Microstructure Effect on Fracture of Stainless Steels

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Abstract: Duplex stainless steels specimens were heat treated at 475°C for different times and pulled to failure. Fracture toughness testing was performed according to BS 7448, , clip gauge , to monitor specimen displacement. In addition, the direct current potential drop(DCPD) technique was used to monitor the crack propagation. The Crack Tip Open Displacement (CTOD) was evaluated. Computational data, Shear model, were fit to the experimental ones. Discrepancy was observed between the experimental data and the computational ones. The model was able to expect the crack tip open displacement (CTOD), experimental data , only within a certain range of the material hardness i.e. microstructure . In addition, the direct current potential drop technique was more sensitive to detect the crack propagation process than that observed for the clip gauge. This work aims to study the fracture mechanism during cracking of duplex stainless steels.

Keywords: Stainless steels, Fracture toughness, 475°C embrittlement, CTOD, DCPD.

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1 Introduction

Duplex stainless steels (DSS) may be defined as a family of steels having a two phase, ferritic-austenitic or austenitic-ferritic, microstructure, the components of which are both stainless. They combine good properties of ferritic steels alloyed with chromium and nickel, which provide excellent resistance to pitting and stress corrosion, and ,in addition, from the mechanical point view a high degree of flexibility, resistance to fracture ,good tensile strength. Accordingly, duplex stainless steels are attractive materials for oil and gas applications, particularly offshore where there is the added complication of corrosion by seawater. Fracture of stainless steel parts can be contributed by their embrittlement which takes place during the process of thermal treatment. High-chromium stainless steels normally become harder when they are held for long periods of time at temperatures in the range of 400-500ºC i.e. 475ºC embrittlement. Embrittlement of duplex stainless at 475°C is accompanied by an increase in both the ferrite hardness and the ductile to brittle transition temperature [1]. Overall, the fracture toughness is reduced by the development of this phenomenon [2].

1.1 Fracture Toughness Evaluation:

The fracture toughness of a material is conventionally assessed in terms of the critical value of some crack tip field characterizing parameters (K_{IC}, J, CTOD) for unstable crack growth [3] or the value of those parameters at the beginning of stable crack growth [4] (K_{init}, Ji, CTODi). The crack tip open displacement for unstable crack growth (CTOD) may not a reliable parameter to measure the intrinsic material fracture toughness in age-hardened duplex stainless steels. That is due to large (CTOD) that could be obtained due to stable crack growth which cannot be detected without using crack monitoring techniques such as direct current potential drop (DCPD). The crack initiation toughness (CTODi), which characterizes the stable crack onset, is the parameter which best describes the intrinsic fracture toughness of the material. Fracture toughness testing of welds and ferritic steels in the brittle to ductile region often show a phenomenon called *pop-in* defined in the ASTM standard test method [5] as *discontinuity* in the load vs. clip gauge displacement record. The record of a pop-in shows a sudden increase in displacement and, generally, a decrease in load. Pop-in is a common feature of fracture testing in DSS [6]. A graphical procedure based on the elastic compliance change during pop-in may be used for the fracture data analysis . The standard BS 7448 [7] for fracture toughness assessment assess each pop-in , in the load vs displacement draw, separately. The load drop , dn%F , at the each pop-in is measured according to the following equation

$$d_n\%F_1 = 100[1-\frac{D_1}{F_1}(\frac{F_n - y_n}{D_n - x_n})]\% \qquad (1)$$

where n is the number of the considered popin i.e. 1st,2nd , 3rd etc. Yn and Xn are the resultant change in load and COD respectively. The other parameters in equation (1) are defined in the BS 7448 [7] . Pop-ins having load drop value less than 5% are ignored. If higher, the ratio $F_{max}/ F_{pop\text{-}in}$ is used to assess the validity $F_{pop\text{-}in}$ for the calculation of K_{IC}. If this ratio is higher than 1.1, the Fpop-in then is considered to be invalid for the calculation of K_{IC} and should be used for CTOD assessment instead. Fracture toughness testing can be performed using a clip gauge to monitor specimen displacement. In addition, the direct current potential drop (DCPD) technique is used to monitor the crack propagation. In this technique a constant D.C current passes across the tested specimen. An electric field is produced and distributed across the specimen material. As the crack propagates, the flow area is reduced which causes a change in the potential distribution. Crack propagation therefore gives a measurable change in the voltage measured across the crack. Good sensitivity could be obtained if the two inputs were located close to the cracking plane.

