The ϵ Phase Formation Range of Fe-C-Mn Alloys under Quenched Conditions*

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The ϵ phase formation range of Fe-C-Mn alloys under quenched conditions has been investigated by the dilatation test, the magnetic test and X-ray analysis. The alloys of transitional compositions, changing the structure from a complete α phase into a complete γ phase under quenched conditions, show two clear expansions at about 200°C and 500°C in the thermal dilatation curves. These phenomena have been considered the transformation of retained austenite into α in these two temperature ranges in a way similar to some Cr steels. But, in the present work, some anomalies are observed at about 200°C in the dilatation curves. Consequently, it is evident that this expansion is not caused by the simple retained austenite transformation into α , but either the $\epsilon \rightarrow \gamma$ transformation or the two concurrent transformations, such as the retained austenite transformation and the $\epsilon \rightarrow \gamma$ transformation. Low manganese content alloys transforms only at the lower temperature range in a way similar to that of the plain carbon steel, and high manganese content alloys transforms only in the higher temperature range. However, alloys, containing 1% C and 2~3% Mn, show a two-stage transformation of retained austenite into α in both dilatation and thermo-magnetic curves. The manganese content enough to form the ϵ phase in the quenched Fe-Mn alloys is gradually lowered with an increase of the carbon content, and its content range decreases to a narrower range with the increasing carbon content. The ϵ phase is formed in 1.0%C-2~4%Mn alloys under quenched conditions.

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I. Introduction

The ϵ phase is known to occur in Fe-Mn binary alloys with 9~25% Mn. The ϵ phase formation range of Fe-Cr-Mn ternary alloys^{(1)~(3)} has also been reported⁽³⁾. However, only a few reports deal with the formation of the ϵ phase in Fe-C-Mn alloys. Even in high Mn steels forming a complete austenite phase after solution treatment, the ϵ phase is observed occasionally at room temperature after isothermal heating at any temperature, where a large quantity of carbide precipitates can be formed^{(4)~(5)}. It has also been clarified that the ϵ phase takes place in the cold-worked Hadfield steel with stacking faults⁽⁶⁾. Formation of the ϵ phase under quenched conditions has been considered only in connection with high manganese content alloys, but it appears that nothing has been studied on low manganese alloys containing carbon. In a previous paper⁽⁷⁾ the present author investigated the effect of carbon on the phase relations in the quenched and cold-worked 12% Mn-Fe alloy, and made clear that the effect of carbon on the formation of the ϵ phase in the quenched Mn-Fe alloy depended upon the stability of austenite because of the character of the ϵ phase as the transitional phase during the transformation process of $\gamma \rightarrow \alpha$, and the carbon content, enough to form the ϵ phase gradually moved to the higher carbon

side with the increasing working ratio due to the phase transformation of $\gamma \rightarrow \epsilon \rightarrow \alpha$ by cold-working.

In the dilatation curves of low manganese alloys which show the transitional structure between the α phase and the γ phase under quenched conditions, an anomalous expansion was observed, and it was inferred that the ϵ phase is formed even in such low manganese alloys. This paper is intended to chiefly report the ϵ phase formation range in Fe-C-Mn ternary alloys under the quenched conditions.

II. Materials and Experimental Methods

The electrolytic iron and the electrolytic Mn were melted in a high frequency furnace and cast in 2-kg ingots containing various amounts of C and Mn. According to a previous report⁽⁷⁾, the maximum quantity of the ϵ phase in the 12% Mn-C-Fe alloy under the quenched condition has been observed with 0.2% C. It has been reported on Fe-Mn binary alloys, that the maximum quantity of the ϵ is found with about 17% Mn⁽⁸⁾. Therefore, the ϵ phase formation in the quenched Fe-C-Mn ternary alloy is expected in the range of higher C and lower Mn alloys than in Fe-Mn binary alloys. Three series of specimens, 1.0% C and 0~18% Mn, 0.5% C and 5~15% Mn, and 0.3~1.0% C and 6% Mn, were prepared. The results of chemical analysis of C and Mn of the specimens are shown schematically in each figure. The nitrogen contents of the alloys are min. 0.004-max. 0.011% with the mean value of 0.007%, and the effect of nitrogen on the experimental results may be neglected.

