Mechanisms for cleavage fracture in duplex stainless steels

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Abstract Duplex stainless steels that have two phases with different mechanical and physical properties exhibit micro deformation heterogeneity and localized cracking or fracture in nature. In this study, the cleavage fracture behaviors in two super duplex stainless steels have been investigated in the following three conditions: at low temperatures, the material with spinodal decomposition and that with other precipitates and defects. By both impact toughness and fracture toughness tests, the ductile to brittle transition and cleavage behaviors have been investigated. The fracture is analyzed using a SEM with EBSD. The fracture mechanisms are focused; mainly the parameters or factors that cause the occurrence of cleavage in the materials. Since the austenitic phase in the material in these conditions is still tough, a coupling effect will be discussed to explain the cleavage in the austenitic phase observed. Local approach to fracture is used to assess or discuss the influence of local hardness, precipitates and stress concentration on the formation of cleavage fracture in the materials in different conditions.

Keywords Duplex stainless steel, cleavage, local approach method, spinodal decomposition, fracture

1. Introduction

Multiphase materials due to their microstructures and excellent properties are becoming more attractive for both engineering applications and academic interests. Duplex stainless steels (DSS) are a group of steels that consist of approximately equal volume of austenite and ferrite. Due to a good combination of excellent corrosion resistance and high mechanical properties, they are increasingly employed in various industries [1-4].

Duplex stainless steels have two phases with different mechanical and physical properties such as modulus of elasticity, yield strength and deformation hardening rate, and therefore exhibit micro deformation heterogeneity [5-13]. As a result, both stress and strain are not uniformly distributed at the phases and the actual load sharing on the microscopic scale is dependent on the property mismatch and microstructural features. It is believed that the difference in the elasto-plastic properties between the phases and the coupling effect, i.e., the load and strain sharing between the phases, is largely responsible for the varying elasto-plastic deformation mechanisms with varying plastic strain ranges in DSSs [8]. The phase-specific stresses, i.e., the total stresses that the constituent phases are subjected to is the sum of macrostresses, corresponding directly to the applied stresses, and microstresses due to micromechanical responses. The importance of micromechanical interactions under mechanical load has been recognized [8, 9]. Recently, the micro yielding and damage behavior of the austenitic and ferritic phases in duplex stainless steels have been studied by in-situ X-ray and neutron diffraction and multiscale modelling [8-13]. Fracture in duplex stainless steel is a local process in nature. Several works on local approach to fracture have been done to discuss the fracture in duplex stainless steels [14-17]. However, less

investigation has been done on the factors that affect the ductile to brittle transition behavior. In this paper, the influences of the factors such as temperature, ferrite content, cold deformation, phase size and different precipitates on the ductile to brittle transition and cleavage behaviors of a super duplex stainless steel are discussed with the purpose to increase the understanding on the micro fracture behaviors in duplex stainless steels and provide the information for the reliability and integrity to use the material.

2. Material and experimental

The material used is super duplex stainless steel (SDSS): UNS S32750 (Sandvik SAF 2507) with a nominal chemical composition as shown in Table 1:

Materials	C _{max}	Si	Mn	Cr	Ni	Mo	N	Dimension	Rp0.2
								(mm)	(MPa)
SAF 2507	0.03	0.8	1.2	25	7	4	0.3	φ260x14	595
Filler 25.10.4.L	0.02	0.3	0.4	25	9.5	4	0.24	φ2.4	537

Table 1 Nominal chemical compositions and information of DSS used

Two types of materials were used. One was a tube with an outer diameter of 260 mm and a wall thickness of 14mm. The other type of material was all-weld metal produced with TIG-welding using super duplex filler Sandvik 25.10.4.L. The welding was carried out in a V-groove using 13mm plates in SAF 2507 with 10° beveling and a back strip 6 mm in thickness in SAF 2507. Distance between the plates was 14 mm. Arc energy varied between 0.69 and 0.82 kJ/mm and the shielding gas consisted of Ar + $2\%N_2$. Totally 32 runs were used to build up the all-weld metal. Interpass temperature was below 150°C. Figure 1 shows the typical microstructure of the base material (Fig. 1a) and the weld metal (Fig. 1b).



Figure 1 Typical microstructure of SAF 2507; (a). Base material, X200, (b). Weld metal.

Two types of toughness tests (impact toughness and CTOD) have been performed in a temperature range from -196°C up to room temperature (RT). Two to three samples/temperature were tested and

an average value was used in the toughness versus temperature or DBT curves. For impact toughness testing, standard sample with a dimension of 50x10x10mm (SS-EN 10045-1) was used. For CTOD sample, SENT type of sample with a dimension of 90x20x10mm was used and tested according to BS 7448, 1991.

