

Original Paper

Factors Affecting the Temper Embrittlement in HAZ of Cr-Mo Steel

Koreaki TAMAKI, Jippei SUZUKI and Kohji RYOKE
(Department of Mechanical Engineering)

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A new type of temper embrittlement is possible to occur in the HAZ of 1 1/4Cr-1/2Mo steel. The factors influencing this embrittlement would be the tempering- time and temperature, SR-treatment before tempering and the stress during tempering. Those factors were examined using the simulated-HAZ specimen. This specimen (HAZ specimen) was tempered at 773 to 873K for 5 to 2000 hours with and without loading. Charpy impact test was carried out on the tempered specimen. The temper embrittlement occurs most intensively at 823K when the tempering time exceeded 200 hours at 823K. The embrittlement still occurred in the SR-treated specimen when tempering was prolonged. The stress intensified the tendency of embrittlement when tempering time exceeded 200 hours at 823K. SEM observation informed that the embrittlement was caused by the weakening of grain boundary of the prior-austenite.

Key words: temper embrittlement, Cr-Mo steel, tempering condition, effect of stress, SR-treatment, intergranular fracture

1. Introduction

The term "temper embrittlement" was used originally for the embrittlement phenomenon occurring in medium-carbon, low-alloy steels when they were subjected to the intentional heat treatment of "quench-and-tempering". The temper embrittlement has an essential nature of the weakening of grain boundary.

Recently, the phenomena resembling the temper embrittlement were found in low-carbon, low-alloy steels used for boilers and pressure vessels. Those phenomena, which may be named as "the temper embrittlement in a wide sense", were observed in two cases. The first case is "the SR embrittlement" in 2 1/4Cr-1Mo, 1.4Mn and HT80 steels of the QT-type[1,2]. It occurs in the base metal area when a thick plate is welded and stress-relieved. The second case is "the embrittlement in service" occurring in the QT-type 2 1/4Cr-1Mo steel during high-temperature, long-time operation of vessels [3].

Other than those, the temper embrittlement is possible to occur in HAZ of steels of the non-QT type. In this case "quenching" is made by the weld-thermal-cycle.

The weakening phenomenon of grain boundary has been observed in the tempered HAZ in cases of the reheat cracking [4,5] and the creep-embrittlement [6-8]. These phenomena occur most intensively in 1 1/4Cr-1/2Mo steel [4-8]. Those data suggest that this steel will be sensitive also to the temper embrittlement [1-3].

The main object of this research is to examine the factors influencing this new type of temper embrittlement in the HAZ of 1 1/4Cr-1/2Mo steel. The factors selected by the authors were the tempering-time and temperature, SR-treatment before tempering and the stress during tempering.

"The HAZ specimen" which simulates the microstructure of real HAZ was prepared. The intensity of embrittling was shown by the shift of transition temperature of the specimen. The fracture surface obtained by impact test was observed by a SEM, and the mechanism of temper embrittlement was discussed from the view point of the weakening of grain boundary.

2. Specimen and experiment

2.1. Preparation of specimen

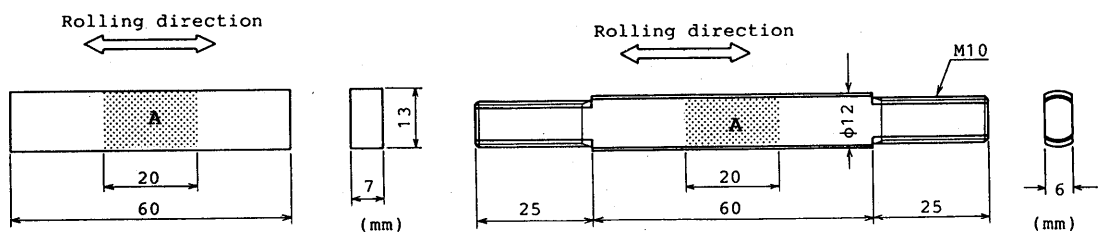
1 1/4Cr-1/2Mo steel plate (JIS CMV 3) of 13 mm in thickness was used; its chemical composition is shown in Table 1.

The microstructure of HAZ given by bead-welding was reproduced in a steel bar by using a weld-thermal-cycle simulator. The bead-welding was made by the following welding parameters: arc voltage 24V, welding current 180A, welding speed 15cm/min, heat input 17.3kJ/cm, without preheating.

The HAZ contained 80% bainite and 20% martensite; its hardness was 380 in Hv. The simulated-HAZ obtained by the heating- and cooling conditions as follows: heating rate 92K/sec (by HF-induction coil of 30mm in length), maximum temperature 1623K, cooling rate 21K/sec (by nitrogen gas). The size of simulated-HAZ specimen is shown in Fig.1(a); the center portion of 20mm in length

Table 1 Chemical compositions of 1 1/4Cr-1/2Mo steel (wt%)

C	Si	Mn	P	S	Cu	Ni	Mo	Cr	As
0.15	0.74	0.60	0.005	0.003	0.01	0.03	0.52	1.40	0.002



(a) Specimen for general use

(b) Specimen used for tempering under stress

Fig.1 Simulated HAZ specimens (A: portion of a uniform microstructure)

reserved an uniform microstructure.

2.2. Heat treatment

The simulated-HAZ specimens (briefly written as "HAZ specimens") were subjected to three types of heat treatments.

Tempering: The HAZ specimen of Fig.1(a) was tempered at 773, 823 and 873K for 5 to 2000 hours in a sealing steel-container placed in an electric-resistance furnace. 10 to 14 specimens were used for each set of time-temperature condition.

SR-treatment and tempering: SR heat treatment was given to HAZ specimens as; heating rate of 200K/hr, keeping at 923K for 2 hours, cooling rate of 200K/hr. The SR-treated specimens were tempered by each set of tempering conditions shown above.

