VOL.1, NO.1, JUNE 2006 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

CRITICAL ASSESSMENT OF THE DEGREE OF TEMPER EMBRITTLEMENT IN 2.25Cr-1Mo STEEL

M.A. Islam
Materials and Metallurgical Engineering Department
Bangladesh University of Engineering and Technology, Dhaka-1000, Bangladesh
E-mail: urzsai@gmail.com

ABSTRACT

Reversible temper embrittlement has been frequently observed in many different low alloy steels, serving at high temperatures, due to segregation of trace elements at grain boundaries and/or carbide/matrix interfaces. This type of impurity element segregation can severely deteriorate the toughness and fatigue properties of the steel. In general, increase in the hardness and tensile strength also increases the fatigue life of the steel. So, fatigue lives of steels are sometimes assessed by these parameters. In this research work 2.25Cr-1Mo steel, before and after temper embrittlement, was characterized by microstructure observation, hardness measurement, tensile, fatigue and fracture toughness tests at room temperature in air. Experimental results revealed that temper embrittlement hardly modify the room temperature hardness values and tensile properties, although the fatigue and fracture behaviours of this steel are significantly changed.

Keywords: Low alloy steel, Temper embrittlement, Tensile strength, Fatigue, Fracture toughness, Intergranular fracture.

INTRODUCTION

The 2.25Cr-1Mo steel has many advantages for both ambient and high temperature service. The potential uses of this steel are in the electrical power generation plants and petrochemical industries, because of its good mechanical properties at elevated temperatures. For high temperature applications, one common problem for low alloy steels is the temper embrittlement (TE) due to segregation of P and S when the steel is held for a prolonged time at temperatures of order of 500^{o}C (1-4). In practice, before entering service, steel in the quenched condition is fully tempered (stress relieved) in order to obtain optimum mechanical properties throughout the whole section. The fracture properties of these quenched and tempered steels are, however, adversely affected by the segregation of impurity elements to prior austenite grain boundaries and carbide/matrix interfaces during high temperature service. This type of in-service degradation in mechanical properties of the product can, however, lead to a catastrophic failure causing a huge loss and shutdown of the industry. So, temper embrittlement-induced deterioration in toughness must be checked on routine basis with proper mechanical test that can characterize it more critically keeping in mind that accident is always a very critical issue. In the present work, aging of quenched and tempered (unembrittled condition, UE steel) steel has been carried out at 520^{o}C for different time periods. Then the associated embrittlement effect has been characterized by microstructural observation, Vickers hardness, tensile, fracture toughness and fatigue crack propagation tests at room temperature in the air.

MATERIALS AND METHODS

Material and Heat Treatment

The material used in this study was a commercial grade of 2.25Cr-1Mo pressure vessel steel. The chemical composition of the steel is given in Table-1.

Table-1. Chemical composition of the steel (mass%).

Element	2.25Cr-1Mo Steel
C	0.15
Si	0.22
Mn	0.51
P	0.013
S	0.023
Ni	0.11
Cr	2.27
Mo	0.91
Cu	0.16

At first specimens of this steel were austenitised for 2 hours at a temperature of 1100^{o}C and quenched in oil. These oil quenched specimens were then tempered at 650^{o}C for 2 hours to obtain stress free unembrittled (UE) samples. To induce different level of temper embrittlement quenched and tempered samples were aged at 520^{o}C for different time periods as 24 (light embrittlement, LE condition), 96 (medium embrittlement, ME condition) and 200 hours (heavy embrittlement, HE condition).

Metallography

Metallographic samples from all heat treatment conditions were prepared and etched in 2% nital in order to reveal the microstructures. The etched specimens were observed in optical and scanning electron microscopes and photographed.

Hardness Measurement

Hardness measurements were performed using a Vickers hardness testing machine. Indents were made on the polished surfaces using a 20kg load and a 10sec dwell time. Hardness measurements were made randomly (up to thirty measurements for each heat treatment condition) and the average was taken as a representative hardness in each heat treatment condition.

