KAWASAKI STEEL TECHNICAL REPORT
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Synopsis :
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The body can be viewed from the next page.

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Shingo SATO ** Teiichi ENAMI ** Susumu MATSUI **
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An investigation has been made of the influence of silicon, manganese, phosphorus and austenitizing temperature on the temper embrittlement of $2\frac{1}{4}$Cr-1Mo steel which is well known for its higher susceptibility to the embrittlement among Cr-Mo pressure vessel steels. According to this investigation the following tendency is observed.

(*1*) *Decrease in silicon, manganese or phosphorus content lowers the susceptibility. However, lowering of silicon or manganese content decreases the strength.*

(*2*) *Though raising of austenitizing temperature promotes the susceptibility, this procedure increases the strength.*

It is concluded that Cr-Mo steels with high strength and low susceptibility to temper embrittlement can be obtained by lowering phosphorus content sufficiently or quenching from higher austenitizing temperature under the condition of low silicon, manganese and phosphorus contents.

1 Introduction

Temper embrittlement is one of the most important characteristics in the quality of Cr-Mo steels for pressure vessels. Temper embrittlement signifies the phenomenon of deterioration in toughness which occurs when the steel is heated in the temperature range about 350 to 550°C for a long period of time, or when it is slowly cooled through this temperature range. The embrittlement is characterized by the development of intergranular fractures in the brittle fracture of surface. Cr-Mo steels are frequently employed in a temperature range which causes temper embrittlement; and therefore, for maintenance reasons, great attention is paid to the embrittlement.

Research has been done on temper embrittlement since long ago, and recent developments in analysis technology, such as the Auger electron spectrum analysis, have furthered detailed research, but a unified interpretation of its mechanism has not been found as yet. Practical measures towards reduction of susceptibility to embrittlement, however, have resulted from these researches.

Our company has taken up several of these measures, and has succeeded in producing various Cr-Mo steels, especially $2\frac{1}{4}$Cr-1Mo steel, which possess strong resistance to temper embrittlement. This report will describe the embrittlement in various Cr-Mo steels, and the influences of chemical composition on embrittlement of $2\frac{1}{4}$Cr-1Mo steel, highly susceptible to embrittlement in the Cr-Mo steels, as well as influence of austenitizing temperature which also affects the strength of the steel.

2 Temper Embrittlement of Various Cr-Mo Steels

Embrittlement of Cr-Mo steel occurs when used as pressure vessels in the temperature range which causes embrittlement, for a long period of time. It takes a long time to confirm characteristics of embrittlement caused by such retention of constant temperature. Therefore, many treatments for embrittlement acceleration, called step cooling, are employed in evaluating the characteristics of embrittlement in a short period of time. In this report, step cooling condition indicated in **Fig. 1** has been employed in embrittlement treatments unless otherwise stated. After the above treatment is applied, the fracture appearance transition temperature (${}_{V}T_S$) of $2\frac{1}{4}$Cr-1Mo steel in the charpy impact test almost equals the ${}_{V}T_S$ after 2 000 h of retention at 450°C, as shown in **Fig. 2**. Further, in this case, the ${}_{V}T_S$ hardly changes in the range between 2 000 h and 5 000 h.

Cr and Mo contents vary according to grades of Cr-Mo pressure vessel steels. Uses for these various types are determined according to their resistance to hydrogen[1)] and their elevated temperature strength.

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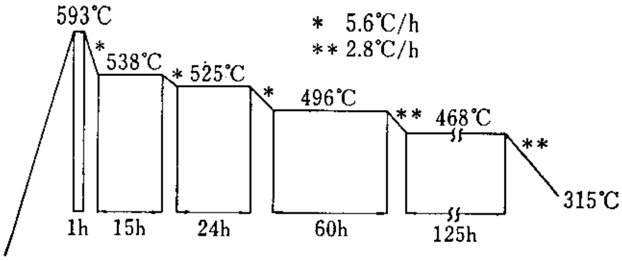

Fig. 1 Step cooling condition for the experiments

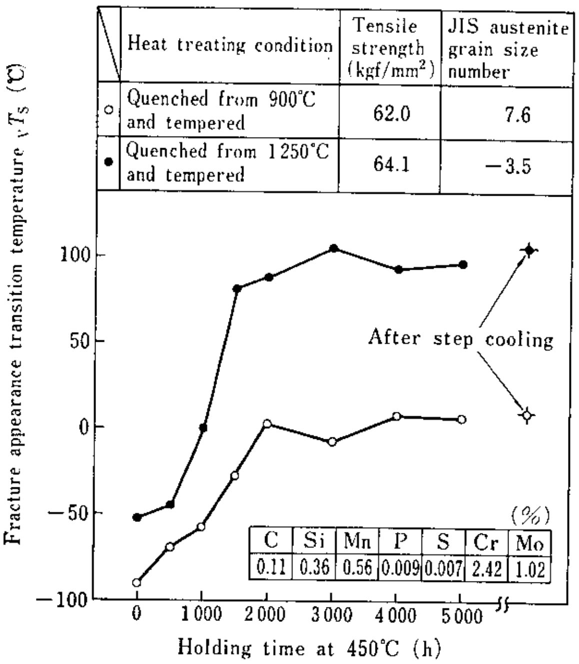

Fig. 2 Change in fracture appearance transition temperature during holding at 450°C compared with step cooling treatment

A comparison of temper embrittlement among such steels as carbon steel and Cr-Mo steels is shown in **Fig. 3**. Increment of the ${}_{V}T_{S}$ by step cooling ($\Delta_{V}T_{S}$) is greatly influenced by not only the composition but also microstructure of the steel. It is generally highest in 2¼Cr-1Mo steel with bainitic structure, and tends to be lower in either Cr-Mo steels with higher or lower Cr content. On the other hand, a low ${}_{V}T_{S}$ after step cooling can be obtained in bainitic 2¼Cr-1Mo steel, 3Cr-1Mo steel, and 5Cr-½Mo steel.

