Effect of TiN Inclusion on Fracture Toughness in Ultrahigh Strength Steel

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The investigation was conducted on 6 specimens of an identical composition except for N and Ti contents, the specimens were remarkably free of nonmetallic inclusions other than TiN inclusion.

The quasi-dynamic method has been used to investigate the condition of nucleation, growth and propagation of microcrack from TiN inclusion.

In summarizing the present study the following conclusion can be made:

(1) Fracture toughness K_{IC} is decreased rapidly with increasing volume fraction of inclusion (f_v) for $f_v < 0.1$ % and becomes insensitive when $f_v > 0.1$ % where the percentage of cracked-TiN reaches a limiting value.

(2) The percentage of cracked-TiN is determined by the true fracture strain ϵ_f and its propagation depends mainly on the critical true fracture strain ϵ_f^* . The ϵ_f^* can be computed by using both McClintock's model and Wall-Hill method as follows:

$$\epsilon_f^* = 0.0345 \ln (\bar{d}_t/\bar{a})$$

Thus the ϵ_f^* can be correlated with the inclusion spacing ($\bar{d}_t$) and size ($\bar{a}$).

(3) Based on the testing results, we get a similar relation to the ductile fracture model proposed by Rice, that is:

$$K_{IC} = (E \cdot \sigma_y \cdot \bar{d}_t)^{1/2}$$

KEY WORDS: fracture toughness; inclusion; ultrahigh strength steel; titanium nitride; quasi-dynamic method.

1. Introduction

The toughness of ultrahigh strength steels is an important property for their use as structural materials. Some catastrophic accidents have been caused by fracture under lower stress. It is confirmed from fracture mechanics that the crack size in the structural material is larger than the critical size. Cracks can cause a high concentration of stress in the localized region at their tips and the likelihood of complete fracture of the material depends on their propagation. Brooksbank and Andrews¹⁾ have shown that stress concentrations around inclusions in steel may be formed due to a difference in thermal compression between the inclusion phases and the steel matrix. Birkle *et al.*²⁻⁹⁾ have given the mechanism of microcrack nucleation. McClintock¹⁰⁾ and Rice¹¹⁾ have also proposed the models for microcrack growth. Thomson and Hancock¹²⁾ have been summarized and made the scientific researches on the ductile failure

by void nucleation, growth and coalescence. Since inclusions play an important role in ductile failure, it is important to determine the metallurgical and physical parameters which control microcracks nucleation. The main purpose of this paper is to investigate both the condition of microcrack nucleation, growth and propagation from inclusions and the relationship between inclusions and fracture toughness in an ultrahigh strength steels.

2. Experiments

The investigation was conducted on 6 steels with similar compositions except for Ti and N contents. The specimens were remarkably free of nonmetallic inclusions other than TiN inclusion. Their compositions are shown in Table 1.

The tensile tests was carried out on standard round tensile specimens using a ZDM-30T machine loaded by hydrostatic press. The evaluation of the fracture

Table 1. Composition of specimens. (wt%)

No.	Composition										
	C	Si	Mn	Ni	Mo	Cr	S	P	(O)	Ti	N
1	0.43	0.24	0.57	0.69	0.97	0.97	0.016	0.005	0.0009	0.07	0.0043
2	0.45	0.23	0.65	0.70	0.99	1.00	0.015	0.005	0.0012	0.11	0.0091
3	0.40	0.23	0.73	0.65	0.92	0.98	0.016	0.005	0.0009	0.19	0.0120
4	0.42	0.26	0.45	0.82	1.07	1.06	0.013	0.005	0.0027	0.17	0.0094
5	0.45	0.27	0.76	0.82	1.04	1.08	0.013	0.005	0.0026	0.19	0.0170
6	0.48	0.26	0.48	0.86	1.06	1.06	0.012	0.005	0.0016	0.26	0.0377

toughness K_{IC} was made on single-edge notched (SEN) three point bending specimens of $15 \times 30 \times 140$ mm in dimensions cut with the long axis along the rolling direction.