Tensile Tests

Tensile tests were carried out on a universal testing machine (UTM) using BS M8 type specimens (Figure-1) at room temperature in the air. All tensile tests were performed at a crosshead displacement of 1mm/min and load-elongation diagrams were obtained automatically from the computer. From the recorded load vs. elongation charts yield stress σy, UTS (ultimate tensile strength) and %elongation were calculated.

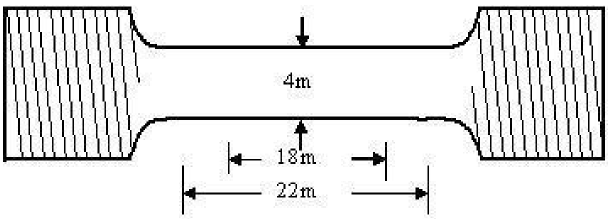

Figure-1. Schematic diagram of tensile specimen geometry.

Fatigue and Fracture Toughness Testing

Using single edge notch bend (SENB) specimens and three point bend configuration, Figure-2, fatigue crack propagation tests were performed on Vibrophore at high frequency (65Hz) at room temperature in air with R ratio (P_{min}/P_{max}) of 0.4. This test was only carried on unembrittled (UE) and heavily embrittled (HE) specimens.

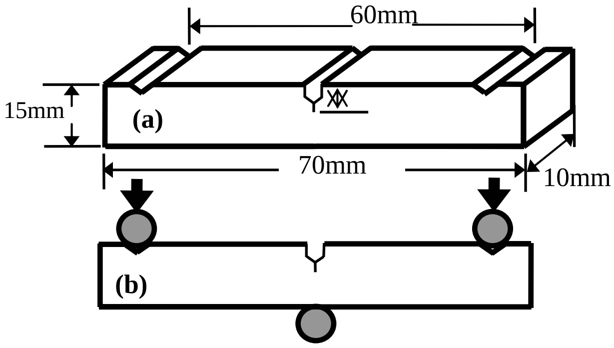

Figure-2. Schematic representation of (a) single edge notch bend specimen and (b) three point bend configuration.

Fracture toughness tests of samples (fatigue precracked SENB samples) of UE, ME and HE conditions were carried out in a UTM of 200kN capacity in accordance with BS 7448 (5).

Fractography

After mechanical tests (tensile, fatigue and fracture toughness tests), specimens were cut ~10mm behind the fracture surfaces to provide samples suitable for observation in the scanning electron microscope (SEM). The fracture surfaces of all specimens were then carefully observed and photographed in the SEM with an accelerating voltage 20kV and a stage tilt of 0^o.

RESULTS AND DISCUSSION

Microstructural Characterization

The micrographs on the polished and nital etched specimens under all heat treatment conditions were produced using optical and scanning electron microscopes. Austenitising at a temperature of 110^o0C for 2 hours and quenching in oil and tempering at 650^oC for two hours (UE condition) produced a tempered martensitic microstructure, which is typical in this steel, Figure-3a. When UE samples were aged at 520^oC, the size and distribution of carbide particles were found to change. Some other investigators also mentioned similar changes in the carbide morphologies (6,7).

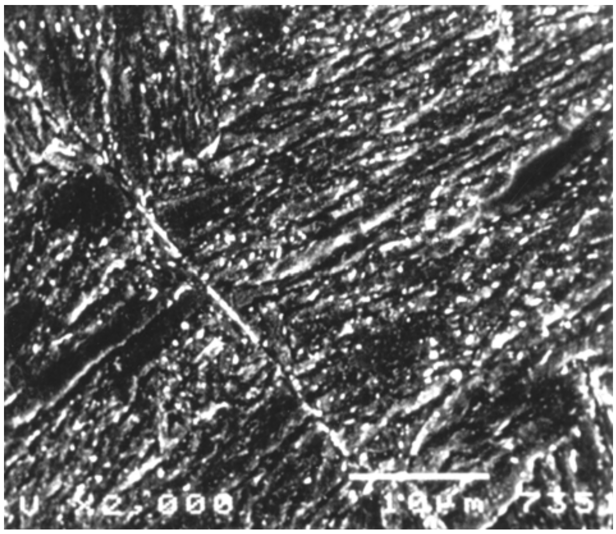

Figure-3a. SEM micrograph on polished and nital etched specimen under UE conditions.