Tempering under stress: The influence of the stress on the embrittlement was estimated by the experiment under creep-loading. 20 creep-test machines of the lever type were constructed in the laboratory [8]. The HAZ specimen of Fig.1(b) was adopted in this experiment. The specimen was tempered at 823K for 20 to 2000 hours with the stress of 73.5 and 98MPa. Some experiment of the authors has predicted that the time to final fracture would be about 5000 hours at the stress of 98MPa [8].

Heating of base metal: The steel plate in as-received condition (the base metal plate) was heated with the tempering conditions same as the HAZ specimen, and the result was referred to that of the latter.

2.3. Charpy impact test

The half-size specimen shown in Fig.2 was used in this experiment as the maximum load of the creep-test machine was limited to 9800N (135MPa for the specimen of Fig.1(b)). The Charpy impact test machine of the maximum energy of 98J (10kgf-m) was used.

2.4. Observation of fracture surface

Immediately after the impact test, the specimen was dipped in a alcohol bath of room temperature. It was dried and used for the observation by a scanning electron microscope with the operating parameters; accelerating voltage of

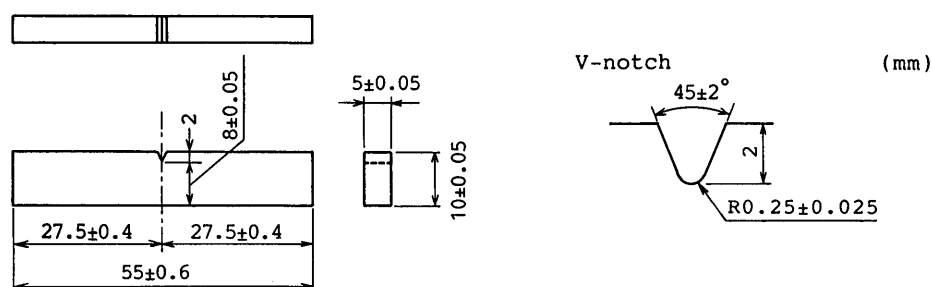


Fig.2 Specimen for Charpy impact test

30 kV, cathode current of 60 μ A.

3. Change in toughness during tempering

3.1. Original toughness of base metal and HAZ specimens

Fig.3(a) and (b) show the transition curves of the absorbed energy and the percentage of brittle fracture, respectively, for the base metal in as-received condition and HAZ specimen. Each curve was drawn in approximation to a curve given by the equation;

$$Y=a/[1+\exp((b-X)/c)],$$

a, b and c are chosen optionally.

The transition temperatures TrE and TrS are the temperatures at which a half of the upper-shelf energy and 50% of brittle fracture surface are given, respectively. TrE and TrS base metal are as low as 200 and 220K.

The TrE and TrS of HAZ specimen are 280 and 285K, respectively, as shown in Fig.3. That is, the TrE and TrS of the base metal are raised by 80 and 65K by weld-thermal-cycle. The upper-shelf energy Eus itself is also decreased from 98 to 59J.

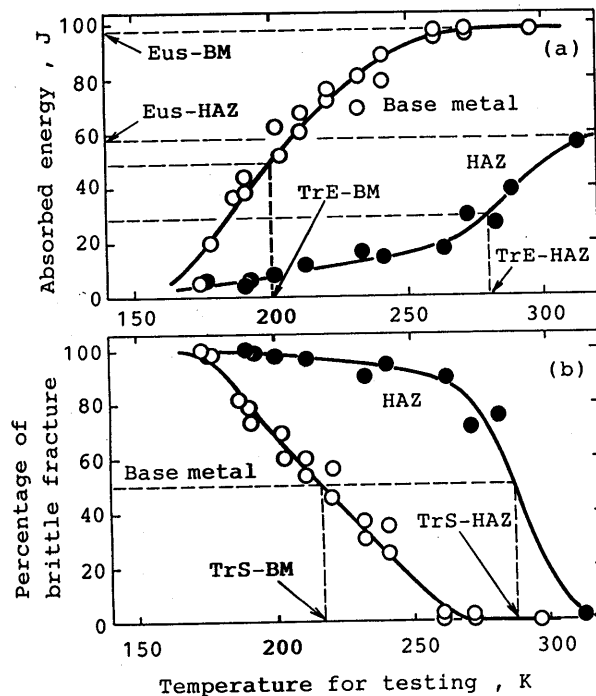


Fig.3 Transition curves of base metal in as-received condition (a) and HAZ specimen (b)

3.2. Embrittlement in the HAZ specimen

The whole shape of transition curve for each tempering condition was compared by using two principal values, TrE and Eus. As the TrS value of each specimen tested is closely dependent on the TrE, as shown in Fig.4, the TrS is left out in the succeeding figures.

Fig.5 shows the changes of the transition temperature, TrE and the upper-

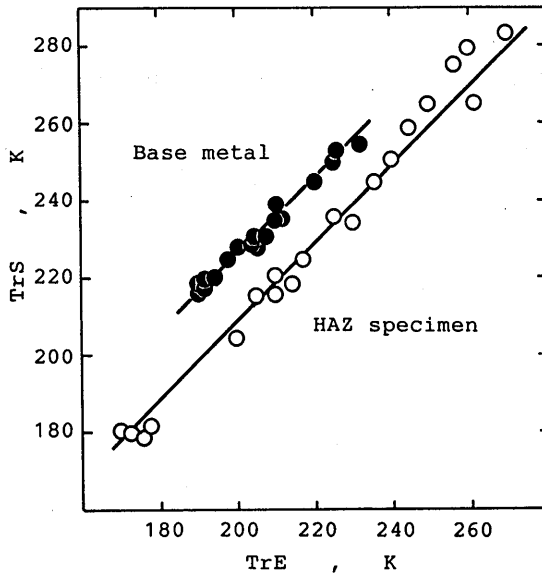


Fig.4 Dependence of TrS upon TrE

shelf energy, Eus during isothermal tempering at 773, 823 and 873K.

