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Weldability of Fe-36%Ni Alloy (Report VI)†

— Further Investigation on Mechanism of Reheat Hot Cracking in Weld Metal —

Yue-Chang ZHANG*, Hiroji NAKAGAWA** and Fukuhisa MATSUDA***

Abstract

Stress concentrator, such as grain-boundary precipitates, grain-boundary ledges and/or serrated grain-boundary, necessary for cavity formation by grain-boundary sliding was studied to complete the mechanism of the reheat hot cracking in weld metal of Fe-36%Ni alloy Invar. Main conclusions obtained are as follows: (1) The temperature dependencies of ductility and wavy feature of intergranular fracture surface were correlated well to the densities of precipitates on intergranular fracture surface and the serrated grain-boundary, respectively. (2) Thus, it was considered that the cavities were formed by the cooperation of the grain-boundary precipitates and the serrated grain-boundaries. (3) Grain-boundary precipitation was promoted near the intersection with dendritic subboundaries by the solidification micro-segregation, and this is one of the reason why the weld metal is very sensitive to the cracking comparing HAZ. (4) Comprehensive model are proposed for the mechanism of reheat hot cracking.

KEY WORDS: (Hot Cracking) (Controlled Expansion Alloys) (Containers) (GTA Welding)

1. Introduction

In the previous paper[1)], the authors have revealed that grain-boundary sliding and its resultant cavity formation are prerequisite to the ductility trough between about 600 and 1100°C relating to the reheat hot cracking in weld metal of Fe-36%Ni alloy Invar, and that grain-boundary migration and dynamic recrystallization are improving factors. However, the previous paper[1)] was not enough to make the mechanism of the cracking clear, because it is generally known that not only grain-boundary sliding but also such stress concentrator as grain-boundary precipitate or grain-boundary ledge are necessary for the cavity formation. Concerning this, it is reported recently[2)] that serrated grain-boundary is effective enough for the cavity formation in Al-Mg alloy containing no precipitates. Therefore, the first purpose of this paper is to study whether there are grain-boundary precipitates or not, and the morphology of grain-boundary in relation to the cavity formation.

The second purpose is to study why the reheat hot cracking predominantly occurs in the weld metal comparing HAZ. This question can be expressed in other word; is only the large grain size the cause, or does dendritic segregation have any role? The authors have treated in this report the above subjects, and finally comprehensive expression on the mechanism of reheat hot cracking in weld metal of Fe-36%Ni alloy.

2. Materials Used and Experimental Procedures

2.1 Materials used

The chemical compositions of five tentative Fe-36%Ni alloys used are shown in **Table 1**, where the Items except Item Nos. 25 and 26 follow those in the Report II[3)]. Sulphur and aluminum contents were increased in Item Nos. 25 and 26, respectively. The materials of high and low crack susceptibilities were prepared. The thickness was 3 mm except Item No. 10 of 1.5 mm.

2.2 Experimental Procedures

Simulated hot ductility test was done to study fracture

Table 1 Chemical compositions of materials used.

Item No.	Chemical composition (wt.%)								
	C	Si	Mn	P	S	N	O	Al*	Ni
1	0.031	0.20	0.50	0.002	0.0011	0.0008	0.0034	0.009	36.25
10	0.032	0.20	0.36	0.001	0.008	0.0011	0.0018	0.001	36.24
12	0.033	0.19	0.35	0.003	0.005	0.0035	0.0021	0.001	36.05
25	0.009	0.21	0.33	0.003	0.0249	0.0015	0.0133	0.002	35.29
26	0.013	0.25	0.34	0.001	0.0007	0.0016	0.0024	0.516	36.23

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surface, precipitation and feature of grain-boundary. The procedure of the simulated hot ductility test was completely the same as that in the previous paper[1)]. Besides, effect of solution treatment for weld metal prior to the hot ductility test on ductility was studied. The solution treatment was done at 1350°C for 180 sec and 540 sec in Ar atmosphere.

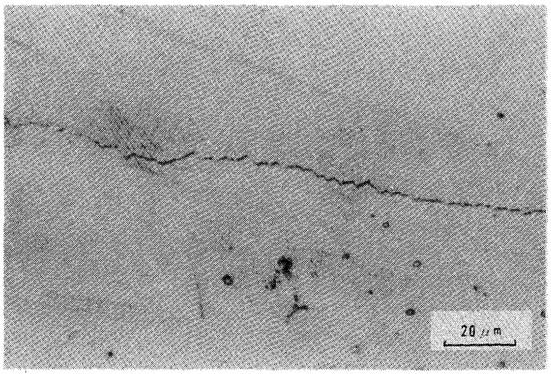
Actual weld hot cracking test was done with U-form hot cracking device[4)] for Item No. 10. Both the first and the second pass welding was done with GTAW without filler metal under the following conditions; welding current of 60 A, arc voltage of 9–11 V and welding speed of 100 mm/min. The Cross-bead cracking test[3)] with this device was done in a welding chamber filled with 99.99% Ar gas after evaquation to 5×10^{-5} torr in order to prevent the oxidation of the fracture surface in weld metal.

To observe the precipitates on fracture surface and polished surface, the specimens were etched with SPEED method[5)]. Well, it is said[6)] that the number of precipitates on grain-boundary is decisive for high temperature embrittlement. Therefore in this paper, the density of precipitates on the fracture surface ρ_p (number/100 μm^2), was measured by counting the number of precipitates in arbitrary about ten SEM fields in × 15,000 with the aid of EDX to check the composition.

