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A New Explanation for Role of Delta-Ferrite Improving Weld Solidification Crack Susceptibility in Austenitic Stainless Steel[†]

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Abstract

The reason why delta-ferrite improves the weld solidification crack susceptibility of austenitic stainless steels has been investigated, applying fractographic technique to solidification cracks induced with the Varestraint test. Then a new explanation for the beneficial effect of delta-ferrite has been proposed. Main conclusions obtained are as follows:

(1) Segregation of impurities during the solidification is decreased in the weld metal containing a small amount of delta-ferrite, because delta-ferrite having a greater solubility for impurities solidifies as the primary phase.

(2) Propagation of solidification crack in the weld metal containing a small amount of delta-ferrite is arrested by the peritectic/eutectic reaction, because the residual liguids are confined as liguid droplets inside austenite grains formed on the occasion of this reaction. This (2) is the new explanation proposed by the authors.

KEY WORDS: (Austenitic Stainless Steels) (Solidification) (Hot Cracking) (Ferrite) (Fractography)

1. Introduction

It is well known that weld metals of fully austenitic stainless steels are very susceptible to solidification cracks. On the other hand, it is also well known that weld metals of austenitic stainless steels containing a small amount (more than about 5%) of delta-ferrite are much more resistant to solidification cracks.

As regards the reason why delta-ferrite prevents the crack, there are several explanations so far^{1-6} : (1) Ferrite has a greater solubility than austenite for harmful elements such as sulphur and phosphorus and thus the segregation of these elements at the grain boundary is decreased. (2) Total amount of grain boundaries is enlarged due to the presence of ferrite-austenite boundary together with austenite grain boundary. (3) Austenite grain size would be refined in a two phase alloy containing ferrite. (4) Compositions forming some ferrite have a smaller solidification range than fully austenitic composition. (5) Contraction stress is reduced because of a smaller coefficient of thermal expansion of ferrite. (6) Liquid films are dispersed by the existence of ferrite, and ferrite strengthens the grain boundary or prevents the formation or propagation of crack. (7) Ferrite suppresses polygonization process. Among them, the first explanation is widely accepted and also is experimentally proved by the authors⁷⁾. However, judging from the complicated solidification mode⁷⁾ of austenitic stainless steels containing a small amount of ferrite, the authors think that this problem is not so simple and that there must be also another factor.

On the other hand, the authors showed in a previous paper⁸⁾ that application of fractographic technique to the solidification crack induced with the Varestraint test is very useful to study the behavior of crack vs. the crack-opening temperature. Therefore, the present work was undertaken to reveal the role of delta-ferrite improving the crack susceptibility by using mainly this fractographic technique.

2. Materials Used and Experimental Procedure

2.1 Materials Used

Materials used are 25Cr-20Ni type fully austenitic stainless steel SUS310S[☆], 17Cr type ferritic stainless steel SUS430 and 18Cr-8Ni type austenitic stainless steel SUS304. The chemical compositions are shown in **Table 1**. As regards SUS304, three commercial sheets were used in order to confirm that the results obtained would have

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[☆] SUS is the designation for stainless steel in Japan Industrial Standard (JIS).

SUS310S, 430 and 304 correspond to AIS1 310S, 430 and 304 respectively.

Table 1 Chemical compositions of materials used

Material	Composition (wt. %)							
. Waterial	C	Si	Mn	P	S	Cr	Ni	
SUS310S	0.08	0.94	1.58	0.022	0.007	24.7	20.4	
SUS430	0.060	0.61	0.52	0.021	0.006	16.54	0.13	
SUS304 (A)	0.072	0.52	0.91	0.030	0.005	18.12	8.74	
SUS304 (B)	0.070	0.56	1.50	0.034	0.010	18.45	8.59	
SUS304 (C)	0.070	0.70	1.18	0.027	0.010	18.26	8.44	
SUS304+S	0.050	0.56	0.94	0.025	0.22	18.50	9.10	

generality. Further, SUS304+S in Table 1 is extraordinary SUS304 to which about 0.2% sulphur was added in order to study the behavior of sulphur-enriched residual liguids at the last stage of solidification of SUS304. The thickness of each material was 3mm.

2.2 Thermal Analysis

Thermal analysis of each sample was made to determine the liquidus temperature and, if present, the other reaction temperature during solidification. To do this, cooling curve was measured with a Pt-Pt/13%Rh thermocouple of 0.5mm diameter after melting the sample of about 100g in an alumina crucible using an electric furnace of argon atmosphere. Then, the cooling curve was converted into inverse rate curve.