1.3 Fracture toughness modeling

Ritchie and Knott [8] studied the criteria of brittle fracture in mild steels. They proposed that unstable cracks propagate when the local tensile stress (σ_{yy}) exceeds a critical tensile stress value (σ_f) over a critical distance (X_0) determined as twice the grain size. This criterion can be expressed as follows;

$$\sigma_{yy} \le \sigma_f \quad \text{and} \quad X \le X_0 \qquad (2)$$

Curry [9] combined the model proposed by Ritchie and Knott [8] with the analysis of the stress field and obtained the relationship between fracture toughness (K_{IC}) and the critical distance for cleavage fracture as follows:

$$K_{IC} = \beta^{-\frac{(N+1)}{2}} X_0^{\frac{1}{2}} \frac{\sigma_f^{\frac{N+1}{2}}}{\sigma_y^{\frac{N-1}{2}}} \qquad (3)$$

where N and β are material constants .The critical tensile stress model was proposed [8] to apply for materials where inclusions and carbide particles serve as crack nuclei. Plastic deformation easily cracks those particles and fracture is a propagation-controlled process. This model is not applicable for duplex stainless steels since it is clean material with a few inclusions, and crack initiation is difficult i.e. fracture in DSS is a crack initiation controlled process. Accordingly the critical shear stress model for fracture of duplex stainless steels was more convenient [9]. In this model, the fracture criteria assume critical shear stress (τ_f) acting over a critical distance (X_0). That is

$$\sigma_{12} > \tau_f \quad \text{and} \quad x < x_0$$

(4)

and

$$K_{IC} = \beta^{-\frac{(N+1)}{2}} X_0^{\frac{1}{2}} \frac{\tau_f^{\frac{N+1}{2}}}{\sigma_y^{\frac{N-1}{2}}} \qquad (5)$$

$$CTOD_{init} = 0.717\frac{K_{IC}^2}{E\sigma_y} \qquad (6)$$

$$\tau_f = \tau_i + \tau_s \qquad (7)$$

and

$$\tau_s = \frac{G}{2\pi(1-\nu)}\sqrt{\frac{3.67b}{D}} \qquad (8)$$

Where

τi is the friction stress.

τs is the critical shear stress for crack nucleation.

G is the shear modulus.

ν is Poisson's ratio.

b is the Burger's length.

D is the length of dislocation pile-up.

2. Experimental Procedure:

The aim of these experiments was to investigate the interaction between the microstructure and the propagation of stable cracks i.e. the effect of microstructure on the fracture toughness (CTOD) of duplex stainless steels. In addition, the fracture mechanism was to be studied to determine if any transition took place.

2.1 Materials:

The as-received material was in the form of extruded bars solution heat treated at 1100ºC for 105min and water quenched. The material chemical composition is shown in table 1. Specimens from the as-received material were cut perpendicular to the bar axis. Two phases were present in the as-received microstructure with 50:50 ratio ; the ferrite phase and austenite phase. This microstructure was observed to be free of sigma phase and with a hardness of 258Hv. As illustrated in Fig.1 , specimens were machined according to BS 7448 [7] as straight notch compact tension (CT) specimens . In order to introduce a sharp crack in front of the notch tip, specimens were fatigued for a few millimeters. Specimens were then heat treated, to introduce brittleness to the ferrite phase, at475ºC for the following aging times2h ,5h ,13h,24h,49h,72h,166h,and118h.That was in order to obtain different levels of hardness. Finally, specimens were allowed to air-cool to room temperature.