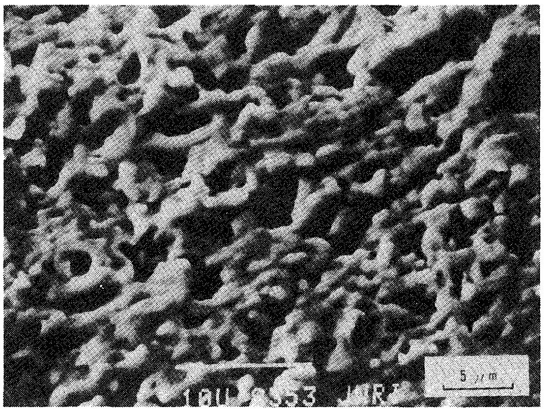
3. Experimental Results and Discussions

3.1 Correlation between grain-boundary serration and wavy feature of intergranular fracture surface

An example of the serrated grain boundary in the weld metal after actual weld cracking test and its intergranular fracture surface are shown in **Fig. 1 (a)** and **(b)**, respectively. It is observed clearly that the grain-boundary migrated from the original position at solidification was serrated, and that the feature of intergranular fracture surface was wavy as seen in Fig. 8 in the previous paper[1)]. Comparing the serrated grain-boundary and the wavy feature of the fracture surface, it is understood that the period of serration is nearly the same as the unit size of the wave. This suggests an intimate correlation between the grain-boundary serration and the wavy intergranular fracture surface.



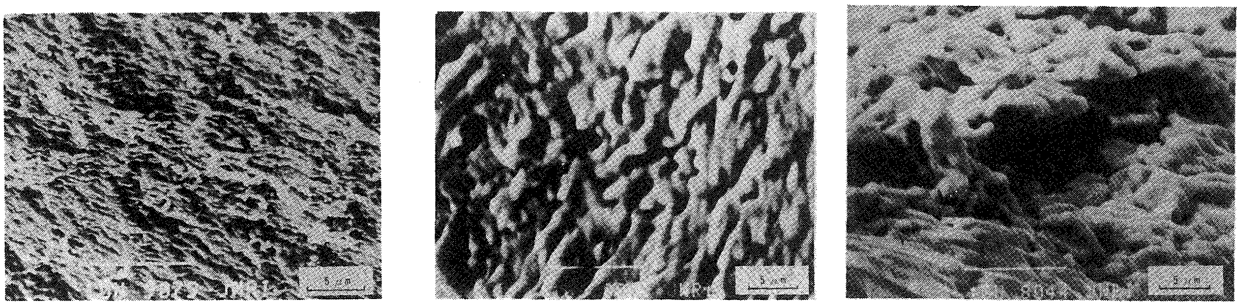
(a)



(b)

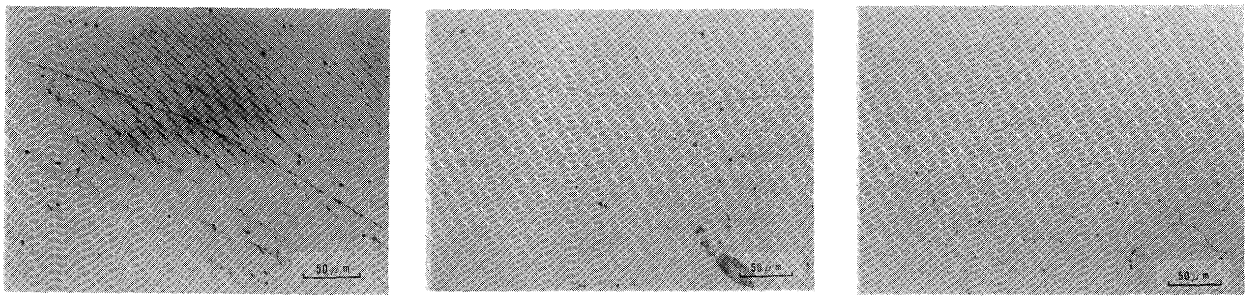
Fig. 1 Features of migrated grain-boundary (a) and intergranular fracture surface (b) after actual weld cracking test for Item No. 10.

The same behaviors were observed in the hot ductility test, and thus studied in detail. Features of the intergranular fracture surface under various testing temperature are compared in **Fig. 2**. The wavy feature is generally seen from 600 to 1100°C, and the unit size of the wavy pattern was varied a little depending on the testing temperature. The size was small at 600°C as seen in (a), and became larger a little with the increase in the testing temperature as in (b) and (c). Examples of serrated grain-boundary under various testing temperature are shown in **Fig. 3**. Nearly amooth grain-boundary is observed below



(a) 600°C (b) 900°C (c) 1100°C

Fig. 2 Relation between testing temperature and feature of intergranular fracture surface in Item No. 9. crosshead speed (C.H.S.): $\dot{d}_m$.



(a) 500°C (b) 900°C (c) 1100°C

Fig. 3 Relation between testing temperature and feature of serrated grain-boundary in Item No. 12. C.H.S.: $\dot{d}_m$.

the testing temperature of 500°C as in (a), but the serration was obvious above about 700°C, and the typical example is shown in (b) for 900°C. On the other hand, the period of serration was somewhat wider at 1100°C as in (c).

Moreover, high crosshead speed caused not only finely serrated grain-boundary but also finely wavy fracture surface at 900°C.

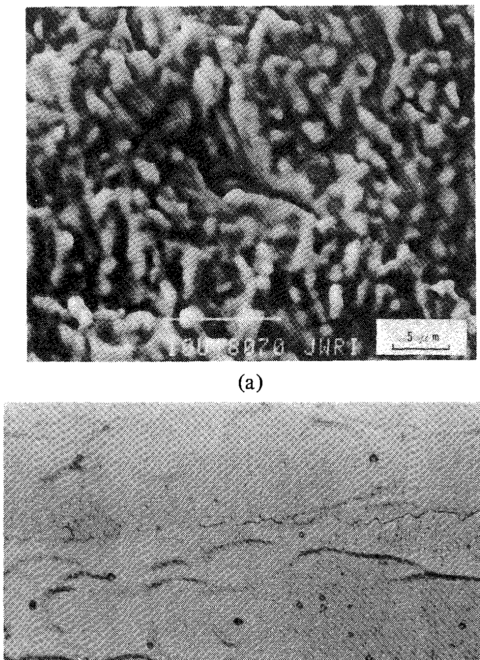
These prove that the wavy feature of intergranular fracture surface was correlated well with the serrated grain-boundary and that both were influenced by testing conditions. Though Item No. 1 is insensitive to the hot cracking, it also showed the wavy intergranular fracture surface and the serrated grain-boundary as shown in **Fig.4**.