The heat treatment of specimens consisted of normalizing at 1173 K in salt bath, re-austenitizing for 30 min at 1153 K followed by quenching in oil, and tempering for 2 h at 783 K followed by cooling in air. The tempering treatment was intended to provide a similar microstructure.

The quasi-dynamic process of nucleation and propagation of microcrack from inclusions was observed in plane tensile specimens. The dimension of plane tensile specimen is shown in Fig. 1.

At first, the central part of a plane-specimen was polished; the strain was measured and the number of cracked inclusions was counted by a metallographic microscope.

3. Results

The specimens had the similar microstructure of Sorbite (Fig. 2) and grain size was Grade No. 11 based on ASTM Standard (Fig. 3).

Content, number, size and spacing of inclusions were determined by AIA Quantimet-720. The frequency distribution of nitride inclusions size and area percent are shown in Figs. 4 and 5, respectively.

Fig. 1. Dimension of plane specimen.

Fig. 2. Microstructure showing Sorbite. ($\times 500$) ($\times 3/4$)

Fig. 3. Microstructure showing grain size. ($\times 400$) ($\times 3/4$)

Fig. 4. Size distribution of TiN inclusion. (Numbers in the figure show specimen number.)

Fig. 5. Size and area percent of TiN inclusions. (Numbers in the figure show specimen number.)

Table 2. Quantitative data on TiN inclusion.

Specimen No.	1	2	3	4	5	6
Vol. fraction, f_v (%)	0.062	0.049	0.071	0.089	0.103	0.149
Spacing, d_{ts} (μm)	87.42	74.79	64.7	55.77	53.16	50.86
Average TiN size (μm)	1.77	1.78	2.09	1.99	2.00	2.25

Table 3. Tensile properties and fracture toughness.*

No.	σ_y (MPa)	σ_b (MPa)	σ_f (MPa)	δ_5 (%)	Ψ (%)	ϵ_f (%)	K_{IC} (MPa $\sqrt{m}$)
1	1448	1488	2292	12.7	53.1	75.8	90.9
2	1431	1507	2332	11.9	53.0	75.6	87.3
3	1395	1428	2343	13.3	50.2	69.9	84.9
4	1461	1521	2300	11.8	51.6	72.6	82.7
5	1439	1529	2130	10.3	48.5	66.6	81.1
6	1444	1541	2253	11.3	47.0	63.5	80.4

* Each data is the average value of 3 specimens.

$$\bar{d}_t = \left(\frac{A}{N} \right)^{1/2}$$

where, A : the total measured area

N : the total number of inclusion.

The K_{IC} values which validity was judged by the thickness of specimens $B \geq 2.5(K_{IC}/\sigma_{ys})^2$ and smooth tensile properties are given in Table 3.

The observation of the quasi-dynamic process of nucleation and propagation of a microcrack in a TiN-inclusion is shown in Fig. 6 for a plane tensile specimen.

The type and the chemical composition of the inclusions were analyzed by X-ray diffraction and software based X-ray microanalysis system, respectively.

Fig. 6. Microcrack nucleation in a TiN inclusion as a function of strain and stress during tensile testing.

The results obtained showed that the structure of inclusion was the same as that of TiN.

The elastic modulus and Poisson's ratio were determined by the use of 3 specimens. The values are given in Table 4.

4. Discussion

4.1. Effect of the Volume Fraction of TiN-inclusions on Fracture Toughness

The area fraction of inclusions which is equivalent

to the volume fraction f_v is listed in Table 2. The relation between K_{IC} and f_v shows in Fig. 7. There was a linear relationship between f_v and K_{IC} when $f_v < 0.1\%$. However, K_{IC} decreases slowly with increasing f_v in the range over 0.1% . This behavior was related to the percentage of the number of cracked TiN. It increased with increasing strain before necking point, but the percentage of microcracked-TiN was kept as nearly constant level after necking point (Figs. 8 and 9), i.e., the number of microcracked TiN had a limiting value. Alternatively, the number of microcracked TiN inclusion was proportional to the volume fraction (f_v) of TiN inclusion, and was controlled by strain. Therefore, K_{IC} values were found to decrease rapidly as f_v increased up to 0.1% and become insensitive when $f_v > 0.1\%$ where the percentage of microcracked TiN reached a limiting value.

