Intergranular Fracture on Fatigue Fracture Surface of 2.25Cr-1Mo Steel at Room Temperature in Air

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Low-alloy steels serving for a long time at high temperature (~500 °C) are very sensitive to temper embrittlement due to segregation of various trace elements at prior austenite grain boundaries and/or carbide/matrix interfaces. This type of segregation in combination with various environmental effects can adversely affect the fracture resistance and fatigue crack propagation rate with subsequent change in the fracture morphology of low-alloy steels. The present work describes the effects of heat treatments on impurity element segregation and its subsequent effects on fatigue fracture behavior of 2.25Cr-1Mo steel under different environmental conditions and temperatures. It has been found that either prior impurity element segregation caused during the heat treatment or hydrogen-induced embrittlement due to the presence of water vapor in laboratory air alone cannot produce intergranular fracture on the fatigue surfaces of 2.25Cr-1Mo steel at room temperature in air. The occurrence of intergranular fracture on the fatigue surfaces results from the combined effect of impurity element segregation-induced grain boundary embrittlement and hydrogen-induced embrittlement, and that the proportion of intergranular fracture is a function of prior impurity element segregation provided that the grain boundary segregation level exceeds a certain critical value.

Keywords embrittlement, fatigue fracture, fracture behavior, heat treatment

1. Introduction

Metal fatigue is well recognized as an important cause for failure or the early retirement of engineering structures. The performance of the structure can be further degraded by the interactions of fatigue loading with the external (service) environment. An important feature of fractography of fatigue surfaces obtained from long-crack propagation experiments made on the quenched and tempered, and quenched, tempered, and embrittled low-alloy steels at room temperature in laboratory air is the presence of intergranular facets, which is thought to be associated with prior austenite grain boundaries. The facets are present only at an intermediate value of stress intensity ΔK (Ref 1).

In the case of 2.25Cr-1Mo steel for fast fracture, the brittle fracture behavior can change from its typical transgranular cleavage fracture to intergranular decohesion if the impurity element segregation level at prior austenite grain boundaries exceeds a certain critical value (Ref 2-5). As for brittle fast fracture, impurity element segregation due to classic temper embrittlement can change the fracture mode in fatigue and enhance the crack growth rate (Ref 6, 7). The aim of the present investigation was to discuss the importance of water vapor (at relative humidity levels associated with normal laboratory air)

M.A. Islam, Materials and Metallurgical Engineering Department, Bangladesh University of Engineering and Technology (BUET), Dhaka-1000, Bangladesh; and **P. Bowen** and **J.F. Knott**, the School of Metallurgy and Materials, The University of Birmingham, B15 2TT Edgbaston, Birmingham, U.K. Contact: e-mail: aminulislam@ mme.buet.ac.bd. and a certain level of impurity element segregation at grain boundaries to change the fracture morphology of 2.25Cr-1Mo steel at room temperature in air.

2. Experimental

2.1 Materials and Heat Treatment

The material used in this investigation was a commercial grade of 2.25Cr-1Mo steel the composition of which is given in Table 1. In this investigation, the following microstructures were considered:

- As-quenched martensitic microstructures: oil-quenched microstructures after austenitizing at 1100 °C for 2 h (asquenched condition).
- Quenched and embrittled martensitic microstructures: quenched microstructure tempered at 520 °C for 96 h (QE condition).
- Quenched and lightly tempered martensitic microstructures: quenched microstructures tempered at 650 °C for 20 min (QLT condition).
- Quenched and fully tempered martensitic microstructures: quenched microstructures tempered at 650 °C for 2 h (QT condition).
- Heavily embrittled QT martensitic microstructures: QT condition embrittled for 210 h at 520 °C (QTHE condition).

Table 1 Chemical composition of the steel

Composition, wt.%										
С	Si	S	Р	Mn	Ni	Cr	Мо	V	Cu	Al
0.15	0.22	0.023	0.013	0.51	0.11	2.27	0.91	0.01	0.16	0.03

2.2 Fatigue Crack Growth Testing

Using the three-point bend configuration, fatigue crack propagation tests were performed on a Vibrophore (Russenberger Prufmaschinen AG, Rheinfall, Switzerland) at high frequency (65 Hz), whereas for low frequency (0.25 Hz) a Universal testing machine (Instron, High Wycombe, Bucks, UK) was used. For all cases, an *R* ratio of 0.1 was used (where *R* is defined as P_{\min}/P_{\max}).

