The fracture mechanisms in duplex stainless steels at sub-zero temperatures

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Abstract

The aim of the thesis was to study the susceptibility for brittle failures and the fracture process of duplex stainless steels at sub-zero temperatures (°C). In the first part of the thesis plates of hot-rolled duplex stainless steel with various thicknesses were used to study the influence of delamination (also known as splits) on the fracture toughness. The methods used were impact and fracture toughness testing. Light optical microscopy and scanning electron microscopy were used to investigate the microstructure and fracture surfaces. It was concluded that the delaminations caused a loss of constraint along the crack front which resulted in a stable fracture process despite the presence of cleavage cracks. These delaminations occurred when cleavage cracks are constrained by the elongated austenite lamellae. The pop-in phenomenon which is frequently observed in duplex stainless steels during fracture toughness testing was shown to occur due to these delaminations. The susceptibility for pop-in behaviour during testing increased with decreasing plate thickness. The toughness anisotropy was also explained by the delamination phenomenon.

In the second part of the thesis duplex stainless steel weld metals from lean duplex and super duplex were investigated. For the lean duplex weldments with different nickel contents, tensile, impact and fracture toughness testing were conducted from room temperature to sub-zero temperatures. The result showed that increased nickel content decreased the susceptibility for critical cleavage initiation at sub-zero temperatures. The super duplex stainless steel weldment was post weld heat treated. The fracture sequence at low temperature was critical cleavage fracture initiation after minor crack-tip blunting and ductile fracture. Energy-dispersive X-ray spectroscopy investigation of the weld metals showed that substitutional element partitioning is small in the weld metal. However, for the post weld heat treated weldments element partitioning occurred which resulted in decreased nickel content in the ferrite.

Keywords: Duplex stainless steel; Weldments; Delamination; Fracture toughness; Impact toughness; Cleavage fracture; Nickel
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List of appended papers


Author contribution

Paper A
The author of this thesis made the fracture mechanical experiments, performed the fractographic study, examined the microstructure, evaluated the results and wrote the manuscript.

Paper B
The author of this thesis made the fracture mechanical experiments, performed the fractographic study, examined the microstructure, evaluated the results and wrote the manuscript. The impact toughness testing was performed by Outokumpu Avesta Research Centre.

Paper C
The author of this thesis performed the EBSD study, evaluated the results and wrote the manuscript.

Paper D
The author of this thesis made the fracture mechanical experiments, performed the fractographic study, examined the microstructure, evaluated the results and wrote the manuscript. The tensile testing was performed by P. Casarotto and the impact toughness testing was performed by Outokumpu Avesta Research Centre.

Paper E
The author of this thesis made the fracture mechanical experiments of the weldment. H. Sieurin performed the fracture mechanical experiments on the base metal. The author
performed the fractographic study, examined the microstructure, evaluated the results and wrote the manuscript. The EBSD study was performed by Sandvik Materials Technology.
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1 Introduction

Duplex stainless steel (DSS) is defined as consisting of the two crystallographic phases ferrite (\(\alpha\)) and austenite (\(\gamma\)). The phase composition can vary greatly with the type of production, i.e. cast, wrought or welded steel and on the requirements and type of application. Optimum phase composition for mechanical and corrosion properties is a 50-50 phase composition for wrought materials. The major alloying elements are chromium, manganese, nickel, nitrogen and molybdenum. The chromium, molybdenum and nitrogen are added for the corrosion resistance while the nickel and manganese are added for austenite stability and thus giving the duplex phase structure [1]. Tungsten and copper addition for further corrosion resistance can also be found in certain DSS grades [1]. The advantage of mixing ferrite and austenite is that the resulting material has good retained properties from the two phases. Two examples are the resistance to stress corrosion cracking in the ferrite and the high toughness in the austenite [2].

