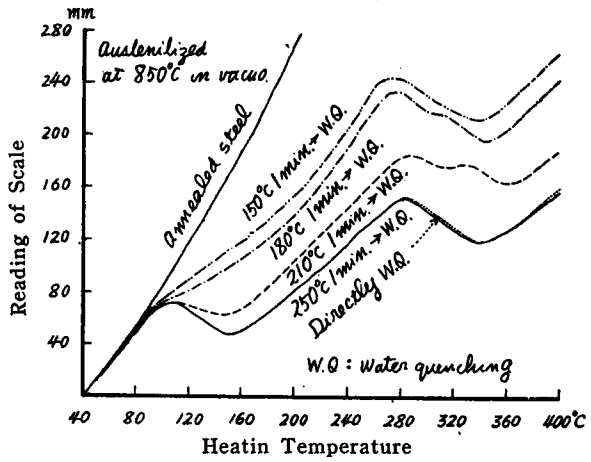

Fig. 3. Chage of the dilatometric curves of the interupted quenching specimens

It is seen from the dilatometric curves that the lower the temperature of isothermal heat-treatment, the less the primary shrinkage. That this primary shrinkage is due undoubtedly to the formation of tempered martensite by the isothermal heat-treatment is evident from Photos 2, 3 and 4 (light etching). The dilatometric curve obtained by re-heating the water-quenched specimen, is similar to that of the specimen quenched in the bath held at 250°C. and treated isothermally since these specimens show the structure consisting entirely of martensite as shown in Photo 1 (deep etching). The above results suggest the possibility of determining the Ms temperature by the delatometric method of analysis. The authors, therefore, conducted the following experiment for the more accurate measurement of the Ms temperature.

The Determination of the Ms Temperature.

The experiment was carried out in the same way as before except that the rotating needle used was 0.4 mm in diameter thereby increasing the magnification of delatation to 4000. The specimens were similarly austenitized at 850°C. as before, quenched in the salt-bath maintained at 250°, 245°, 240°, 237°, 235°, 230°, 325°, and 220°C. respectively and held at these temperatures for a half minute. They were subsequently quenched in water.

The dilatometric curves of these specimens obtained on heating are all shown in Figure 4. From the curves in this figure, it is seen that the Ms temperature lies between 240°–235°C. from what has been described above. To clarify this point, the specimen isothermally heat-treated at 237°C. was examined in particular.

The lessening of the primary shrinkage in connection with the temperature of isothermal treatment was plotted from the scale-readings and the result is given in Figure 5. From this figure, the Ms temperature is confirmed to be 237°C.

Photos 5, 6, 7 and 8 are the micro-photographs of the specimens isothermally heat-treated at 240°, 237°, 235° and 230°C. respectively. Tempered martensite is not observable in Photo 5 and also in Photo 6. In Photo 6, however, are regions which are about to change into tempered martensite. In Photos 7 and 8 tempered martensite is distinctly observable. The results of these observations agree quite well with those of the dilatometric experiments.

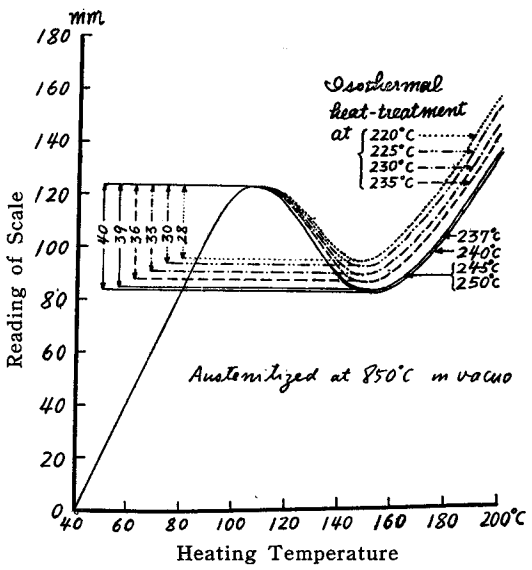

Fig. 4. Change of 1st. shirinkage in the dilatometric curves of the interupted quenching specimens which were kept isothermally for 30 seconds and again quenched into water

Examination of Retained Austenite.

In the above experiments, austenite was not considered to be retained in the specimen when quenched but to change entirely into martensite. Austenite, however, must exist in the specimen together with martensite and the amount of retained austenite in relation to that of tempered martensite has to be determined. For this particular purpose, differential dilatometric experiments were conducted.

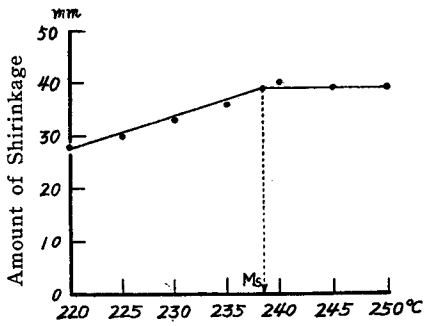

Fig. 5. Relation between the amount ofthe 1st. shrinkage and the temp. of isothermal heat-treatment

In the differential dilatometric analysis, the magnification was 2000 as the rotating needle was 0.8 mm in diameter.