In this paper, two methods have been used to determine a ductile to brittle transition temperature (DBTT). One is to determine the transition temperature, T_{50} , by Eq. 1 with 50% probability from a ductile to brittle transition curve [18, 19].

$$KV = \frac{\left(KV_{\max} - KV_{\min}\right) \times \exp\left(\frac{2 \times (T - T_{50})}{C}\right)}{1 + \exp\left(\frac{2 \times (T - T_{50})}{C}\right)} + KV_{\min}$$
(1)

Where KV is the toughness at temperature T, KV_{max} is the maximum toughness, KV_{min} is the minimum toughness, and T_{50} is the temperature with 50% probability of brittle fracture, C is a constant.

Another is though fracture analysis using a scanning electron microscopy (SEM). DBTT is the temperature where no cleavage could be observed on the fracture of an impact sample. In this investigation, no cleavage could be observed on the fracture of the sample with impact toughness higher than 90 joules. Therefore, T_{90J} is another definition of DBTT for this material.

In order to study the influence of factors on cleavage behaviors in DSS, some special tests were arranged. One is the effect of spinodal decomposition. The samples were aged at 450°C for up to 300 hours. The second is the effect of intermetallic phase, mainly sigma phase. The samples were tempered at 850°C for up to 10 minutes. The third is the effect of hydrogen or hydrogen induced stress cracking, HISC. The experimental details are described in reference [21]. The effect of other defects such as inclusion and Al nitride were also investigated.

3. Results and discussion

3.1 Influence of temperature on DBT behavior

Figure 2 shows the DBT curves from the CTOD and the impact toughness tests. As expected, both CTOD and impact toughness decrease with decreasing temperature. The modeling curves using Eq. 1 with C=35 were used to determine T_{50} and T_{90J} . Table 2 shows a summary of the influences of the factors such as amount of ferrite, cold deformation and phase size on the DBTT. The influence of phase size on the DBTT is relatively small. This result is quite different from that of the weld material where the DBTT (T_{27J}) increases significantly with increasing grain size [20]. An increase in ferrite content will raise the DBTT. This is due to the fact that it is the ferrite that causes a DBT. Cold deformation that introduces dislocations in the material can cause a decrease of impact toughness and consequently increase the DBTT since cold deformation increases both the strength of the material and the density of dislocation, which will promote the tendency for cleavage [21].



Figure 2 DBT behavior of UNS S32750 SDSS, (a). CTOD (b). Impact toughness.

DBTT	Reference	Ferrite content (%)			Cold deformation (%)				Austenite spacing (µm)		
(°C)		45	61	68	0	5	10	15	18	23	32
T ₅₀	-86	-74	-58	-41					-74	-76	-72
T _{90J}	-100	-92	-74	-54	-85	-76	-58	-52	-92	-94	-90

Table 2 Influences of the different factors on DBTT

As known, the tendency for cleavage in the ferritic phase can increase when temperature decrease [22]. At -75°C, isolated cleavage fracture can be observed (Fig. 3a). It is in the transition regime as shown in Fig. 2. At -130°C, cleavage fracture is now dominant (Fig. 3b). It seems that brittle fracture could have also occurred in the austenitic phase. As discussed in [15, 16], the ferrite that has a BCC structure obeys the weakest link theory and shows a brittle fracture. For the austenitic phase, the weakest link theory can not be applied, but it follows the coupling effect. The effect of crack front length on fracture toughness is now practically absent when the hardness of austenitic phase is high enough. A sharp cleavage crack in the ferrite can lead to a stress concentration that can be higher than the critical shear stress for a cleavage fracture in the austenitic phase. In this investigation, quasi-cleavage or cleavage fracture can be observed in the austenitic phase. The cleavage in the ferrite shows multiple lines, not converge but are vertical to single big line like a "river" (Fig. 3c). The cleavage in the "river" may nucleate at some nuclei (Fig. 3c). A small cracking inclusion can be one of them (Fig. 3d).



Figure 3 Influence of temperature on fracture in duplex stainless steel, (a). At -75°C, impact toughness 46J, (a). At -130°C, impact toughness 14J, (c). Enlarged cleavage fracture at -75°C, (d). Enlarged cleavage in weld metal at -100 °C, a cracking inclusion is a Griffith crack.

3.2 Influence of cluster on DBT behavior

Duplex stainless steel can suffer from a spinodal decomposition at temperatures between 300-500°C where the ferritic phase undertakes a miscibility gap that gives rise concentration variations with Fe-rich (α) and Cr-rich (α ') regions (Fig. 4a), which leads to the formation of Fe-rich and Cr-rich clusters or phases [1, 2, 5]. Spinodal decomposition leads to an increase in the hardness of the ferritic phase, but not the austenitic phase (Fig. 4b). It was found that an increase in hardness of the ferritic phase promotes the occurrence of cleavage or DBT (Fig. 4c). Using Eq. 1, the hardness at T₅₀ can also be determined as shown in Fig. 4d. This type of curve provides useful information for a quick evaluation of the influence of spinodal decomposition on the brittleness of the material.