Characteristics of temper embrittlement are evaluated according to susceptibility to embrittlement as indicated by $\Delta_{V}T_{S}$, or the toughness of the steel after it has received embrittlement treatment. Thus, it is necessary to increase the toughness before embrittlement and reduce the susceptibility to embrittlement simultaneously.

3 Temper Embrittlement of 2¼Cr-1Mo Steel

Temper embrittlement is mainly influenced by the chemical composition and heat treatment conditions. Within these heat treatment conditions, however, the cooling rate from the austenitizing temperature is restricted according to the thickness of the steel, and conditions for tempering and post-weld heat treatment are restricted according to the grades, thicknesses of steel, and levels of strength required; thus, selection of heat treatment conditions is very limited. This chapter will deal with research results concerning influences of chemical composition, mainly Si, Mn, and P, on 2¼Cr-1Mo steel, which has comparatively high susceptibility to embrittlement. This chapter will also deal with measures for reducing

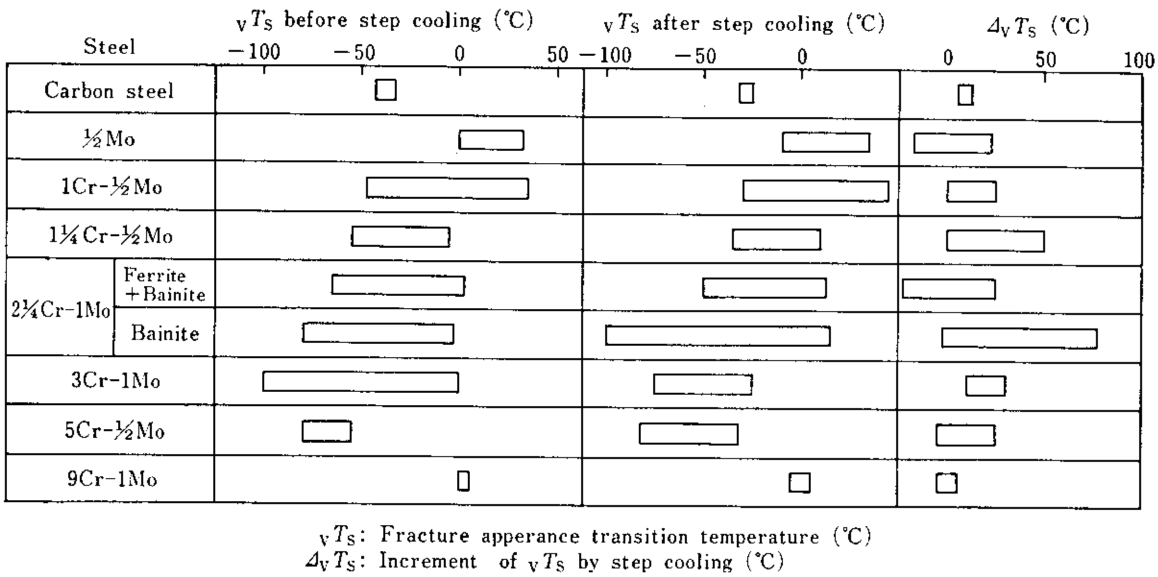

Fig. 3 Temper embrittlement of Cr-Mo steels

Table 1 Chemical compositions of steels tested

(%)

Steel	C	Si	Mn	P	S	Cr	Mo	Al	N	V	Nb
1	0.13	0.02	0.50	0.008	0.006	2.25	1.04	0.010	0.0085	—	—
2	0.12	0.02	0.50	0.014	0.007	2.25	1.07	0.011	0.0083	—	—
3	0.11	0.02	0.49	0.021	0.003	2.16	1.00	0.017	0.0076	—	—
4	0.11	0.16	0.50	0.009	0.003	2.20	1.03	0.014	0.0073	—	—
5	0.12	0.16	0.51	0.015	0.003	2.19	1.05	0.016	0.0080	—	—
6	0.12	0.26	0.51	0.009	0.003	2.22	1.05	0.016	0.0067	—	—
7	0.12	0.25	0.51	0.016	0.007	2.21	1.04	0.014	0.0076	—	—
8	0.12	0.36	0.50	0.005	0.004	2.20	1.04	0.013	0.0077	—	—
9	0.10	0.45	0.54	0.009	0.010	2.58	1.04	0.003	0.0019	—	
A	0.14	0.07	0.50	0.009	0.004	2.42	1.00	0.013	0.0050	0.014	—
B	0.12	0.07	0.50	0.009	0.004	2.41	1.01	0.012	0.0058	0.052	—
C	0.12	0.07	0.50	0.009	0.004	2.41	1.01	0.012	0.0058	0.083	—
D	0.13	0.27	0.50	0.009	0.004	2.42	1.00	0.012	0.0048	0.014	—
E	0.13	0.26	0.50	0.010	0.003	2.40	1.01	0.012	0.0047	0.015	0.046
F	0.13	0.26	0.10	0.010	0.003	2.42	1.00	0.013	0.0054	0.013	—
G	0.12	0.26	0.30	0.010	0.003	2.42	1.00	0.013	0.0054	0.013	—
H	0.14	0.27	0.69	0.010	0.004	2.40	1.00	0.013	0.0046	0.014	—
I	0.13	0.27	0.97	0.010	0.004	2.40	1.00	0.013	0.0046	0.014	—
J	0.13	0.46	0.11	0.010	0.003	2.41	1.00	0.013	0.0065	0.013	—
K	0.14	0.28	0.49	0.008	0.013	2.29	1.01	0.011	0.0055	0.010	—

embrittlement, and adjustment of austenitizing temperature for obtaining high strength and low susceptibility to embrittlement, especially when steel thickness is increased. Steel ingots of 50 kg produced in a vacuum induction furnace have been employed for the above research. Their chemical compositions are given in **Table 1**.