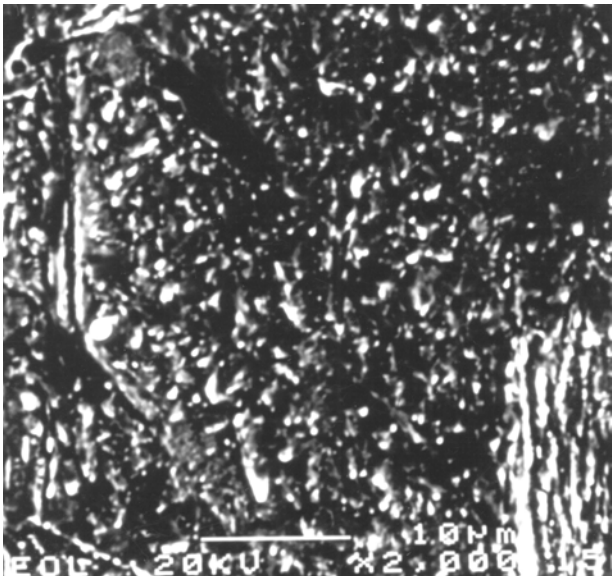

Figure-3b. SEM micrograph on polished and nital etched specimen under HE conditions.

Hardness Measurement

Vickers hardness measurement on samples under all heat treatment conditions were performed at room temperature. The results of the room temperature hardness values are shown in Table-2. From Table-2, it is clear that the as-quenched specimen showed the highest hardness. Tempering at 650°C for 2 hours (UE condition) exhibited a sharp decrease in hardness (from 397 to 255 Hv). When UE specimens were subsequently aged at 520°C for 24 hours (LE condition) or 96 hours (ME condition), no significant change in hardness values was observed. However, for long time exposure, e.g., 210 hours at this temperature (HE condition), a slight decrease in hardness was observed. The highest hardness in the as-quenched specimen seems to be consistent with the heat treatment condition. Quenching directly from the austenitising temperature retains carbon and other alloying elements in the solid solution. Moreover, as-quenched steel usually contains a very high level of dislocation density. Plausibly, as-quenched specimen showed the maximum hardness value. On tempering, carbon and alloying elements come out from the solid solution and form different carbides depending on the tempering temperature and time (8). At the same time, tempering causes the recovery of martensite lath structures and lath boundaries disappear gradually (8,9). As a result, the hardness of as-quenched specimen decreases with tempering. UE specimens were further aged at 520°C for different time periods, e.g., 24 hours (LE condition), 96 hours (ME condition) and 200 hours (HE condition). No marked change in the hardness of UE specimens was observed for either LE or ME condition. Yu and McMahon (10) also found no change in hardness during aging of the same steel at 520°C. This may be due to the balance between the softening effect caused by carbide coarsening along with recovery of martensitic structures and the secondary hardening effect of fine M_2C carbide formation due to somewhat higher temperature aging. The 200 hours aging at 520°C (HE condition) caused a slight decrease in hardness. This effect is thought to be due to a carbide coarsening effect along with further recovery of martensitic structure, as seen in Figure-3b.

Table-2. Room temperature average Vickers hardness values with standard deviation (SD).