Table 1 . The chemical composition of the as-received materials

Element	Wt%
C	0.02
Si	0.22
Mn	0.58
P	0.021
S	0.001
Cr	25.12
Mo	3.55
Ni	6.90
W	0.54
Cu	0.59
Fe	Bal

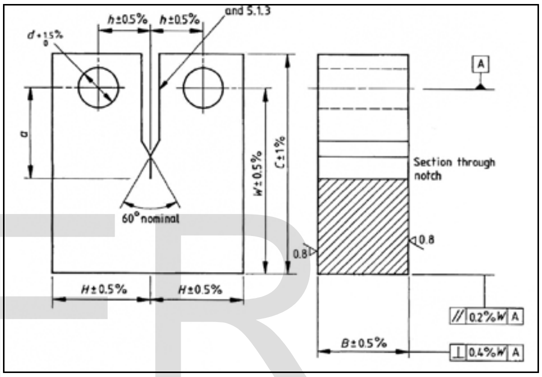

Fig1.Specimendimensionsforfracture toughness testing.(CT)

2.2 Fracture toughness testing :

Fracture toughness testing was performed according to BS 7448 using a clip gauge to monitor specimen displacement. In addition, the direct current potential drop (DCPD) technique, of constant current of 10A , was used to monitor the crack propagation. A millimeter was connected to monitor the potential, typically 1.36mV, at the test start across the specimen. During loading, the load vs. time and potential change across the specimen were recorded by a connected chart recorder. That was to determine the load when the DCPD was observed to change by a fixed amount, representative of a critical amount of crack propagation. Another chart recorder , connected to the clip gauge , was used to record the relationship between the load and COD during loading stage. Specimens finally were loaded to failure at 1mm/min cross head speed.

Data analysis was carried out according to BS 7448 [7] first for K_{IC} measurement validity . The CTOD value was calculated, in the case where data were invalid for K_{IC} measurement, according to the following [7]:

$$\mathrm{CTOD} = [\frac{F}{BW^{0.5}} f'(\frac{a_0}{W})]^2 [\frac{(1-\upsilon^2)}{2\sigma_{YS}E} + \frac{0.46(W - a_0)V_P}{0.46W + 0.54a_0 + (C - W) + Z}] \quad (9)$$

Where:

F the load at pop-in/fracture.

σys the yield stress.

E Young's modulus.

Vp the plastic component at the pop-in/final fracture considered.

B, a0, W and C as defined in Fig. 1

Z the Knife edges thickness.

ν Poisson ratio.

Poisson's ratio was taken as 0.3, E as 203 GPa. The yield stress (σys) was taken as the 0.5% proof stress. The force Fdcpd, at which a stable crack growth took place, was determined from load vs. dcpd chart obtained, considering the dcpd increase. The dcpd increase for the determination of F_{dcpd} was selected with sensitivity gives a crack growth increment of 1% . The voltage change equivalent to 1% increase in the crack length was calculated according to the following equation (ASTM E1290) [10] ;

$$\frac{V}{V_r} = A_0 + A_1(\frac{a}{W}) + A_2(\frac{a}{W})^2 A_3(\frac{a}{W})^3 \quad (10)$$

$$0.24 \leq \frac{a}{W} \leq 0.7 \quad (11)$$

Where;

V = the measured electric potential difference (EPD) voltage, Vr = the reference crack voltage corresponding to a/W = 0.241, a = the crack size, W = the specimen width, A0 = 0.5766, A1 = 1.9169, A2 = -1.0712 and A3 = 1.6898

Voltage change of 0.006±0.001mV was observed to be equivalent to 1% increase in the crack length. The values of the load F_{dcpd} and V_{dcpd} were determined in the load vs. displacement graph and the $CTOD_{dcpd}$ value was calculated using equation (9). The term Vdcpd is the plastic component of the equation . The tested specimens were of 0.5% proof stress in the range of 829-946 MPa. Fracture surfaces of three specimens of different proof stress i.e. 829,841, and 862 KN/mm2 were examined using Philips type (SEM) . That was in order to examine any transition in the fracture mechanism as the proof stress goes to a lower value. The fracture mechanism was expected to change from brittle fracture to ductile failure with decreasing hardness.