Tempering at 823K: There are two stages in the TrE-time curve at 823K in Fig.5. The TrE falls at first in the tempering time shorter than 200 hours, and then it rises. The fall of TrE in the first stage is brought about by softening and toughening due to tempering of martensite. The phenomenon of temper embrittlement appears for the first time when the tempering time exceeds 200 hours. This stage seem to continue to more than 2000 hours at 823K.

Tempering at 873K: The TrE falls at first by tempering for a short time at 873K as shown in Fig.5. It rises slightly in the second stage (20 to 200 hours), and finally it falls approaching a minimum value of TrE (about 145K). In the second stage, two opposite actions will take place; the action of depressing TrE by toughening the steel matrix, and that of raising it by embrittling the grain boundary.

The specimen which has once been embrittled in the second stage will be "de-embrittled" [9] in the final stage of longest tempering time.

Tempering at 773K: In the case of 773K, only the first stage was observed. Any embrittling tendency is not recognized to the extent of the tempering time of 2000 hours.

3.3. Influence of SR-treatment on embrittlement

As-SR condition: The shifts of TrE and Eus brought about by SR heat treatment are shown on the ordinate of Fig.6. The TrE of HAZ specimen fall down by 120K by SR heat treatment.

Tempering at 823K: The result on the specimens SR-treated and tempered at 823K are shown in Fig.6. The TrE in as-SR condition is originally very low but it rises up greatly when the tempering time exceeds 20 hours. The TrE approaches closely to that of HAZ specimen (dotted line) in the time range longer than 200 hours. The Eus also approaches that of HAZ specimen (dotted line) in the same

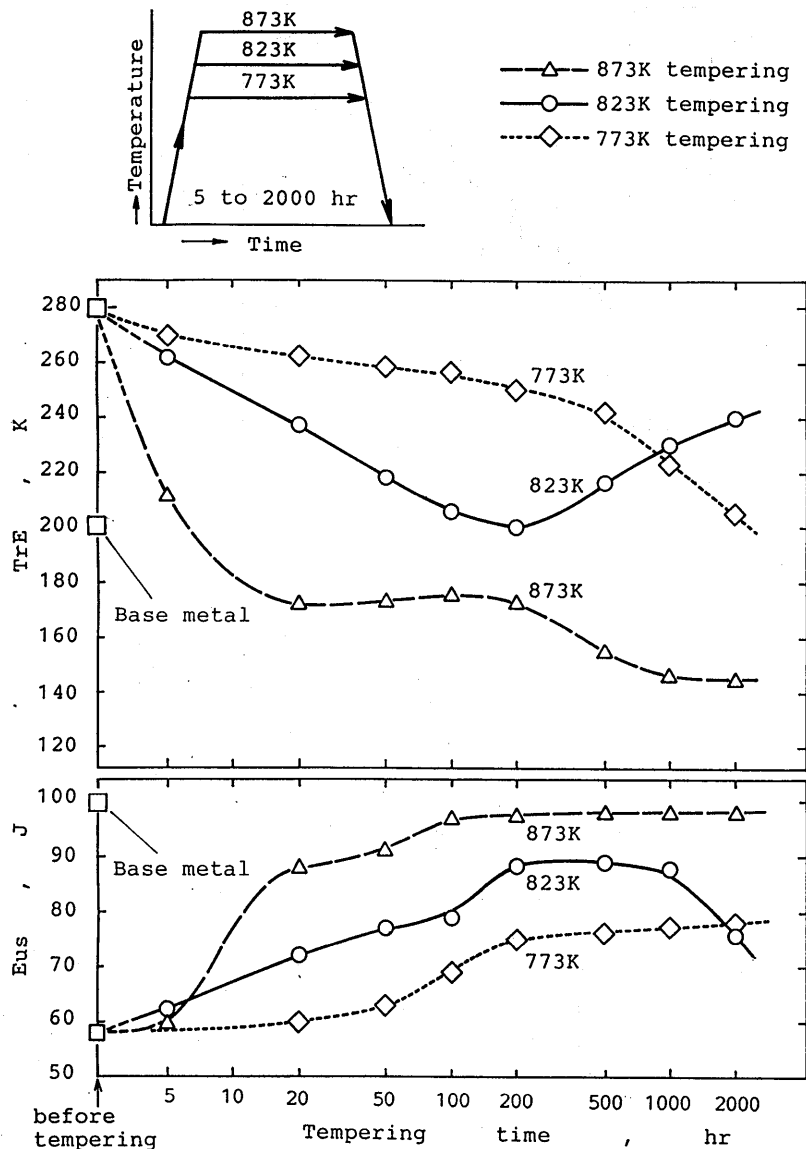


Fig.5 Changes of transition temperature and upper-shelf energy during tempering of HAZ specimen

time range.

It is concluded that the temper embrittlement still occurs in the SR-treated specimen; tempering of a long time period raises the TrE approaching that of the HAZ specimen.

Tempering at 873K: The TrE of as-SR specimen rises by tempering at 873K for 20 hours and then falls approaching a constant value (Fig.6). This value is same as that of de-embrittled HAZ specimen (dotted line). The Eus approaches also that of HAZ specimen.

Although the temper embrittlement is still observed in case of the tempering at 873K, it appears only in a short time range.

