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Review

Improved fracture toughness of ultrahigh strength steel through control of non-metallic inclusions

Y. TOMITA
Department of Metallurgical Engineering, College of Engineering, University of Osaka Prefecture, 1-1 Gakuen-cho, Sakai, Osaka 593, Japan

In recent years, ultrahigh-strength steels, which can be employed successfully at yield strengths of 1400 MPa or higher, have been used increasingly for critical structural applications in aircraft and aerospace vehicles. Most recently, there has been increased demand, however, for ultrahigh-strength steel with superior plane-strain fracture toughness, K_{IC}, and for the steels suitable for large-sized structural applications; isotropy regarding the property has especially been required. One potential solution to this problem is to control non-metallic inclusions of the steels. This review concentrates on recent topics concerning improved K_{IC} of ultrahigh-strength steels, i.e. low-alloy and highly alloyed secondary hardening steels, through control of non-metallic inclusions. The major factors controlling the property are discussed for each of the techniques.

1. Introduction

In recent years, the use of ultrahigh-strength steels for critical structural applications in aircraft and aerospace vehicles has increased. These steels can be classified [1, 2] into three types, as shown in Table I: low-alloy steel containing medium carbon of lightly tempered martensitic structure (e.g. AISI 4340 and 300M) (Type I); highly alloyed secondary hardening steels containing low or medium carbon (e.g. AISI HY180 and AF1410) (Type II); and precipitation hardening steels containing significant low carbon (e.g. AISI 15-5PH and C250) (Type III).

The ultrahigh-strength steels can be employed successfully at yield strengths of 1400 MPa or higher. However, these steels may often develop fatigue and stress-corrosion cracks during service; consequently, catastrophic fracture may occur and lead to serious consequences which can cause financial loss, environmental contamination, or loss of life. To minimize the risk of failure, during the last decade, considerable research has been directed toward improving plane-strain fracture toughness, K_{IC}, which is the critical value of the stress-intensity factor and is used as a fracture criterion in crack growth of the ultrahigh-strength steels. So far, most of these studies have been focused on developing the K_{IC} of the steels with microstructural control. Among these, for example, are thermal and thermomechanical treatments [3–27] and new alloying designs [28–33].

Most recently there has been increased demand for ultrahigh-strength steels with superior K_{IC} and for steels suitable for large-sized structural applications and, in particular, isotropy regarding the property has been required. Although modification through microstructural control has benefits for dramatically developing the K_{IC}, it is not very promising for large-scale applications and for modifying the isotropy regarding the properties. In such situations, one potential solution to this problem is to control the non-metallic inclusions in the ultrahigh-strength steels. Therefore, research has been directed towards dramatically improving the K_{IC} of the ultrahigh-strength steels through control of the non-metallic inclusions.

This review concentrates on recent topics concerning improved K_{IC} of the ultrahigh-strength steels, i.e. low-alloy and highly secondary hardening steels, through control of non-metallic inclusions. The major factors controlling the property are discussed for each of the techniques.

2. Improved fracture toughness of low-alloy steel through control of non-metallic inclusions

The low-alloy steels are of medium carbon (0.25–0.50 wt %) content and contain variable amounts of chromium, molybdenum, nickel, silicon and vanadium (see Type I, Table I). The 4340 steel has been developed since 1950 and has been used in commercial practice since 1955. This steel is usually tempered at or close to 473 K. At present, it is widely used in aircraft, aerospace, and atomic power applications. The 300M

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TABLE I Chemical composition of typical AISI ultrahigh-strength steel for aircraft and aerospace applications

Type	Steel	Chemical composition (wt %)										
		C	Mn	Si	Ni	Co	Cr	Mo	V	Cu	Al	Ti
I	4340	0.40	0.7	0.2	1.8	–	0.85	0.25	–	–	–	–
	300M	0.40	0.7	1.6	1.8	–	0.85	0.40	0.1	–	–	–
	D6AC	0.48	0.7	0.2	0.6	–	1.0	1.0	–	–	–	–
	4330Si	0.30	0.7	0.6	2.0	–	1.2	0.45	–	–	–	–
	4330V	0.30	0.8	0.2	1.8	–	0.9	0.45	0.1	–	–	–
	HY-TUF	0.25	1.4	1.6	1.8	–	0.3	0.45	–	–	–	–
II	HP9-4-20	0.20	0.15	0.2	9.0	4	0.8	1.0	0.08	–	–	–
	HP9-4-30	0.30	0.15	0.1	9.0	4	1.0	1.0	0.08	–	–	–
	HY180	0.10	0.15	–	10	8	2.0	1.0	–	–	–	–
	AF1410	0.16	–	–	10	14	2.0	1.0	–	–	–	–
III	15-5PH	0.04	0.25	0.4	4.6	–	15	–	–	3.3	–	–
	PH13-8	0.04	–	–	8.0	–	13	2.2	–	–	1.1	–
	C-250	0.005	–	–	18	8	4.8	–	–	0.1	–	0.4