4.2. Inclusion Size vs. Microcracked Nucleation

From the observation of plane tensile specimens the true fracture strains for different size TiN-inclusions were different. The larger size inclusion cracked initially at a strain less than 1% , but TiN below $0.5\ \mu\text{m}$ in size did not crack until necking occurred. Inclusion size (a) was related to true fracture strain as given in Table 5.

The calculated strain, $\varepsilon_c\%$ in Table 5, is based on Tanaka's model (Energy Criterion)⁹⁾ relating to the void formation condition on spherical inclusion interface. In comparison with the two strain values (ε_f and ε_c) in Table 5, it is revealed that the both values are in good agreement. For example, the $\varepsilon_f\%$ and $\varepsilon_c\%$ were 0.657% and 0.68% , respectively when TiN size was $13.2\ \mu\text{m}$. Therefore, the true fracture strain (ε_f) measured by strain gauge is in correspondence

Fig. 7. Effect of TiN volume fraction on fracture toughness.

Table 4. Young's modulus E , shear modulus G , and Poisson's ratio ν of specimens.*

No.	4	5	6	Average
E (GPa)	213	212	211	212
G (GPa)	80.9	80.4	81.3	80.8
ν	0.32	0.32	0.30	0.31

* Each datum is the average value of two specimens.

Fig. 8. Relation between percentage of cracked TiN and strain. (before necking point)

Fig. 9. Relation between percentage of cracked TiN and strain. (after necking point)

Table 5. TiN size and true fracture strain (ε_f)

TiN size, a (μm)	13.2	5.8	1.6	1.0
Fracture strain,* ε_f (%)	0.68	1.2	2.4	4.32
Calculated strain,** ε_c (%)	0.657	1.0	1.89	2.39

* The values are measured by strain gauge.

** The material constants was used for calculating ε_c :

$E(\text{TiN})$ 317 GPa, $E(\text{Matrix})$ 212 GPa,
 $\nu(\text{TiN})$ 0.19, $\nu(\text{Matrix})$ 0.31.

with critical strain of microcrack nucleation (ϵ_c), so that the TiN-size (a) is in correspondence with critical size a_c , and a_c varies inversely with ϵ_c . It is possible to extent Tanaka's model from spherical to non-spherical inclusion interface.

4.3. Inclusion Spacing and Size vs. Critical True Fracture Strain

The microcrack nucleation in TiN inclusion is controlled by critical strain ϵ_c . The crack propagation occurs through the microcrack coalescence, and growth. When all the TiN inclusions in an aggregative area are torn off, the crack will be propagated. However, the tear of aggregative area may be controlled by true fracture strain ϵ_f^* . Since ϵ_f^* is a micro-parameter, it can not be determined from the mechanical testing on a macroscale. The ϵ_f^* was assumed to $\epsilon_f/3$ by Hahn-Rosenfield,¹³⁾ $2\epsilon_f/3$ by Osborne-Embury¹⁴⁾ and ϵ_f by the others, where ϵ_f represents the true fracture strain under the uni-axial tension.

The critical true fracture ϵ_f^* can be computed using McClintock's void coalescence model for elliptical cavities, i.e.,

$$\epsilon_f^* = \frac{(1-n) \ln l_0/2r}{\sin h \left[(1-n) \times (\sigma_{11} + \sigma_{22}) / \frac{2\bar{\sigma}}{\sqrt{3}} \right]} \quad \text{.....(1)}$$

where, l_0 : the initial void spacing

r_0 : the initial micro semi-axis of the pre-existing voids

$\bar{\sigma}$: the effective stress

σ_{11}, σ_{22} : the stress parallel to the major and minor axes of the elliptical void, respectively.