Fatigue tests at -196 °C were carried out on the Vibrophore, but the crack growth test at 110 °C with 0.25 Hz frequency was carried out on the Universal testing machine equipped with heating facilities. The test temperature was maintained at 110 ± 2 °C. The fatigue test conditions used for this present investigation are summarized in Table 2.

Table 2Detailed fractographic features observed onfatigue surfaces of specimens under different heattreatment conditions

	Test	
HT code	temperature	Fractographic features
As-quenched	RT	100% transgranular at all levels of ΔK
QE	RT	100% transgranular on all levels of ΔK
QLT	RT	Isolated IG over a narrow ΔK range and a maximum 5% IG fracture at ΔK level of ~20 MPa \sqrt{m}
QT	RT	IG facets over ΔK range ~12-32 MPa \sqrt{m} and a maximum ~20% IG facets at ~20 MPa \sqrt{m}
QTHE	RT	IG facets over ΔK range ~10-34 MPa \sqrt{m} and a maximum ~50% IG facets at ~20 MPa \sqrt{m}
	110 °C	100% transgranular at all levels of ΔK
	−196 °C	100% transgranular at all levels of ΔK

HT, heat treatment; IG, intergranular fracture

Note that before fatigue crack growth testing, a fatigue precrack was formed in all specimens under all heat treatment conditions at room temperature in air.

2.3 Metallography

A detailed microstructural characterization of specimens under different heat treatment conditions used in this investigation has been performed; details can be found elsewhere (Ref 2).

2.4 Fractography

After the growth of fatigue crack, fracture surfaces were cut off the ends of specimens. These fracture surfaces were then cleaned in an ultrasonic cleaner using acetone as the solvent, dried and mounted on aluminum stubs. Fractographic observations were then carried out in a scanning electron microscope (SEM) operating at 20 kV and 0°. Using these SEM micrographs, area fractions of intergranular fractures were measured either manually or automatically on a Quantimet-500 image analyzer computer (Leica, Cambridge, UK).

3. Results

3.1 Metallography

The optical micrographs of specimens under various heat treatment conditions mainly exhibited a lath martensitic microstructure and exhibited no noticeable difference between the unembrittled and temper-embrittled conditions (Ref 2).

3.2 Fractography

A fatigue crack growth test of an as-quenched specimen was performed over the ΔK range of 13 to 24 MPa \sqrt{m} . Throughout



Fig. 1 Fatigue surfaces observed for the as-quenched specimen over the ΔK range 13 to 24 MPa \sqrt{m} at room temperature in air for a frequency 65 Hz. The arrow indicates the direction of crack growth.

this stress intensity range, no intergranular fracture was found (Fig. 1). After 96 h of tempering at 520 °C (QE condition), no change in the fracture morphology was observed (i.e., the QE specimen again showed 100% transgranular fracture) (Fig. 2). The tempering of the quenched specimen at 650 °C for 20 min (QLT condition), however, resulted in some isolated intergranular fracture (maximum ~5%) for intermediate ΔK levels (Fig. 3).

With an increase in the tempering time (e.g., 2 h) at 650 °C (QT condition), a substantial increase in the proportion of in-

tergranular fracture was observed. In this case, intergranular facets were observed over a total ΔK range of 12 to 32 MPa \sqrt{m} . For ΔK values of <12 MPa \sqrt{m} , no intergranular fracture was found (Fig. 4). After this, the proportion of intergranular fracture increased with the increase in ΔK level, and a maximum of 20% intergranular fracture was obtained at a ΔK level of ~20 MPa \sqrt{m} (Fig. 5). A further increase in the ΔK level resulted in a decrease in the proportion of intergranular fracture, and above a ΔK level of 32 MPa \sqrt{m} , no intergranular fracture was observed (Fig. 4). Below a ΔK level of 12 MPa \sqrt{m} and above



Fig. 2 Fatigue surfaces observed for the QE specimen over the ΔK range 14 to 24 MPa \sqrt{m} at room temperature in air for a frequency 65 Hz. The arrow indicates the direction of crack growth.