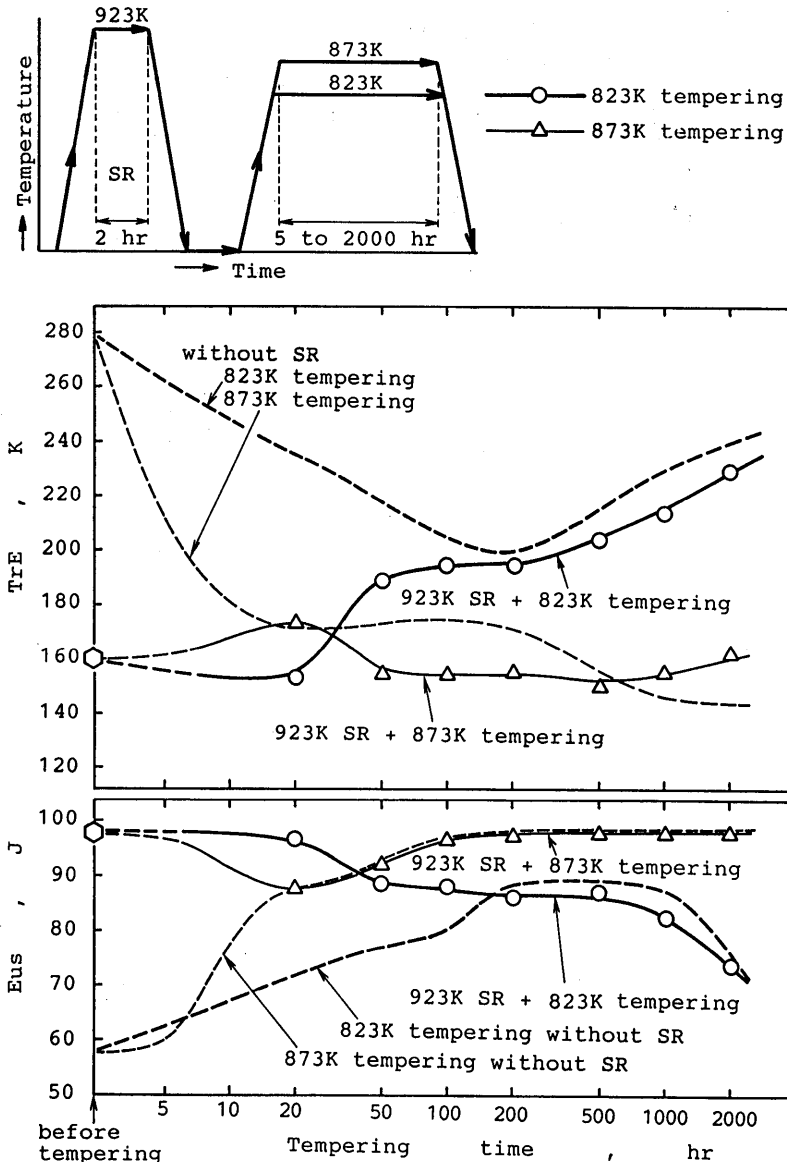


Fig.6 Changes of transition temperature and upper-shelf energy during tempering of SR-treated specimen

3.4. Influence of stress on embrittlement

The result on the specimens tempered with stress is shown in Fig.7. The shape of TrE-time curve itself is similar to that without stress (dotted line) which is consisted of two stages (Fig.5). The TrE value in the first stage is not affected by the stress, but that in the second stage is raised with an increasing stress level as shown in Fig.7. The effect of stress is more clearly seen as the fall of Eus.

It is concluded that the stress affects little the process of martensite decomposition, but does very much the process of impurity segregation, and therefore, the process of embrittlement.

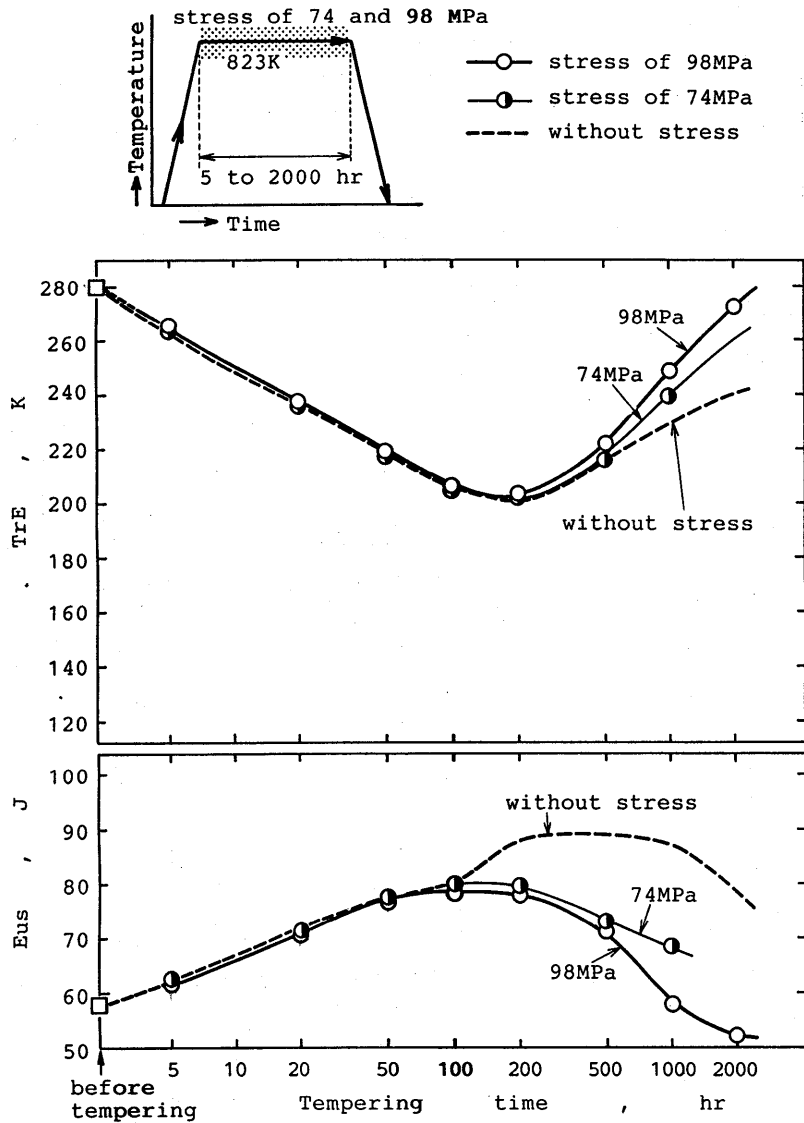


Fig.7 Influence of stress on the transition temperature and upper-shelf energy of tempered HAZ specimen

4. Influence of heating on the toughness of base metal

The base metal in as-received condition was heated at 773 to 873K for 2000 hours in maximum. The heating at 873K for 2000 hours raises the TrE by 30K, but that at 773K and 823K does a little as shown in Fig.8. However, the heating does not affect the Eus value in as-received condition (98J; 10kgf-m).