Typical examples of cavities formed are shown in **Fig. 5**, where the spacing of cavities nearly corresponded to the period of grain-boundary serration.

Recently, Horiuchi et al.$^{2)}$ have studied high temperature embrittlement of Al-Mg alloy, which had no precipitates, and concluded that cavities are formed by serrated grain-boundary. They showed that the stress distribution originated by the serrated grain-boundary during grain-boundary sliding could make the maximum stress concentration coefficient up to 2.5–5.0, which satisfies the condition of cavity formation at the grain-boundary. Therefore, it is considered that also the serrated grain-boundary in the weld metal of Invar has an important role in the formation of cavities.

3.2 Correlation between grain-boundary precipitation and hot ductility

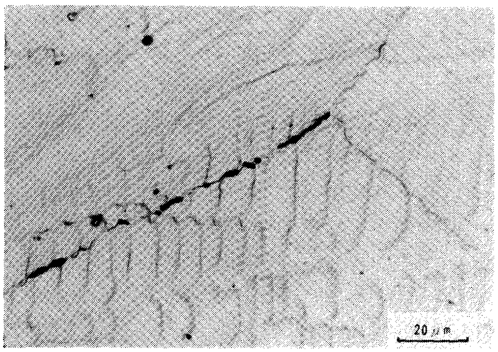
SPEED method$^{5)}$ applied to the intergranular fracture



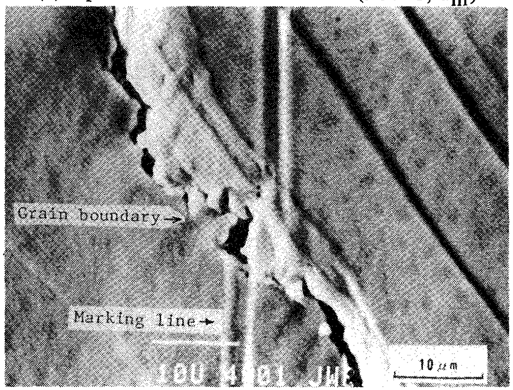
(a)

(b)

Fig. 4 Correspondency of features of intergranular fracture surface (a) and serrated grain-boundary (b) in Item No. 1 insensitive to cracking.



(a) polished and etched surface (900°C, $\dot{d}_m$)



(b) specimen surface (800°C, $\dot{d}_m$)

Fig. 5 Relation between feature of grain-boundary and site of cavity formation in Item No. 12.

surface in the weld metal after the actual weld cracking test showed clearly the existence of precipitates as in **Fig. 6**, enriched with S, Si and Mn. Thus, the behavior of intergranular precipitates was studied in detail for the specimen after the hot ductility test. Precipitates on the fracture surface under various testing temperature are shown in **Fig. 7** for Item No. 12 sensitive to the hot cracking. It is understood that there were many precipitates at 900°C as in (b), which gave nearly the minimum hot ductility. On the other hand, there were comparatively less precipitates at 600°C and 1100°C as in (a) and (c), respectively, which both gave a tendency to recover the ductility. The precipitates on the fracture surface at 900°C for Item No. 1 insensitive to the hot cracking, for Item No. 25 containing higher sulphur and for Item No. 26 containing higher aluminum are shown in **Fig. 8**. It should be noted that the ductility ΔW/W at 900°C is 5.5 in No. 12, 20 in No. 1, 3.3 in No. 25 and 3.0 in No. 26. Therefore it seems that the amount of precipitates has good correlation with the difference in the ductility among materials. That is to say, more the precipitates are on the intergranular fracture surface, lower the ductility is.

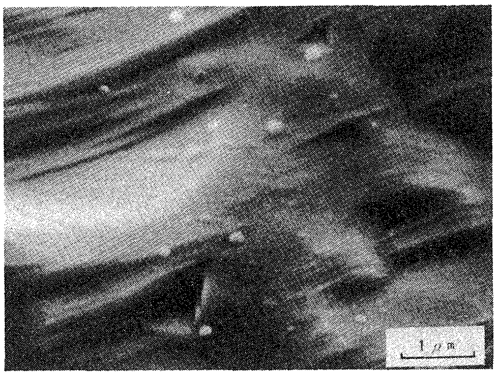
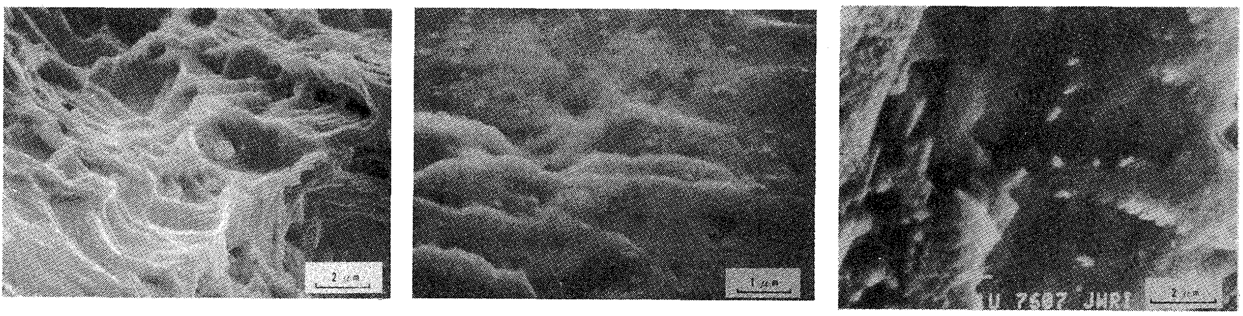


Fig. 6 An example of precipitates on intergranular fracture surface after actual cracking test for Item No. 10.