TABLE II Chemical composition of 0.4C–Cr–Mo–Ni steel [37]

Steel	Chemical composition (wt %)								
	C	Si	Mn	P	S	Cr	Mo	Ni	Ca
Heat 1	0.40	0.17	0.70	0.013	0.016	0.75	0.23	1.71	–
Heat 2	0.40	0.18	0.71	0.012	0.002	0.76	0.22	1.72	–
Heat 3	0.40	0.19	0.71	0.012	0.002	0.76	0.21	1.72	0.0061

TABLE III Inclusion data of 0.4C–Cr–Mo–Ni steel (Al_2O_3 inclusions also present, but no data evaluated) [37]

Steel	Inclusion	f_v[a] (%)	l[b] (μm)	w[c] (μm)	MAR[d]	R_0[e] (μm)
Heat 1	MnS	0.168	35.9	2.3	15.6	–
Heat 2	MnS	0.038	5.5	2.2	2.5	–
Heat 3	CaS	0.068	–	–	–	1.4

[a] Volume fraction.
[b] Mean length.
[c] Mean width.
[d] Mean aspect ratio (l/w).
[e] Mean diameter.

steel, mechanical properties of which are modified by adding silicon and vanadium alloying elements, has been extensively employed in critical components of the landing gear in aircraft since 1966. The silicon addition allows the steel to be tempered at temperatures higher than 473 K, increasing the yield strength and toughness compared with 4340 steel. If strength was not so important, but high toughness was required, e.g. the landing gear in helicopters and light transport planes, 4330Si or 4330V steel would be then used in preference to 4340 or 300M steel.

2.1. Desulphurizing treatment

In recent years, the theory and practice of desulphurization employed to produce a reduced sulphur content, have been significantly advanced and this is reflected in the definite tendency towards significantly low-sulphur content in steels now produced in high-quality grades. Rice and Johnson [34] have shown that the upper shelf fracture toughness of ultrahigh-strength steels is sensitive to both inclusion spacing and inclusion volume fraction. Cox and Low [35] have demonstrated that reducing the inclusion content increases the resistance to void nucleation and is also effective in improving the fracture toughness of 4340 ultrahigh-strength steels. Leslie [36] has also suggested that reducing the sulphur content may be a means of achieving reasonable fracture toughness of ultrahigh-strength steels.

From this point of view, Tomita [37] has recently studied the effect of desulphurizing treatment on the fracture toughness of 0.4C–Cr–Mo–Ni steel (4340 grade). Desulphurized steel containing 0.002 wt % S (Heat 2, Table II) compared with steel of commercial sulphur content (0.16 wt %) (Heat 1, Table II) decreased dramatically the MnS inclusion contents and its mean aspect ratio (Table III). This suppressed the lamellate fracture and improved mechanical isotropy as shown in Table IV. Unfortunately, intergranular failure occurred in the fracture toughness test so there was little difference in the K_{IC} of Heat 2 and that of

TABLE IV Effect of desulphurizing treatment on mechanical properties of 0.4C–Cr–Mo–Ni steel in longitudinal (L) and transverse (T) orientations [37]

Steel	σ_{YS}[a] (MPa)	σ_{UTS}[b] (MPa)	ε_f[c]	K_{IC}[d] (MPa m$^{1/2}$)	E_{ch}[e] (J)
Heat 1-L	1621.3	1800.6	0.36	51.2	21.8
Heat 1-T	1612.5	1798.6	0.16	36.5	8.2
Heat 2-L	1618.3	1820.9	0.39	51.6	20.3
Heat 2-T	1615.3	1825.6	0.33	44.9	16.3

[a] 0.2% yield strength.
[b] Ultimate tensile strength.
[c] True strain at fracture.
[d] Plane-strain fracture toughness.
[e] Charpy impact energy.