As shown in **Fig. 4**, there is a good relationship between the strength at room temperature and that at an elevated temperature, which is one of the important properties when the steel is used at temperatures below approximately 500°C. Hereinafter, strength at room temperature is given for the evaluation of strength in general.

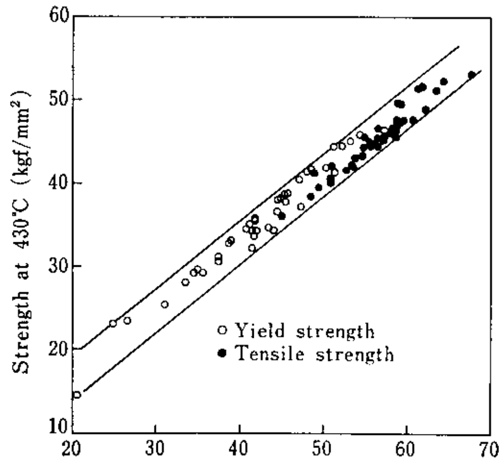

Fig. 4 Relation between strength at 430°C and the one at room temperature for $2\frac{1}{4}$Cr-1Mo steel

3.1 Influence of Chemical Composition

3.1.1 Silicon and phosphorus

P, Sn, Sb and As are tramp elements well known for promoting embrittlement, but the amounts of Sn, Sb, and As can be sufficiently reduced through selection of raw materials used for steelmaking; so, P is the only element which remains in question[2].

Si is also well known for promoting embrittlement[3], and decrease in Si content is often used as one of the measures for reducing embrittlement in steel. Concerning the effect of Si on embrittlement, Inoue et al.[4] found that the intergranular segregation of P is not altered by Si, but that Si increases susceptibility to

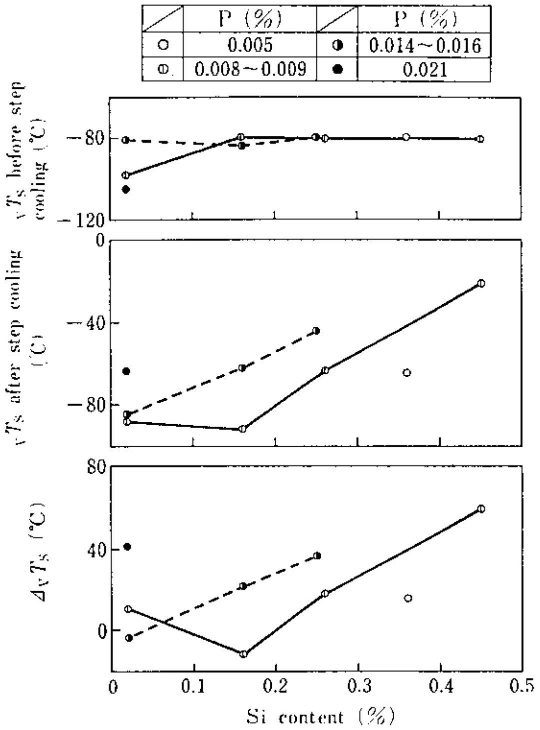

Fig. 5 Influence of silicon and phosphorus on temper embrittlement of 2¼Cr-1Mo steel quenched from 900°C with cooling rate of 500°C/min and tempered at 690°C for 17 h

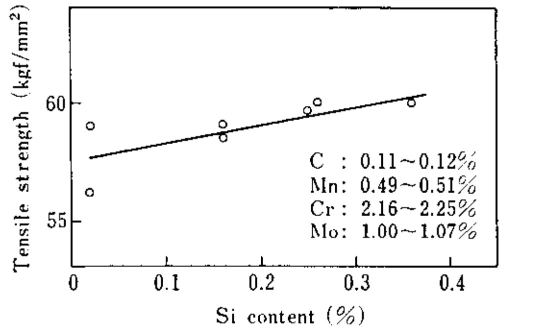

Fig. 6 Influence of silicon on tensile strength at room temperature for 2¼Cr-1Mo steel. Heat treating condition used is the same as in Fig. 5

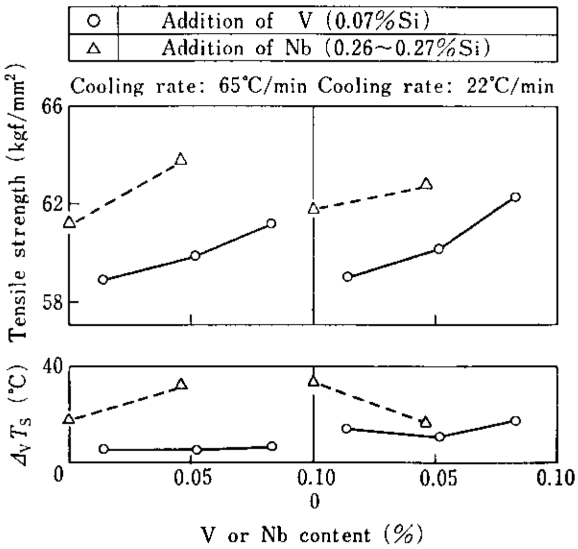

Fig. 7 Influence of vanadium or niobium on tensile strength at room temperature and temper embrittlement of 2¼Cr-1Mo steel quenched from 950°C with cooling rates of 65 and 22°C/min and tempered at 690°C for 7.5 h

embrittlement even when the intergranular segregation of P remains the same.