As mentioned above, the influence of tempering (heating) on the toughness of base metal, which did not experience any quenching process, is quite different from that of HAZ specimen.

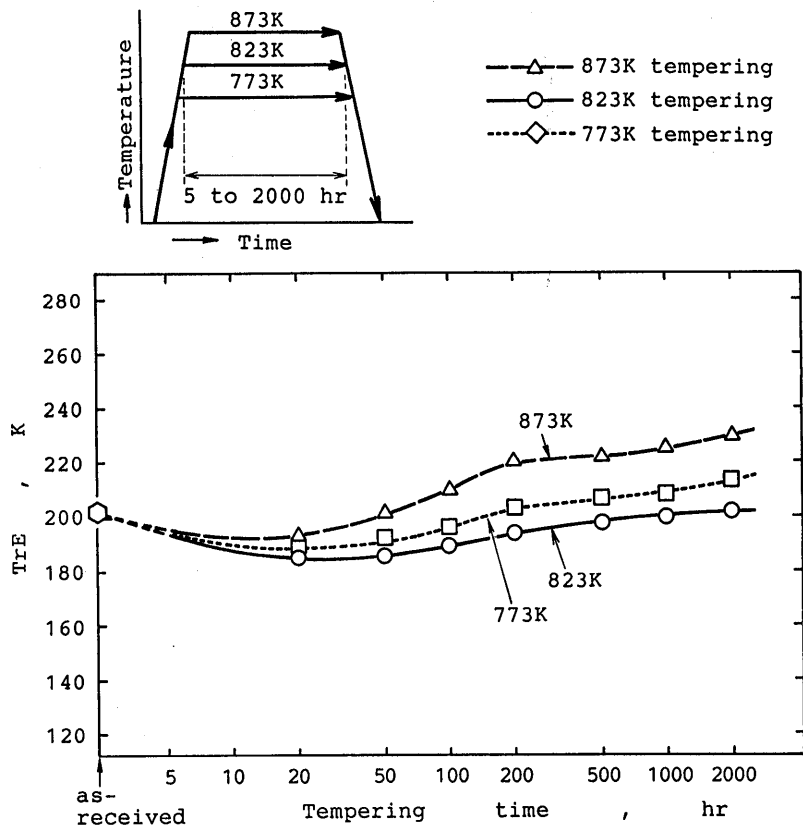


Fig.8 Changes of transition temperature during tempering of base metal

5. Observation of the fracture surface

After impact test, the fractured surface was observed by a scanning electron microscope, and the fracture mode was discussed referring to the embrittling behavior at each tempering temperature.

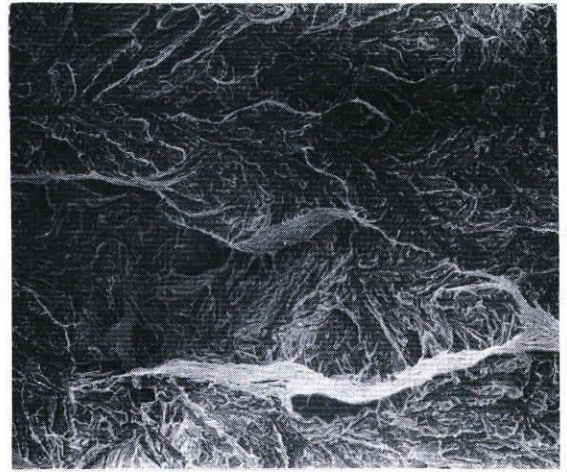
5.1. Tempered HAZ specimen

Table 2 summarizes the fracture mode of all the specimens tested at 158K. The fracture modes are classified in the table as [IG]: intergranular fracture, [CL]: transgranular fracture of cleavage mode and [DI]: transgranular fracture of dimple mode.

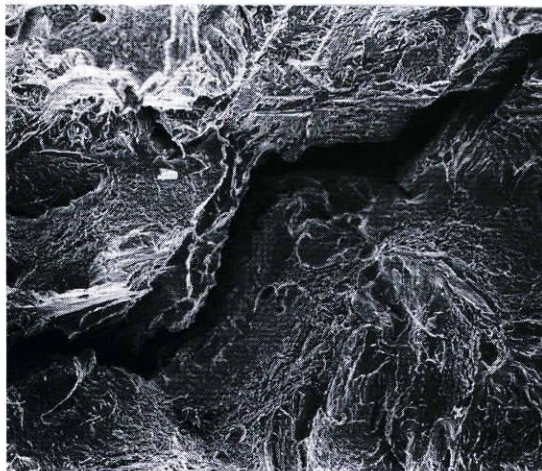
Tempering at 823K: Photo 1 shows the fractured surfaces of the specimens tempered at 823K and tested at 158K. The fracture mode of each specimen is shown beside each photo. In the tempering time shorter than 200 hours, the fracture occurs in the ferrite grains with the cleavage mode (Photo 1(a) to (c)). The intergranular fracture occurs with the tempering time longer than 500 hours (Photo 1(d) to (f)). The grain boundary in this case corresponds to that of prior-austenite which was produced by the weld-thermal-cycle [5]. The specimens in which intergranular fracture is observed correspond well to the specimen in