TABLE V Effect of calcium treatment on mechanical properties of 0.4C–Cr–Mo–Ni steel [37]

Steel	σ_{YS} (MPa)	σ_{UTS} (MPa)	ε_f	K_{IC} (MPa m$^{1/2}$)	E_{ch} (J)
Heat 2-L	1618.3	1820.9	0.39	51.6	20.3
Heat 2-T	1615.3	1825.6	0.33	44.9	16.3
Heat 3-L	1620.8	1830.3	0.63	79.5	26.9
Heat 3-T	1624.7	1823.7	0.51	73.8	23.4

Heat 1 (Table IV). The intergranular failure was attributed to the overheating phenomenon [38–46], in which manganese and sulphur, which are taken into solution in the high rolling temperature range, segregate to the grain boundaries and reprecipitate there as small globular MnS particles. Thus, when desulphurizing treatment is used alone, it does not always meet the current stringent requirements of K_{IC} of ultrahigh-strength steels.

2.2. Calcium treatment

Tomita [37, 47] has suggested calcium treatment whereby the overheating phenomenon disappears and K_{IC} is dramatically improved. Calcium modification has already been applied where low-temperature toughness is required or in deep-water applications where high reliability is demanded [48, 49]. He demonstrated that for desulphurized 4340 steel, addition of calcium to the desulphurized melt (Heat 3, Table II) is very effective for improving the fracture toughness, whereas calcium addition to the melt of steel containing sulphur at a commercial level, such as Heat 1, is not effective in modifying the morphology of the inclusions to improve the property. As can be seen from Tables III and V, calcium feeding ($Ca/S \simeq 3$) using Ca–Si wires (Ca:Si = 60:30) modified the morphology of the inclusions from stringer MnS to CaS particles and dramatically improved the K_{IC} independent of testing orientations. Also this improved the ductile–brittle transition temperature of the same type of steel [50]. Tomita suggested [37] that this beneficial effect can be attributed to the presence of the spherical CaS inclusions, resulting in crack extensions by a fibrous mode in which the main crack proceeds by the formation of voids at CaS inclusion sites. These voids grow and eventually link up as a result of rupture of intervening ligaments by localized shear [51].

2.3. Decreased hot-rolling reduction treatment

As noted above, the role of the modified inclusions to improved K_{IC} has been found to suppress the lamellate fracture as much as possible and then to result in the fibrous mode fracture. Thus, other treatments should cause the same effects, provided they modify the morphology (shape, size and distribution) of the inclusions. The obvious alternative treatment is modification of inclusions through decreased hot-rolling reduction treatment (DT). This has a great advantage in that the method is more economical in commercial practice. Also, there is an added advantage that, unlike chemical means such as calcium modification, such mechanical treatments do not require highly developed techniques. From this view point, Tomita [52] has investigated the effect of DT on modification of the morphology of MnS inclusions and the K_{IC} of 4340 ultrahigh-strength steel. The microstructural results are summarized in Table VI: (1) decreasing the hot-rolling reduction from 98% to 80% modified the shape of MnS inclusions from stringer (average aspect ratio = 17.5) to ellipsoid (average aspect ratio = 3.8); (2) the volume fraction of MnS inclusions is similar; (3) prior austenite grain size and retained austenite contents obtained after 80% hot-rolling reduction are similar to those obtained after 98% hot-rolling reduction. The mechanical properties obtained for the same type of steel similarly processed are given in Table VII. The results are summarized as follows. (1) In the longitudinal testing orientation, the K_{IC} improved (by $\sim$ 20 MPa m$^{1/2}$) as the hot-rolling reduction was decreased, whereas only small differences in tensile properties and Charpy impact energy were obtained. This could be a result of the ellipsoid MnS inclusions separating from the matrix during plastic deformation thereby producing large voids which, during testing, act to blunt and arrest cracks propagating across the specimen that would otherwise cause failure. (2) In the transverse testing orientation, the K_{IC} value increased by ~ 17 MPa m$^{1/2}$ as the hot-rolling reduction was decreased. The ductility and Charpy impact energy also increased, but there was little appreciable change of strength. This can be attributed to suppression of lamellar fracture, which occurs in a brittle manner along the interfaces of the MnS inclusion/matrix at the crack tip, as a result of the change of the shape of the MnS inclusions from stringer to ellipsoid. A series of experiments [53–55] revealed that the improvement in K_{IC} can be achieved by means of this approach independent of strength levels of heat-treated low-alloy steels.