We take the TiN cracked as a void and let the length of TiN as a void diameter, i.e., $\bar{a} = 2r_0$, and the inclusion spacing d_t as the initial void spacing. Since strain hardening exponent n is very small in ultrahigh strength steel, the solution of stress distribution in front of a precrack tip in the non-hardening material can be obtained by using the Wall-Hill method; thus

$$\begin{aligned} \sigma_{11} &= 2\tau_0 \ln \left(1 + \frac{2x}{2V_c} \right) \\ \sigma_{22} &= 2\tau_0 \left[1 + \ln \left(1 + \frac{2x}{2V_c} \right) \right] \quad \text{.....(2)} \\ \sigma_{33} &= 2\tau_0 \left[\frac{1}{2} + \ln \left(1 + \frac{2x}{2V_c} \right) \right] \end{aligned}$$

where, τ_0 : the shear yield stress

$2V_c$: the critical crack opening displacement

x : the distance from the crack tip.

It is expected to be maximum triaxiality at a distance of x is equal to $1.96 (2V_c)$ from crack tip. We will obtain the following results from Eq. (2);

$$\sigma_{11} = 2\tau_0 \ln 4.92$$

$$\sigma_{22} = 2\tau_0 (1 + \ln 4.92) \quad \text{.....(3)}$$

$$\sigma_{33} = 2\tau_0 \left(\frac{1}{2} + \ln 4.92 \right)$$

$$\begin{aligned} \text{so } \bar{\sigma} &= \left\{ \frac{1}{2} [(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2] \right\}^{1/2} \\ &= \sqrt{3} \tau_0 \end{aligned}$$

$$\frac{\sigma_{11} + \sigma_{22}}{\bar{\sigma}} = \frac{2\tau_0 [\ln 4.92 + (1 + \ln 4.92)]}{\sqrt{3} \tau_0} = \frac{8.373}{\sqrt{3}}$$

Substituting the above results into Eq. (1), we obtained:

$$\epsilon_f^* = 0.0345 \ln \frac{\bar{d}_t}{\bar{a}} \quad \text{.....(4)}$$

Furthermore, when the results shown in Tables 2 and 3 are substituted into Eq. (4), a series of ϵ_f^* is obtained as shown in Table 6.

It shows that ϵ_f^* values are smaller than about 6 times as small as ϵ_f . Why the ϵ_f^* became so smaller than ϵ_f ? We obtained some data given in Table 7 using the Figs. 10 to 12 taken from the aggregative area of TiN inclusions in specimen No. 6. This method which was used to get the data was that:

The distance between two TiN will be changed from l_0 to l_1 when the specimen was loaded, we get $\epsilon_{p1} = (l_1 - l_0)/l_0$ and $\epsilon_{p2} = (l_2 - l_0)$ so $\Delta\epsilon_p = \epsilon_{p2} - \epsilon_{p1}$. There are a lot of TiN in the aggregative area, so we can get a series of $\Delta\epsilon_p$. These data in Table 7 are an evidence that the ϵ_f^* decreases by the inclusions in the aggregative area. Therefore, the specimen containing inclusions breaks easily from aggregative area

Table 6. Calculated and experimental values of fracture strain.

Specimen No.	1	2	3	4	5	6
Spacing, d_t (μm)	87.42	74.97	64.7	55.77	53.16	50.86
TiN size, $\bar{a}$ (μm)	1.7	1.7	2.1	1.9	2.0	2.2
Fracture strain, ϵ_f	0.76	0.75	0.70	0.73	0.67	0.63
Calculated strain, ϵ_f^*	0.136	0.131	0.118	0.116	0.113	0.108

Table 7. Ratio of localized strain increment, $\Delta\epsilon_p$, and macro strain increment, $\Delta\epsilon$, in the aggregative area. (%)

Before necking			After necking					
$\Delta\epsilon_p$	$\Delta\epsilon$	$\Delta\epsilon_p/\Delta\epsilon$	$\Delta\epsilon_p$	$\Delta\epsilon$	$\Delta\epsilon_p/\Delta\epsilon$	$\Delta\epsilon_p$	$\Delta\epsilon$	$\Delta\epsilon_p/\Delta\epsilon$
3.09	2.8	1.10	7.94	2.8	2.83	10.29	2.96	3.48
3.17	2.8	1.13	8.24	2.8	2.40	12.56	2.96	4.24
4.13	2.8	1.47	8.95	2.8	3.20	13.59	2.96	4.59
5.26	2.8	1.88	9.37	2.8	3.35	15.00	2.96	5.07
5.26	2.8	1.88	11.24	2.8	4.00	15.20	2.96	5.14