Furthermore, the melting temperature of sulphidematrix eutectic of SUS304+S was measured by direct observation with a hot-stage microscope.

2.3 The Trans-Varestraint Test

The Trans-Varestraint test was performed to induce solidification cracks while TIG-arc bead-on-plate welding without filler metal was being carried out in conditions of 100A, 12-13V(DCSP) and 150mm/min. The temperature distribution along the center of weld metal was measured with a W/5%Re-W/26%Re thermocouple of 0.3mm diameter. The detail of the test was the same as in a previous paper⁹).

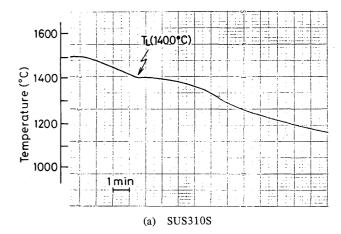
2.4 Observation of Cracked Surface

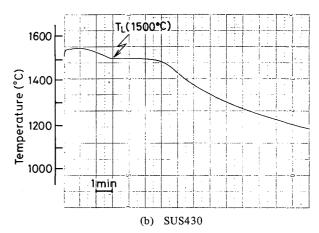
The welded specimen, to which an augmented strain of 3.75% was applied with the Varestraint test, was fractured in room temperature using an aid of mechanical vice in order to expose the cracked surface. Then, the cracked surface was observed with a scanning electron microscope (SEM), and elements on the cracked surface were analyzed with an energy-dispersive type analyzer equipped with the SEM.

3. Experimental Results and Discussion

3.1 Thermal Analysis

Cooling curves for the commercial meterials are shown





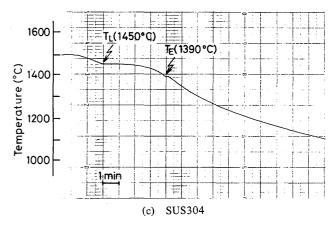


Fig. 1 Comparison of cooling curves among SUS310S, 430 and 304

in Fig. 1. Smooth cooling after the liquidus temperature T_L in Figs. 1(a) and (b) means that the solidification modes of SUS310S and 430 are similar to that of an alloy forming a simple solid solution*. On the other hand,

^{*} As regards SUS430, it is generally said that a peritectic reaction occurs at the last stage of solidification. For our SUS430, however, any fluctuation due to the peritectic reaction was not observed even in the inverse rate curve, perhaps owing to somewhat lower carbon content. After the solidification our SUS430 also, of course, must have transformed partly to austenite and further to martensite.

there is a crooked point at 1390°C on the cooling curve of SUS304 in Fig. 1(c). This is caused by a peritectic/eutectic reaction, judging from Fe-Ni-Cr phase diagram¹⁰. The peritectic reaction is; liquid plus delta-ferrite austenite. The eutectic reaction is; liguid austenite. It is obscure which of them acts at the crooked point in Fig. 1(c), because the detail of Fe-Ni-Cr phase diagram is incomplete and also SUS304 is not a simple Fe-Ni-Cr alloy. There must be some possibility that the both reactions act. By the way, in SUS304+S both the liquidus temperature and the crooked point lowered by about 20°C compared with those of SUS304.

3.2 Ductility Curve

Ductility curves for the commercial materials obtained with the Trans-Varestraint test are shown in Fig. 2. The

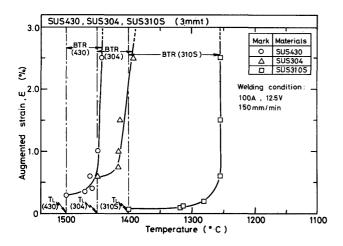


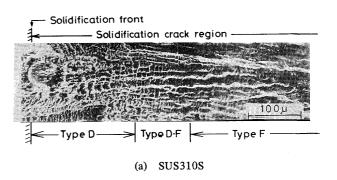
Fig. 2 Comparison of ductility curves obtained with the Trans-Varestraint test among SUS310S, 430 and 304.