A new approach [56] has been suggested by Tomita which consists of combing decreased hot-rolling reduction and short-term isothermal transformation (DT–IT). The mechanical properties of 0.4C–Cr–Mo–Ni steel (4340 steel grade) obtained using this approach are shown in Fig. 1. In this figure, the results of those steels obtained by high-temperature austenitizing treatment (HAT) and controlled rolling technique (CRT), are given for comparison. The DT–IT steel can be seen to have an improved combination of mechanical properties. This can be attributed to the cumulative effect of modification of the morphology of sulphide inclusions through decreased hot-rolling reduction and improved microstructure obtained via short-term isothermal transformation.

3. Improved fracture toughness of highly alloyed secondary hardening steel through control of non-metallic inclusions

If strength were the only criterion, and cost and availability were of importance, low-alloy steels would be used in preference to other ultrahigh-strength steels. However, the use in severe environments of low-alloy steels has often been limited by their low fracture toughness. For the use of these levels, the highly alloyed secondary hardening steels might be preferred. The highly alloyed secondary hardening steels are Fe–10Ni alloyed steels containing low or medium carbon and variable amounts of cobalt, chromium and molybdenum (see Type II, Table I). The HP-9-4-20 and HP-9-4-30 steels have been developed by the American Republic Steel Company since 1962 and 1965, respectively. At present, the HY-180 and AF1410 steels, mechanical properties of which are modified by reducing the carbon content and increasing the cobalt and chromium alloying elements, have been extensively employed as aircraft and aerospace vehicles. These steels are usually austenitized at 1080–1100 K followed by oil quenching and subsequent ageing at 783–838 K. Excellent combination of strength and toughness of these steels has been achieved by the fact that complex alloying carbide, $(Cr, Mo)_2C$, is dispersed in highly nickel-alloyed martensitic matrix. The cobalt alloying element acts as a driving force to disperse homogeneously the complex alloy-carbides in the martensitic matrix and is capable of retarding the dislocation rearrangements produced

TABLE VI Inclusion data of 4340 steel processed by hot-rolling reduction, HR (Al_2O_3 inclusions also present, but no data evaluated) [52]

Steel	Inclusion	f_v (%)	l (μm)	w (μm)	MAR
80% HR	MnS	0.113	23.8	6.2	3.8
98% HR	MnS	0.112	38.4	2.2	17.5

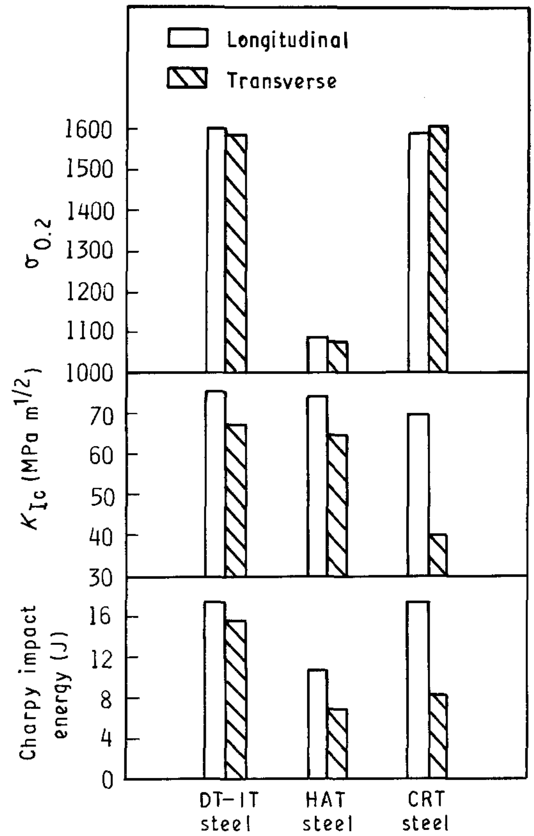

Figure 1 Effect of combined decreased hot rolling reduction and short-term isothermal transformation treatments (DT–IT), high-temperature austenitizing treatment (HAT) and controlled rolling technique (CRT) on 0.4C–Cr–Mo–Ni steel, tempered at 473 K [56].

