On the Toughness of Ni-Cr-Mo Steels in As Quenched Conditions*

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Synopsis

The fracture toughness of Ni-Cr-Mo steels was estimated for as quenched conditions in relation to the prior austenite grain size. The fracture toughness was estimated from the experimentally determined critical J -value, using small sized three-point bending specimens.

The results obtained are summarized as follows:

(1) The critical stress intensity factor calculated from the critical J -value is in satisfactory agreement with the measured plane strain fracture toughness.

(2) While the tensile properties deteriorate with an increase in the prior austenite grain size, the fracture toughness remains nearly constant, over a wide range of the prior austenite grain size, showing a somewhat decreasing tendency. In the range of the larger austenite grain size, the fracture toughness decreases abruptly, corresponding to the change of the fracture surface appearance from transgranular to intergranular dimple fracture.

(3) The fracture toughness of the steels is found to have a good correlation with the size of dimples that were originated at small second phase particles. This fact may exclude the effects of the prior austenite grain size larger than the distance between second phase particles responsible to dimples on the fracture toughness.

I. Introduction

There is an increasing demand for high strength low alloyed steels with superior fracture toughness. The development of reliable testing method to obtain an accurate value for plane strain fracture toughness has stimulated the investigations to design such alloyed steels, correlating the fracture toughness to the metal-lurgical variables of the steels.

The fracture toughness testing procedure¹⁾ based on the path independent J -integral²⁾ has been proposed recently and can be considered to be useful to evaluate the fracture toughness of relatively tough materials for the above mentioned purpose.

This investigation was conducted to define the fracture toughness of as-quenched Ni-Cr-Mo steels, using the critical J -value, as a function of the prior austenite grain size, and then to understand the reasons of the relationships between these two variables. Also, the availability of the critical J -value for the fracture toughness evaluation in relation to the crack opening

displacement and the critical stress intensity factor.

II. Materials and Experimental Procedures

1. Materials

The chemical composition and the transformation temperature M_s of the steels used are given in Table 1. The steels were melted in a vacuum induction furnace and forged and rolled at 1 200°C, after solution-treated at 1 200°C for 4 hr, into bars of 20 mm in diameter, and some portions of these steels into plates of 25 mm in thickness. The tensile, bending and compact tension specimens as shown in Fig. 1 were machined from the bars and the plates normalized at 890°C for 25 min. In order to obtain different austenite grain sizes, the specimens were austenitized at 850°, 950°, 1 050°, 1 150° or 1 250°C for 15 min and then quenched to 850°C. After being held at the temperature for 10 min they were oil-quenched.

2. Mechanical Tests

The tensile specimens were tested in an Instron machine of 10⁵ N capacity, attached with a strain gage extensometer and the load-elongation curves were recorded. The cross head speed was 1.0 mm/min.

A precrack was developed in all bending specimens at the base of the machined notch by a Vibrophore fatigue machine (10×10⁴ N capacity), using three-point bending load. The number of loading cycles necessary to achieve a desired fatigue crack length of about 2.0 to 4.5 mm was of the order of 5×10⁵. The static bending tests were conducted with the Instron machine at a constant cross head speed of 0.5 mm/min. The recorded curves with a multi-pen X-Y plotter during the test consisted of: a load-crack opening displacement ($P-V_o$) and a load-deflection ($P-D$) curve. The crack opening displacement V_o was measured with a standard clip gage mounted by a pair of knife edges at the notched surface of a specimen. The deflection was defined here as that of the specimen measured at the loading point in the loading direction.