All the ingots were hot rolled to 6 mm ϕ bars, and these bars were machined to the diameter of 5 mm and

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(6) Z. Nishiyama : Metal Physics, Japan, 7 (1961), No.3, 107.

(7) Y. Imai and T. Saito : J. Japan Inst. Metals, 26 (1962), No.5, 283.

(8) M. Gensamer, J. F. Eckel and F. M. Walters : J. ASST, 19 (1935), 589.

(9) T. Honma : Denki Seiko, Japan, 30 (1958), 319.

the length of 70 mm. Then, all the specimens were solution treated at 1100°C for 1/2 hr by water quenching, and electropolished to remove the surface layer. The dilatation specimens were heated up to 800°C in the differential dilatometer at the rate of 3°C/min, using pure Cu as the neutral body. The ϵ phase formed in each specimen was detected mainly by the expansion caused by the $\epsilon \rightarrow \gamma$ transformation during heating, and when the presence of ϵ cannot be detected by the dilatation curves, the alloys were further tested by the ballistic magnetometer at a magnetic field of about 1300 Oe or by X-ray analysis. For the X-ray analysis, needle-shape test pieces were cut from the dilatation specimens, and electropolished to avoid any phase change during the working process. The Fe target was used.

III. Experimental Results and Considerations

Fig. 1 shows the differential dilatation curves of the alloys containing about 1.0% C and various amounts of Mn under the quenched condition. The hardness of each alloy is appended to every curve. In the dilatation

Fig. 1 Thermal dilatation curves for quenched alloys.

curves of the low manganese alloys, the first stage of contraction, the second stage of expansion and the third stage of contraction are clearly observed in a way similar to the behaviour of the tempered martensite in carbon steel. The first stage of contraction gradually decreased with the manganese content, and disappeared at about 6% manganese. This phenomenon was caused by the decrease of the martensite content in the alloys under the quenched condition. On the other hand, the second stage of expansion caused by the decomposition of retained austenite, increased gradually with the manganese content, showing a maximum at about 3% manganese, then decreased again. The temperature at which the maximum peak was observed became higher from the initial temperature of 280°C with increasing of manganese content, but it became constant in the vicinity of 350°C in the alloys containing more than 2% manganese. Moreover, the second stage of expansion began at about 230°C in the 0.41~2% manganese alloy as in the plain carbon steel, but at 200°C in the 2.81% Mn alloy which showed the highest expansion and at 180°C in the 4.37% Mn alloy which showed a comparatively small expansion. It appears that the peak temperature of the second stage of expansion increases gradually and finally becomes constant in the vicinity of 350°C with the increasing Mn content. On the other hand, the expansion began at a constant temperature in the low Mn alloys, but in the high Mn alloys it began at a low temperature. Also, the width of the peak gradually increased with the Mn content, as clearly shown in Fig. 1.

The second stage of expansion with a peak at about 570°C was observed in the dilatation curve of the 4.37% and 2.81% Mn alloys and slightly, in the 2.23% Mn alloy, but not observed in the lower Mn content alloys. In this paper, the expansion beginning at about 230°C is called the low temperature range expansion and that at about 500°C the high temperature range expansion. A large contraction at about 700°C due to the $\alpha \rightarrow \gamma$ transformation decreased with the increasing Mn content.

There is some problem in the decomposition behaviour of retained austenite which is shown in Fig. 1. The retained austenite in the quenched alloys gradually increased with the Mn content, so that the expansion caused by the decomposition of retained austenite increased and the decomposition temperature gradually, became higher. It seemed that in the alloys with more than 2.23% Mn, the decomposition of retained austenite was not completed in the low temperature range, and reappeared at about 500°C. As mentioned above, some questions in the first stage decomposition remains unexplained, such as the rise of the peak temperature, the increase of the width, and the lowering of the temperature for the initial expansion.