TABLE VII Effect of hot-rolling reduction on mechanical properties of 4340 steel [52]

Steel	σ_{YS} (MPa)	σ_{UTS} (MPa)	ε_f	K_{IC} (MPa m$^{1/2}$)	E_{ch} (J)
80% HR-L	1475.6	1876.0	0.48	64.7	26.3
80% HR-T	1530.4	1874.5	0.14	54.7	15.8
98% HR-L	1486.9	1900.3	0.52	44.3	26.7
98% HR-T	1521.0	1840.0	0.38	38.4	6.3

TABLE VIII Chemical composition and gas analysis of AF1410 steel [59]

Steel	Chemical composition and gas analysis (wt %)										
	C	Ni	Co	Cr	Mo	Mn	S	Al	La	O[a]	N[a]
Heat 1	0.16	10.1	14.0	2.1	1.0	0.03	0.001	0.003	0.008	9	3
Heat 2	0.16	9.97	14.0	2.0	1.0	0.003	0.001	0.003	0.002	10	9

[a] p.p.m.

TABLE IX Inclusion data of AF1410 steel [59]

Steel	Inclusion	f_v (μm)	R_0 (μm)	X_0[a] (μm)
Heat 1	La_2O_2S	0.042	0.64	7.6
Heat 2	CrS	0.034	0.18	2.3

[a] Mean inclusion spacing, $X_0 = 0.89 R_0 f_v^{-1/3}$.

during the recovery process of martensitic substructure. However, the cobalt element contributes somewhat to solid-solution strengthening. Most recently, manufacturers and designers have sought highly alloyed secondary hardening steels with excellent resistant to stress-corrosion cracking at a tensile strength equivalent to that of 300M steel. Therefore, at present, research is being directed towards dramatically improving the mechanical properties of the AF1410 steel by increasing carbon and chromium contents.

3.1. Lanthanum treatment

Garrison and Moody [57] have investigated the effect of the ageing temperature and the inclusion morphology on K_{IC} of AF1410 steel. They have suggested that K_{IC} depends on the microstructural parameter, $[X_0(R_V/R_I)]$ [58], where X_0 is the inclusion spacing and (R_V/R_I) is the parameter relating to void growth, and the larger is the value of $[X_0(R_V/R_I)]$, the higher is K_{IC}. Based on the above ideas, Handerhan *et al.* [59] investigated the effect of lanthanum treatment on K_{IC} of AF1410 steel, consequently succeeding in significantly developing K_{IC} of AF1410 steel. The results are summarized in Tables VIII–X. They demonstrated that addition of lanthanum to vacuum-melted AF1410 (Heat 2, Table VIII) produces stabilized complex-inclusion, La_2O_2S at 0.042 vol % levels, consequently resulting in larger inclusion spacing (7.6 μm), whereas no addition of lanthanum to the same melt (Heat 1, Table IX) produces CrS inclusion at similar volume fraction levels, consequently resulting in smaller inclusion spacing (2.3 μm). Mechanical tests revealed that Heat 1 compared with Heat 2 increased K_{IC} by 32.3 MPa $m^{1/2}$ at similar Charpy impact energy levels at 693 K ageing, and improved K_{IC} by 68.4 MPa $m^{1/2}$ increased the Charpy impact energy level at 783 K ageing (Table X). They concluded that the improved K_{IC} can be attributed to the fact that the mean spacing of La_2O_2S inclusions appearing in Heat 1 is larger than that of CrS inclusions present in Heat 2. They also suggested that the large difference in K_{IC} after ageing at 693 and 783 K is attributed to microstructural changes; an important microstructural difference between the two ageing temperatures is the type and size of intra-lath carbides, i.e. coarser cementite obtained at 693 K and small needle M_2C carbides at 783 K. As noted above, modifying the inclusion spacing by the lanthanum treatment has been found to lead to improved K_{IC}. However, some difficulty may be encountered in commercial steel-making practice [60]: (1) the rare-earth metals (REMs) such as lanthanum are apt to react with activated oxygen [O] dissolved in the molten steel, refractory materials and the atmosphere; (2) there is the disadvantage that REM oxide and oxysulphide accumulate at the bottom of the ingot and deteriorate ductility and toughness of the steel; (3) REM injection requires highly developed techniques at the commercial heat level. Therefore, further fundamental studies are required using more materials, before the treatment is applied to improved K_{IC} of the highly alloyed secondary hardening steels in commercial practice.