Table 1. Chemical composition (wt%) and transformation temperature (°C, austenitized at 900°C) of the steels used

Sample No.	C	Mn	Si	Ni	Cr	Mo	V	Nb	S	O	N	A_f	A_s	M_s
1	0.28	0.56	0.29	1.91	0.78	0.24	—	—	0.006	0.0030	0.0034	775	720	362
2	0.24	0.49	0.24	1.86	0.79	0.24	0.061	—	0.006	0.0024	0.0026	787	720	376
3	0.28	0.57	0.25	1.93	0.78	0.24	—	0.062	0.006	0.0021	0.0030	796	727	377
4	0.27	0.52	0.25	1.85	0.78	0.24	0.072	0.060	0.005	0.0023	0.0025	786	718	363

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a) Tensile specimen
b) Three-point bending specimen, tested after fatigue precracked
c) Compact tension specimen, tested after fatigue precracked

Fig. 1. Testing specimen designs

The preliminary tests were carried out to correlate the initiation of the crack propagation to the behavior of the load-crack opening displacement curve by observing the fracture surface of the specimens fractured at -196°C using a Charpy test machine after interrupted at different stages of the loading, and it was confirmed that the initiation of the crack propagation corresponds to an remarkable change of the slope in the load-crack opening displacement curve. From this observation, the deflection Δ^{crit} was obtained at which the crack propagation initiated, as shown later in Fig. 5.

The compact tension specimens of 20 mm in thickness were precracked by tension-tension cyclic loading and tested in 5×10^5 N capacity tensile machine according to the standard testing method³⁾ for plane strain fracture toughness.

III. Experimental Results

1. Prior Austenite Grain Size

Figure 2 shows the relationship between the austenitizing temperature and the prior austenite grain size obtained. The relation for the steels containing a small amount of Nb differs from that for the steels free from Nb. A small amount of V, as used for the present experiment, shows no effects on the growth of the austenite grain.

2. Tensile Test

The relationships between the tensile properties and the prior austenite grain size are shown in Fig. 3 for all steels tested. The yield (0.2% proof) stress σ_y and

Fig. 2. Relationship between austenitizing temperature and the prior austenite grain size d_γ of the steels

Fig. 3. Effect of the prior austenite grain size d_γ on the tensile properties of the steels

UTS σ_B decrease gradually with an increase in the prior austenite grain size d_γ . The decrease in fracture strength σ_F is abrupt, corresponding to a decrease in the reduction of area ϕ with an increase in the prior austenite grain size d_γ . However, a large difference in the tensile properties among the steels used can not be recognized, comparing those at a given prior austenite grain size. Figure 4 shows the relationship between the yield stress σ_y and the prior austenite grain size d_γ for all steels tested on the basis of the Hall-Petch relation $\sigma_y = \sigma_0 + k_y d_\gamma^{-1/2}$. The value of k_y is found to be about $17 \text{ N/mm}^{3/2}$, which is smaller than those reported previously for the ferrite grain size as 20 to $25 \text{ N/mm}^{3/2}$. It can be assumed from this fact that the strengthening effect of the prior austenite grain boundaries on the martensitic structure is not so strong as that of the ferrite grain boundaries on the ferrite matrices.

3. Bending Test

A typical example of the load-clip gage displace-

ment ($P-V_g$) and the load-deflection ($P-\Delta$) curve recorded during the bending test is reproduced in Fig. 5. V_g^{crit} and $\Delta_{a/w=0.502}^{\text{crit}}$ denote respectively the clip gage displacement and deflection where the propagation of the crack is considered to initiate, as mentioned in the previous section. The apparent fracture toughness was calculated by the following equation,³⁾ using the value of the load P_Q that corresponds to the above-mentioned displacement V_g^{crit} or the deflection Δ^{crit} :

$$K_Q = \frac{3P_Q S}{2BW^{3/2}} [1.93(a/w)^{1/2} - 3.07(a/w)^{3/2} + 14.53(a/w)^{5/2} - 25.11(a/w)^{7/2} + 25.80(a/w)^{9/2}] \quad (1)$$

where, S , a , B and w denote span length, crack length, specimen thickness and specimen width respectively. Figure 6 shows the value of K_Q as a function of the prior austenite grain size d_T . The value of the apparent fracture toughness K_Q is nearly constant independently of the prior austenite grain size d_T . As the

Fig. 4. Relationship between the reciprocal square root of the prior austenite grain size $d_T^{-1/2}$ and yield (0.2% proof) stress σ_y , showing $k_y = 17 \text{ N/mm}^{3/2}$ in the Hall-Petch equation

Fig. 5. Load-displacement curves for a three-point bending test. V_g and Δ denote the crack opening displacement measured on the specimen surface with a standard clip gage and the deflection of the specimen in the loading direction at the loading point respectively.

values of the K_Q do not satisfy the conditions of

$$B, a, (w-a) \geq 2.5 \left(\frac{K_Q}{\sigma_y} \right)^2 \quad (2)$$

K_Q is not considered to be plane strain fracture toughness in the ASTM sense.