The fracture structure in Fig. 4c is different from that in Fig. 3b. In Fig. 4c, the ferritic phase has a cleavage fracture, but not the austenitic phase. The austenitic phase is still ductile, and dimples can be observed. This indicates that the stress concentration at the cleavage crack in the ferritic phase is not high enough to initiate a quasi-brittle fracture as that in Fig. 3b since the fracture toughness in the austenitic phase is still high enough. The above discussion shows that local approach to

fracture should be considered when a fracture in duplex stainless steel is involved, but the mechanical behaviors of both phases in the considered environment should be considered.



Figure 4 Influence of spinodal decomposition on DBT of DSS, (a). Formation of cluster, (b). Influence of spinodal decomposition on the hardness of individual phases, (c). Fracture, (d). Correlation between hardness and impact toughness; α is the ferritic phase or Fe rich cluster, γ is the austenhitic phase, α' is the Cr rich cluster or phase.

3.3 Influence of precipitates and other defects on DBT behavior

Classically, a second phase particle induced cleavage is treaded as the formation of cracks or Griffith cracks in the particle because of plastic strain in the surrounding matrix. If the stress ahead of the crack is sufficient, it will propagate into the matrix, causing failure by cleavage [21]. For duplex stainless steels, the following cases can be considered. It was found that the influences of different precipitates on the DBT or cleavage behaviors are different. Figure 5 shows the force versus time curves from an instrumental impact toughness testing for SAF 2507 material with different conditions. For the as received material and the weld joint, both the crack initiation and propagation energies are high, which lead to high impact toughness. For the material containing small amount of Al nitrides, the crack initiation energy is high, the propagation energy is also high, but lower than that the as received and welded material. This indicates that crack propagation rate in this case can be higher.

For the material containing sigma phase, both the crack initiation and propagation energies are low, which lead to a low impact toughness.



Figure 5 Force versus time curves from the instrumental impact toughness testing for SAF 2507 material with different conditions

The different cleavage behavior from different precipitates depends on the nature of precipitates and fracture mechanisms. Sigma phase has a P4₂/mnm structure and is brittle. They precipitate mainly along grain or phase boundaries. These particles become easily cracking during plastic deformation. The cracking usually occurs usually through the particle (Fig. 6a). These sharp microcracks become then stress raisers or Griffith cracks that provide a local stress concentration that initiate cleavage fracture in the ferritic phase (Fig. 6b). High amounts of sigma phase will increase number of microcracks or Griffith cracks formed during plastic deformation, the hardness of the ferritic phase is also increased. This leads to an easier cleavage fracture with smaller cleavage plans (Fig. 6c).



Figure 6 Influence of sigma phase on cleavage in duplex stainless steel at RT. (a). Cracking of sigma phase, (b). Cleavage fracture in the material with about 1% sigma phase, (c). Cleavage fracture in the material with about 10% sigma phase.

As shown in Fig. 5, the presence of small amount of Al nitrides has a small effect on the impact toughness comparing with sigma phase. However, at low temperature, Al nitride can also initiate cleavage fracture as shown in Fig. 7a. This does not happen for an Al oxide that is round and has a larger size (Fig. 7b). The results show that needle type of Al nitride can create a stress concentration that is higher than the critical stress for cleavage fracture in the ferritic phase. The morphology of the second particle can also play an important role to cleavage fracture.



Figure 7 Influence of precipitate and defect on cleavage in duplex stainless steel. (a). Al nitride, (b). Al oxide at -130°C.

Recently, hydrogen induced stress cracking (HISC) in duplex stainless steel is concerned [22]. Diffusion of hydrogen into the material can lead to an increase in hardness, and on the other hand hydrogen can accumulate at phase and grain boundaries, which causes the formation of hydrogen pore or voids [22], and consequently the formation of stress concentration. When the hydrostatic pressure in the void reaches a critical value or a critical shear stress, the void can develop into a small crack or Griffith crack, and eventually cleavage initiation (Fig. 8a). Cleavage fracture propagates discontinuously due to the austenitic phase that acts as a hinder for crack propagation, which creates an unusual top-valley fracture (Fig. 8b). The cracks in the ferritic phase are mainly classical brittle cleavage. The herringbone pattern of fracture with the cleavage plane of {100} and two growth directions from <110> is the most common one (Fig. 8c). The situation here is similar to that due to the spinodal decomposition as discussed above.



Figure 8 Cleavage initiation and propagation in hydrogen enriched duplex stainless steel, γ –austenite, α -ferrite.

The results and discussion above indicate that fracture initiation and propagation in duplex stainless steel are local processes but also have coupling effects. They can behave very differently in different environments. Local approach to fracture should be applied by considering the heterogeneous mechanical behaviors in these two phases.

4. Concluding remarks

Duplex stainless steels have heterogeneous mechanical and fracture behaviors in the ferrit and austenitic phases. The cleavage behavior of the material depends strongly on the microstructure and environments. Influences of small variations of the microstructure such as phase size, ferrite content and cold deformation on the DBTT are small. Spinodal decomposition and precipitates can significantly raise the DBTT. The cleavage propagation in duplex stainless steel can be discontinuous since the austenitic phase behaves as a stopper.

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