Fig. 3 Fatigue surfaces observed for the QLT condition at room temperature in air for a frequency of 65 Hz and ΔK range 14 to 24 MPa \sqrt{m} . The arrow indicates the direction of crack growth.



Fig. 4 Fatigue surfaces observed for the QT specimen over the ΔK range 8.6 to 36 MPa \sqrt{m} at room temperature in air for a frequency of 65 Hz. The arrow indicates the direction of crack growth.



Fig. 5 Fatigue surfaces observed for the QT specimen (Fig. 4) showing the intergranular fracture at the peak region (at ΔK value ~20 MPa \sqrt{m})

32 MPa \sqrt{m} , the fracture surface was almost 100% transgranular (Fig. 6).

When quenched and tempered specimens were isothermally embrittled at 520 °C for 210 h (QTHE condition), intergranular facets were observed over a wider range of ΔK values than those for the QT condition. In such a case, the formation of intergranular facets started from a ΔK level of 10 MPa \sqrt{m} and ended above 34 MPa \sqrt{m} (Fig. 7). Now, the maximum proportion of intergranular fracture of the QT specimen has increased from 20% to about 50%. At some regions, a very high proportion of intergranular fracture was also observed (Fig. 8). Interestingly, the peak proportion of intergranular facets was found at a ΔK level similar to that for the QT specimen (Fig. 4, 7).

When the QTHE specimen was tested at a lower frequency (0.25 Hz), the maximum proportion of the intergranular facet



Fig. 6 Fatigue surfaces of the QT specimen (Fig.4) showing the transgranular fracture features at a ΔK level of 34 MPa \sqrt{m}

was found to increase slightly (50 to 55%). At the same frequency, the QTHE specimen produced no intergranular fracture when the test temperature was increased from room temperature to 110 °C (Fig. 9). Another interesting observation is the absence of intergranular facets when the test temperature was reduced from room temperature to -196 °C (Fig. 10), even though the same specimen produced about 70% intergranular fracture when it was broken under monotonic loading at the same temperature (Fig. 11).

4. Discussion

To investigate the occurrence of intergranular fracture on the fatigue surfaces of 2.25Cr-1Mo steel, single end notched



Fig. 7 Fatigue surfaces observed on QTHE specimen over the ΔK range of 8.6 to 38 MPa \sqrt{m} at room temperature in air for a frequency of 65 Hz. The arrow indicates the direction of crack growth.



Fig. 8 Fractograph showing a very high proportion of intergranular fracture (~75%) observed at the peak position on the fracture surface of the QTHE specimen tested at room temperature in air

beam (SENB) specimens were prepared under five different heat treatment conditions. For similar test conditions, the current study clearly demonstrates the presence of a substantial proportion of intergranular facets on fatigue surfaces of the QT and QTHE specimens (Fig. 4, 7). Some isolated intergranular facets have also been found on the QLT specimen (Fig. 3), whereas fatigue surfaces of the as-quenched and QE specimens are completely free of intergranular facets (Fig. 1, 2). The individual heat treatment conditions are now considered to explain the reasons for the presence and absence of intergranular facets on fatigue surfaces.

4.1 As-Quenched Condition

From Fig. 1, it is clear that the as-quenched specimen exhibited no intergranular facets at any ΔK level, which is in agreement with findings of Nishioka and Knott (Ref 1) for 9Cr-1Mo steel. For this condition, a SENB specimen encapsulated in a silica glass tube was austenitized at 1100 °C for 2 h and was quenched directly in oil to room temperature. This treatment resulted in an autotempered martensitic structure with an average prior austenite grain size of $\sim 120 \mu m$. In the as-quenched steel, the maximum proportion of alloying and impurity elements remain dissolved in the matrix. In this situation, one would expect no, or very little, impurity element segregation at the prior austenite grain boundaries. In several studies, the role of hydrogen atoms (formed by the decomposition of water vapor present in the atmospheric air) to produce intergranular facets at room temperature is well established, but the absence of intergranular facets on the fracture surfaces of the as-quenched specimen suggests that the combined effect of any (slight) grain boundary impurity element segregation and hydrogen atoms could not produce intergranular facet on fatigue surfaces at room temperature in air (Ref 8, 9).