The specimens were heated at 850°C. in vacuo, quenched in the salt-bath at 250°, 200° and 150°C. respectively, kept at these temperatures isothermally for 30 seconds and subsequently quenched in water. To have a specimen with larger amount of martensite, another specimen was quenched in the salt-bath at 250°C., kept at this temperature for 30 seconds and finally cooled in air.

The dilatometric changes in them on heating were measured similarly. Figure 6 shows the results of the measurement. It is seen from the figure that the extent of

the primary shrinkage is almost inversely proportional to the amount of tempered martensite. The amount of retained austenite can also be estimated by the degree of the expansion which is observed before the beginning of the secondary shrinkage on the dilatometric curve.

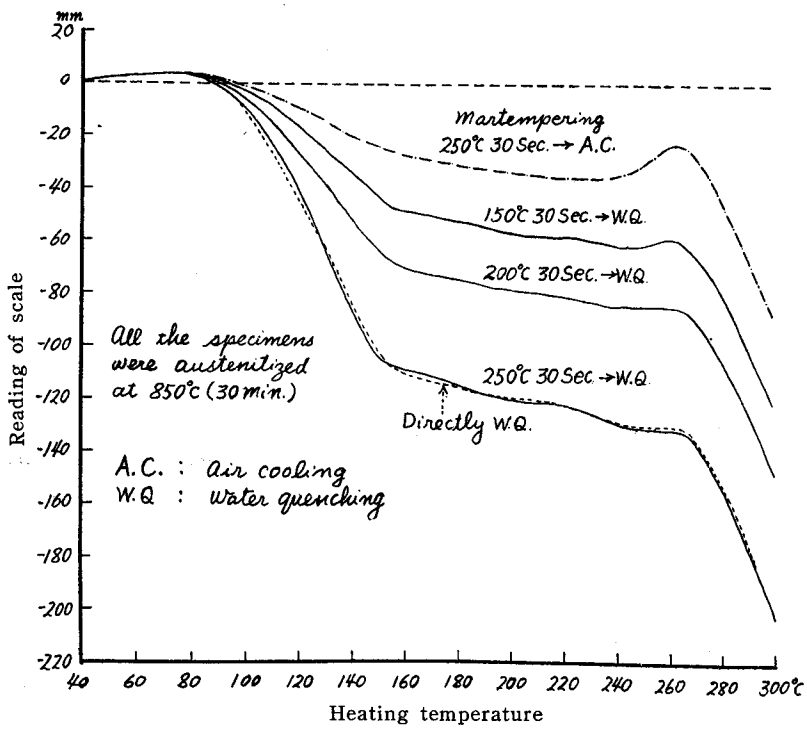

Fig. 6. Behaviour of retained austenite in the differential dilatometric curves of the interupted quenching specimens

In the present experiment, it was found that the amount of retained austenite becomes larger with the increase of tempered martensite. From Figure 6, it seems that the primary shrinkage observable on the dilatometric curve, decreases with the increase of tempered martensite and retained austenite coexisting at the same time. It may be said conclusively from this fact that the presence of retained austenite has no contradictory influence to the determination of the Ms temperature.

From the experimental results, the authors conclude as follows:

(1) The martensite formed isothermally below the Ms temperature transforms into so-called tempered martensite. This tendency decreases as the temperature of the isothermal treatment is lowered. The tempering effect, however, does not give any trouble to the determination of the Ms temperature as described above.

If the Ms temperature is too low to enable martensite to change into tempered

martensite, neither will be the martensite formed isothermally below the Ms temperature tempered in the salt-bath. In such a case, the darkening (tempering) treatment, by which the isothermally formed martensite is tempered without changing retained austenite, must be given to the specimen before it is used for the determination of the Ms temperature by dilatometric method as A. B. Greninger and A. R. Troiano[1] gave to their specimen in the determination of the Ms temperature by microscopic method.

(2) For the determination of the Ms temperature, direct dilatometric measurement is sufficient as described above, but in case of low carbon steel, differential dilatometric method must be employed for the accurate measurement of the primary shrinkage in the specimen as the dilatometric change in it is not so distinct.

(3) The specimen for the determination of the Ms temperature, must not be kept in the salt-bath long lest the bainite reaction should take place.

(4) The especimen cooled in air after the isothermal heat-treatment at 250°C., contained the largest quantity of tempered martensite, from which it can be said that the internal strain of martempered steel is very small owing to the existence of tempered martensite in great quantity.

(5) Retained austenite increases as the quantity of tempered martensite becomes greater. This fact agrees with so-called stabilization of austenite below the Ms temperature.[2]

Conclusion.

The Ms temperature of a steel can be determined accurately from the results of dilatometric measurement on heating the specimens martempered at various temperatures.