Fig. 2 shows the differential dilatation curves of the alloys containing 6% Mn and various C contents, and 0.5% C and various Mn contents. In both curves of the 0.6% C-5.56% Mn and 0.46% C-5.67% Mn alloys, a two-stage expansion, at 200°~400°C and at 450°~550°C, is observed. The dilatational behaviours of these alloys are quite similar to those of the 0.91% C-4.37% Mn alloy shown in Fig. 1 and the same questions as mentioned above remain unexplained. In the dilatation

curve of higher C% alloys, such as the 0.69% C-5.56% Mn alloy and the 0.82% C-6.07% Mn alloy, expansions are observed only at 200°~400°C. This is considered to be due to the $\epsilon \rightarrow \gamma$ transformation rather than the decomposition of retained austenite.

Fig. 2 Thermal dilatation curves for quenched alloys.

Fig. 3 Thermal dilatation curves for quenched alloys.

Fig. 3 (curves a and c) shows the remeasured dilatation curves of both 0.91% C-4.37% Mn and 1.02% C-5.93% Mn alloys, which show different dilatational behaviours from those in Fig. 1, with good reproducibility. The curve (b) shows the dilatation curve of the

0.91% C-4.31% Mn alloy, which was held for a long time at the beginning temperature of expansion to complete the expansion at that temperature. As the expansion rate was too slow by holding at 200°C, the specimen was held at 250°C after holding for 4 hr at 200°C. At 250°C, a large expansion was observed after 21 hr of holding, and the specimen was heated up again at the heating rate of 3°C/min. Comparing with the expansion at about 550°C in the curves (b) and (a), the expansion in the curve (b) apparently decreases, suggesting the expansion during the isothermal holding at 250°C to be only due to the decomposition of retained austenite. The curve (d) shows the dilatation curve of subzero-treated 1.02% C-5.93% Mn alloy held for 20 min at the liquid nitrogen temperature. The decrease of the thermal expansion coefficient in the curve (d) and the increase of the hardness (Hv 216 $\rightarrow$ 306) were caused during the transformation by subzero-treatment. The configuration of the curve (d) is quite similar to the curve (a), and two expansions appear and they are inferred to be due to the two-stage decomposition of austenite.

From the above results, it has been clarified that two remarkable expansions at about 200°C and 500°C take place in the dilatation curves of the alloys containing intermediate carbon or manganese contents. This may show that the transitional structure between the complete α phase and the complete γ phase is formed after quenching. It appears that these expansions are caused by the same decomposition of retained austenite as that of some chromium steels. However, there remains an unexplained question for the low temperature range expansion. To clarify it, the specimens were tested by magnetic and X-ray analyses.

The thermal magnetic intensity curves of the three alloys which showed the two-stage expansion in their dilatation curves, are shown in Fig. 4. The increase in

Fig. 4 Thermal magnetic intensity curves for quenched alloys.

the intensity of magnetization is observed only at about 500°C, corresponding to the high temperature range expansion. But no change is observed at about 200°C, corresponding to the lower temperature range expansion. Namely, it becomes evident that the two-stage expansion observed in the dilatation curves of these alloys is not caused by the two-stage decomposition of retained austenite, and that only the expansion beginning at about 500°C corresponds to the decomposition of retained austenite. However, it remains unknown whether the

two-stage decomposition of austenite takes place in the Mn steel, or in what process there occurs the change in the decomposition temperature of the retained austenite from the lower temperature range of about 230°C in plain carbon steel, to the higher temperature range of

perature gradually raises with increasing of the manganese content. The two-stage decomposition of retained austenite is observed in the 2~3% Mn alloys, and only the first stage decomposition is observed in the lower Mn content alloys as in the plain carbon steels, and also

only the second stage decomposition is observed in the higher manganese content alloys. It is inferred that the decomposition of retained austenite in the low temperature range is discontinuous to that of the high temperature range, because the gradient of the first decomposition curve is negligibly small and the temperature difference between the first and the second decompositions is considerably large. That is, the thermally stable retained austenite is formed and gradually increases with the manganese content, and finally the complete retained austenite becomes thermally stable. The alloys containing a larger manganese content than the one which shows the two-stage decomposition of austenite under the quenched condition, are almost or wholly austenitic. Therefore, it may be appropriate to name the small amount of martensite retained in alloy the "retained martensite (M_R or α_R)" rather than the retained austenite (γ_R). It is considered that the decom-