Steels 1–9 in **Table 1** were heated to 900°C, then cooled at an average cooling rate from a temperature of 800°C to 400°C (simply abbreviated as "average cooling rate" hereinafter) of 500°C/min, then tempered at 690°C for 17 h. **Fig. 5** shows the results of an investigation into the influence of Si and P on temper embrittlement. In **Fig. 5**, austenite grains had to be fine, with a grain size number of 7.9 to 9.9 according to JIS (Japanese Industrial Standards), and the microstructure was dominantly martensite. Before step cooling, $_vT_s$ depends very little on Si or P content, but Δ_vT_s or $_vT_s$ after step cooling raises with an increase in amounts of Si and P. The relation between Si content and Δ_vT_s in steels with a P content of 0.008%–0.009% and those with a P content of 0.014%–0.016% shows that increment of Δ_vT_s with Si is hardly influenced by the amount of P. Therefore, there are two different systems for reducing embrittlement: that in which Si content is lowered, and that in which high Si content is accompanied by ultra low P content.

Steel with a low Si content is advantageous in creep-rupture strength under limited conditions of Larson-Miller's parameter[5], but it shows lowers strength at room temperature as indicated in **Fig. 6**. Therefore, when strength at room temperature, or at high temperatures plays an important role, strengthening measure must be considered.

One example of such strengthening measures employs V or Nb. **Fig. 7** shows strength and embrittlement in relation to amounts of V and Nb, when steels A–E of **Table 1** are quenched from 950°C with average cooling rates of 65 and 22°C/min, then tempered at 690°C for 7.5 h. Both V and Nb have the effect of increasing strength without raising Δ_vT_s. It is necessary, however, to give sufficient consideration to weldabilities in fixing the permissible content of these elements.

No such problem exists when steel possesses a high

Si content and an ultra low P content. The refining process for achieving ultra low P steel is shown in **Fig. 8**. This process makes the production of steel with a P content of 0.005% or less possible. **Fig. 9** indicates the strength and temper embrittlement of 0.35% Si-0.003% P steel[6] produced using the above process, in relation with a tempering parameter of, $T(20 + \log t') \times 10^{-3}$. A high degree of strength and resistance to temper embrittlement can be gained.

3.1.2 Manganese

Like Si, Mn is an element also well known for promoting embrittlement. Unlike Si, however, Mn greatly influences hardenability. The degree to which Mn content can be reduced becomes limited as thickness of material increases, since it is required to ensure hardenability, that is, to hinder the formation of ferrite. In order to observe this tendency, the

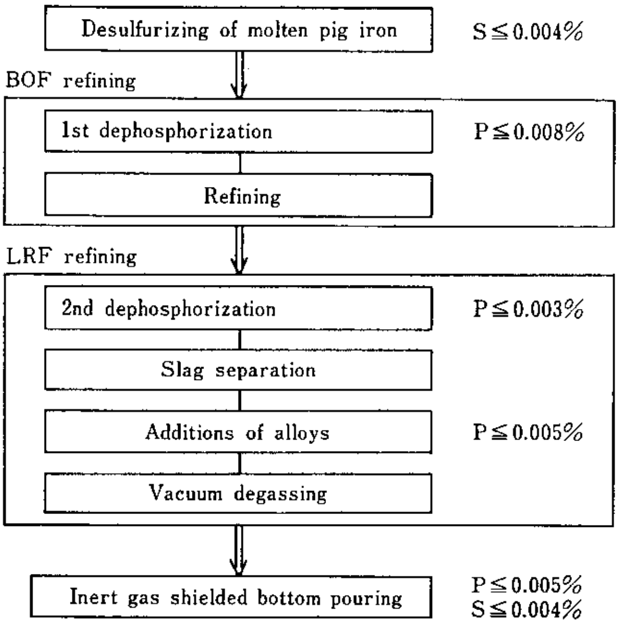

Fig. 8 Refining process of ultra low phosphorus steel using BOF and LRF

(%)

C	Si	Mn	P	S	Cr	Mo	Al
0.14	0.35	0.56	0.003	0.004	2.18	1.02	0.018

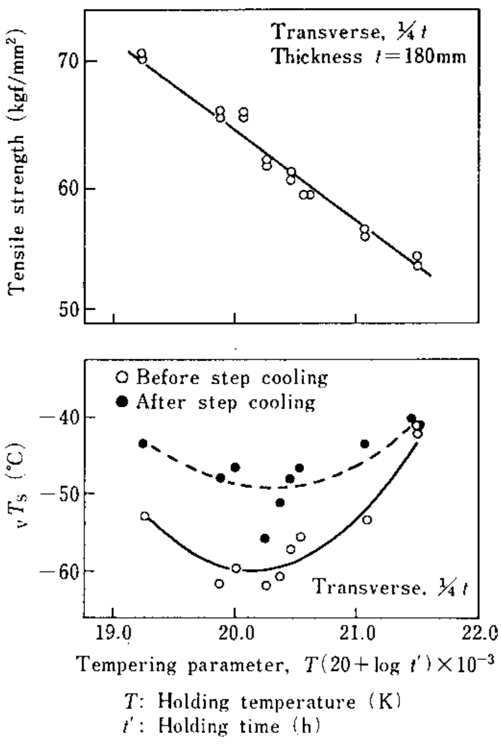

Fig. 9 Changes in tensile strength at room temperature and temper embrittlement with tempering parameter, $T(20 + \log t') \times 10^{-3}$, for $2\frac{1}{4}$Cr-1Mo steel plate with high silicon and ultra low phosphorus content, water quenched from 930°C