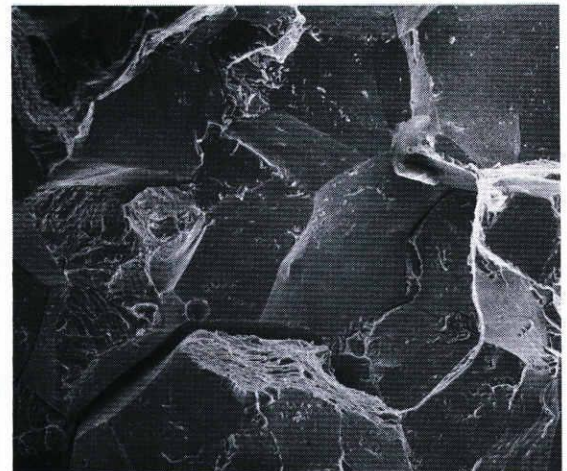
(a) 50 hr [CL]



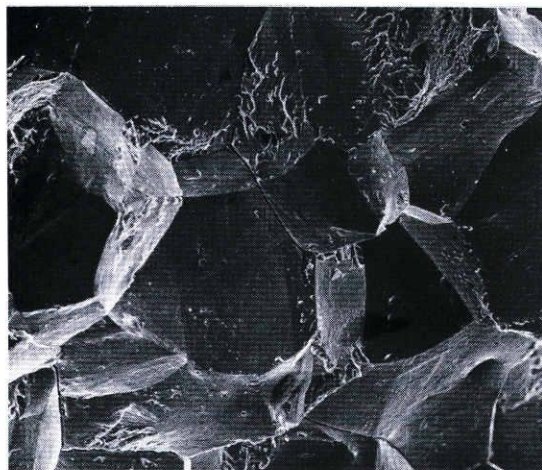
(b) 100 hr [CL]



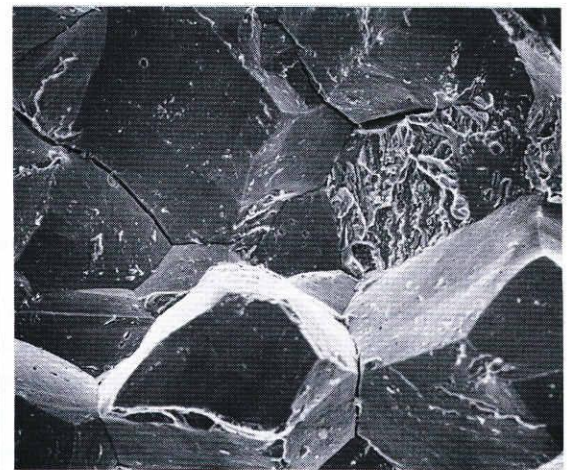
(c) 200 hr [CL]



(d) 500 hr [IG]



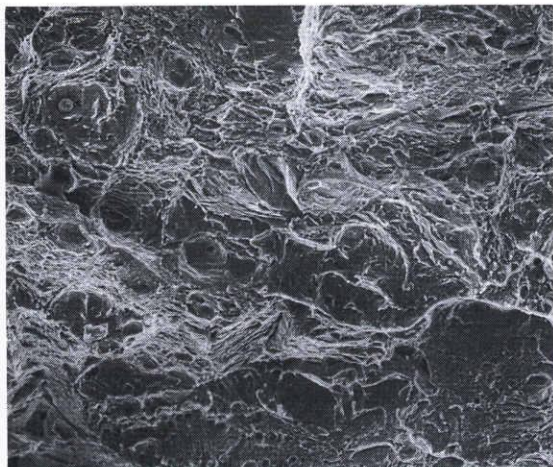
(e) 1000 hr [IG]



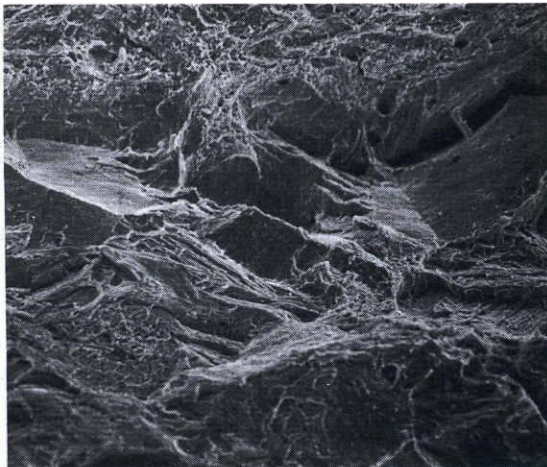
(f) 2000 hr [IG]

100μm

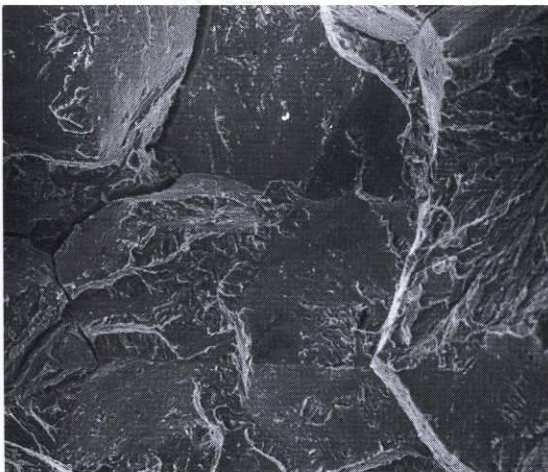
Photo 1 Change of fractured surface with the lapse of tempering time at 823K;
HAZ specimen impact-tested at 158K



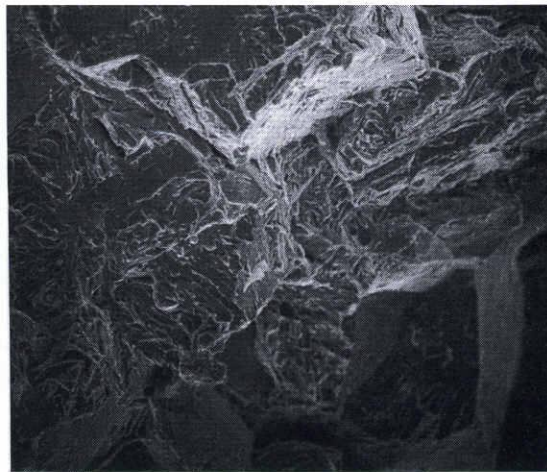
(a) 50 hr [CL]