4.2 Quenched and Embrittled Condition (QE Condition)

The QE specimen was prepared by tempering the asquenched specimen at 520 °C (which is the peak embrittling temperature for reversible temper embrittlement in this steel) for 96 h and then quenched in oil. After this heat treatment, the fatigue fracture surface under the same test conditions as those used for the as-quenched specimen was found to be free of intergranular facets (Fig. 2). To explain the fracture behavior of the QE specimen, the work of Baker and Nutting (Ref 10) on carbide formation during tempering is revisited. According to the standard diagram (Fig. 12) for carbide formation in quenched 2.25Cr-1Mo steel, 96 h of tempering at a temperature of 520 °C produces only iron-based carbide M_3C . Its formation, therefore, has a very small effect on the content of dissolved alloying elements, especially on the dissolved molybdenum (Mo) content. Due to the higher interaction energy between Mo and phosphorus (P), which has been found to be the main embrittling element for this steel under the present investigation, the dissolved Mo keeps the P atoms within the grains and retards its segregation at grain boundaries (Ref 2, 3, 5).

Metallographic observations suggested that the grain

boundary segregation level of impurity elements for the QE specimen is somewhat higher than that for the as-quenched specimen (Ref 2). This slightly higher level of segregation in the QE condition is not unexpected, as such specimens have experienced long time exposure at 520 °C, for which some undissolved impurity elements may migrate to the grain boundaries. Moreover, the iron-based Fe₃C carbides may also contain a proportion of Mo atoms (Ref 11, 12) Dissolution of Mo may then release P atoms, which eventually migrate to the grain boundaries. Interestingly, QE specimens exhibit no intergranu-



Fig. 9 Fatigue surfaces observed for the QTHE specimen over the ΔK range of 14 to 24 MPa \sqrt{m} at 110 °C in air for a frequency of 0.25 Hz. The region precracked at room temperature in air showed a noticeable proportion of intergranular facets, whereas fatigue fracture surfaces at 110 °C in air are completely transgranular. The arrow indicates the direction of crack growth.



Fig. 10 Fatigue surfaces observed for the QTHE specimen over the ΔK range of 12 to 26 MPa \sqrt{m} at -196 °C in air for a frequency of 65 Hz. The region precracked at room temperature in air showed many intergranular facets, but the fracture surface at -196 °C in air is almost transgranular. The arrow indicates the direction of crack growth.



Fig. 11 Fractograph of brittle fracture region ahead of the fatigue crack tip (marked by arrows). High-volume fraction of the intergranular facet (~70%) can be seen in the region of the brittle fracture, whereas the fatigue surface is completely transgranular.



Fig. 12 Isothermal diagram showing the sequence of carbide formation on tempering of quenched 2.25Cr-1Mo steel (Ref 10).

lar facets on the fatigue surfaces tested at room temperature in air. This fractographic observation indicates that the level of grain boundary segregation in combination with hydrogen cannot produce intergranular fracture during fatigue at room temperature in air.

4.3 Quenched and Lightly Tempered Condition (QLT Condition)

For this condition, fractographic observation revealed some isolated intergranular facets on the fatigue surfaces (Fig. 3). Because the tests were carried out under identical conditions, one would expect a level of hydrogen availability at the crack