HT Code	Hardness, Hv (kg/mm2)	SD
As-quenched	397	±8
UE	255	±5
LE	253	±7
ME	254	±6
HE	242	±5

Tensile Properties

For all the heat treatment conditions used, no true yield point was observed in the tensile tests. As a result, the yield stress (σ_y) was taken to be the stress at 0.2% offset, Table-3. As can be seen from Table-3 that temper embrittlement (TE) has virtually no effect on the 0.2% proof stress and ultimate tensile strength of the unembrittled steel at room temperature. During TE impurity elements segregate to prior austenite grain boundaries and carbide/matrix interfaces. This type of segregation weakens grain boundaries and carbide/matrix interfaces and produces the failure at lower strains. In the present work, however, no marked effect of segregation on 0.2% proof stress, ultimate tensile strength or elongation was found. For En30A steel, King (11) also found no effect of temper embrittlement on the 0.2% proof stress of the unembrittled samples. It is now well accepted that impurity element segregation reduces the grain boundary cohesive strength of the material. If segregation reduces the grain boundary strength, arguably it will also affect the carbide/matrix interface strength. So, the possible reason of identical fracture behaviour of unembrittled and embrittled condition is that segregated carbide/matrix interfaces are probably as good as non-segregated carbide/matrix interfaces with respect to impeding motion of dislocations at low strains (0.2% proof stress) even at moderate strains (UTS) and that the overall failure strain (and %R.A.) may still be dominated by the distribution of larger inclusion-initiated voids rather than the smaller voids created by carbide particles. This possibly happens due to that during tensile test whole cross section of the specimen comes under similar level of stress and that a large numbers of inclusions can dominate in the fracture process simultaneously. In this situation, effect of fine carbide particles on fracture process becomes less dominating.

VOL.1, NO.1, JUNE 2006 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

Table-3. Tensile properties of steel after different heat treatments.

0.2% Proof Stress, MPa				UTS, MPa				% Elongation			
UE	LE	ME	HE	UE	LE	ME	HE	UE	LE	ME	HE
710	698	716	705	837	818	815	795	20	20	19	20

At room temperature, specimens under all other heat treatment conditions failed by cup and cone separation. Low magnification scanning electron micrographs showing the typical macroscopic appearances of the fracture surfaces is presented in Fig.4. Similar type of ductile cup and cone fracture morphologies of unembrittled and embrittled steels also suggest that tensile test can not critically assess the embrittlement effect in the steel. The possible reason behind this is the stress distribution during uniaxial tensile test. During tensile testing, smooth specimens experience almost similar level of stress under loading. In this situation, stress triaxiality is not so severe because of stress relaxation throughout the whole outer surface of the loaded specimen.

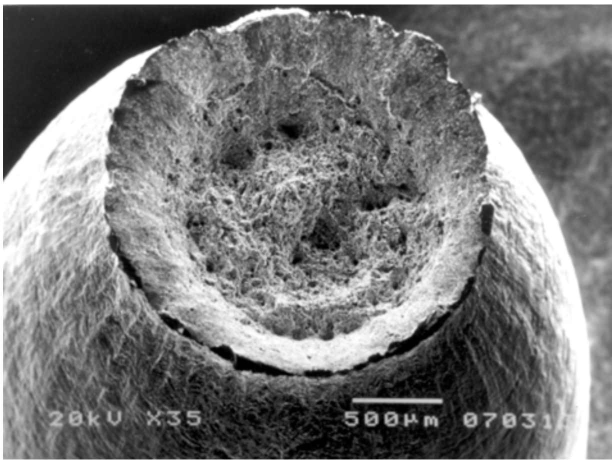

Figure-4a. SEM micrograph showing the ductile cup and cone fracture on UE specimen; cup half.

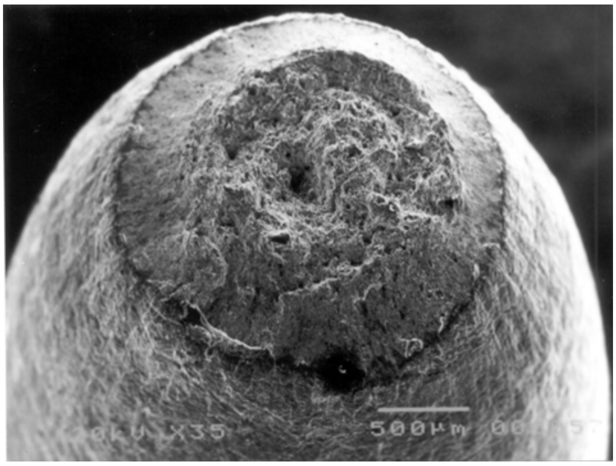

Figure-4b. SEM micrograph showing the ductile cup and cone fracture on UE specimen; cone half.