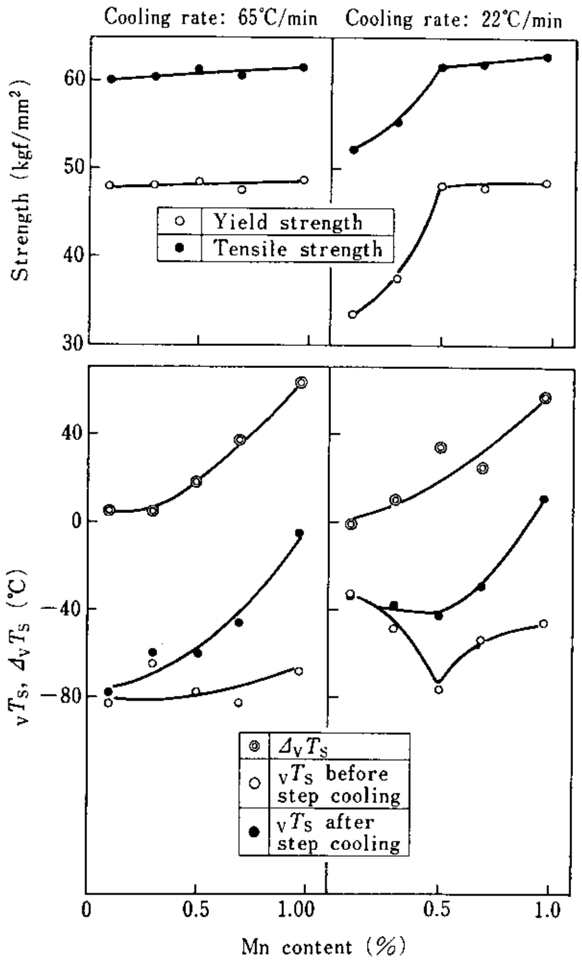

Fig. 10 Influence of manganese on strength at room temperature and temper embrittlement of $2\frac{1}{4}$Cr-1Mo steel quenched from 950°C with cooling rates of 65 and 22°C/min and tempered at 690°C for 7.5 h

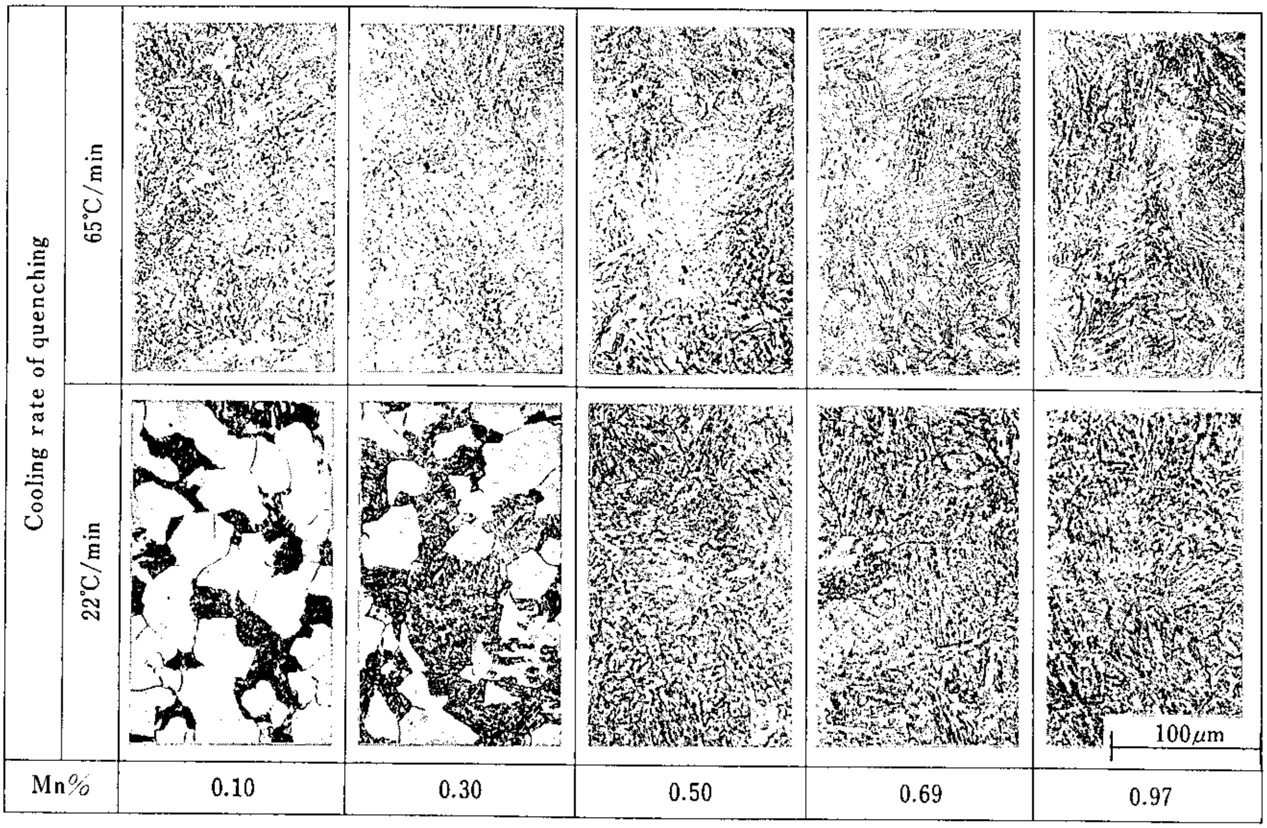

Photo. 1 Optical micrographs of $2\frac{1}{4}$Cr-1Mo steels with various manganese contents

influence of Mn on strength and embrittlement was studied by applying heat treatment under the same conditions as shown in **Fig. 7** to steels A, D, and F–J of **Table 1**. Further comparison was made between the effects of Si and Mn.

The influence of Mn on strength and embrittlement is shown in **Fig. 10**, and the influence on the microstructure is shown in **Photo. 1**. In cases where the microstructure consisted fully of bainite, the decrease in strength by lowering the Mn content was extremely small. This lowering of the Mn content decreases $\Delta_V T_S$, and also somewhat decreases the ${}_VT_S$ before step cooling. In particular, $\Delta_V T_S$ can be made extremely low by reducing Mn content to 0.3% or less. On the other hand, a large amount of ferrite is formed when steels with Mn content of 0.3% or less are cooled at an average cooling rate of 22°C/min. In this case strength decreases sharply and the ${}_VT_S$ before step cooling goes up, even though $\Delta_V T_S$ remains extremely low. Thus, it can be said that if Mn content is kept down to the minimum amount required to hinder formation of ferrite, characteristics of embrittlement may be improved without much decrease in strength.

