

Photo. 10.

Transmission electron micrographs of structures in Fe-C alloys treated isothermally under high pressure

- (a) Aggregate; Fe-0.99%C alloy, 29 kbar, 450°C×1 min
 (b) Aggregate; Fe-0.42%C alloy, 38.5 kbar, 450°C×2 min
 (c) Aggregate; Fe-0.28%C alloy, 29 kbar, 500°C×30 sec
 (d) Columnar bainite; Fe-0.28%C alloy, 29 kbar, 300°C×3 hr

the data at atmospheric pressure are also shown for comparison.

Since the experiments were carried out at every 50°C, the temperatures of the occurrence of change structures were not exactly determined, and actually of these temperatures were not so clear because the mixed structures were often observed. The structures observed at higher temperatures above the nose were generally those which could be expected from the effects of pressure on the equilibrium phase diagram. However, in the A-9 alloy and SK3 steel containing higher carbon than others, some singular structures were observed while the pearlite structure does not appeared through the whole temperature range at both high pressures.

Photograph 10 shows the transmission electron micrographs of the "aggregate" and the "columnar bainite" structures observed in temperature ranges below the nose; Photos. 10 (a), (b), and (c) are the "aggregate" structures obtained in three kinds of Fe-C alloys and Photo. 10 (d) is the "columnar bainite" structure in the A-2 alloy. With an increase of carbon content, the appearance of carbides in these bainitic structures become increasingly lamellar, but the individual area of these structures is generally small and its morphology is rather cellular than lath-like. On the other hand, it is reported²³⁾ that the carbides in upper bainite obtained at 1 atm generally arrayed along the longitudinal axis of the lath. Therefore, it is possible to think that the "aggregate" structure is different from the upper bainite because the arrangement of carbides in the former is more complex than the latter. Furthermore, it is observed that each of the carbide lamellas in the "columnar bainite" arranged more regular and developed wider than in the "aggregate" structure.

IV. Discussions

1. TTT Diagrams

The forms of the TTT diagrams obtained under

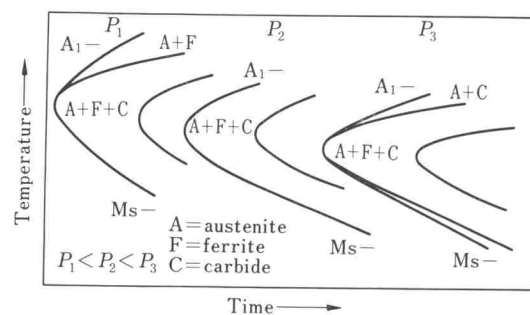


Fig. 5. Schematic illustration to the change of TTT diagram of a hypoeutectoid steel due to high pressure. Hydrostatic pressures increase from P_1 to P_3

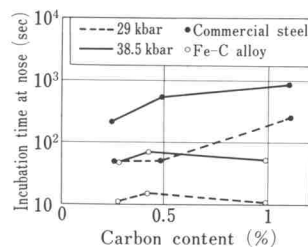


Fig. 6. Effect of pressure on the incubation time at the nose in TTT diagrams of Fe-C alloys and commercial steel

high pressure were not essentially different from that at 1 atm, and the transfer of curves with the change of carbon content or pressure could be explained on the basis of the change of phase diagram under high pressure. The temperature at the nose decreased in a same extent with a decrease of the eutectoid temperature caused by pressure. The incubation time before transformation was extended with an increase of pressure, and such retardation became more remarkable in a lower temperature range.

The representative behaviours of a TTT diagram under high pressure are illustrated schematically in Fig. 5. When the pressure increases from P_1 to P_3 , the characteristics of a hypoeutectoid type TTT diagram change to eutectoid or hypereutectoid type accompanying with the increasing pressure. Additionally, the incubation time increases and oppositely

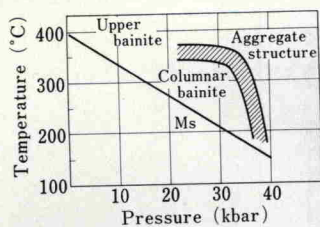


Fig. 7. Effect of pressure and temperature on the changes among various bainitic structures of Fe-0.42%C alloy

the temperature of the nose decreases at the same time. Although the decrease of the nucleation rate for the decomposition of austenite is reported⁵⁾ to be far greater than that of the growth rate thereafter, when the pressure increases up to P_3 , namely when the form of isothermal transformation changes to hyper-eutectoid type, it is anticipated that the decrease of the nucleation rate would be lessened by the precipitation of carbides providing nucleation sites suitable to start the transformation.