3.2. Titanium treatment

Speich *et al.* [61] have investigated the effect of inclusions and microstructure on K_{IC} in Fe–10Ni–8Co–2Cr–1Mo alloy steel. They suggested that K_{IC} will be improved if crack initiation at inclusion sites is significantly suppressed. Based on this idea, Maloney and Garrison [62] have suggested a titanium treatment which precipitates inclusions having a strong coherency for the matrix, whereby K_{IC} of HY180 is dramatically improved at 1200–1250 MPa strength levels. The results are summarized in Tables XI–XIII. As seen from Tables XI and XII, addition of titanium to the HY 180 steel (Heat 2, Table XI) produces the complex inclusion, Ti_2CS, at the 0.019 vol % level, while no addition of titanium (Heat 1, Table XI) mainly precipitates MnS inclusions at similar volume fraction levels. Mechanical tests revealed that Heat 2 compared with Heat 1 dramatically improved K_{IC} at increased Charpy impact energy levels (Table XIII). Unfortunately, the significant improvement in K_{IC} cannot be explained by the inclusion spacing, because inclusion spacing of Heat 2 compared with that of Heat 1 is small, as can be seen in Table XII. So, they investigated the relationship between microvoid initiation parameter (MIP)* at inclusion sites (ratio of microvoid to inclusion volume fraction) and true plastic strain at fracture, ε_f, in both Heats 1 and 2. It was

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* If microvoid initiation does not occur at inclusion site, the MIP equals to 1.

TABLE X Effect of ageing temperature on mechanical properties of AF1410 steel [59]

Ageing temperature (K)	Steel	σ_{YS} (MPa)	σ_{UTS} (MPa)	ε_f	K_{IC} (MPa m$^{1/2}$)	E_{ch} (J)
693	Heat 1	1354	1749	0.96	118.5[a]	30
	Heat 2	1398	1757	0.93	86.2	28
783	Heat 1	1503	1659	1.16	196.6[b]	88
	Heat 2	1527	1699	1.16	128.2[b]	61

[a] K_Q value.
[b] $K_{IC} = [J_{IC}E/(1 - \nu^2)]^{1/2}$.

TABLE XI Chemical composition and gas analysis of HY180 steel [62]

Steel	Chemical composition and gas analysis (wt %)										
	C	Ni	Co	Cr	Mo	Mn	S	Ti	Al	O[a]	N[a]
Heat 1	0.10	9.86	7.96	1.98	1.02	0.31	0.002	0.004	0.002	6	3
Heat 2	0.11	9.88	8.07	1.99	1.00	0.01	0.001	0.021	0.003	4	1

[a] p.p.m.

TABLE XII Inclusion data of HY180 steel [62]

Steel	Inclusion	f_v (%)	R_0 (μm)	X_0 (μm)
Heat 1	MnS	0.021	0.16	2.39
Heat 2	Ti_2CS	0.019	0.10	1.60

TABLE XIII Mechanical properties of HY180 steel [62]

Steel	σ_{YS} (MPa)	σ_{UTS} (MPa)	ε_f	K_{IC}[a] (MPa m$^{1/2}$)	E_{ch} (J)
Heat 1	1208	1343	1.40	254	174
Heat 2	1241	1369	1.58	474	267

[a] $K_{IC} = [J_{IC}E/(1 - \nu^2)]^{1/2}$.

found that for Heat 2 the value of the MIP remains as 1 until ε_f attains 1.30, whereas for Heat 1 the MIP exceeds 2 when ε_f reaches 0.75. This suggests that for Heat 2, microvoid initiation does not occur at inclusion sites until the value of ε_f attains 1.30, whereas for Heat 1, microvoids are already initiated in the inclusion sites at 0.75 of ε_f. From these results, they concluded that the dramatic improvement of K_{IC} for Heat 2 can be attributed to the fact that Ti_2CS inclusions, compared with MnS inclusions, have a strong coherency with the matrix.

4. Conclusions

An attempt has been made to present recent topics on control of non-metallic inclusions to improve K_{IC} of the large-sized ultrahigh-strength steels. The studies mentioned here have suggested that inclusion control is most important for developing fracture toughness of large-sized ultrahigh-strength steels. It is believed that this review will contribute to provide new suggestions for further development of new materials in the future.

Most recently, there has been increased demand for improving not only K_{IC} but also resistance to stress corrosion cracking of the large-sized ultrahigh-strength steels in severe environments. One potential approach is to stabilize impurities segregated along grain boundaries, e.g. phosphorus and sulphur, to fine complex inclusions. Studies have been made of stabilizing impurities by means of rapidly solidified powder metallurgy (e.g. [63]). At present, however, there are insufficient data to develop such a steel in commercial practice, but it is hoped that a solution to the problem will be found from on-going fundamental studies.

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