Fatigue Properties

The fatigue crack growth rates measured for the UE and HE specimens at room temperature in air are shown in Fig.5. From this figure, it is clear that reversible temper embrittlement clearly increased the crack growth rate throughout all the stress intensity range.

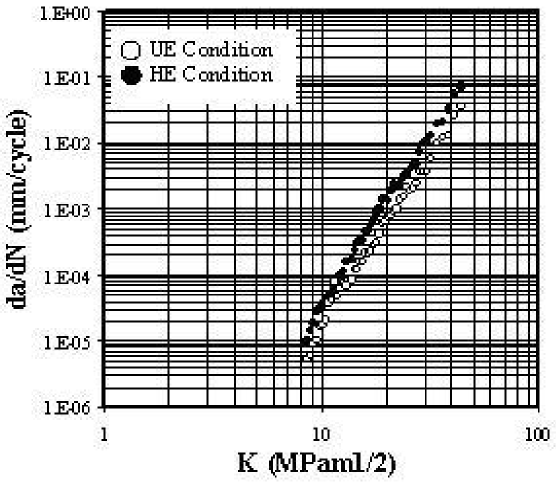

Figure-5. Fatigue crack growth curves of UE and HE samples.

Here it is to be noted that there is also a significant change in the fatigue fracture morphologies. For the UE samples the fracture mode was almost completely brittle transgranular type. However, a significant amount of intergranular fracture was observed on fatigue surfaces of HE samples, Fig.6. This higher proportion of intergranular fracture eventually affected the crack growth rate of HE sample. For tests in pure hydrogen, Hippsley (12) also found increased crack growth rates for embrittled specimens compared with those for the unembrittled specimens of 2.25Cr-1Mo steel. He also mentioned the increased fatigue crack rate of embrittled steel to be due to the higher proportion intergranular cracking.

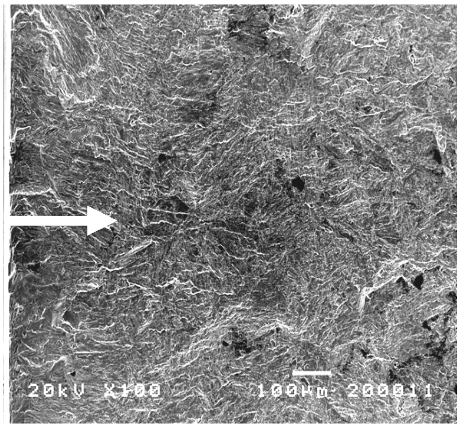

Figure-6a. Fracture features on UE sample at room temperature in the air. Arrow is indicating the direction of crack propagation.

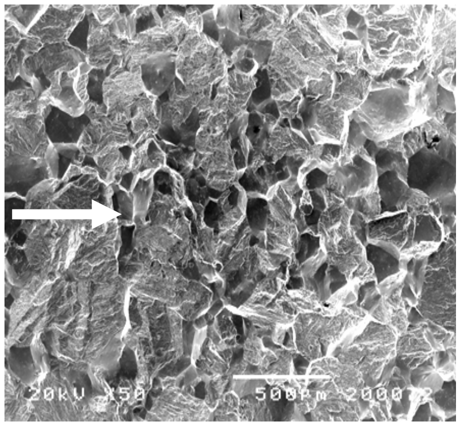

Figure-6b. Fracture features on HE sample at room temperature in the air. Arrow is indicating the direction of crack propagation.