3 Results and Discussion :

The specimens hardness and yield stress were observed to increase with ageing time. This is may be attributed to 475ºC embrittlement which took place in ferrite. Specimen proof stress that was selected was for 0.5% strain, since the 0.1% and 0.2% proof stress showed significant scatter compared to the 0.5% proof stress. This scatter may be due to the early yielding and the work hardening of the softer austenite, giving a non-linear behavior at low strains. Near the crack tip, the critical tensile stress for crack propagation is already satisfied. Consequently, the cracking criterion is nucleation-controlled. That is by availability of nucleation sites ahead of the crack tip characterized by a critical distance, which in turn depends on ferrite grain size. The standard BS7448 [7] was followed to check specimen validity for fractured toughness assessment by direct determination of K_{IC}. In most cases, specimens were invalid for K_{IC} measurement as. The crack tip opening displacement fracture toughness (CTOD) was therefore used .The fracture toughness for stable crack growth (CTODi) decreased with increasing specimen proof stress Fig.(2). This may be understood as a result of 475ºC embrittlement as reported in literature. Dislocation movement is retarded by both factors increasing ferrite cleavage, and lower fracture toughness is predicted. The fracture toughness (CTODi) data showed agreement between the two techniques for higher proof stress Fig.(2). At lower yield stress, the two techniques showed discrepancy in the fracture toughness data. For proof stress value below 850MPa, higher CTOD values were observed using BS7448 [7] . The interpretation is that stable crack growth was detected by dcpd technique but not by BS744. This is since detection of stable crack growth by dcpd technique is subject to achievement of 1% increase in crack length, equivalent to 0.006±0.001mV voltage increase, regardless of specimen behavior during loading. Consequently, higher (CTODi) values using BS7448 technique are expected. For specimen aged for longer ageing times, higher proof stress, the CTOD data was in agreement using the two techniques. This may be because that ferrite cleavage , Fig.(3), is encouraged by embrittlement of the ferrite. As demonstrated, the tendency for ferrite cleavage increases with ageing time. This will encourage unstable brittle fracture. The two procedures are equivalent only when single pop-ins are assessed. This implies that CTODi measurement by dcpd monitoring produces a better measure of toughness in small specimens than BS7448 clip gauge method. In Figure 4 , the data obtained was fitted to the critical shear stress model for brittle fracture in duplex stainless steels. The elastic modulus value was 200GPa , $\beta = 0.59$ and N=13 taken from Rice and Johnson 11. The critical shear stress for nucleation (τ_f), depends on specimen yield stress, and was estimated from the data reported by Marrow et al. [12] . The critical distance, (X0) in equation (5) , was selected as20µm to fit the experimental data to the critical shear stress. As a physical meaning, the critical distance (X0) represents the availability of twins, for crack initiation, a head of the crack tip. The measured grain size, equivalent to the distance between the centers of two adjacent austenite grains, was 50µm. The experimental data was not in good agreement with the model. This implies that 20µm, as a critical distance (X0), is not a good value for fitting the experimental data to the critical shear stress model. As illustrated in Fig.2, the obtained fracture toughness data (CTODi) was in good agreement with the critical shear model only at relatively higher proof stresses i.e. proof stress higher than 850MPa. Below this proof stress value, the (CTODi) value obtained, either using BS7448 or dcpd technique, was higher than that predicted by the model. This is may due to that a transition in fracture mode which took place at/near this proof stress value. It is well documented that 475°C embrittlement may change the cracking mode from ductile failure to brittle cleavage [13]. Brittle fracture nucleation is related to deformation twinning. Below the proof stress of 850MP, higher (CTODi) value was not predicted by the critical shear stress model. This is attributed to the transition being not taken into account by the critical shear stress model. Below the transition, the critical tensile stress for crack propagation may be satisfied i.e. already exceeded near the crack tip, but not the critical shear stress for crack nucleation i.e. crack nucleation by deformation twinning is more difficult to satisfy than the condition for crack propagation. Brittle fracture of embrittled DSS can be modeled using the shear stress model only if ferrite fails by cleavage.