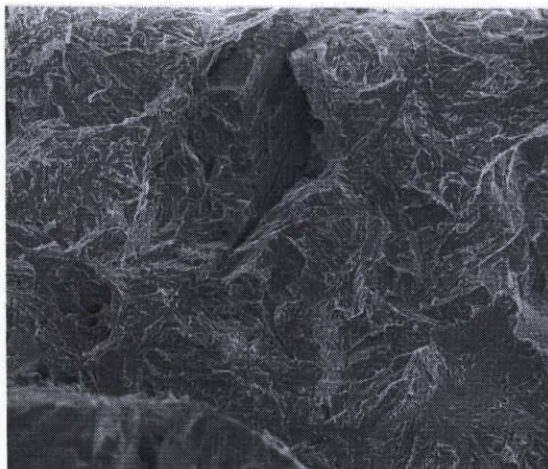
(b) 100 hr [CL+IG]



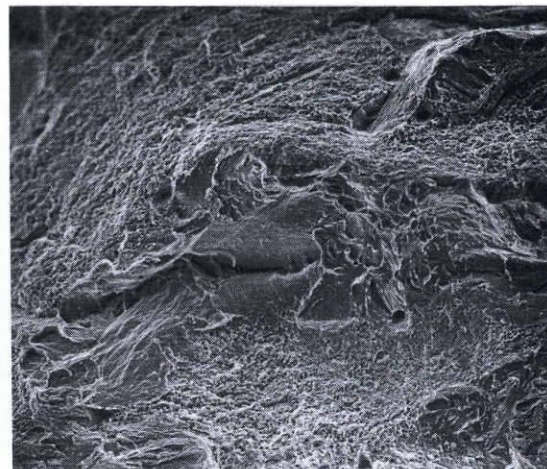
(c) 200 hr [CL+IG]



(d) 500 hr [CL+IG]



(e) 1000 hr [DI+CL]



(f) 2000 hr [DI+CL]

100μm

Photo 2 Change of fractured surface with the lapse of tempering time at 873K;
HAZ specimen impact-tested at 158K

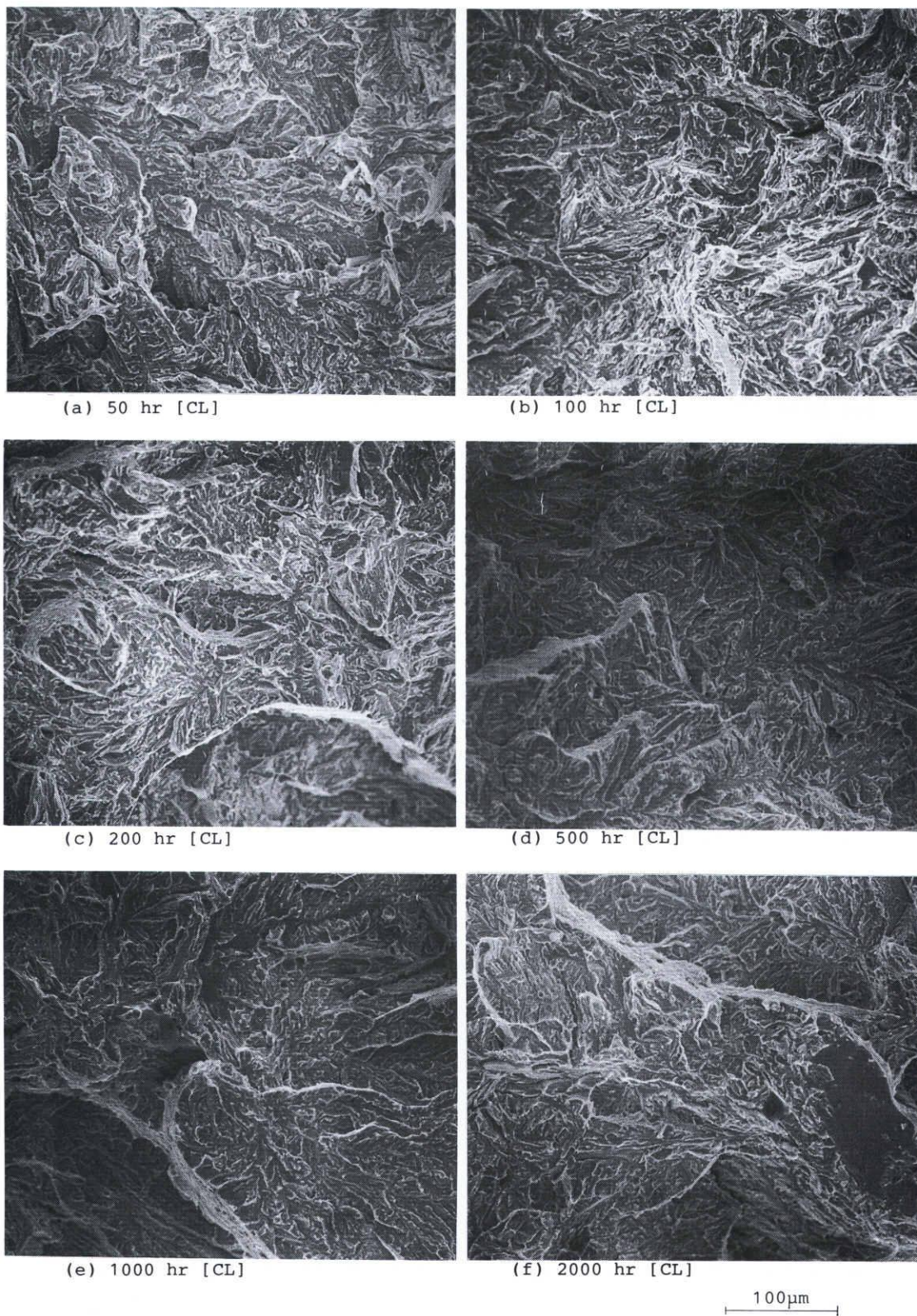


Photo 3 Change of fractured surface with the lapse of tempering time at 773K;
HAZ specimen impact-tested at 158K