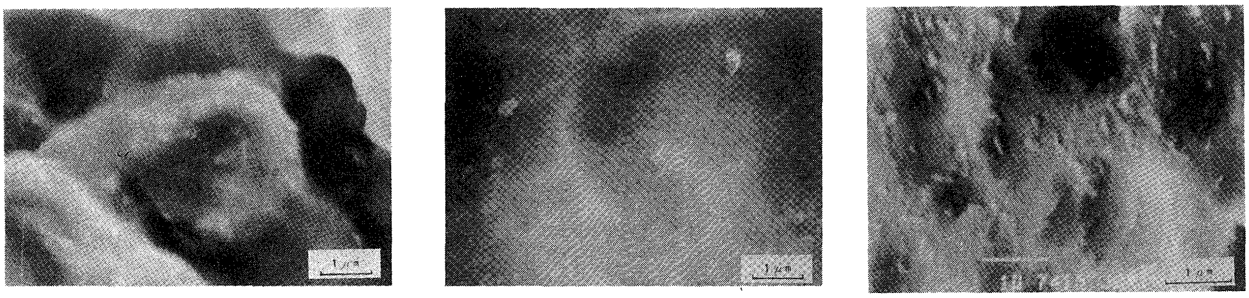


(a) 600°C (b) 900°C (c) 1100°C

Fig. 7 Comparison of precipitates on intergranular fracture surface among different testing temperature in Item No. 12.

Thus, the density of precipitates on the intergranular fracture surface of Item Nos. 1 and 12 were measured by SEM, and the results are shown in **Fig. 9**, where the hot ductility curves are also shown by broken lines. It is seen that the density of precipitates in Item No. 12 depended on the testing temperature, and the temperature of the maximum density corresponded well to that of the minimum ductility. The density in Item No. 1 had similar characteristic, but was fairly less than Item No. 12.

Then, the microstructure of weld metal polished and etched by SPEED method was observed by SEM. **Figure 10** shows the microstructure of Item No. 12 before the hot ductility test, and **Fig. 11** does that after the hot ductility test. Precipitates were formed at migrated grain-boundary both in Figs. 10 and 11, but the weld metal reheated during the hot ductility test had generally more precipitates than that before the reheating. Therefore, it is understood that precipitation occurs even during cooling after welding, but is increased by the reheating. This is



(a) Item No. 1 (b) Item No. 25 (c) Item No. 26

Fig. 8 Comparison of precipitates on intergranular fracture surface at 900°C among different materials.

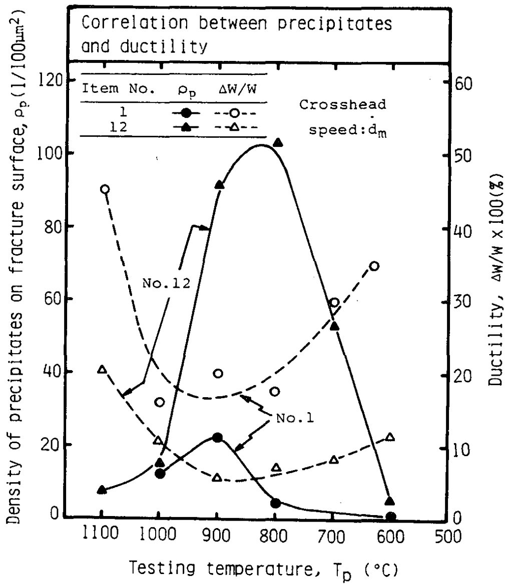
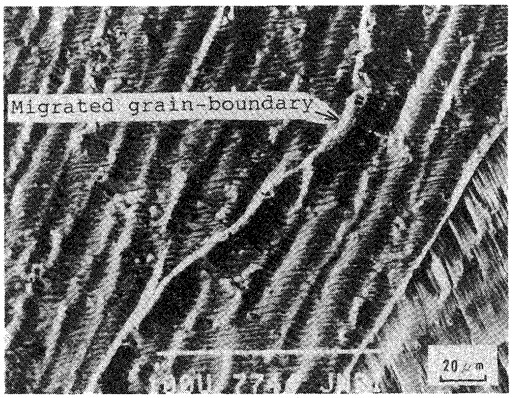
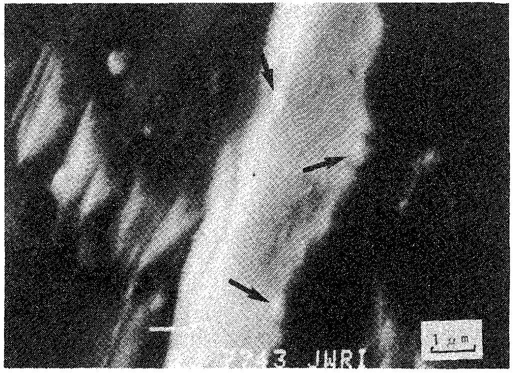


Fig. 9 Density of precipitates and ductility vs. temperature.

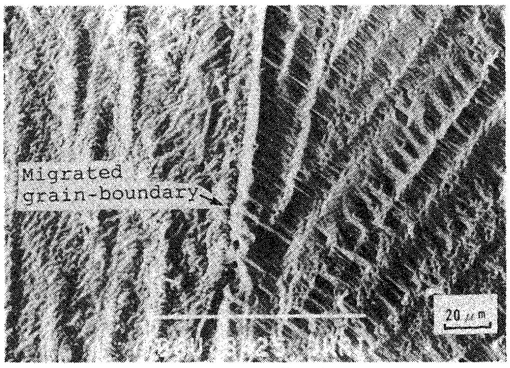


(a) low magnification

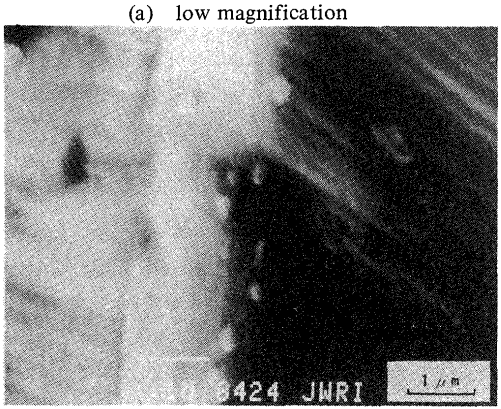


(b) high magnification (arrows show precipitates)

Fig. 10 Precipitates on migrated grain-boundary after welding, Item No. 12.