In earlier it has been mentioned that TE has no marked effect on microstructure, hardness and room temperature mechanical flow behaviour of the quenched and tempered steel. The effect of embrittling treatments is to produce the impurity segregation at prior austenite grain boundaries and carbide/matrix interfaces. This has been identified in the present investigation by AES analysis, Figure-7.

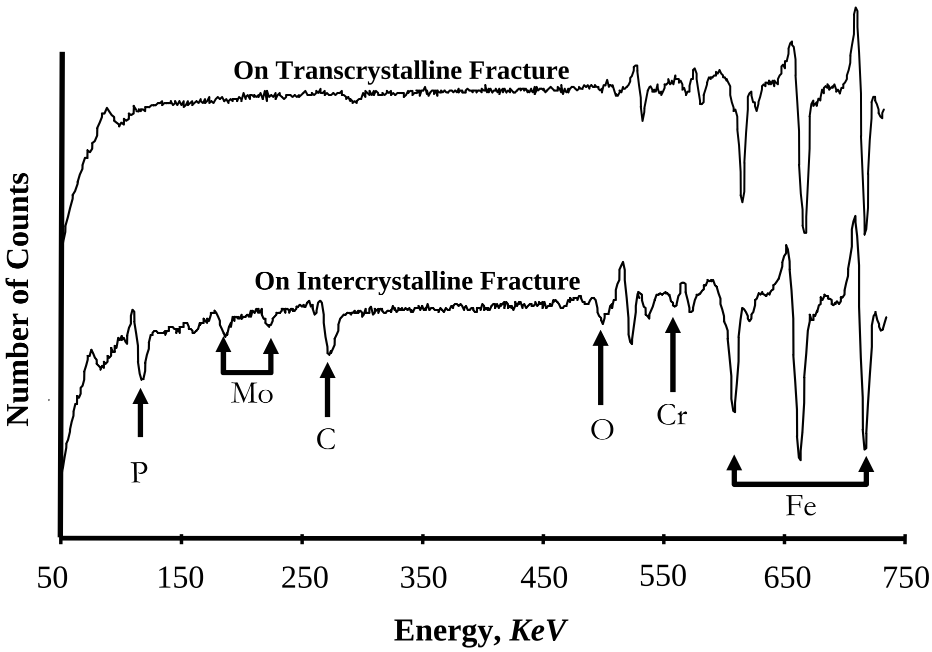

Figure-7. AES spectra transcrystalline and intercrystalline fractures of 2.25Cr-1Mo steel.

VOL.1, NO.1, JUNE 2006 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

As per the AES results, phosphorus has been found to be the main embrittling element. When phosphorus segregates to prior austenite grain boundaries, it weakens the local bonding and enables interfaces to break open at lower strain. The higher crack growth rate of the HE specimen compared with that of the quenched and tempered specimen is arguably due to the grain boundary phosphorus coverage. However, having the similar amount P segregation at grain boundaries, no significant and consistent effect of embrittlement was found on tensile strength and fracture morphology. This important point will now be discussed. For fatigue test, SENB specimen was used, Figure-2a. This specimen was precracked from the central notch.

During precracking load was maintained in such a way that the formed crack could be very sharp (see ref.5). This precracked SENB specimen was then used for fatigue growth test in three point bending configuration, Fig.2b. Under this configuration, the geometrical compliance function, i.e., the stress concentration effect is very high. At the same time, the load is applied at the very sharp crack tip, which can be considered as just a line. Materials along this line experience direct loading effect and any induced stress below this line is resisted by the materials there. In this situation, materials in the loaded region experience a severe stress triaxiality and thus the materials show more brittle behaviour (13). As the materials behave as a brittle manner, the heavily segregation grain boundaries are cracked well before the start of transgranular cracking. These secondary intergranular cracks induce more stress concentration effect at their tips. As a result, cracks are forced to grow inside the matrix and cause more crack growth rate. For unembrittled steel, intergranular cohesive strength is higher than that of cleavage plane. As a result, during fatigue crack growth, no intergranular cracking takes place and that no additional stress concentration effect inside the matrix is induced in the unembrittled steel that can accelerate the crack growth rate. So, fatigue crack growth test can assess the impurity element segregation induced material degradation more accurately. In contrast, whole section of the tensile specimen experiences almost similar level of stress and that severe local stress triaxiality (like fatigue crack growth test) can not be achieved because of stress relaxation from whole surface area. So, tensile specimens under all heat treatment conditions showed ductile fractures of similar nature and tensile strength values were indistinguishable.