Table 2 Changes of fracture mode with the lapse of tempering time;
Temperature of impact test: 158K

Experiment	Tempering temperature ,K	Tempering time , hr						
		20	50	100	200	500	1000	2000
Tempering of HAZ specimen	873	CL	CL	CL + IG	CL + IG	CL + IG	DI + CL	DI + CL
	823	CL	CL	CL	CL	IG	IG	IG
	773	CL	CL	CL	CL	CL	CL	CL
Tempering of SR-treated specimen	823				CL	CL	CL	CL
Tempering with stress of 98MPa	823	CL	CL	CL	IG	IG	IG	IG

Note; CL: transgranular fracture of cleavage mode

IG: intergranular fracture

DI: transgranular fracture of dimple mode

the embrittled stage.

The dimple mode of fracture appears by the test at the higher temperature (323K), but the intergranular mode still remains predominantly in the full-embrittled specimens.

Tempering at 873K: Photo 2 shows the specimen tempered at 873K. In the tempering time shorter than 50 hours, the fractured surface exhibits the cleavage mode (Photo 2(a)). The specimens in the embrittled state (100 to 500 hours; the second stage in Fig.5) are fractured with the intergranular mode as shown in Photo 2(b) to (d). In the de-embrittled state (1000 to 2000 hours), the intergranular fracture does not occur as shown in Photo 2(e) and (f).

Tempering at 773K: Only the cleavage mode appears in all the specimens tempered at 773K and test at 158K as shown in Photo 3.

5.2. SR-treated specimen and stress-loaded specimen

The intergranular fracture appeared in the specimens which were SR-treated and tempered in the embrittling stage (Fig.6), as shown in Table 2.

The stress of 98MPa shortened the time period at which the intergranular fracture began to appear as shown in Table 2.

5.3. Base metal specimen

The observation was made also on the fractured surface of base metal specimen heated at 873K for 20 to 2000 hours. All the specimens tested at 172K exhibited the fracture of cleavage mode and those tested at 303K did the dimple mode.

6. Influence of phosphorus on temper embrittlement

The results obtained by impact test and observation on fracture surface are discussed in this section from the view point of segregation of impurity elements.

In the preceding reports, the authors have informed the segregation behavior of some impurity elements, such as phosphorus [10,11]. The phosphorus concentration in the grain boundary was semi-quantified by the grain boundary etching method; its concentration is proportional to "the depth of etched grain boundary, d " [12]. A set of implant test specimen was made from 1Cr-1/2Mo-0.068P steel. The welded specimen was set in the implant testing machine and tempered with or without loading [10]. Fig.9 shows the influences of tempering time and temperature on the depth of etched grain boundary, d . The maximum value of d appears in the area around 823K and 5 hours in both the case of with and without loading. The stress of 196MPa increases the d very significantly (Fig.9(b)).

The time at which phosphorus segregates (5 hours; Fig.9) does not meet that at which the temper embrittlement occurs (200 hours; Fig.5). This difference in the experimental results may come from the difference in phosphorus content of the specimens. Nevertheless, the result of Fig.9 proposes useful informations on the relation between the impurity segregation and the temper

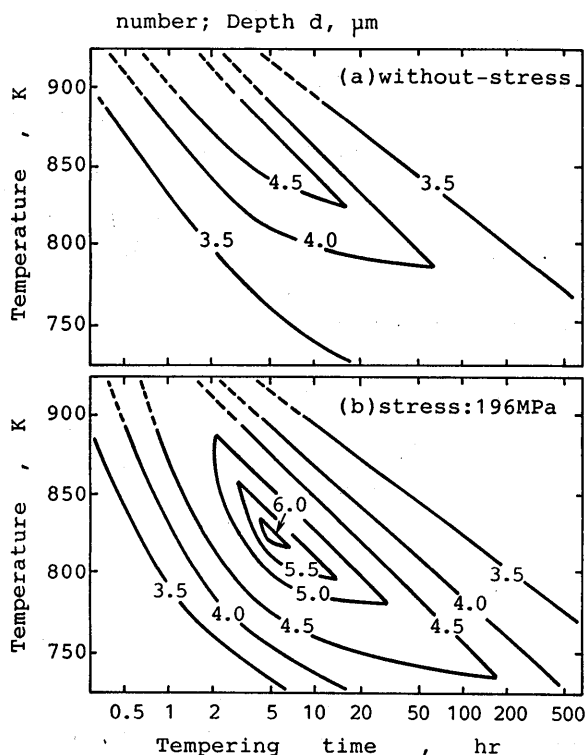


Fig.9 Depth of etched grain boundary shown in the tempering time-temperature diagram; HAZ of 1Cr-1/2Mo-0.068P steel

embrittlement, as;

- (1) Temper embrittlement occurs most intensively at 823K at which the phosphorus concentration of grain boundary reaches a maximum value. Phosphorus will weaken the grain boundary and assist the intergranular fracture to occur.
- (2) Temper embrittlement occurs as 873K only in a short time; this is explained by that phosphorus segregates once in the grain boundary but it can easily diffuse away from there at this temperature.
- (3) The stress increases the intensity of temper embrittlement by the role of increasing the concentration of phosphorus in the grain boundary.

7. Conclusions

A new type of temper embrittlement which is possible to occur in the HAZ of 1 1/4Cr-1/2Mo steel was investigated, and obtained the following experimental results.

- (1) In the HAZ specimen, the embrittling stage appeared after 200 hours of tempering at 823K.
- (2) The embrittled specimen was fractured by the impact test along the grain boundary of prior austenite where some impurity elements were concentrated.
- (3) Temper embrittlement occurred also in the SR-treated specimen.
- (4) The stress intensified the temper embrittlement. It will assist the segregation of impurity elements.

Acknowledgments

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