Figure 7 shows the relationship between critical COD Φ_c^t and the prior austenite grain size d_T . Φ_c^t is calculated by the simplified equation of the form,⁴⁾

$$\Phi_c^t = V_g^{\text{crit}} / [1.0 + (a+z)/r(w-a)] \quad (3)$$

where, z is the thickness of the knife edge, r rotational factor which is defined to be equal to 1/3 for this experiment. The value of r is, however, not constant over a wide range of V_g^{crit} , in particular the relation between V_g^{crit} and Φ_c^t is not linear as shown in Eq. (3) for relatively small values of V_g^{crit} .^{5,6)} This limits the validity of Φ_c^t calculated by Eq. (3), as in the case of the apparent fracture toughness K_Q , as a quantitative toughness value of the steel.

In order to estimate the fracture toughness accurately, attempts were made to obtain the critical J -value using small sized bending specimens as reported by J. A. Begley and J. D. Landes.¹⁾ Figure 8 shows an example of the relationship between crack length a and work done to the unit thickness of the bending speci-

Fig. 6. Relationship between the prior austenite grain size d_T and the apparent stress intensity factor K_Q at the beginning of the crack propagation

Fig. 7. Relationship between the prior austenite grain size d_T and critical crack opening displacement Φ_c^t , calculated on the assumption of a constant rotational center

Fig. 8. Work done to the unit thickness of the specimen U/B as a function of the crack length a for the specimens of the steel No. 1, austenitized at 850°C and oil quenched. The open and solid marks denote the displacement range of $\Delta < J^{crit}$ and $\Delta \geq J^{crit}$ respectively.

men U/B , which was planimetrically obtained from the load-deflection (P - Δ) curve recorded. As shown in Fig. 8, the relation between U/B and a for a given deflection Δ is approximated to be linear in the range of the measurements. From such relations the critical J -value can be obtained as

$$J^{crit} = - \frac{\partial(U/B)}{\partial a} \Big|_{\Delta/a/w=0.5}^{J^{crit}} \quad \dots\dots\dots(4)$$

and was plotted as a function of the prior austenite grain size d_T in Fig. 9. The general tendency of the relation in Fig. 9 is quite similar to that in Fig. 6 and Fig. 7. The fracture toughness of the steels seems to be improved at a given prior austenite grain size by adding a small amount of V and/or Nb.

IV. Discussion

1. Relations between Critical J -value and Apparent Fracture Toughness or Critical COD

J. A. Begley and J. D. Landes¹⁾ showed on the basis of their experimental results that the following equation correlates the linear elastic plane strain fracture toughness K_{Ic} measured by the ASTM standard testing method to the critical J -value termed by J_{Ic} that was obtained on small sized specimens under full scale yielding condition,

$$J_{Ic} = \frac{K_{Ic}^2}{E} (1 - \nu^2) \quad \dots\dots\dots(5)$$

where, E and ν denote Young's modulus and Poisson's ratio. Using Eq. (5) and the critical J -values obtained in the present experiment, the values of K_{Ic} were calculated to be 3 240 and 2 240 N/mm^{3/2} for the steel No. 1, 850°C OQ specimens and the steel No. 1, 1 250°C+850°C OQ specimens respectively.

Fig. 9. Relationship between the prior austenite grain size d_T and critical J -value

Fig. 10. Relationship between the prior austenite grain size d_T and plane strain fracture toughness K_{Ic} calculated from the critical J -value

The values of plane strain fracture toughness K_{Ic} obtained from compact tension tests according to the standard testing method were 3 140 and 2 500 N/mm^{3/2} respectively for the above mentioned specimens. The obtained values by these two different methods agree within the experimental errors. Figure 10 shows the relationship between the K_{Ic} -values calculated by Eq. (5) from the critical J -values and the prior austenite grain size d_T .