The first commercial DSS product dates from the late 1920s [2]. For the following 50 years the interest for this type of steel was lukewarm [3]. This was partially due to the requirement of having very high control of the chemical composition and production processes for avoiding the precipitation of detrimental phases [3]. Another issue was the high ferrite content in the heat-affected zone (HAZ) which resulted in low toughness and corrosion resistance [4]. During the late 1960s and early 1970s the vacuum and argon oxygen decarburisation (VOD and AOD) processing techniques were introduced which made it possible to produce steel with more narrow composition limits and having lower carbon, sulphur and oxygen content [2, 3]. At the same time the volatility of the nickel price called for development for alternative to the austenitic stainless steels and the offshore oil industry demanded stainless steels suitable for seawater applications [2, 3]. This resulted in advancement of the duplex stainless steel and in the beginning of the 1980s the second generation duplex stainless steels were introduced with improved weldability, which led to the breakthrough for the DSS. Today the duplex family range from lean duplex (lower molybdenum and nickel content) to the highly alloyed super and nowadays also hyper duplex stainless steels. The duplex stainless steels represent about 1 % of the stainless steel market and the use of DSS grows rapidly [5]. The entire stainless steel market represents about 1 % (in mass) of the total steel market [5].

The DSS are often used in applications that require high corrosion resistance and mechanical strength. The main applications for the DSS are found in the petrochemical, storage, transportation and the chemical process industry [5]. Nowadays there is an increasing use of duplex stainless steel for other structural applications due to the maintenance cost savings that come with the corrosion resistance [5]. The lean duplex grades with lower nickel and molybdenum content give a competitive cost and are anticipated to be increasingly used for structural applications [6].

With decreasing temperature, the fracture mechanism for ferrite goes from ductile with large plastic deformation to brittle with low fracture deformation. The shift from one fracture mechanism to the other is called the ductile to brittle transition, see Figure 1. The cause for
this change in fracture behaviour for the ferrite has been explained by the inability for cross-slip to occur at lower temperatures [7, 8, 9, 10]. The inability for cross-slip is likely explained by the higher temperature sensitivity for the Peierls stress in the ferrite due to the body-centred cubic crystal structure [11]. The duplex stainless steel with its ferrite content has therefore a ductile to brittle transition temperature (DBTT) region. The temperature for the transition from ductile to brittle fracture depends on the chemical content, microstructure, existence of detrimental phases and precipitations. Design against brittle failure is an important concept for structural applications and for the DSS there has previously been reservation against full utilisation of DSS at sub-zero temperatures due to lack of knowledge and technical experience. However, modern design rules have now been developed and implemented in the European Pressure vessel Code EN 13445 [12, 13, 14]. This allows the use of DSS down to low temperatures if appropriate welding methods are used.

![Diagram showing ductile to brittle transition in ferritic steels](image.png)

**Figure 1.** The ductile to brittle transition in ferritic steels [11]. The plateau region at lower temperatures is called the lower shelf while the plateau region at higher temperatures is called the upper shelf. The temperature region between these two plateaus is the transition region.

### 1.1 Aim of work

Fracture mechanical testing shows that both the base metal and weldments of DSS can have quite satisfactory fracture toughness at sub-zero temperatures [15, 16, 17, 18, 19, 20, 21, 22]. For hot-rolled plates of DSS a delamination phenomenon, also known as splits, occurs for the base metal during fracture and impact toughness testing. This delamination phenomenon has not received much attention in previously published works. The purpose of the first part of the work was to gain better understanding on how the delamination phenomenon in the base metal affects the fracture behaviour and the evaluated fracture toughness. The cause for the delamination of hot-rolled DSS was also covered.

The nickel content is very important for the toughness of DSS. The nickel addition increases the austenite phase content [2, 23] and the toughness of the ferrite phase [7, 9]. Filler metal with enhanced nickel content is usually used when welding DSS. The increased nickel content helps the austenite formation during cooling so higher austenite phase composition can be achieved with much reduced risk for detrimental phases and
precipitations that come with slower cooling times [2]. Since nickel is an expensive alloying element, it is desirable to use as little as possible. The purpose of the second part of the work was to examine the influence of the nickel content on the weld metal susceptibility for unstable fracture. The examination of the fracture toughness of a super duplex stainless steel was also included.
2 Fracture toughness

The fracture toughness is the materials resistance to fracture in the presence of a sharp crack. There are various definitions for the fracture toughness, e.g. $K_I$ and $J_I$ and in the ideal case it is a material property independent of shape and size of the cracked body. More of this in the subsequent chapters.