Reference

1) A. B. Greninger and A. R. Troiano: Trans. A. S. M. 28 (1940) 537.
2) W. J. Harris and M. Cohen: Trans. A. I. M. E. 180 (1949) 447.

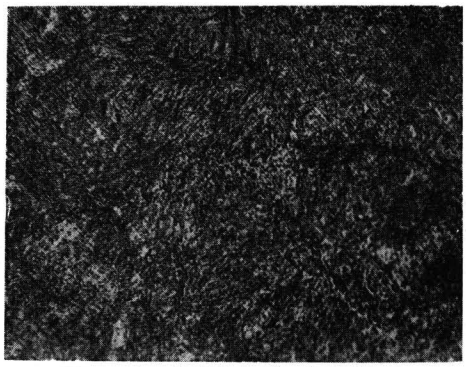

Photo. 1. 250°C 1 min. keeping
deep etching ×200

Photo. 2. 210°C 1 min. keeping
shallow etching ×200

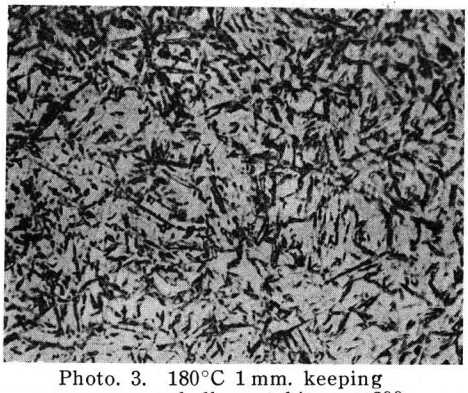

Photo. 3. 180°C 1 mm. keeping
shallow etching ×200

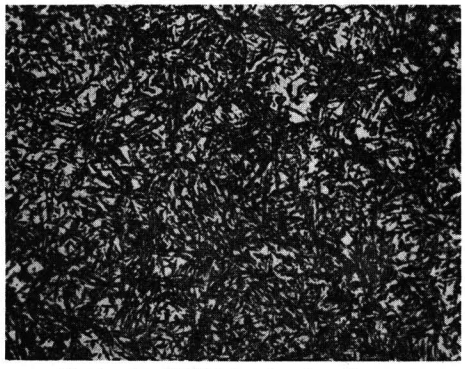

Photo. 4. 150°C 1 min. keeping
shallow etching ×200

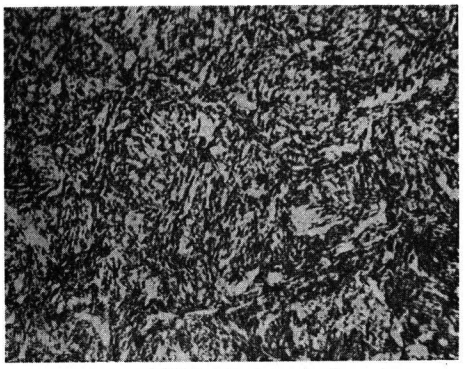

Photo. 5. 240°C 30 seconds keeping
deep etching ×400

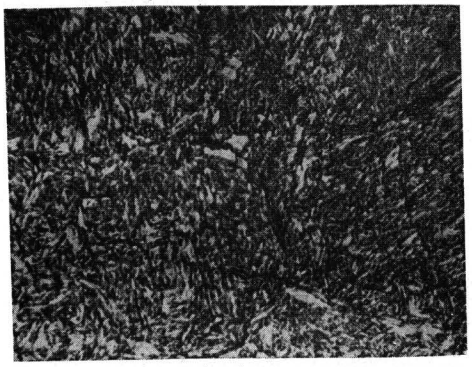

Photo. 6. 237°C (Ms) 30 seconds keeping
deep etching ×400

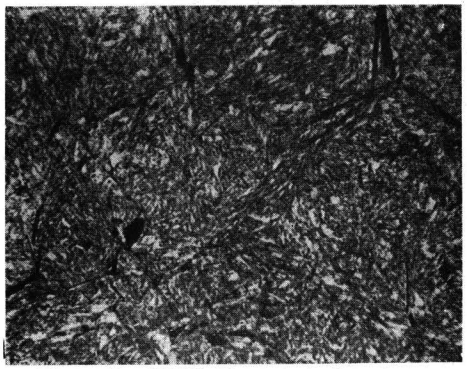

Photo. 7. 235°C 30 seconds keeping
deep etching ×400

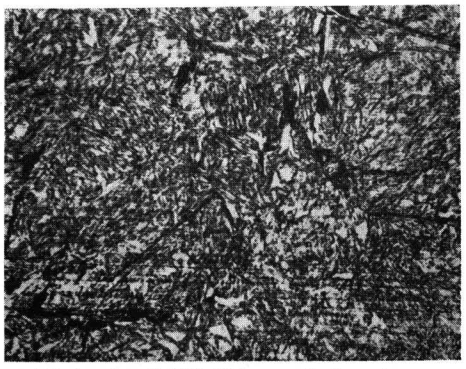

Photo. 8. 230°C 30 seconds keeping
deep etching ×400