Figure 6 shows the effect of pressure on the incubation time at the nose in TTT diagrams of Fe-C alloys and commercial steels. In this figure, it is shown that the incubation time is always longer in the commercial steels than in the high purity alloys, and this tendency is entirely the same with that in the case of atmospheric pressure. With an increase of pressure from 29 to 38.5 kbar, the time before the start of transformation at the nose temperature increased by approximately five times for both materials, and there could not be detected any significant difference between these materials on the pressure dependence of the retardation. From the fact that the activation volumes for the growth of pearlite had different values between the Fe-C alloys and the commercial steels,⁷⁾ it had been anticipated that the pressure dependence of the retardation on these two kinds of the materials would be different. However, as the nucleation rate would affect the start of transformation, and as the decrease of the nucleation rate is larger than that of the growth rate under high pressure, it is thought that the resultant retardation caused by an increase of pressure showed the similar tendency in both materials after set off other factors.

2. Microstructures

The microstructures obtained under high pressure were those which corresponded generally to the changes of the phase diagram caused by pressure. In place of the acicular bainite structure commonly observed at atmospheric pressure, some singular structures were obtained after the isothermal transformation at relatively lower temperature under high pressure. Namely, the structure changed to "columnar bainite" or "aggregate" structure with the changes of pressure and temperature. Figure 7 is the summary of the results of Radcliffe *et al.*,⁵⁾ Nilan,⁶⁾ and the present study, and shows the relationship between pressure and temperature on the structure changes of the A-4 alloy. The boundaries among these structures are

not clear, and are thought to be changed gradually as would be understood from Photo. 4 which showed the combined effects of pressure, temperature and composition on the change of "columnar bainite" structure. Although Nilan⁶⁾ pointed out that the effect of pressure on the temperature at which "columnar bainite" would appear nearly corresponded to the one on the A_1 temperature, it is not possible to explain that such structural transition was observed with an increase of carbon content as obtained in the present study. As the increase of carbon and the presence of impurities in commercial steels shifted the Ms temperature lower, and as the increase of pressure also depressed it, it is possible to think that the increase of carbon or some impurities has a similar effect to the increase of pressure. By reason of the above, the structural transition from "columnar bainite" to "aggregate" would occur with an increase of carbon content.

In regard to a temperature range which was classified in the present study as the one in which "aggregate" structure would be expected, Nilan noted in his paper that the upper bainite structure had been observed in that range. However, the transmission electron microscopy showed that the structure obtained in the present study was quite similar to the "aggregate" structure which was shown by Radcliffe *et al.* Such difference in structures may be caused by the difference in the experimental procedures.

Namely, Nilan used a tetrahedral anvil type apparatus, and on the other hand, Radcliffe *et al.* and the present authors used a piston-cylinder type and a girdle type apparatus respectively. In general, tetrahedral type is said to be suitable to obtain better hydrostatic pressure, but in the present study, no structural difference was observed between two principal directions of a specimen, parallel and perpendicular to the loading axis of the press.

When the commercial SK3 steel was treated at 29 and 38.5 kbar, the shape of the precipitated carbide changed from spherical or columnar in the case of 29 kbar to acicular in 38.5 kbar. On the contrary such change was not observed in the high purity A-9 alloy, namely, the shape of the carbide precipitated in this alloy was spherical or columnar at all these examined pressures. It is obvious from the result of Radcliffe *et al.* and of the additional experiments of the present authors that this fact cannot be attributed to the difference of carbon content in these two kinds of specimens. The exact reason is not obvious yet, but one of the cause to show the difference is possibly due to the effect of impurities contained in the SK3 steel.

As the preparation of thin foils for transmission electron microscopy was very difficult for the specimens in which the carbides were precipitated, those carbides were extracted by 5% HNO_3 -ethanol solution and were determined the crystallographic structures by X-ray diffraction. As the result, both spherical and acicular carbides showed the same diffraction pattern of Fe_3C . This result would be understood from the fact that the phase Fe_3C has the largest density compared with other carbides and so that it must be