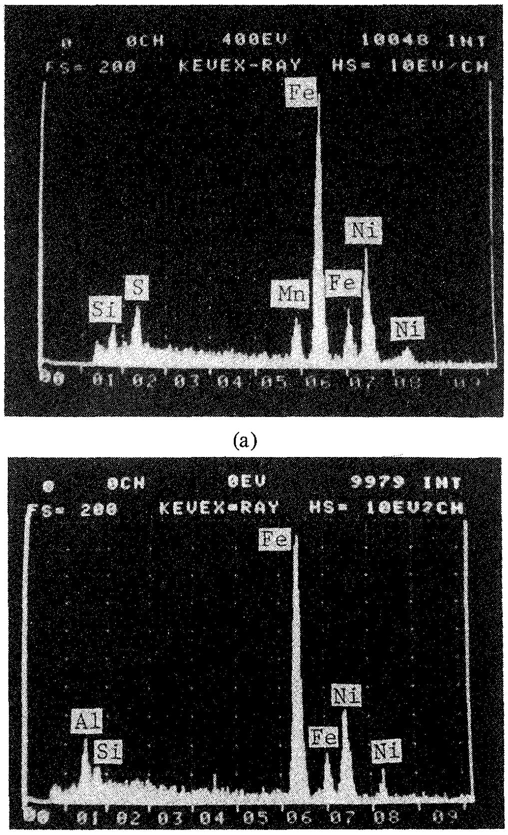


(a) low magnification



(b) high magnification

Fig. 11 Precipitates on migrated grain-boundary after hot ductility test, Item No. 12.



(b)

Fig. 12 Examples of EDX analyses on precipitates on intergranular fracture surface and migrated grain-boundary.

considered to be the reason why the reheated weld metal is more sensitive to the cracking than as-welded weld metal.

From the precipitates on both the fracture and the eched surfaces, S, Si, Al and Mn were detected by EDX as shown in **Fig. 12**. Precipitates on the etched surface were identified as MnS, MnO, AlN and Al_2O_3 by TEM using carbon extraction replica, but nickel sulphide known to make eutectic of low melting point was not identified. Any precipitate consisting of Si, such as oxide or nitride, was not identified by TEM. It is considered that Si may form amorphous compounds.

Therefore, it is proved that another reason for the ductility trough is the grain-boundary precipitation composed of sulphides, oxides and nitrides.

3.3 Comparison of effect on ductility trough between grain-boundary serration and grain-boundary precipitation

From the results mentioned in the previous two sections, it is understood that both the grain-boundary serration and the grain-boundary precipitates are the causes of the ductility trough, but it is not clear which is more effective controlling factor, or whether there is any cooperation between them. These are discussed in this section.

Figure 13 shows an example of rare intergranular dimple, whose size was nearly the same as that of cavity shown in Fig. 5(b). Therefore, such dimple is considered to be a cavity, but different from usual ones in the meaning that the surroundings of the cavity was ductile. It is noticed, however, that there were several precipitates inside of the dimple.

As well known, intergranular fracture at elevated temperature is usually caused by the initiation, growth and coalescence of integranular cavities, and the cavities are generally initiated at second phase particles in the grain-boundary, because it is said that the particles are often poorly bonded to the matrix, and because grain-boundary sliding produces stress concentration at the matrix-particle interface. However, Raj$^{7)}$ studied the fracturing behavior of bicrystals of Cu-0.1%Si alloy which was internally oxidized to obtain dispersed silica particles, and showed that cavities did not originate at silica particles when the size of cavities was considerably different from the average spacing of the silica particles. Nearly the same behavior was also observed in the weld metal of Invar, that is, the average spacing of the precipitates in Figs. 7(b), 8(b) and (c) was less than about 1 μm, while the average size of cavities was about 2 to 5 μm as in Fig. 5. This means that the cavity formation can not been explained fully by the effect of precipitates.

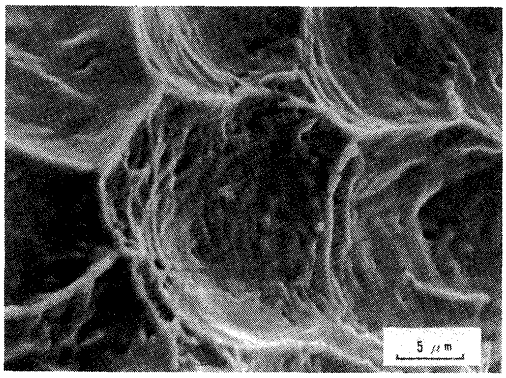


Fig. 13 An example of intergranular dimple including several precipitates, Item No. 1, 900°C.

On the other hand, the temperature dependency of the average period of the serrated grain-boundary shown in Fig. 3 had the same tendency as that of the average size of wavy feature of the intergranular fracture surface shown in Fig. 2. Moreover, these period and size agree nearly with each other. Therefore, according to only the feature of the fracture surface, the serrated grain-boundary may be more effective for the cavity formation.