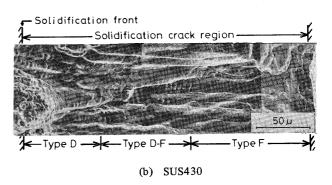
ductility curve of SUS304 is fairly superior to that of SUS310S, and further somewhat superior even to that of SUS430. That is to say, the brittleness temperature range (BTR) of SUS304 is narrower than that of SUS430 in the augmented strain less than about 2%, and the minimum ductility of SUS304 is higher than that of SUS430. It should be noticed that BTR at the augmented strain of about 0.5%~1% is nearly equal to that at 2.5% for SUS310S and 430, while the former is about half as wide as the latter for SUS304. The reason will be mentioned later.

3.3 Fractography of Solidification Crack

3.3.1 SUS310S

Microfractograph of the solidification crack of SUS310S induced with the Trans-Varestraint test is shown in Fig. 3(a). This was discussed in detail in the previous





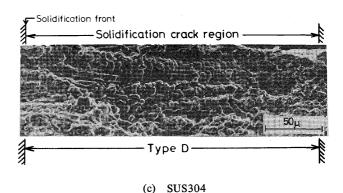


Fig. 3 Comparison of microfractographs of solidification cracks induced with the Trans-Varestraint test among SUS310S, 430 and 304, the augmented strain ε =3.75%.

paper⁸⁾, and the fracture mode of the cracked surface was classified into three types, namely, Types D, D-F and F. In Type D seen in the highest temperature region in the BTR, many protuberances are remarkably observed and correspond to the dendrite arms. In Type D-F seen in the medium temperature region, the protuberances of secondary arms become gradually obscure. In Type F seen in the lowest temperature region, the protuberances of primary arms also become obscure gradually and consequently a flat surface becomes dominant. This change is mainly caused by gradual decrease in residual liguids in the interdendritic sites.

Moreover, observation in much higher magnification revealed the existence of non-metallic inclusions enriched with phosphorus, sulphur or oxygen on the cracked surface.

3.3.2 SUS430

Microfractograph of the solidification crack of SUS430 is shown in Fig. 3(b), which gives a feature consisting of Types D, D-F and F similar to that of SUS310S. The crack, however, is very small and non-metallic inclusions are seldom observed on the cracked surface. Small crack length and few non-metallic inclusions must be caused by the larger solubilities or distribution coefficients of sulphur, phosphorus, etc. in ferrite in comparison with those in austenite. In addition, the very narrow solidification range at about 17%Cr in Fe-Cr phase diagram¹¹⁾ may be another reason for small crack length.

By the way, the features of solidification cracked surfaces of aluminum alloys 1070, 5052 and 5083 also consist of Types D, D-F and F^{12}). This suggests that this feature giving Types D, D-F and F is intrinsic to an alloy where a solid solution is largely formed on the occasion of completion of the solidification.

3.3.3 SUS304

An example of microfractograph of the solidification crack of SUS304 is shown in Fig. 3(c), which gives a feature consisting of only Type D. Namely, SUS304 is very different from SUS310S and 430 in that the solidification crack surface doesn't indicate the feature of Type F. Observing in detail, the rugged feature in the lower half in temperature, that is, in the right half of Type D seems to be somewhat finer than that in the higher half. It is considered that Type D in the higher half is similar to that of SUS430, because SUS304 solidifies as ferrite phase until the peritectic/eutectic reaction. Therefore, the reason why SUS304 is insensitive to solidification crack seems to be related to the existence of Type D in the lower half. This feature of cracked surface is always observed in all SUS304 used and even in SUS304+S.

Moreover, non-metallic inclusions are seldom observed on the cracked surface except SUS304+S.

3.3.4 Distribution of Chromium on Cracked Surface

It was recognized in the previous paper⁸⁾ that element distribution on the cracked surface gives important information to study the characteristics of the crack formation. Thus, in this work the distribution of chromium of the cracked surface was also measured. The results are shown in Fig. 4. The Cr counts of SUS310S, which were quoted from the previous paper⁸⁾, increase gradually from Type D toward Type D-F and then decrease gradually in Type F. The increase can be

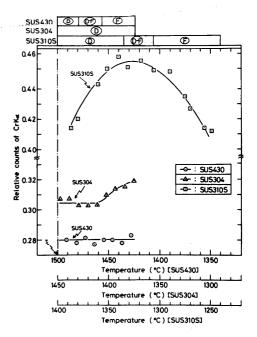


Fig. 4 Comparison of distributions of relative counts of CrK_{α} on cracked surface among SUS310S, 430 and 304. $\varepsilon = 3.75\%$.

explained from a viewpoint of the accumulation of segregation as the solidification advances, and the decrease can be explained from the fact that grain boundary migration occurs in the parts where the solid bridging has already completed⁸).