The two most common specimen types for measuring fracture toughness are the compact tension (CT) and the single-edge notched bend bar SE(B), see Figure 2a and b. Both of these two types are practically and economically feasible and most importantly exhibit high constraint along the crack front. The advantage of the CT-specimen is the low material consumption; the drawbacks are higher cost for specimen manufacturing, more demanding set-up (specimen alignment and contact issues between the loading pins and the specimen).

For the SE(B)-specimens the advantages are lower cost for specimen manufacturing and easier experimental set-up but the drawback is the much higher material consumption for the specimens.

The specimens contain a notch which ends in a sharp crack which for metallic materials consists of a fatigued pre-crack as shown in Figure 2a.

![Figure 2](image.png)

**Figure 2.** The two commonly used specimen types for fracture toughness measurement, a) compact tension [24], b) single-edge notched bend bar [25].

To measure the fracture toughness two physical quantities are recorded, the force on the specimen and the displacement of the specimen. The force on the specimen is measured by a load cell mounted on the test rig. The specimen displacement is nowadays most common measured as the crack-mouth opening displacement by a clip-gauge, see Figure 2.

The specimen is subjected to a constant displacement speed (m/s) until unstable fracture occurs (either by total failure of the specimen or the fracture arrests, i.e. pop-in occurs). If the
material is expected to be ductile (no unstable fracture) the test is stopped after sufficient

\[ \text{crack growth. The crack extension during the testing can in that case be measured by the} \]

compliance method \([26]\), the potential drop method \([27]\) or by optical measuring. The

specimen is usually loaded in mode I, see Figure 3.

2.1 The stress intensity factor \(K\)

A crack-tip in an arbitrary body is shown in Figure 4. If one assumes isotropic linear elastic

material behaviour for the body it can be shown that the stress field around the crack-tip

(polar coordinates) can be described as:

\[
\sigma_{ij} = \left( \frac{k}{\sqrt{r}} \right) f_{ij}(\theta) + \sum_{m=0}^{\infty} A_m r^m g_{ij}(\theta) \quad [\text{Pa}]
\]

where \(\sigma_{ij}\) is the stress tensor, \(r\) is the distance to the point of interest, \(\theta\) the angle to the point

of interest, \(k\) is a constant and \(f_{ij}\) is a dimensionless function of \(\theta\). The higher order terms after

the addition sign depends on the geometry of the body and can be considered to take into

account remote boundary conditions.

Figure 4. Definition of the coordinate axis ahead of a crack-tip \([28]\).
The stress field close to the crack-tip can be written as:

\[
\lim_{r \to 0} \sigma_{ij} = \left(\frac{k}{\sqrt{r}}\right)f_g(\theta) + \sum_{m=0}^{\infty} A_m r^{-\frac{m}{2}} g^{(m)}(\theta) = \left(\frac{k}{\sqrt{r}}\right)f_g(\theta) \quad \text{[Pa]} \tag{2}
\]

When \( r \) approaches 0 the leading term in equation (1) goes to infinity whereas the higher order terms remain finite or go to 0. Thus, the governing term for the stress field is \( 1/\sqrt{r} \) close to the crack-tip, i.e. a stress singularity exists in the crack-tip vicinity, see Figure 5.