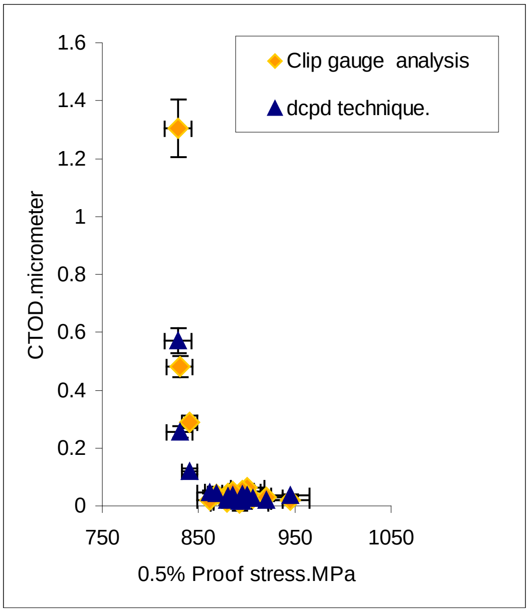

Figure2.The CTOD vs. yield stress observed for (CT) specimens tested for fracture toughness.

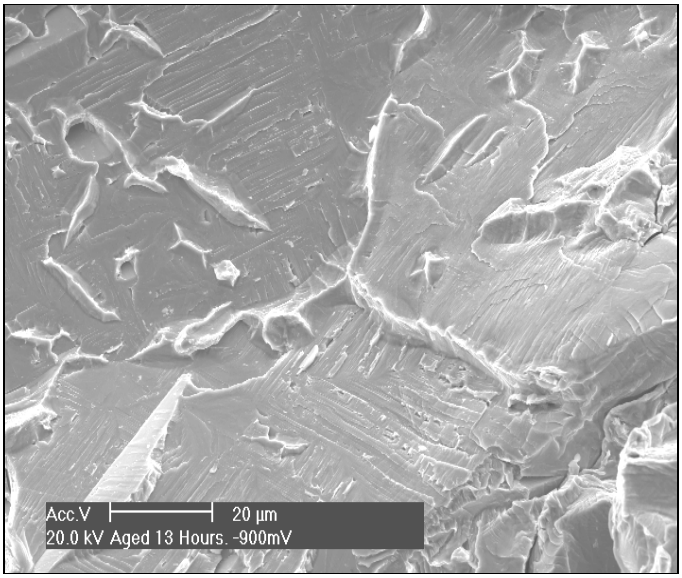

Fig 3 SEM for ferrite cleavage . Specimen aged for 13h at 475°C tested at $K_{applied}$ = 90MPa√m

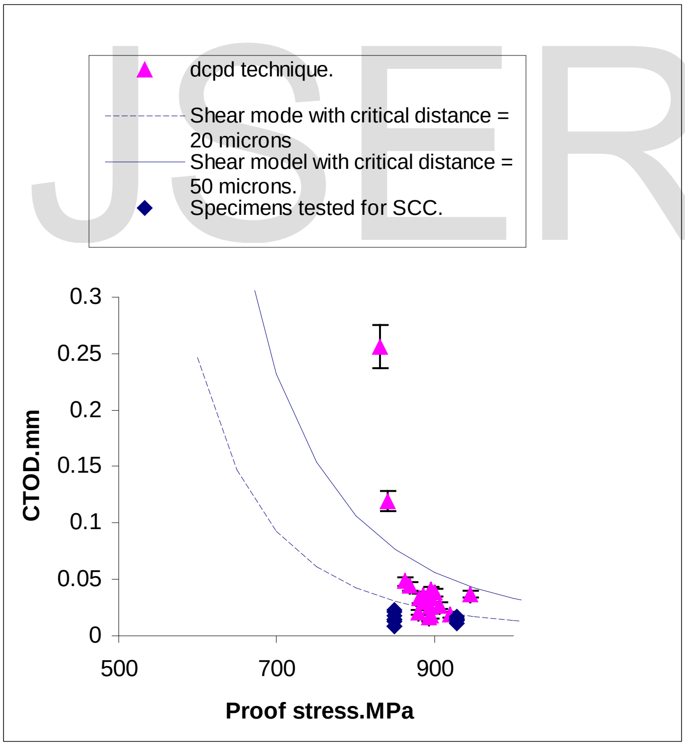

Fig 4. Measured fracture toughness compared to that predicted by critical shear stress model.

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