The Cr counts of SUS430 in Fig. 4 are nearly constant regardless of Types D, D-F and F. This can be explained from a viewpoint that the distribution coefficient of chromium during the solidification of ferrite is nearly unity and thus the segregation is little since chromium is a ferrite-stabilizer. In SUS430 the grain boundary migration occurred partly as well at the last stage of solidification, but its effect on the distribution of Cr counts is negligible because of the little segregation of chromium at the solidification grain boundary.

The Cr counts of SUS304 in Fig. 4 are nearly constant in the higher half in temperature and increase gradually in the lower half. The behavior in the higher half is considered to be explained by the same cause as that mentioned for SUS430. It is noteworthy that the temperature from which the Cr counts begin to increase agrees approximately with the temperature of the crooked point of the cooling curve in Fig. 1(c).

3.4 Crack Path in SUS304 Weld Metal

It was already revealed for SUS304 in the previous paper⁷⁾ that delta-ferrite is situated in the core of cellular dendrite and that austenite phase occupies the boundaries between the cellular dendrites and between the columnar

grains of delta-ferrite. Thus, if the microstructure containing solidification crack is observed in room temperature, the crack must essentially pass through the austenite phase.

Now, a microstructure in the lower temperature region of the solidification crack of SUS304 induced by the Varestraint test is shown in Fig. 5. It should be

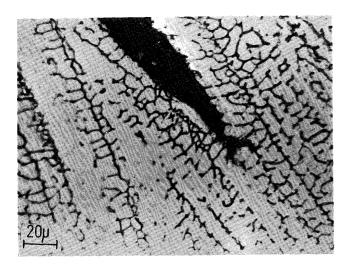


Fig. 5 Microstructure around a solidification crack of SUS304 induced with the Trans-Varestraint test. ε = 3.75%.

noticed that the crack passes along ferrite-austenite boundary. For reference the content of delta-ferrite is 3.0% according to a Ferrite Scope.

Fig. 6 shows a pair of cracked surfaces of SUS304 etched with Kalling's reagent which are opposite to each other taken from the same crack. It is known that Kalling's reagent etches preferably delta-ferrite. According to the general view of Figs. 6(a) and (b), both of the opposite cracked surfaces are not etched in the higher half in temperature, namely in the left half. That is to say, the both cracked surfaces are occupied by austenite. This means that the solidification crack must have passed along delta-ferrite's grain boundary filled with liquids. and that the liquids were transformed into austenite. However, the both in the lower half are partly etched and partly not etched as seen in Figs. 6(c) and (d) which are outlined areas in Figs. 6(a) and (b) respectively. Moreover, the detailed observation of Figs. 6(c) and (d) shows that the part opposite to an etched part on one of the paired cracked surfaces is not etched. This means that the solidification crack in the lower temperature region passes along ferrite-austenite boundary as already seen in Fig. 5.

Now, why does the feature of the cracked surface of SUS304 in Fig. 3(c) give Type D even in the lower temperature region? This can be explained as follows: It

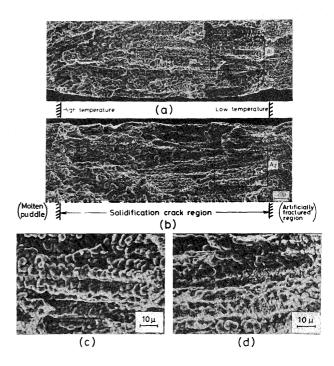


Fig. 6 Microfractograph of solidification crack of SUS304 etched with Kalling's reagent. €=3.75%.

- (a) general view of cracked surface
- (b) cracked surface opposite to (a)
- (c) outlined area A₁ in (a)
- (d) outlined area A2 in (b)

has been shown¹³⁾ that the austenite formed by the peritectic/eutectic reaction grows into the delta-ferrite in Widmanstätten-like mode in rapid cooling condition. Thus, the ferrite-austenite boundaries are very rugged and the cracked surface reflects this rugged feature. Therefore, Type D of SUS304 is caused both by the cellular dendrites in the higher temperature region and by the ferrite-austenite boundaries in the lower temperature region.

Moreover, the increase in Cr counts on the cracked surface of SUS304 in the lower temperature region as shown in Fig. 4 can be explained by the existence of delta-ferrite on the cracked surface, because chromium is somewhat more enriched into delta-ferrite than austenite⁷).