Concerning this, Raj$^{7)}$ showed that there were copious slip bands on the fracture surface when the fracture time was short (30 sec) under high loading level. He suggested that the fracture in this case was caused by the incompatibility of the matrix slip at the grain-boundary. From this viewpoint, it is considered that the reheat hot cracking in the weld metal of Invar may be caused by the incompatibility of the matrix slip at the grain-boundary, because weld thermal cycle is generally very short. For example, **Fig. 14** shows that slip bands are seen both on the specimen surface and the fracture surface. Moreover, **Fig. 15** shows an interesting phenomenon that the slip bands on the specimen surface continue to the ledges on the

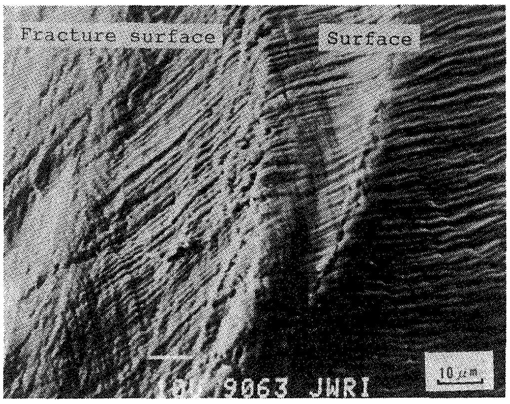


Fig. 14 Correlation between slip bands on specimen surface and feature of intergranular fracture surface in Item No. 12 (900°C, $\dot{d}_h$)

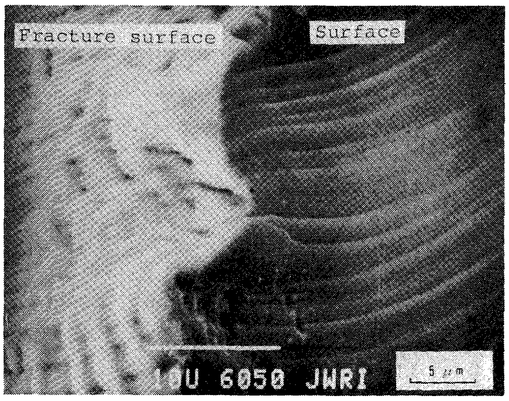


Fig. 15 Correlation between slip bands on specimen surface and feature of intergranular fracture surface in Item No. 1 (1100°C, $\dot{d}_m$)

wavy fracture surface. It is noted that the spacing of the slip bands was nearly the same as the size of the wavy feature in Fig. 15. This intimate relation between the slip bands and the grain-boundary serration was also seen at grain-boundary steps on the surface of weld metal after the actual weld cracking test as shown in **Fig. 16**.

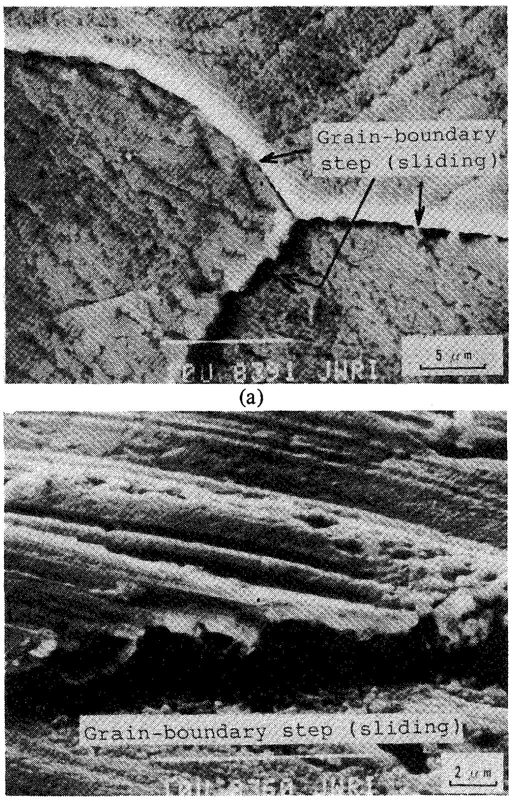


Fig. 16 Examples of grain-boundary step (sliding), slip bands and grain-boundary ledges after actual weld cracking test for Item No. 10.

However, the comparison between Item No. 1 insensitive to the cracking with Item No. 12 sensitive to the cracking suggests very strongly that the serrated grain-boundary itself without the aid of the grain-boundary precipitation is not enough to cause cavity formation, because both materials showed similar serrated grain-boundary and similar wavy intergranular fracture surface. This is supported by the fact that the temperature giving the minimum ductility was nearly independent of the strain rate as shown in Report III[8)], because Horiuchi[9)] showed that the temperature giving the minimum ductility must shift to a higher value with the increase in strain rate if only the serrated grain-boundary is the factor of cavity formation.

Therefore, it may be concluded that the cavity formation in the reheat hot cracking is caused by the cooperation of the serrated grain-boundary and the grain-boundary precipitation, as follows: At the beginning, grain-boundary is serrated by grain-boundary sliding and grain-boundary migration[10)], and this produces periodically concentrated plus and minus stress distribution along the grain-boundary. The coefficient of the stress concentration maybe depends on the morphology of the serrated grain-boundary influenced by testing temperature and strain rate, and the maximum stress maybe depends on tensile stress and the coefficient of stress concentration under various testing temperature. If there are precipitates on the serrated grain-boundary, the stress concentration due to the precipitates must be superposed, and the interface between the matrix and the precipitates are generally weak. Consequently, cavities will be easily formed at precipitates. The cavities, however, soon grow in their half period of plus stress, as seen in Fig. 5, and thus this characteristic may cause the wavy feature of the integranular fracture surface. On the other hand, if there are little precipitates, the level of the stress concentration is not enhanced enough, cavity formation is not easy, and thus the ductility is high. The feature of the fracture surface, however, is also wavy, because the overall stress concentration is distributed by the serrated grain-boundary. Once the cavities are formed in the both cases, the cracking will be formed by the growth and coalescence of the cavities.

3.4 Effect of dendritic segregation on reheat hot cracking

It was shown in Report I[11)] that the reheat hot cracking occurs predominantly in the weld metal, but does hardly in HAZ. This is very interesting from the viewpoint of the mechanism of this crack. **Table 2** assures this using Item No. 12, namely the hot ductility ΔW/W at 900°C is extremely low in the weld metal comparing those in the base metal and the synthetic HAZ. Also interestingly in Table 2, the density of precipitates on the intergranular fracture surface at 900°C is very high in the weld metal comparing those in the base metal and the synthetic HAZ. This implies that the high sensitivity of the weld metal to the crack may be attributed to dendritic segregation causing many grain-boundary precipitates, although of course large grain size in the weld metal may be another reason.