Fig. 5 Thermal magnetic intensity curves for quenched alloys.

about 500°C with an increase of the Mn content. Fig. 5 shows the thermo-magnetic curves of the same series of the 1.0% C alloys with various manganese contents whose dilatation behaviours are given in Fig. 1. From the figure it can be known that the magnetization intensity of the low Mn alloys increases at about 230°C due to the decomposition of retained austenite in a way similar to that of the plain carbon steel, and that the increment gradually increases and the peak temperature raises to a higher range with the increasing manganese content. It also appears that the increment of the magnetization intensity over the higher temperature range gradually decreases with a further increase of the Mn content, and the peak disappears in the alloys containing more than 8% Mn. In the thermo-magnetic curves of the transitional composition alloys, the increases of the magnetization intensity is observed at two temperatures, a large increment at about 350°C and a small increment at about 600°C. From these results, it is confirmed that the two-stage decomposition of retained austenite occurs in these alloys.

Fig. 6 shows the relations between the transformation temperature of retained austenite into α measured by the thermo-magnetic curves and the manganese content. The first decomposition of retained austenite begins at about 200°C in the Mn free alloy, and linearly raises to higher temperature range with the increasing manganese content, but it begins at such a low temperature as 300°C, even in the 3% Mn alloy containing the largest amount of manganese among the tested specimens. On the other hand, the second decomposition of the retained austenite begins above 500°C in all the alloys. The second decomposition is observed at the lowest temperature in the 4.47% Mn alloy, but the decomposition tem-

position of austenite is generally caused by the heating whose curve passes across the transformation range in the isothermal transformation diagram. The isothermal transformation diagrams of some Cr steels show the two-stage decomposition of austenite⁽¹⁰⁾, that is, the bainitic

Fig. 6 Relations between $\gamma_R \rightarrow \alpha$ transformation temperature and manganese contents in 1.0% C-Mn-Fe alloy under quenched conditions.

transformation in the low temperature range and the pearlitic transformation in the high temperature range. However, Mn steels show a simple C curve similar to that of the carbon steel, even in the Hadfield steel containing a comparatively high Mn content⁽¹¹⁾⁽¹²⁾, and the C curve moves to a prolonged range with the increasing

(10) M. Cohen : Trans. ASM, 41 (1949), 485.

(11) Y. Imai : J. Japan Inst. Metals, 9 (1944), 259.

(12) Y. Imai and T. Saito : J. Japan Iron Steel Inst., 47 (1961), No. 7, 929.

Mn content⁽¹¹⁾⁽¹³⁾. Therefore, it may be reasonable to recognize that the decomposition temperature of austenite in both high and low temperature ranges proceeds to the higher temperature range with an increase of the Mn content. However, in the low Mn alloys the decomposition temperature of austenite in the low temperature range coincides with the beginning temperature of the first expansion in the dilatation curve shown in Fig. 1, whereas in the dilatation curves of the alloys containing more than 2.81% Mn the expansion begins at a lower temperature than the decomposition temperature of austenite. Therefore, it is probable that, in the dilatation curves of these transitional composition alloys, the two expanding phenomena are due to the simultaneous decomposition of austenite and the $\epsilon \rightarrow \gamma$ transformation.

In Mn steels, the $\epsilon \rightarrow \gamma$ transformation are observed in a temperature range of 200°~400°C, and the ϵ phase may be formed in the alloys which have comparatively less α and much γ , because the ϵ phase is a transitional phase which occurs during the transformation process of γ into α ⁽⁷⁾. Photo. 1 shows the X-ray diffraction pat-

And the expansion in the high temperature range seems to be due to the decomposition of austenite. It is known that the decomposition of retained austenite is a diffusional transformation, but the transformation of ϵ into γ is martensitic and has some temperature range. Therefore, a clear-cut expansion caused by the decomposition of retained austenite takes place in the dilatation curve and can be completed by an isothermal heating at the beginning temperature of the expansion, but the temperature range for the expansion caused by the $\epsilon \rightarrow \gamma$ transformation become extended and cannot be completed by the isothermal holding. Photo. 1 (c) and (d) show the X-ray diffraction patterns of both quenched and subzero-treated 1.02% C-5.93% Mn alloys corresponding to the thermal dilatation curves in Fig. 3 (c) and (d). The presence of the ϵ phase is expected by the configuration of the dilatation curve in the subzero-treated alloy, but no ϵ phase can be observed in the X-ray photograph.