Now, there is an important matter which should be emphasized to avoid misunderstanding the behavior of crack. This is that the path along ferrite-austenite boundary in the lower temperature region of the crack is observed only in such an abnormally large augmented strain as 3.75%. As seen in Fig. 2, the crack of SUS304 in the augmented strain not larger than 2.5% is always stopped at a temperature not lower than 1390°C which is the crooked point in Fig. 1(c). Therefore, it should be rather said that the peritectic/eutectic reaction arrests the crack propagation. As regards SUS310S and 430, the

features of cracked surfaces in $0.5\sim1\%$ augmented strain are the same as those of 3.75% as expected from the shape of ductility curves in Fig. 2.

3.5 Behavior of Residual Liquids in SUS304 Weld Metal

Then, the behavior of residual liquids after the beginning of the peritectic/eutectic reaction becomes an interesting problem. For the purpose of revealing this, the microstructure of the weld metal of SUS304+S was examined in detail. The microstructure in Fig. 7, where

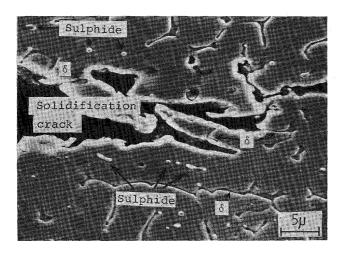


Fig. 7 Microstructure around a solidification crack of SUS304+S induced with the Trans-Varestraint test. $\varepsilon = 3.75\%$.

the right direction is lower in temperature, shows globular sulphides (Mn, Cr)S, the network of delta-ferrites and solidification crack. It is observed that the crack passes along ferrite-austenite boundaries. On the other hand, observation of the melting of the sulphide-matrix eutectic with a hot-stage microscope indicated that the melting occurred at about 1300°C. Since the parts showing the microstructure similar to Fig. 7 are situated between about 1330 and 1370°C at the moment of crack formation, the sulphides must have been still liquids. Therefore it should be natural to expect that the crack propagates along the row of the sulphide-liquid droplets. On the contrary, the crack propagates along ferrite-austenite boundaries as seen in Fig. 7.

The reason why the crack does not propagate along the row of the sulphide-liquid droplets is considered to be that any grain boundary between austenite grains is not observed along the row of the droplets in Fig. 7. In other words, because the residual liquids after the beginning of peritectic/eutectic reaction are confined as liquid droplets inside the austenite grains, it is difficult for crack to propagate along the row of the liquid droplets. The crack must be compelled to propagate along the ferrite-austenite boundaries in an extremely large augmented strain,

because grain boundary and inter-phase boundary such as ferrite-austenite boundary are well known to be generally weak in high temperatures. It may be also suspected that ferrite-austenite boundary is weakened by the enrichment of sulphur, etc. because supersaturated sulphur, etc. in the austenite is rejected toward the ferrite during the growth of austenite.

Besides, as regards the fact mentioned in Fig. 7 that any grain boundary between austenite grains is not observed along the row of the droplets, it was also confirmed by an etch pit method¹⁴) that the shape of etch pit, namely the crystallographic orientation is uniform around the sulphides formed in the grain boundary

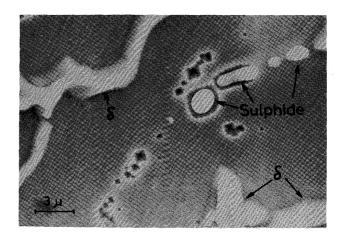
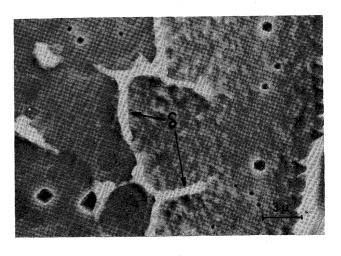


Fig. 8 Etch pits around sulphides in weld metal of SUS304+S

as shown in Fig. 8. Furthermore, the etch pit method revealed that austenite grains adjacent to each other with different orientations are bounded by delta-ferrite as seen in Fig. 9(a). At the place where delta-ferrite is disconnected, austenite grain boundary is formed so as to link the delta-ferrites as seen in Fig. 9(b).