Thus, the effect of solution treatment to diminish the dendritic segregation was studied. The solution treatment was done at 1350°C for 180 sec and 540 sec, and then the hot ductility test was done at 900°C, which is illustrated

Table 2 Comparison of ductility and density of precipitates on intergranular fracture surface at 900°C among base metal, synthetic HAZ and weld metal.

	$\Delta W/W$ x100(%)*	ρ_p (1/100μm²)**
Base metal	22	7
Synthetic HAZ (peak temp.:1350°C)	19	19
Weld metal	5.5	91

*: ductility at 900°C

**: density of precipitates on intergranular fracture surface at 900°C

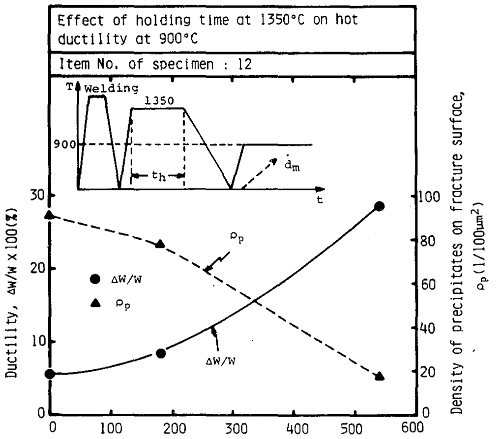


Fig. 17 Effect of solution treatment at 1350°C for weld metal on ductility and density of precipitates on intergranular fracture surface at 900°C.

in **Fig. 17** together with the results of ductility ΔW/W and density of precipitates on the fracture surface. Figure 17 means that the solution treatment for 180 sec had only a little effect on the recovery of the ductility, but the solution treatment for 540 sec had noticeable effect on the recovery of the ductility, and this tendency corresponds well to the decrease in the density of precipitates.

Figure 18 shows the solidification substructure of these specimens. Dendritic substructure remained a little after the treatment for 180 sec, but disappeared almost completely after the treatment for 540 sec. Thus, it is guessed that the dendritic subboundary promotes the grain-boundary precipitation and thus accelerates the ductility trough. Then, this behavior was checked microscopically. **Figure 19** shows an example of the microstructure before the hot ductility test, and it was generally seen that there are more precipitates at the site of the migrated grain-boundary intersecting with the dendritic subboundary as seen in (c) than other general sites as seen in (b).

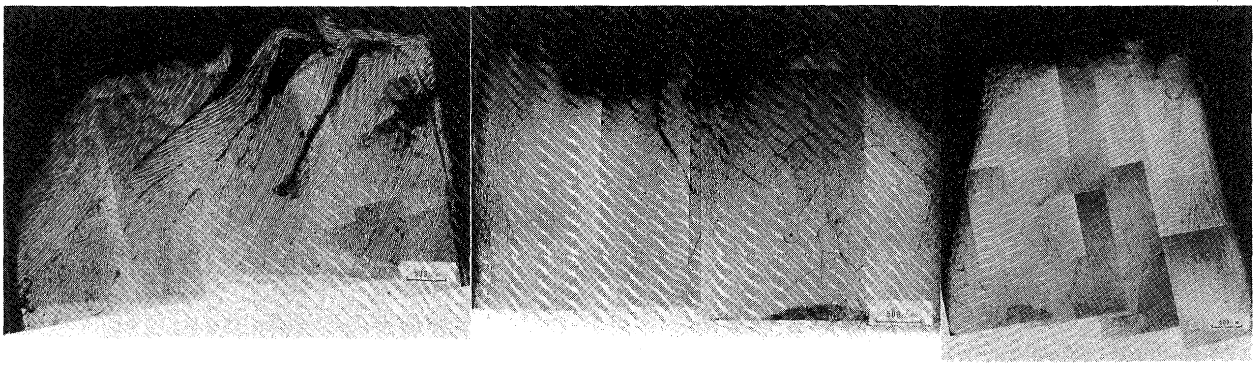
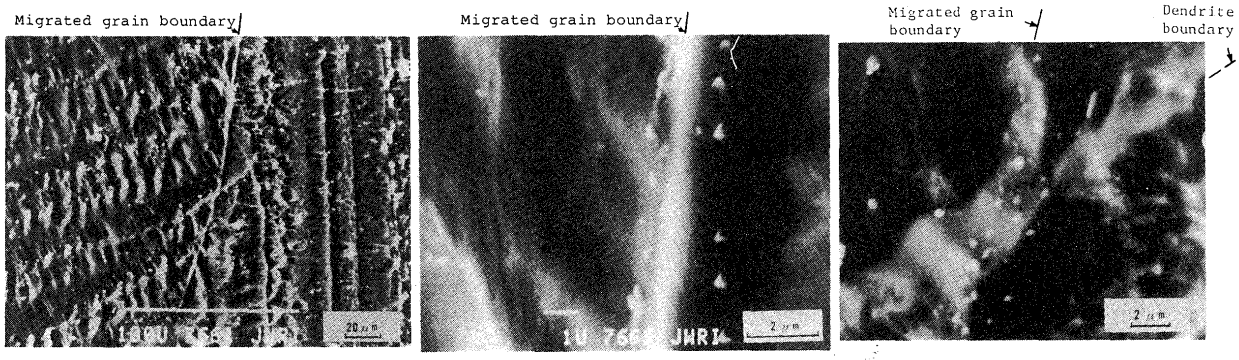

(a) without solution treatment (b) 1350°C X 180 sec (c) 1350°C X 540 sec

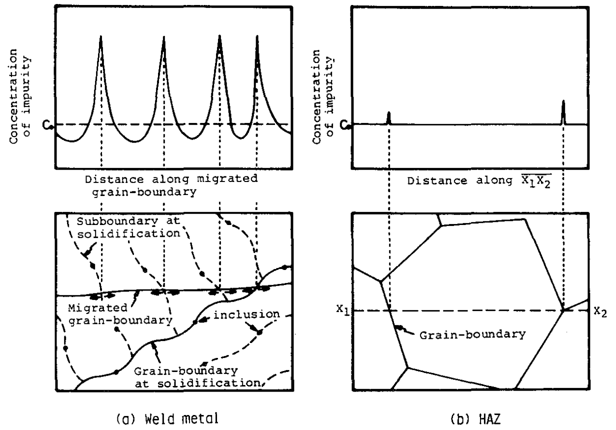
Fig. 18 Effect of solution treatment at 1350°C on solidification substructure.