If one substitute \( k \) with \( K = k\sqrt{2\pi} \) in equation (2) and rearrange the terms, the stress intensity factor (SIF) \( K \) can be defined as:

\[
K_I = \lim_{r \to 0} \sigma_{ij} \sqrt{2\pi r} f^{-1}_g(\theta) \quad \text{[Pa}\sqrt{\text{m}]} \tag{3}
\]

A very important feature of equation (3) is that the stress and displacement field around the crack-tip is fully characterized by the stress intensity factors and they always have the same distribution as long as the singularity zone is dominating [28, 29]. This is one of the most important concepts in what is called linear elastic fracture mechanics (LEFM) [28]. Outside the singularity zone, equation (3) quickly loses accuracy and the higher order terms in equation (1) start to be dominating.

\[\text{Figure 5. The stress distribution normal to the crack plane in mode I [28].}\]

Equation (3) can be rewritten in the general form (mode I):

\[
K_I = \sigma_{app} \sqrt{\pi a} f \quad \text{[Pa}\sqrt{\text{m}]} \tag{4}
\]

where the \( \sigma_{app} \) is the applied stress to the body, \( a \) is the crack length and \( f \) is the geometrical function that depends on the geometry of the body and crack. The \( f \) function can be
analytically found for simpler geometries. More complicated and realistic geometries are nowadays analysed with finite element analyses (FEA).

Very few materials exhibit a perfect elastic behaviour and for most real materials, non-linear deformations (crack-tip blunting, plastic deformation and crack growth) occur at the crack-tip vicinity under the impact of high stresses \[29\]. It can be shown however, that if the non-linear deformation (i.e. the plastic zone) around the crack-tip is small enough to be contained by the singularity zone, the outer boundary conditions influence on the crack-tip can be included in the singularity zone. Thereby can the SIF still be used to characterise the crack-tip conditions even though the actual crack-tip condition is unknown \[28, 29\]. This is called small scale yielding (SSY) and is an important concept in LEFM. SSY is illustrated in Figure 6.

![Figure 6](image)

**Figure 6.** The concept of small scale yielding (SSY). The conditions at the crack-tip should be the same for the test specimen and the structure as long as the plastic zone is small compared to all relevant dimension \[28\].

The size of the plastic zone can be estimated with the assumption that the boundary between the plastic zone and the singularity zone occurs when a yielding criterion is satisfied. Consider the stresses directly ahead of the crack-tip (\( \theta = 0 \)) and assume that the material is perfectly plastic with a yield strength \( \sigma_{YS} \) \[11, 28, 29\]. Then from dimensional consideration one can conclude from equation (3) that the size of the plastic zone, \( r_p \), takes the form:

\[
    r_p \propto \left( \frac{K_I}{\sigma_{YS}} \right)^2 \quad [m]
\]

(5)

The size of the plastic zone depends also on the stress state (plane stress or plane strain), the angle \( \theta \) and the model used for describing the stresses in the crack-tip region \[11\].

2.1.1 The linear elastic plane-strain fracture toughness $K_{Ic}$

The fracture toughness parameter $K_{Ic}$ characterises the resistance of a material to fracture in the presence of a sharp crack during SSY conditions and with the crack front in plane-strain stress condition [26]. A valid $K_{Ic}$ result is a material property independent of the size and geometry of the cracked body. LEFM may still be valid if plane-strain is not present but the measured fracture toughness depends on the size or geometry of the cracked body [28].

The experimental procedure for determine the $K_{Ic}$ can be briefly summarised by the following steps:

- The specimen, e.g. a CT or SE(B), is fatigued for creating a sharp crack. The total crack length (notch + fatigue crack) over the specimen width ($a/W$) has to satisfy the condition (6).

$$0.45 \leq a/W \leq 0.55 \quad [m/m]$$

The fatigue crack also has to fulfil certain restrictions on straightness and maximum used SIF during the fatigue cracking.

- The specimen is loaded until maximum load is reached. A secant line is constructed with 95 % of the slope from the initial linear load-displacement. The intersection with this secant line and the load-displacement curve is denoted $P_Q$ and is assumed to be the critical load [26, 28, 29]. In Figure 7 three results for the loading is shown. For type I the load-displacement curve deviates from linearity until maximum load is reached. This non-linearity can be due to plasticity, crack growth or both [28]. For type II a limited unstable crack extension occurs prior to the maximum load, this is called a pop-in. For type III the load-displacement curve is more or less linear until the maximum load. Regardless of which type of the load-displacement curve the relation between $P_Q$ and $P_{max}$ is constrained by equation (7) for valid $K_{Ic}$ result.

$$P_{max} \leq 1.10P_Q \quad [N]$$
The tentative fracture toughness, $K_Q$, is qualified as $K_{IC}$ if the requirements of equation (6) to (8) are fulfilled [26].

$$B, b_0, a, W \geq 2.5 \left( \frac{K_Q}{\sigma_{YS}} \right)^2 [m]$$

In equation (8), $B$ is the specimen thickness, $W$ is the specimen width, $a$ is the initial total crack length and $b_0$ is the remaining ligament ($W-a$). If these requirements are not met the specimen thickness may be increased for increasing the constraint at the crack front.