tip similar to that experienced by either the as-quenched or the QE specimen. The presence of intergranular facets in the QLT condition indicates a somewhat higher level of impurity coverage in this specimen. At least some of the grain boundaries were associated with a segregation level equal to, or above, the threshold level required for intergranular fracture in the presence of water vapor at room temperature in laboratory air. The metallographic observations also support this (Ref 2). This situation can again be explained with reference to the work of Baker and Nutting (Ref 10) and other investigators (Ref 13). From the diagram presented in Fig. 12, it is clear that tempering at 650 °C can produce Mo-rich carbides in as-quenched 2.25Cr-1Mo steel. The formation of this carbide implies the depletion of dissolved Mo from the matrix and the breaking of Mo-P clusters, enabling the release and migration of P atoms to grain boundaries. The tempering time, however, is possibly not enough to produce a substantial amount of Mo-rich carbide and/or migration of the released P atoms to grain boundaries. Moreover, the energy level of all grain boundaries is not the same, which plausibly produces different impurity element segregation rates at different grain boundaries. As a result, some isolated grain boundaries, which are more favorable sites for segregating species, only experience a level of impurity coverage that is sufficient to produce intergranular decohesion in combination with the hydrogen-induced embrittlement. All other grain boundaries (which have impurity element coverage lower than that of the threshold level to cause intergranular decohesion) fail by normal transgranular fatigue fracture.

4.4 Quenched and Tempered Condition (QT Condition)

After quenching in oil, the as-quenched specimen was tempered at 650 °C for 2 h. Specimens in this QT condition produced a considerable proportion of intergranular facets on the fatigue surfaces for tests carried out at room temperature in air. These intergranular facets were found for ΔK ranges of 12 to 32 MPa√m (Fig. 4) with the maximum proportion of intergranular facets equal to about 20% (which is significantly higher than that of QLT condition), which were observed at a ΔK level of 20 MPa \sqrt{m} (Fig. 5). The specimens for both heat treatment conditions (OLT and OT) were tempered at 650 °C, but the tempering time for the QT specimen was six times longer than that for the QLT condition. Due to this longer tempering time, the volume fraction of carbides is higher, and more Mo-rich carbide is formed as per the diagram presented in Fig. 12. The formation of the more Mo-rich carbides implies significant Mo depletion in the matrix with more P atoms released by breaking Mo-P clusters. These released P atoms can randomly redistribute themselves at microstructural sites. Transmission electron microscopy (TEM) observation suggests a somewhat higher concentration of P at grain boundaries (Fig. 13). The higher level of impurity coverage in the QT specimens compared with that of the QLT condition is simply due to the presence of more free P atoms in the QT specimens, and this is, arguably, responsible for the higher proportion of intergranular fracture in these specimens.

Nishioka and Knott (Ref 1) also found a higher proportion of intergranular facets for quenched and tempered 9Cr-1Mo steel at room temperature in distilled water compared with that in laboratory air. The facets produced in distilled water were



Fig. 13 (a) FEG-STEM image of a grain boundary of QT specimen and (b) variation in P concentration across the boundary. Note: grain boundary (marked by arrows) and analysis direction across the grain boundary (marked by a line) can be seen in Fig. 13a.

more "clean-cut" in nature with intergranular decohesion, which was rarely noticed for the same material tested at room temperature in air. Compared with the tests at room temperature in air, the hydrogen availability in distilled water is plausibly higher, and the higher proportion of intergranular fracture found for distilled water again reveals the importance of hydrogen concentration at the crack tip with respect to the production of intergranular fracture.

4.5 Quenched, Tempered, and Heavily Embrittled Condition (QTHE Condition)

The difference between the heat treatment conditions of QT and QTHE specimens is that the latter experienced an additional 210 h of exposure at 520 °C. This additional treatment increased the maximum proportion of intergranular fracture (with respect to the QT condition) from ~20% to ~50%. From these observations and those treated earlier, it is now clear that a synergism between intergranular impurity element segregation and hydrogen-induced embrittlement controls the formation of intergranular facets during fatigue of 2.25Cr-1Mo steel at room temperature in air.