In general, $\Delta_V T_S$ tends to be lower in steel with mixed structures of ferrite and bainite than that with fully banitic structure[7], but the reduction of $\Delta_V T_S$ by lowering Mn content is not by the formation of ferrite. Concerning the role of Mn in promoting embrittlement, Tanaka et al.[7] explain that Mn promotes diffusion of P. But the following results suggest that there is an interaction between Mn and S as a probable factor affecting the embrittlement. Heat treatment under the same conditions as indicated in **Fig. 10** was applied to steels D and K of **Table 1**. Steels D and K have different S contents, and their characteristics of embrittlement are shown in **Fig. 11**. One notices that an increase in S content is accompanied by a rise in the ${}_VT_S$ before step cooling, but that $\Delta_V T_S$ decreases.

Fig. 12 shows the influence of Mn on strength and embrittlement of steels with constant Si + Mn content of approximately 0.57%, although proportions of Si and Mn contents vary in each steel (steels A, G, and J of **Table 1**), after heat treatment was applied to them under the same conditions as those indicated in **Fig. 10**. The ${}_VT_S$ before step cooling increased as Mn content decreased. This tendency was most salient when the average cooling rate was 22°C/min, because formation of ferrite occurs in low Mn-high Si steels. These results indicate that hardenability is affected more by Mn than by Si. On the other hand, $\Delta_V T_S$ tends to rise to a small extent in accordance with

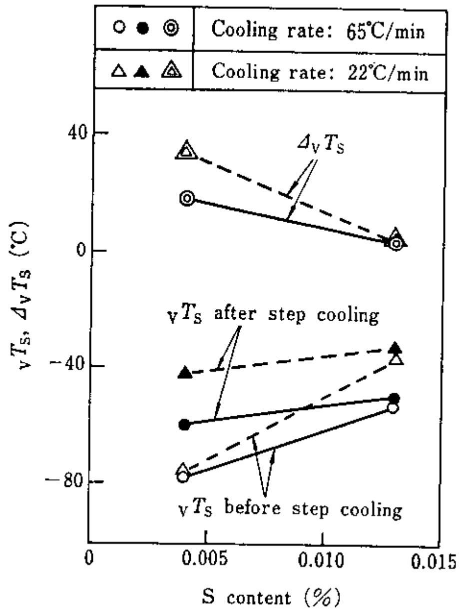

Fig. 11 Influence of sulfur on temper embrittlement of $2\frac{1}{4}$Cr-1Mo steel. Heat treating condition is the same as in Fig. 10

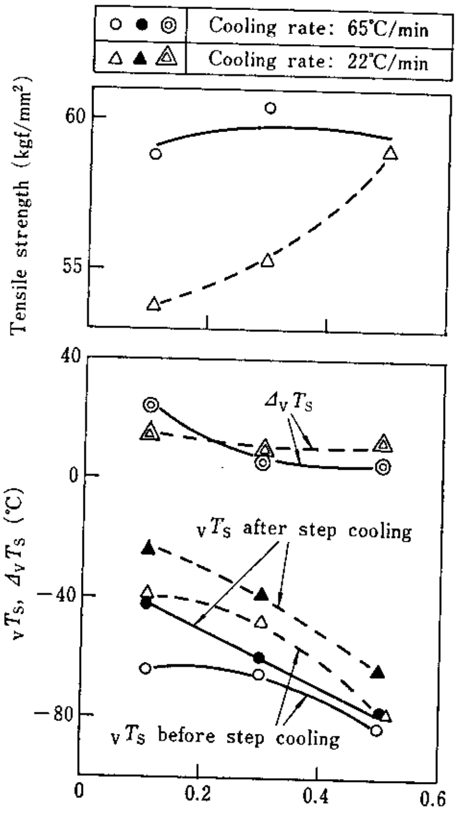

Fig. 12 Influence of manganese on tensile strength at room temperature and temper embrittlement of $2\frac{1}{4}$Cr-1Mo steel when the sum of silicon and manganese content is in the range from 0.56% to 0.57%. Heat treating condition is the same as in Fig. 10

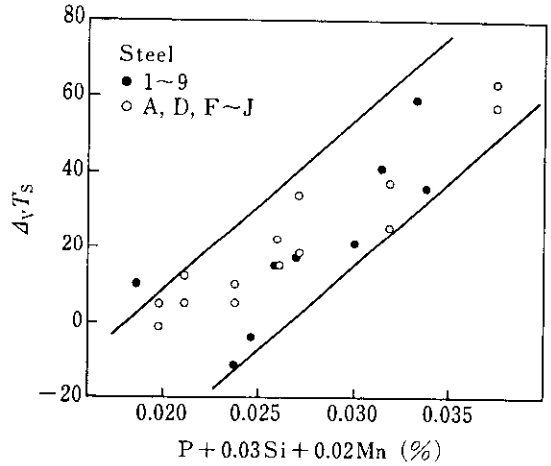

Fig. 13 Influence of phosphorus, silicon and manganese on increment of fracture appearance transition temperature of $2\frac{1}{4}$Cr-1Mo steel

decreases in the amount of Mn, which implies that embrittlement effect is a little smaller in Mn than in Si. Thus, it can be concluded that among steels with constant amounts of Si + Mn, low Si-high Mn steel has a little better balance between strength and susceptivility to embrittlement.