In comparison of Fig. 6 with Fig. 10, the following relationships can be obtained.

In the range of small values of the fracture toughness:

$$K_Q \approx K_{Ic} \quad \dots\dots\dots(6)$$

In the range of larger values of that:

$$K_Q < K_{Ic} \quad \dots\dots\dots(7)$$

These relations (6) and (7) result from the fact that the K type analysis does not include a contribution from crack tip plasticity to the fracture toughness parameter, that is, the increase in the plastic zone size at the crack tip requires more work than if the same load is reached by linear elastic loading. On the other hand, the critical J -values and thereby calculated K_{Ic} -values take account of this contribution.⁷⁾

The value of K_Q approaches to that of K_{Ic} , as the fracture toughness of the steels decreases, that means, with a decrease in the plastic zone size at the crack tip.

The value of the path-independent integral J can be determined for a path which corresponds to the surface of the cohesive zone of the Barenblatt crack model as follows:

$$J^{crit} = \int_0^{\phi_c} \sigma(\phi) d\phi \dots\dots\dots(8)$$

where, $\sigma(\phi)$ is the restraining stress in the cohesive zone. For the Dugdale crack model, where, $\sigma(\phi)$ is constant and given as the yielding stress σ_y , J^{crit} can be obtained by

$$J^{crit} = \sigma_y \phi_c \dots\dots\dots(9)$$

For a plane strain condition, it is pointed out that a constraint factor of the order of 2 to 3 is required to take account of the effect of triaxiality in the vicinity of the crack tip.^{6,8,9)}

Table 2 shows the factor M ;

$$M = J^{crit} / \sigma_y \phi_c' \dots\dots\dots(10)$$

obtained for the present experiment. The value of the factor M is nearly constant except those for the steels with especially low fracture toughness. The constant value can be considered to indicate that the relationship between ϕ_c' and V_g^{crit} is able to be approximated as linear in a relatively narrow range of the

magnitude of V_g^{crit} . $M < 1.0$ shows that the values of ϕ_c' may be overestimated in the range of the non-full scale yielding according to Eq. (3).

2. Relations between Fracture Toughness and Microstructures or Fracture Surface Appearances

As shown in Photo. 1, the microstructure of the steels becomes coarser as the prior austenite grain size increases. Recently it was pointed out by V. F. Zackey, *et al.*¹⁰⁾ that some commercial alloyed steels, such as 4330, 4340 and 300M, showed an improvement in the fracture toughness by a higher austenitizing temperature and rapid quenching, although the reason was not clearly known. However, the present experiment shows that the fracture toughness is nearly constant, showing a somewhat decreasing tendency, over a wide range of the prior austenite grain size and therefore of austenitizing temperature and it decreases abruptly in the range of the larger grain size. The retained austenite was not identified by X-ray diffraction and by the observation with an electron microscope, contrary to the results of V. F. Zackey, *et al.*

The microstructures resulting from austenitization of the steel No. 2 at 850°C followed by oil quenching and austenitization at 1 250°C followed by oil quenching after holding at 850°C are respectively shown in Photo. 2. The structures consist of lath-type martensite auto-tempered during quenching. For the auto-

Table 2. Relationship between the critical J -value and the critical opening displacement ϕ_c'

Treatment	Sample No.	Critical J -value (N mm/mm ²)	$\sigma_y \cdot \phi_c'$ (N mm/mm ²)	M^*
850°C×25 min Oil quench	1	50.0	58.4	0.86
	2	52.0	58.7	0.89
	3	62.4	73.6	0.85
	4	61.8	74.7	0.83
950°C×15 min + 850°C×10 min Oil quench	1	48.0	61.2	0.78
	2	52.8	59.1	0.78
	3	60.8	71.8	0.85
	4	62.0	72.4	0.86
1 050°C×15 min + 850°C×10 min Oil quench	1	48.2	58.6	0.82
	2	—	53.1	—
	3	56.8	70.6	0.80
	4	56.8	69.5	0.85
1 150°C×15 min + 850°C×10 min Oil quench	1	49.4	63.8	0.77
	2	53.6	56.4	0.95
	3	56.2	64.3	0.87
	4	57.8	72.1	0.80
1 250°C×15 min + 850°C×10 min Oil quench	1	23.8	43.5	0.55
	2	21.8	34.5	0.63
	3	42.8	53.9	0.79
	4	40.8	55.3	0.74