4.5.1 Effect of Environment. From the fractographic observations, as-quenched and QE specimens exhibited no intergranular facets on the fatigue surfaces (Fig. 1, 2), whereas for the QLT, QT, and QTHE conditions the maximum proportions

of intergranular facets were found to be approximately 5%, 20%, and 50%, respectively (Fig. 3, 4, 7). According to the metallographic observation of impurity segregation (Ref 2), the degree of grain boundary impurity coverage for specimens under different heat treatment conditions can be ranked in the following way: as-quenched condition < QE condition < QLT condition < QT condition < QTHE condition. From this observation, and the fractographic data presented in this article, it is quite clear that there is a correlation between the proportion of intergranular fracture on fatigue surfaces at room temperature in air and impurity segregation at grain boundaries during heat treatment. Several studies (Ref 1, 9) have also demonstrated the important role of water vapor in encouraging the formation of intergranular facets. To investigate this, fatigue tests in the absence of water vapor were carried out. There are several different ways to achieve an atmosphere free of water vapor: test in vacuum, test at temperatures above 100 °C in air, or test in liquid nitrogen. In the present investigation, fatigue tests were performed at 110 and -196 °C to achieve this condition, and at these temperatures fatigue tests were made on the OTHE specimen, which exhibited the highest level of intergranular fracture.

4.5.2 Fatigue Test at 110 °C. At 110 °C, the QTHE specimen showed no intergranular fracture, whereas the same specimen exhibited a significant amount of intergranular fracture at room temperature (Fig. 9) and a specimen having the same heat treatment condition exhibited a maximum of 50% intergranular

facets (Fig. 7). It has been argued that hydrogen atoms must penetrate to the grain boundaries primarily along the crack tip and that a sufficiently hot bulk specimen would reduce the probability of this happening, even if there was a remnant water vapor pressure in the surrounding atmosphere. This is possibly due to the elimination of condensed water from the crack tip and/or reduced relative humidity surrounding the specimen due to long time exposure at 110 °C. Among all the heat treatment conditions used in the present investigation, the QTHE specimen has the highest level of P coverage at ~18.8% (Ref 1, 3). From this observation, it is clear that prior impurity element segregation at grain boundaries, even if the impurity coverage is significantly high, cannot produce intergranular fracture without further hydrogen-induced embrittlement during fatigue at room temperature in air.

4.5.3 Fatigue Test at -196 °C. As was the case for the fatigue test at 110 °C, the QTHE specimen exhibited no intergranular fracture on the fracture surfaces at -196 °C (Fig. 10). At -196 °C, the penetration of water vapor through the crack tip is prohibited, because the temperature surrounding the specimen is maintained well below the freezing temperature of water vapor: arguably the crack tip of this specimen could not come in contact with water vapor and experience hydrogeninduced embrittlement. The absence of intergranular facets on the fatigue surface at this temperature again supports the fact that prior impurity segregation alone cannot produce intergranular facets, if the water vapor in the air does not come in contact with the crack tip to cause hydrogen-induced embrittlement. The conclusion is simply that temper embrittlement itself is not sufficient to produce intergranular facets at room temperature in air without the interaction of water vapor.

The occurrence of intergranular facets in the low-alloy steels during fatigue is associated with water vapor and, hence, most plausibly with the generation of atomic hydrogen by a reaction mechanism of the sort (Ref 1):

 $Fe + H_2O = FeOH^+ + H^+ + 2e^-$

 $\mathbf{H}^{+} + \mathbf{e}^{-} = \mathbf{H}$

5. Conclusions

 As evidenced from the fractographic observations, either prior impurity element segregation caused during heat treatment or hydrogen-induced embrittlement due to the presence of water vapor in laboratory air alone cannot produce intergranular fracture on the fatigue surfaces of 2.25Cr-1Mo steel at room temperature in air. The occurrence of intergranular fracture on the fatigue surfaces results from the combined effect of impurity element segregation-induced grain boundary embrittlement and hydrogen-induced embrittlement.

• For lightly and fully tempered specimens (QLT and QT conditions, respectively), the maximum proportions of intergranular fractures are ~5% and ~18 to 20%, respectively, whereas 210 h of embrittlement at 520 °C increased the maximum proportion of intergranular fracture of the QT condition to a level of about 50%. This fractographic observation suggests that the proportion of intergranular fracture is a function of prior impurity segregation provided that the grain boundary segregation level exceeds a certain critical level.

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