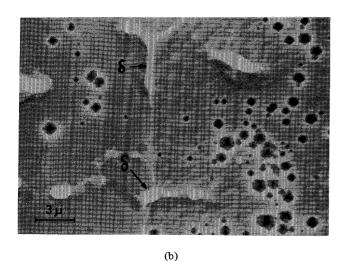


Fig. 9 Relation between etch pits in austenite and delta-ferrite in weld metal of SUS304

3.6 Summary of Solidification Mode and Role of Delta-Ferrite against Solidification Crack in SUS304 Weld Metal

The solidification mode of SUS304 is illustrated in Fig. 10. Delta-ferrite solidifies as the primary phase from the

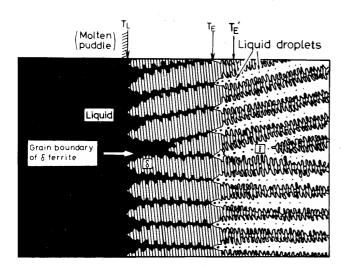


Fig. 10 Illustration of solidification mode of SUS304

liquidus temperature T_L till T_E which is the beginning temperature of peritectic/eutectic reaction. The peritectic/eutectic reaction is completed between T_E and T_E '. The rejection of impurities such as sulphur, phosphorus, etc. into the residual liquids is reduced owing to the primary solidification of ferrite phase, and this is one of the reasons why SUS304 is insensitive to solidification cracks as generally understood. Moreover, the reduced segregation must result in a narrower true solidification range than SUS310S. Additionally the authors wish to say the

following fact in this report as a new explanation for the role of delta-ferrite improving solidification crack susceptibility. From $T_{\rm E}$ austenite solidifies and grows into delta-ferrite in Widmanstätten-like mode. Therefore, the residual liquids on this occasion are confined as liquid droplets inside the austenite grains. Then there is no grain boundary along the row of these residual liquid droplets. Accordingly, if a crack is propagating, it is usually arrested near $T_{\rm E}$, because it is difficult for the crack to propagate along the row of the liquid droplets inside the grains. Moreover, if excess strain is loaded, the crack advances turning to ferrite-austenite boundary.

For the sake of comparison, the solidification mode of SUS310S is illustrated in Fig. 11. Because the rejection of

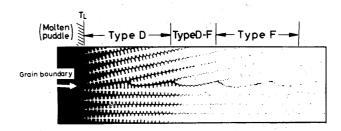


Fig. 11 Illustration of solidification mode of SUS310S

impurities into the residual liquids is remarkable due to the solidification of austenite as the primary phase, there remains residual liquids till fairly low temperature. Moreover, the grain boundaries also remain pinned to the residual liquids because of no transformation, although grain boundary migration occurs in the parts where solid bridging has already completed. Therefore, if a crack should be once initiated the propagation is very easy to low temperature region within the BTR.

4. Conclusions

The reason why delta-ferrite improves the weld solidification crack susceptibility of austenitic stainless steel was investigated by applying the fractographic technique to the solidification crack induced with the Varestraint test. Then the authors proposed a new explanation for the role of delta-ferrite. Main conclusions obtained are as follows:

- (1) The surface of solidification crack of ferritic stainless steel SUS430 gives a feature similar to that of fully austenitic stainless steel SUS310S, namely dendritic Type D, transitional Type D-F and flat Type F, although the crack length is smaller and non-metallic inclusions enriched with sulphur, etc. are less on the cracked surface.
- (2) The surface of solidification crack of austenitic stain-

- less steel SUS304 where there remains a small amount of residual delta-ferrite in the weld metal gives only a feature similar to Type D, although non-metallic inclusions on the cracked surface are few as well as SUS430.
- (3) The solidification crack of SUS304 is usually arrested at a temperature not lower than the beginning temperature of peritectic/eutectic reaction. This is caused by a fact that the residual liquids on the occasion of this reaction are confined as liquid droplets inside the austenite grains formed by this reaction, and thus the crack propagation becomes difficult to advance along the row of the droplets because there is no longer grain boundary there. If excess strain is loaded, the crack propagates along rather ferrite-austenite boundary. In this case the cracked surface gives a feature similar to Type D, because the ferrite-austenite boundary is rugged due to the Widmanstätten-like growth of austenite.
- (4) From the above results, the role of delta-ferrite improving the solidification crack susceptibility are summarized as follows: (i) Segregation of impurities during the solidification is decreased, because ferrite having a greater solubility and distribution coefficient for impurities solidifies as the primary phase. This must result in a narrower solidification range than SUS310S. (ii) Propagation of solidification crack is arrested by the peritetic/eutectic reaction, because the residual liquids are confined as liquid droplets inside austenite grains formed on the occasion of this reaction. This (ii) is the new explanation proposed by the authors.

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