* M : the ratio of the critical J -value to $\sigma_y \phi_c'$

A) Steel No. 1, austenitized at 850°C and oil quenched
B) Steel No. 1, austenitized at 1 050°C and oil quenched after holding at 850°C
C) Steel No. 1, austenitized at 1 250°C and oil quenched after holding at 850°C
D) Steel No. 4, austenitized at 850°C and oil quenched
E) Steel No. 4, austenitized at 1 050°C and oil quenched after holding at 850°C
F) Steel No. 4, austenitized at 1 250°C and oil quenched after holding at 850°C

Photo. 1. Optical micrographs

tempered martensite, the M_s temperature may have some influences on the fracture toughness of the steels; a higher M_s temperature corresponds to a higher tempering temperature. The M_s temperature of the steels used shown in Table 1 are nearly constant and the change of that by the austenitizing temperature is not so large as it may affect the fracture toughness, as shown in Fig. 11.

The abrupt decrease in the fracture toughness in the range of the larger austenite grain size corresponds to the abrupt change of the fracture surface appearances, as shown typically in Photos. 3 and 4, although both are dimple fracture. Photograph 3 shows a

transgranular fracture surface and Photo. 4 an intergranular one.

The influence of the grain size on the fracture toughness should be different between for a dimple fracture and a cleavage one.¹¹⁾ As a dimple fracture goes through the process of void formation and its coalescence, a grain boundary can not necessarily be considered as a barrier to a propagating crack as in the case of cleavage fracture.

As shown in Photos. 3 and 4, the diameter of the dimples is approximately 10 to 5 μm for the transgranular fracture and less than 5 μm for the intergranular fracture. These are smaller than the prior austenite grain size. At the center of the dimples, a small sized second phase particle is observed. This particle is assumed to be an MnS type inclusion.

According to the model by J. R. Rice and R. J. Johnson,¹²⁾ the crack opening displacement is given dependently of the initial size of the void and the true strain on line ahead of a crack at the fracture,

$$\phi_c/X_0 = 1.0 \sim 2.7 \dots\dots\dots(11)$$

A) Steel No. 2, austenitized at 850°C and oil quenched
B) Steel No. 2, austenitized at 1250°C and oil quenched after holding at 850°C

Photo. 2. Electron micrographs

Fig. 11. Relationship between austenitizing temperature and M_s temperature. The specimens were austenitized for 15 min, cooled rapidly to 850°C, held for 10 min at that temperature and then quenched.

Photo. 3. Scanning electron fractograph of bending specimen. Steel No. 2, austenitized at 950°C and oil quenched after holding at 850°C, showing typical dimple fracture surface

Photo. 4. Scanning electron fractograph of bending specimen. Steel No. 2, austenitized at 1250°C and oil quenched after holding at 850°C, showing grain boundary fracture surface with dimple pattern

where, X_o denotes the distance from the crack tip to the nearest point responsible for void formation. K.-H. Schwalbe¹³⁾ has proposed the relationship between the crack opening displacement and stress intensity factor under plane strain conditions by translating the calculated results which was obtained for Mode III Antiplane strain into Mode I Opening, taking account of work-hardening of the type $\tau = \tau_y(\gamma/\gamma_y)^n$ where, τ and γ denote shear stress and shear strain, τ_y and γ_y those at yielding point and n work-hardening exponent. Using this relation, the following equation can be obtained;

$$\phi \approx \frac{30(1-\nu)(1-2\nu)^2}{\pi^2(1+n)\sigma_y^2} \left[\frac{\sigma_y(1+\nu)}{E(1+n)} \cdot \frac{(1+n)^2}{n^{n/(1+n)}} \right]^{(1+n)} \times K^2 \quad \text{.....(12)}$$