Fracture Toughness

After full tempering (2 hours at 650°C), UE specimens were embrittled isothermally at 520°C. Isothermal embrittlement at 520°C for 96 hours (ME condition) reduced the average fracture toughness value compared with that of the UE condition and the fracture toughness values were found to be a function of embrittlement time (ME and HE conditions), Table-4. The reason behind this variation in fracture toughness is due to the variation in grain boundary cohesive strength caused by different level of P segregation. As fracture toughness test was carried out on precracked SENB specimens under three point bend configuration (similar to that of fatigue crack growth test), consistent effect of P segregation on the degradation of toughness of the steel was found.

Table-4. Fracture toughness (MPa√m) of the steel.

UE	ME	HE
116	110	103

It is to be mentioned that the fracture surfaces of fracture toughness specimen were of microvoid coalescence type at room temperature, however, close observation revealed that carbide nucleated voids were more prominent in the embrittled specimen. Actually P segregation takes place not only at the grain boundaries, but also at carbide/matrix interfaces. Similar to grain boundary cohesive strength, P segregation also reduces the bonding strength of the carbide/matrix interfaces. So, during loading microcracks also forms around these interfaces. In order to provide more pronounced effect of carbide/matrix interface bonding, brittle fracture morphologies of UE and HE steels are presented here. From micrographs presented in Fig.8, it is clear that UE steel exhibited completely transgranular cleavage fracture, where HE steel showed intergranular fracture, Fig.8b. Interestingly, many carbide particles were found to be debonded completely, Fig.8b and that this feature is completely absent on UE steel. Here the intergranular fracture is due to decrease in the grain boundary cohesive strength and debonding of carbide particles is due to the weakening of carbide/matrix interfaces.

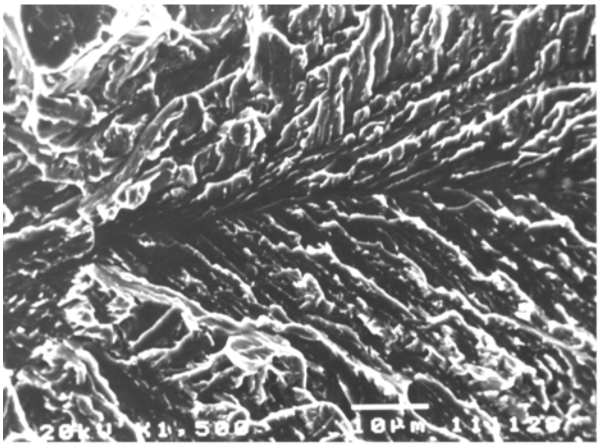

Figure-8a. Fracture surfaces of UE steel.

VOL.1, NO.1, JUNE 2006 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

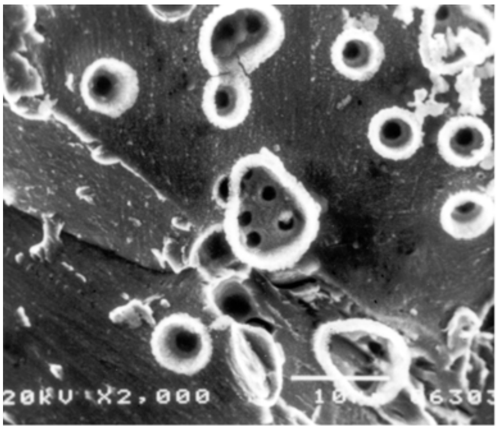

Figure-8b. Fracture surfaces of HE steel. Many carbides particles those were debonded keeping holes at their original positions are seen.