$\epsilon=2.4\%$, $\sigma=157\text{ kg/mm}^2$

Fig. 10. TiN inclusions and microcrack growth.
($\times 500$) ($\times 7/10$)

$\epsilon=8.16\%$, $\sigma=184.6\text{ kg/mm}^2$

Fig. 12. TiN inclusion and microcrack's propagation.
($\times 500$) ($\times 7/10$)

$\epsilon=5.2\%$, $\sigma=178\text{ kg/mm}^2$

Fig. 11. TiN inclusion and microcrack's propagation.
($\times 500$) ($\times 7/10$)

of inclusions.

4.4. Relations of Crack Propagation to Inclusions Spacing and Fracture Toughness

The crack propagation may be controlled by ϵ_f^* deduced from Eq. (4). It has been known that the ϵ_f^* is proportional to $\bar{d}_T$. Therefore, the crack propagation may be also controlled by $\bar{d}_T$. Through the observation of plane tensile specimen (No. 6), we found that the microcracks in TiN inclusion were linked in the aggregative area of TiN inclusion (Fig. 12) resulting in lower fracture toughness for specimen No. 6.

The effect of the inclusion spacing ($\bar{d}_T$), which is determined by metallographs is shown in Fig. 13. There is a good linear relationship between K_{IC} and $\bar{d}_T^{1/2}$. Its linear dependence coefficients is:

$$\gamma_m = 0.9945$$

We get a linear equation by regression analysis as follows:

$$K_{IC} = K(dt)^{1/2} \dots\dots\dots(5)$$

Another ductile fracture model had been proposed by Rice,⁽¹¹⁾ i.e.:

$$K_{IC} = (2E\sigma_y d_T)^{1/2} \dots\dots\dots(6)$$

Fig. 13. Relation between K_{IC} and $d_T^{1/2}$.

Table 8. A comparison between K_{IC} values. (MPam^{1/2})

K_{IC}	Specimen No.					
	1	2	3	4	5	6
By testing	90.9	87.3	84.9	82.7	81.1	80.4
Calculated by $K_{IC} = (2E\sigma_y \bar{d}_T)^{1/2}$	142.1	132.6	117.0	116.5	113.3	108.5
Calculated by $K_{IC} = (E\sigma_y d_T)^{1/2}$	100.6	93.7	82.7	82.5	80.1	76.7

where, σ_y , E : constants for known material.

When comparing the two K_{IC} values calculated from Eqs. (5) and (6) with the measured values, we get useful results given in Table 8.

When the constant K in Eq. (5) is equal to $(E\sigma_y)^{1/2}$, the calculated K_{IC} values is closer to the measured K_{IC} values than K_{IC} values calculated from $k=(2E\sigma_y)^{1/2}$ (the Rice Model). Therefore, we propose that the relation of K_{IC} to $\bar{d}_T$ is expressed as follows:

$$K_{IC} = (E\sigma_y d_T)^{1/2} \dots\dots\dots(7)$$

In comparison with the effects of size and number of inclusion of the fracture toughness, in the range of inclusion size of 0~0.55 μm , the inclusion number of specimen No. 2 is larger than that of specimen No. 6, but K_{IC} value of No. 2 is higher than that of No. 6 (Table 3). On the other hand, in the range of inclusion size above 5.7 μm the number of inclusions in No. 6 is larger than that in No. 2. Therefore, it was

confirmed that the effect of inclusion size is more harmful than number of inclusions on fracture toughness.

5. Conclusion

The fracture toughness appears to be markedly effected by TiN inclusion content and spacing. The most effective measures for improve the toughness of steel are to reduce the nitrogen contents and aggregation of inclusion, or to refine the inclusion particles.

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