As stated before, Si, Mn, and P each influence the characteristics of temper embrittlement in a different manner. Therefore, considering the degree of influence of these elements on $\Delta_V T_S$, the parameter indicating $\Delta_V T_S$ has been determined as (%P" + 0.03 (%Si) + 0.02 (%Mn). There is an almost linear relationship between this parameter and $\Delta_V T_S$ as it is indicated in **Fig. 13**, and it is necessary to sufficiently decrease the value in the parameter in order to lower $\Delta_V T_S$. The lowering of Si, however, produces a reduction in strength no matter what thickness the material may be, and lowering of Mn reduces strength especially in extremely thick materials; so, it becomes necessary to consider strengthening measures which will be discussed in the next chapter.

3.2 Influence of Austenitizing Temperature

Raising austenitizing temperature is one of the effective methods of improving strength. The reason for this is that hardenability or resistance to softening by tempering is heightened through coarsening of austenite grains or through unification of alloying elements[8].

Fig. 14 shows transformation characteristics through continuous cooling of steels with 0.10%, 0.30%, and 0.50% Mn contents (steels F, G, and D of **Table 1**) with changes in austenitizing conditions. When the austenitizing temperature is raised, formation of ferrite is delayed. For example, when steel with a

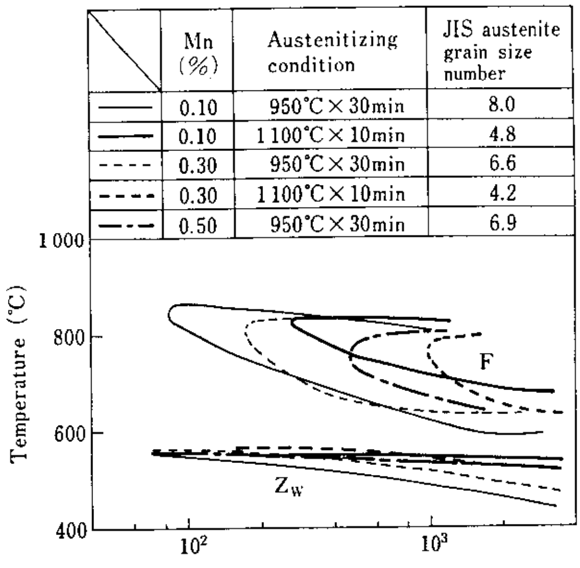

Fig. 14 Change in transformation characteristics with the change of manganese content and austenitizing condition for 2¼Cr-1Mo steel

0.10% Mn content is heated to 1 100°C, the position of ferrite nose may approximate to the steel with a 0.4% Mn content is heated to 950°C. Assuming that improvement in hardenability through raising austenitizing temperature can mainly be attributed to the coarsening of austenite grains, altering the JIS austenite grain size number by one equals changing Mn content by about 0.3%, from the viewpoint of critical cooling rate for formation of ferrite.

Steels 3–8 in **Table 1** were cooled from 1 200°C with an average cooling rate of 500°C/min, and their strengths were tested after tempering under the same conditions as set in **Fig. 6**. In **Fig. 15**, the results of

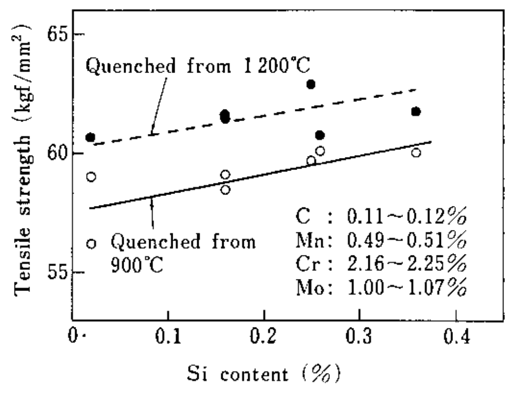

Fig. 15 Influence of austenitizing temperature prior to quenching on tensile strength at room temperature for 2¼Cr-1Mo steel. Cooling rate of quenching and tempering conditions are the same as in Fig. 5

the above experiment are compared with those of **Fig. 6**. Martensite is the main microstructure in both, but material austenitized at 1 200°C has stronger resistance to softening by tempering, and maintains a higher strength after tempering. When steels with a 0.02% Si content are austenitized at 1 200°C, they show the same strength as when steels with a 0.3% Si content were austenitized at 900°C.

By raising the austenitizing temperature, it is possible to increase the strength of the metal. This comes about as a result of improving hardenability in cases of steels with low Mn contents, and heightening resistance to softening by tempering in cases of steels with low Si contents.

It is not very desirable to raise the austenitizing temperature from the viewpoint of temper embrittlement. Raising of the austenitizing temperature induces coarsening of austenite grains, and it has already been proven in **Fig. 2** that coarsening of these grains promotes embrittlement. Two reasons can be given to explain this effect:

(1) As the grains become coarse, increases can be seen in the amounts of intergranularly segregated elements, which promote embrittlement.
(2) Even if the amount of intergranularly segregated elements which promote embrittlement remains the same, intergranular fracture occurs easily as the grains become coarse.

A test piece having gone through the embrittlement treatment in **Fig. 2**, was employed in measuring the maximum value of the ratio between the peak height of P and Fe, I_P/I_{Fe}, using the Auger electron spectrum analysis to determine the degree of intergranular segregation of P. **Fig. 16** shows the relationship among $\Delta_V T_S$, the ratio of intergranular fracture, and maximum value of I_P/I_{Fe} of both fine and coarse grained steel. With fine grains, the actual ratio of intergranular segregation of P should be larger than the measured value, since complete intergranular fracture has not occurred. If step cooling is applied as an embrittlement treatment, the degree of intergranular segregation of P would be smaller in a fine grained steel than in a coarse grained one, even if the degree is assumed to be the same as when the maximum value of I_P/I_{Fe} was divided by the ratio of intergranular fracture, which supports the aforementioned reason **(1)**. When embrittlement treatment is applied at a constant temperature of 450°C, the maximum value of I_P/I_{Fe} for coarse grain material would be approximately the same as that (the actual measured value) for fine grain material; but the ratio of intergranular fracture would be larger, and $\Delta_V T_S$ higher, so the above reason **(2)** may seem more appropriate. In any case, it is necessary to establish a composition which