(a) low magnification (b) general grain-boundary (c) grain-boundary at intersection with dendritic subboundary

Fig. 19 Effect of dendritic subboundary on grain-boundary precipitation after welding, Item No. 25.

Since the solute concentration at the dendritic subboundary is high, the grain-boundary precipitation should easily occur at the intersection with the dendritic subboundary. Moreover, it is supposed that also a certain region of the migrated grain-boundary near the intersection may be enriched by solute elements due to the grain-boundary diffusion from the intersection. These situations are illustrated in **Fig. 20(a)**, where arrows show the grain-boundary diffusion as speedy diffusion path. On the other hand in HAZ, such high segregation as the solidification microsegregation cannot exist as shown in **Fig. 20(b)**. Only grain-boundary segregation induced by the sweeping up[12] of solute element to grain-boundary during grain growth and/or by the driving force of equilibrium grain-boundary segregation should be exist if the effect of inclu-



(a) Weld metal (b) HAZ

Fig. 20 Illustration of segregation behavior on migrated grain-boundary in weld metal and HAZ.

sions is negligible. Therefore, it is concluded that the dendritic substructure due to the microsegregation during solidification is detrimental because of the role in promoting the grain-boundary precipitation.

It may be questioned why the ductility after the solution treatment for 540 sec in Fig. 17 is higher than those of the base metal and the synthetic HAZ in Table 2 inspite of its more precipitates and larger grain size than those in the base metal and the synthetic HAZ. This is considered to be explained by the fact that the size of precipitates after the solution treatment for 540 sec was coarsened.

3.5 Comprehensive process of reheat hot cracking

On the basis of the results in this study and the previous paper$^{1)}$, various factors affecting the reheat hot cracking in the weld metal of Fe-36%Ni alloy Invar are summarized in **Fig. 21**. The reheat hot cracking is connected with the ductility trough occurring between about 600°C and 1100°C as shown in (g), and the ductility trough is caused by the cavity formation by the cooperative effects of grain-boundary sliding (a), grain-boundary precipitation (b) and grain-boundary serration (c). The grain-boundary precipitation is promoted by dendritic segregation. Below about 600°C, not only the grain-boundary sliding but also the grain-boundary precipitates are little. Moreover, the grain-boundary is generally smooth. Thus, the hot ductility is not low. Above about 1100°C, the effect of the grain-boundary sliding is easily relieved by active grain-boundary migration(d). Also the stress concentration to the grain-boundary is not large owing to relatively little precipitates, relatively long period of the serrated grain-boundary and low flow stress (e). Furthermore, strain-concentrated grain-boundary disappears easily by dynamic recrystallization (f).

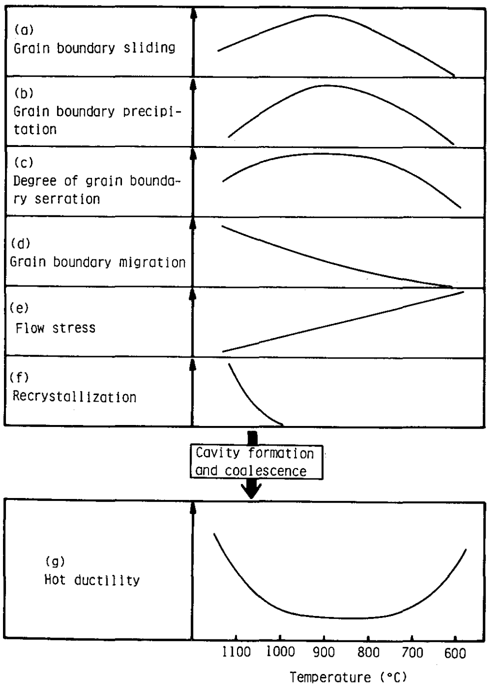


Fig. 21 Comprehensive illustration of factors affecting ductility trough causing reheat hot cracking.

It is sometimes pointed out$^{13,14)}$ that the grain-boundary segregation of sulphur causes the high temperature embrittlement in nickel and nickel-base alloys. However, Auger electron spectroscopic analysis in this study showed no evidence of grain-boundary segregation of sulphur on the intergranular fracture surface in the weld metal of Invar. Although the addition of such element having strong chemical affinity with sulphur as Ca, Mg or Zr is effective to reduce the embrittlement in nickel and nickel-base alloys$^{13,14)}$, the addition of Ti which has also strong chemical affinity with sulphur promotes the reheat hot cracking in Invar$^{15)}$. This also implies that there is little effect of grain-boundary segregation of sulphur on the reheat hot cracking in the weld metal of Invar.

4. Conclusions

Main conclusions obtained are as follows:

1) The characteristic of ductility curve has a good correlation with the temperature dependency of the density of precipitates on the intergranular fracture surface. Moreover, the difference in the ductility between different materials is related to the difference in the density of precipitates. The precipitates identified were MnS, MnO, AlN, Al_2O_3.
2) The size of wave of wavy intergranular fracture surface depending on testing temperature and crosshead speed corresponds well to the period of serrated grain-boundary.
3) It is considered that the cavity formation causing the ductility trough occurs due to the cooperation of the grain-boundary precipitates and the serrated grain-boundary under the process of grain-boundary sliding.
4) Dendritic substructure in which solidification microsegregation exists promotes the grain-boundary precipitation, and is one of the reason why the weld metal is more sensitive to the cracking than HAZ.

5) The process of the ductility trough was represented well as a synthesis of grain-boundary precipitation, serrated grain-boundary, grain-boundary sliding, grain-boundary migration, flow stress and dynamic recrystallization.

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