From these results, it is clarified that there is no anomaly in the thermal dilatation curves of the transitional composition alloys, and the notion that the ex-

Photo.1 X-ray photographs for quenched steels.

terns of the 1.16%C-2.81% Mn alloy and the 0.91% C-4.37% Mn alloy. From the photograph it can be confirmed that the ϵ phase is observed with α and γ phases in the two typical specimens. It is also known that the expansion in the low temperature range in the former alloy is caused simultaneously by the $\epsilon \rightarrow \gamma$ transformation and the decomposition of retained austenite, and that the expansion in the low temperatures range of the latter alloy is caused by only the $\epsilon \rightarrow \gamma$ transformation.

pansion observed in the low temperature range is due to the decomposition of retained austenite, is not warrantable. All questions may be reasonably explained by the consideration that only the $\epsilon \rightarrow \gamma$ transformation occurs or the both the $\epsilon \rightarrow \gamma$ transformation and the decomposition of retained austenite simultaneously occurs in the transitional alloys.

Photo. 2 shows the microstructure of the alloys containing 1% C and various Mn contents. The microstructure of the alloy in which the decomposition of retained

(13) E. C. Bain : Alloying Element in Steel, ASM, (1934).

austenite is observed in the low temperature range only is shown in (a); in (b) the two-stage decomposition is observed, and in (c) and (d) the decomposition is observed in the high temperature range only, while the ϵ phase is formed in (c) and not in (d). In the microstructures

Photo. 2 Microstructures of quenched alloys, electropolished and etched by Nital. ($\times 300$)

of (c) and (d), some strain markings are observed in the austenite grain. As the ϵ phase is not observed in the X-ray diffraction pattern in (d), it is considered that the strain marking observed in the microstructure in (d) is mostly the stacking fault in austenite. It is very dif-

ficult to distinguish the ϵ phase from the stacking fault by optical micrographic observation.

Photo. 3 shows the microstructure of the specimen in which both the decomposition of retained austenite and the transformation of ϵ into γ are simultaneously ob-

Photo. 3 Microstructure of quenched C 1.16%, Mn 2.81% alloy, electropolished and etched by Nital. ($\times 600$)

served. The ϵ phase is occasionally observed in the part surrounded by martensite needles other than strain markings, as shown in Photo. 3. In the microstructure, it is also difficult to distinguish ϵ from α because of the occurrence of the α martensite needles and the ϵ along the (III) trace of γ . But, when a proper electrolytic condition is chosen for the etching of a specimen, only the ϵ phase can be etched, and after further etching with a chemical etchant, the α phase can be observed with ϵ . The photograph shows the etched structure by this method, and it is considered that the gray phase surrounded by white martensite needles is the ϵ phase. From the microstructural observations, it is inferred that the M_s temperature in this alloy is higher than the $\gamma \rightarrow \epsilon$ transformation temperature, and the untransformed γ is compressed by the expansion caused by the formation of martensite in the quenched specimen, whereby the transformation is accelerated. It seems that when the compression force is large enough to act as the driving force of the martensitic transformation, there occurs the transformation $\gamma_R \rightarrow \alpha$ and when the compressional force can not act as the driving force, there occurs the $\gamma_R \rightarrow \epsilon$ transformation and the ϵ phase remains retained. Moreover, it appears that when the internal force is not large enough to act as the driving force of the $\gamma \rightarrow \epsilon$ transformation, only the stacking faults are formed in γ .