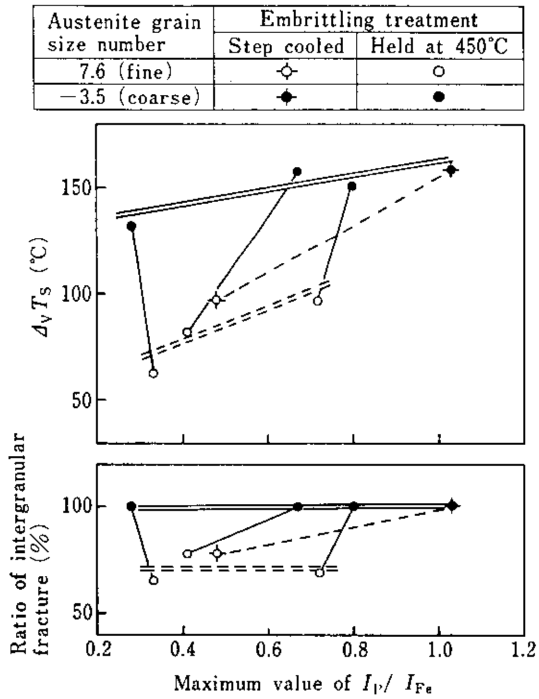

Fig. 16 Relation between $\Delta_V T_S$, intergranular fracture and segregation of phosphorus at boundary of fine and coarse austenite grains. Steel used is the same as in Fig. 2. $\Delta_V T_S$ includes increment of fracture appea rance transition temperature not only by step cooling but also by holding at 450°C. This case is limited only to this figure.

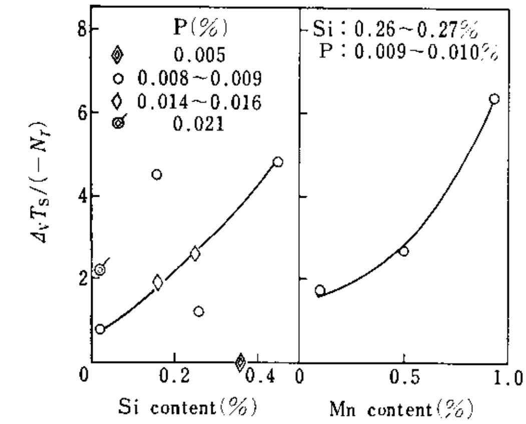

Fig. 17 Influence of manganese, silicon and phosphorus on increment of $\Delta_V T_S$ due to coarsening of austenite grain in $2\frac{1}{4}$Cr-1Mo steel

(%)

C	Si	Mn	P	S	Cr	Mo
0.08	0.06	0.38	0.011	0.004	2.05	0.96

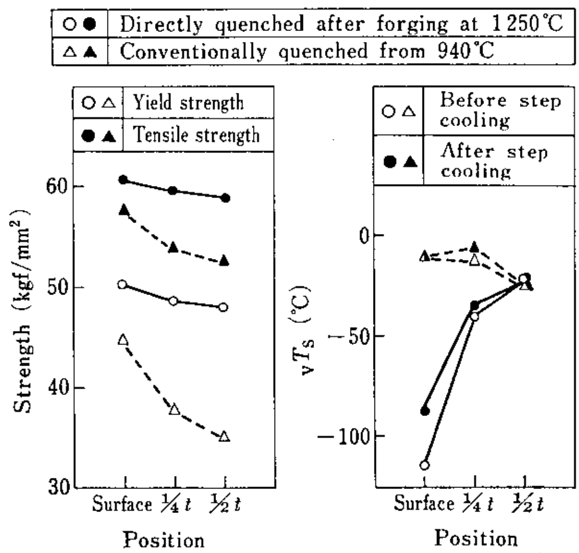

Fig. 18 Strength at room temperature and temper embrittlement of $2\frac{1}{4}$Cr-1Mo steel with low silicon and manganese contents directly and conventionally quenched and tempered at 690°C for 15 h

will inhibit embrittlement even with coarse austenite grains.

On some steels in **Table 1**, $\Delta_V T_S$ was measured when the austenite grain size number was altered by changes in the austenitizing temperature. Then, through linear approximation, the degree of influence of austenite grain size number on $\Delta_V T_S$ and $\Delta_V T_S/(-N_\gamma)$ was obtained, where N_γ means the JIS austenite grain size number. The value is always larger than zero, as can be seen in **Fig. 17**, but the value decreases in accordance with decreases in the amount of either Si, Mn, or P. Therefore, by lowering Si, Mn, or P contents, increases in $\Delta_V T_S$ can be restrained even if austenite grains are coarsened, and production of extremely thick material excelling in both strength and resistance to embrittlement becomes possible by setting a suitable austenitizing temperature.

Based on the above investigation results, 0.06%Si-0.38%Mn steel was quenched directly after forging at 1 250°C, and its strength and temper embrittlement were compared to those of the same steel conventionally quenched from 940°C. The results are shown in **Fig. 18**, and prove that directly quenched steel possesses a higher strength as well as lower susceptibility to temper embrittlement.

4 Conclusion

Various measures have been considered for reduction of embrittlement to date, and it has finally become possible to produce Cr-Mo steels which possess extremely low susceptibility to temper embrittlement.

However, it is not rare for these measures to be limited in application from the standpoint of compatibility with other special characteristics, such as strength. This report has discussed the compatibility of measures for reducing embrittlement with those for improving strength. A combination of decreases in Si, Mn, and P contents and raising of austenitizing temperature is suggested as a suitable measure to maintain a compatible balance between these properties in heavy section steels.

Based on the results obtained from the experiments mentioned above, our company is now producing extra heavy section Cr-Mo steels for pressure vessels which have high strength and low susceptibility to temper embrittlement.

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