Substituting $\phi_c/X_o=1.85$, the mean value of Eq. (12), yields,

$$X_o \approx \frac{16.2(1-\nu)(1-2\nu)^2}{\pi^2(1+n)\sigma_y^2} \left[\frac{\sigma_y(1+\nu)}{E(1+n)} \cdot \frac{(1+n)^2}{n^{n/(1+n)}} \right]^{(1+n)} \times K_{Ic}^2 \quad \text{.....(13)}$$

This equation is similar to that used successfully by H. J. Rack and D. Kalish to explain their experimental results.¹⁴⁾

The calculated results are summarized in Table 3 for the present experiment. The values of X_o are nearly constant for the same fracture mode, i.e., $X_o=5.8$ to $7.9 \mu\text{m}$ ($\bar{X}_o=6.8 \mu\text{m}$) for the transgranular dim-

Table 3. Results of work-hardening exponent n , mean spacing of the points responsible for dimple fracture X_o and calculated critical opening displacement ϕ_c .

Treatment	Sample No.	n^*	X_o (μm)	ϕ_c (μm)
850°C×25 min Oil quench	1	0.179	6.1	11.3
	2	0.171	6.3	11.7
	3	0.169	7.4	13.7
	4	0.165	7.6	14.1
950°C×15 min + 850°C×10 min Oil quench	1	0.162	6.2	11.5
	2	0.179	6.4	11.8
	3	0.189	7.2	13.3
	4	0.170	7.9	14.6
1 050°C×15 min + 850°C×10 min Oil quench	1	0.191	5.8	10.7
	2	—	—	—
	3	0.158	7.5	13.9
	4	0.175	7.3	13.5
1 150°C×15 min + 850°C×10 min Oil quench	1	0.177	6.1	11.3
	2	0.183	6.5	12.0
	3	0.183	7.1	13.1
	4	0.190	7.2	13.3
1 250°C×15 min + 850°C×10 min Oil quench	1	0.176	3.0	5.6
	2	0.189	2.7	5.0
	3	0.159	5.7	10.5
	4	0.177	5.2	9.6

* Measured over the plastic strain range of $\epsilon=0.002$ to 0.004 .

ple fracture and $X_o=2.7$ and $3.0 \mu\text{m}$ for the intergranular dimple fracture. These are in good agreement with the observed dimple diameters in both cases. This agreement indicates the possibilities that the fracture toughness is controlled by the distributions of the second phase particles for dimple fracture, excluding the effects of the prior austenite grain size when it is larger than the distance between these particles.

Finally, the smaller size of the dimples for the intergranular fracture compared with that for the transgranular can be explained as follows; the embrittlement of the prior austenite grain boundaries may take place by the segregation of impurity elements to the boundaries, to which a decrease in the area of grain boundaries due to the grain growth can contribute. Because of such an embrittlement of the grain boundaries, the void can easily form at smaller second phase particles and the deformation during the coalescence of the voids is smaller than that for the transgranular fracture, as is obvious in comparison of Photo. 3 with Photo. 4.

V. Conclusions

The fracture toughness of as-quenched Ni-Cr-Mo steels was investigated as a function of the prior austenite grain size, using the critical J -integral concept. The results obtained are summarized as follows.

(1) The critical J -values measured on small sized bending specimens correspond satisfactorily to the values of the plane strain fracture toughness measured on compact tension specimens that meet minimum size standards for valid K_{Ic} testing. The critical J -value has potentialities in a quantitative evaluation of the fracture toughness in the range of large scale yielding where the crack opening displacement and the plane strain fracture toughness are difficult to be measured.

(2) The tensile properties of the steels deteriorate with coarsening of the prior austenite grain. The fracture toughness, on the other hand, remains nearly constant over a wide range of the prior austenite grain size, showing a gradually decreasing tendency with the grain growth. However, all specimens with largest grain size in each series of the steels show a discontinuous deterioration in the fracture toughness, corresponding to the change of transgranular to intergranular dimple fracture.

(3) The fracture toughness of the steels used is in good correlation with the size of the dimples that were originated at small second phase particles. This fact may exclude the effects of the prior austenite grain size larger than the distance between second phase particles responsible to dimples on the fracture toughness of the steels.

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