Fig. 7 shows the ϵ phase formation range in the Fe-C-Mn alloy under a quenched condition. The data of carbon free Fe-Mn alloys were taken from Hansen's⁽¹⁴⁾, and the data on the 12% Mn alloy with varying carbon contents were taken from a previous report⁽⁷⁾. The complete ϵ phase cannot be obtained in Mn steel, under any condition^{(7)~(9)}, and therefore the coexisting ranges such as $\alpha + \epsilon$, $\alpha + \epsilon + \gamma$ and $\epsilon + \gamma$ are shown in Fig. 7. The curve max- ϵ corresponding to the maximum quantity of ϵ phase, determined by the experimental results,

(14) M. Hansen: Constitution of Binary Alloys, McGraw-Hill, (1958), 664.

is shown in the delta shape ϵ formation range. As can be expected from the previous paper, in the Fe-Mn alloys the ϵ phase is formed in the range of 9~25% Mn, but this range moves to the lower manganese side with the increasing carbon content, and finally the ϵ phase

is no longer formed. It is inferred that any alloying element which stabilizes the austenite in the Mn steel, may result in a similar configuration of the ϵ formation range.

IV. Summary

The anomalous expansions were occasionally observed in the thermal dilatation curves of some Fe-C-Mn alloys under quenched conditions, and it was clarified by dilatometry, and magnetic and X-ray analysis that these expansions were caused by the $\epsilon \rightarrow \gamma$ transformation. The ϵ phase formation range of Fe-C-Mn alloys was determined under quenched conditions. The results obtained were as follows:

(1) Two clear expansions were observed at about 200° and 500°C in the thermal dilatation curves of Fe-C-Mn alloys under quenched conditions, containing intermediate amounts of C and Mn, having a transitional structure between the complete α and the complete γ . It was known that these expansions were not entirely caused by the two-stage decomposition of retained austenite as shown in some Cr steels, but by only the $\epsilon \rightarrow \gamma$ transformation or both the decomposition of retained austenite and the $\epsilon \rightarrow \gamma$ transformation according to alloying composition.

(2) The two-stage decomposition of retained austenite was observed in 2~3% Mn and 1.0% C alloys. In the lower Mn content alloys, the decomposition of retained austenite was entirely the same as that of plain C steel, and in the higher Mn content alloys, only that in the high temperature range was observed.

(3) The ϵ phase formation range gradually moved to the lower Mn side and decreased to a narrow Mn range with the increasing C content.

(4) In the quenched high carbon steel, such as 1.0% C and 3.4% Mn alloys, the ϵ phase unexpectedly appears by the addition of small amount of Mn.

(5) Since the ϵ phase is the transitional phase which appears during the transformation process of $\gamma \rightarrow \alpha$, the ϵ phase formation range after quenching always lies between the complete α phase and the complete γ phase in the low Mn alloys with an increased amount of C, in the same manner when the amount Mn is increased in the high C alloys.

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Fig. 7 Phase relations in quenched Fe-C-Mn alloys.

formation range in the Fe-C-Mn alloys forms a delta shape as shown in Fig. 7. In the 1.0% C-Mn alloy, the ϵ phase is formed in the range of 3~4% Mn, and such a formation of the ϵ phase in such low manganese alloys has never been expected before. However, the configuration shown in Fig. 7 is reasonable, because the ϵ phase is the transitional phase in the process of the $\gamma \rightarrow \alpha$ transformation. It becomes clear that, when the C content is increased, the ϵ phase formation range of the Mn-Fe alloy must be intersected between the complete α phase range and the complete γ phase range in the same manner as that when the Mn content is increased in the low Mn alloy. Also, in the case of a high C steel, attention must be paid to the possible formation of the ϵ phase by the addition of small amount of Mn which has never been expected. Recently, White and Honeycombe⁽¹⁵⁾ reported the results of the X-ray measurement of the phase formed in a Fe-Mn-C alloy. But they were not aware of the above phenomena, because the composition range of their specimens are out of the ϵ formation range which is shown in Fig. 7. In Fe-Cr-Mn alloys, Ono⁽³⁾ reported a similar delta shape of the ϵ formation range. As austenite is stabilized by the addition of Cr in the Mn steel, the above consideration may be war-

(15) C. H. White and R. W. K. Honeycombe: J. Iron Steel Inst., 200 (1962), No. 6, 457.