2.2 The J-integral (this section is mainly based on reference [28])

As stated in the previous section: when the plastic zone is not sufficiently small for SSY to be valid, the stress intensity factor does not fully characterise the crack-tip conditions. Increasing the specimen size (recall equation (8)) for metals with high ductility quickly results in specimen sizes that are neither practically nor economically feasible. Another approach is needed for characterising the fracture toughness for materials that exhibit elastic-plastic behaviour.

If one considers the stress-strain behaviour of monotonically loaded nonlinear elastic and elastic-plastic material the two curves are identical if proportional loading occurs, see Figure 8. Relating the stress to the total strain in an elastic-plastic material to nonlinear elasticity is called deformation theory of plasticity.
Rice published an article in 1968 where he used the deformation theory on a crack in a nonlinear body [30]. With a line integral related to energy, which he defined as $J$, he showed that the value of $J$ is equal to the energy release rate in nonlinear materials and that the integral is path independent. It was later shown that by using a power law relationship between stress and strain that the $J$-integral characterise the crack-tip conditions in a nonlinear elastic material. The $J$-integral can thereby be used as a single-parameter for the fracture toughness as long as the crack-tip is in $J$ dominance (similar to the singularity zone in LEFM). The line integral $J$ can be defined as:

$$J = \int_{\Gamma} \left( \left( w \frac{\partial u_i}{\partial x} \right) ds \right) \quad [N/m]$$

where $w$ is the strain energy density, $T_i$ are components of the traction vector, $u_i$ are components of the displacement vector and $ds$ is a length increment along the contour $\Gamma$ defined in Figure 9. The strain energy density is defined as:

$$w = \int_{0}^{\varepsilon_j} \sigma_{ij} d\varepsilon_{ij} \quad [N/m^2]$$

where $\sigma_{ij}$ is the stress tensor and $\varepsilon_{ij}$ the strain tensor.
The traction is the stress vector normal to the contour and the traction components are given by:

\[ T_i = \sigma_n n_j \quad [\text{Pa}] \]  

where \( n_j \) are the components of the unit vector normal to \( \Gamma \). Experimental measurement following equation (9) can be made by attaching strain gages around the crack-tip. However, this approach is practically cumbersome. From the relationship with the energy release rate in a nonlinear elastic body containing a crack a more suitable expression for the J-integral can be found [28]:

\[ J = J_{el} + J_p = \frac{K(P_i, a_0)^2}{E} + \frac{\eta_{pl} A_{pl}}{B_N b_0} \quad [\text{N/m}] \]  

where \( J_{el} \) is the elastic part of \( J \) and \( J_p \) is the plastic part. \( K \) is the LEFM stress intensity factor at the measured point and \( a_0 \) is the original crack length. \( E' \) is Young’s modulus depending on plane-stress or plane-strain condition. \( \eta_{pl} \) is a dimensionless factor that depends on the specimen geometry, \( A_{pl} \) is the plastic work done (the area under the load-displacement curve minus the elastic part). \( B_N \) is the net specimen thickness and \( b_0 \) is the distance from the crack front to the back of the specimen. Equation (12) is used when crack growth is not considered. If crack growth is accounted for, the expression has to be modified for continuous adjustments of the growing crack:

\[ J(i) = \frac{K(P_i, a_i)^2}{E} + J_{pl}(i) \quad [\text{N/m}] \]  

For plane strain SSY conditions the \( K_{k} \) and critical J at the onset of unstable fracture or ductile initiation fracture (\( J_c \) or \( J_k \), see the following chapters) are related by the following equation:
\[ K_{lc} = \sqrt{\frac{J_{\text{critical}}E}{1-\nu^2}} \quad \text{[Pa}\sqrt{\text{m}]} \]  

(14)

where \( \nu \) is Poisson’s ratio. \( K_{lc} \) from equation (14) can be viewed as \( K_{lc} \) measured from a sufficiently large specimen.