CONCLUSION

(a) Tempering was found to reduce the hardness of the as-quenched steel, but experimental results showed no significant change in hardness of the quenched and fully tempered specimen after a wide range of embrittlement treatments.

(b) At room temperature, temper embrittlement has been found to have virtually no significant and consistent effect on tensile properties of quenched and tempered steel.

(c) Fatigue crack growth and fracture toughness tests showed consistent effect of temper embrittlement. These experimental results suggest that fatigue and fracture toughness tests can assess the temper embrittlement induced degradation of the steel more critically than either hardness or tensile tests and also that by microstructural observation.

ACKNOWLEDGEMENT

The author is highly grateful to ORS Authority of U.K. and also Professor J.F. Knott and Professor P. Bowen of School of Metallurgy and Materials, University of Birmingham, U.K to provide financial help during this research work. He also acknowledges Chris Hardy for her co-operation during AES analysis.

REFERENCES

(1) M.A. Islam. 2001. Intergranular Fracture in 2.25Cr-1Mo Pressure Vessel Steel at Low Temperatures. Ph.D. Thesis, The University of Birmingham, U.K.
(2) M.A. Islam, M. Novovic, P. Bowen and J.F. Knott. 2003. Effect of Phosphorus Segregation on Fracture Properties of 2.25Cr-1Mo Pressure Vessel Steel. Journal of Materials Engineering and Performance, ASM International. Vol. 12, No. 3, p.244-248.
(3) M.A. Islam, J.F. Knott and P. Bowen. 2004. Critical Level of Intergranular Fracture to Affect the Toughness of Embrittled 2.25Cr-1Mo Steels. Journal of Materials Engineering and Performance, ASM International. Vol. 13(5), p.600-606.
(4) C. Naudin, J.M. Frund and A. Pineau. 1999. Intergranular Fracture Stress and Phosphorus Grain Boundary Segregation of a Mn-Ni-Mo Steel. Scripta Mater. Vol. 40, p.1013-1019.
(5) Fracture Mechanics Toughness Tests, Part 1, Method for Determination of K_{IC}, Critical CTOD and Critical J Values of Metallic Materials. 1991. BS 7448. pp.2677-2710.
(6) J. Yu and C.J.Jr. McMahon. 1980. Effect of Composition and Carbide Precipitation on Temper Embrittlement of 2.25Cr-1Mo Steel: Part 1. Effect of P and Sn. Met. Trans. Vol. 11A, pp.277-289.
(7) R.C. Thomson and M.K. Miller. 1998. Carbide Precipitation in Martensite During the Early Stages of Tempering in Cr-and-Mo-Containing Low Alloy Steels. Acta Mater. Vol. 46, No.6, pp.2203-2213.
(8) M. Wada, K. Hosoi and O. Nishiwaka. 1982. FIM Observation of 2.25Cr-1Mo Steel. Acta Metall. Vol. 30, pp.1005-1011.
(9) R.C. Thomson and M.K. Miller. 1998. Carbide Precipitation in Martensite During the Early Stages of Tempering in Cr-and-Mo-Containing Low Alloy Steels. Acta Mater. Vol. 46, No.6, pp.2203-2213.
(10) J. Yu, and C.J.Jr. McMahon. 1980. Effect of Composition and Carbide Precipitation on Temper Embrittlement of 2.25Cr-1Mo Steel: Part 2. Effect of Mn and Si. Met. Trans.Vol. 11A, pp.291-300.
(11) King, J.E. 1979. Fracture Mechanisms in Embrittled Alloy Steel. Ph.D. Thesis. The University of Cambridge, UK.
(12) Hippsley, C.A. 1987. Effect of Environment on the Fatigue Crack Growth of Embrittled 2.25Cr-1Mo steel. Fatigue. pp.1249-56.
(13) J.F. Knott. 1973. Fundamentals of Fracture Mechanics. Butterworths Publication, London.