When excessive plasticity or crack growth occurs the process zone around the crack-tip becomes significant in relation to the size of the body. The J dominance zone gradually becomes invalid due to body boundary interaction with the crack-tip. When this occurs, the process zone around the crack-tip is no longer uniquely characterised by J and thus single-parameter fracture mechanics is invalid. Critical J values then exhibit a size and geometry dependence.

### 2.2.1 The fracture instability toughness \( J_c \)

For materials where the unstable fracture (e.g. cleavage) is preceded by significant plastic flow or crack growth, e.g. in the ductile to brittle transition temperature region for ferritic steels, the LEFM is not suitable to characterise the fracture toughness. The size requirement for LEFM, equation (8), was to limit non-linear material effects in the vicinity of the crack-tip. For the J-integral non-linear plastic deformation is taken into account. However, the non-linear elastic theory assumes small strains at the crack-tip which is a simplification. Large deformation at the crack-tip becomes significant in a zone of the same size as the crack-tip opening displacement. The size requirement for the J-integral is therefore to limit non-linear geometry effects rather than non-linear material effects as in LEFM [29]. The size requirement is [26]:

\[ B_b \geq 100 \frac{J_Q}{\sigma_Y} \quad \text{[m]} \]  

(15)

where \( J_Q \) is the tentative critical J and \( \sigma_Y \) is the effective yield strength (average of the 0.2 % offset yield strength and the ultimate tensile strength). By using equation (8) with equation (15) it can be seen that the size requirement for the non-linear fracture mechanics is less stringent than for LEFM (equation (8)).

The experimental procedure to determine \( J_C \) is similar to \( K_{lc} \) testing except that the point of evaluation is the final point before the unstable fracture. This point is labelled \( J_Q \) and is qualified as \( J_c \) if fracture instability occurred without significant crack growth and that the \( J_Q \) value does not change with increasing specimen size, see equation (15) and (16).

\[ \Delta a_p < 0.0002 + \frac{J_Q}{M_J \sigma_Y} \quad \text{[m]} \]  

(16)

\( \Delta a_p \) is the stable crack extension prior to the unstable fracture. \( M_J \) is a dimensionless constant, which is set to 2 in e.g. the ASTM standard for fracture toughness testing [26]. If the stable
crack extension is equal or greater than equation (16) but valid in accordance with equation (15). \( J_Q \) is qualified as \( J_u \). This is to be considered as the material and specimen size and geometry dependent fracture toughness value.

### 2.2.2 The ductile initiation fracture toughness \( J_{ic} \)

For materials where the unstable fracture occurs after extensive stable crack growth (increasing displacement is needed for advancing the crack) or does not occur at all another parameter has to be used to characterise the fracture toughness. \( J_{ic} \) is defined as the initiation value for \( J \) of stable crack growth in the presence of a sharp crack. Figure 10a shows the evolution of \( J \) in a ductile material, this curve is called a J-R curve where \( R \) stands for resistance against crack extension. The vertical axis displays the J-integral and the horizontal axis the crack extension from the original crack. During the initial loading the crack-tip is blunted which gives a steep curve. For brittle materials blunting occurs too but to a much lesser extent. This blunting is also called the stretch zone width (SZW) and is due to extensive plastic deformation around the crack-tip, see Figure 10b. Eventually, the ability for plastic deformation is exhausted and a stable crack is created.

![Figure 10. a) Schematic J-R curve for a ductile material [28], b) the stretch zone width (SZW) [31].](image)

The crack growth mechanism is usually micro-void and coalescence, see section 3.2. The point on the y-axis when this initiation occurs is very difficult to determine experimentally. The usual practice is to offset the initial slope by 0.2 mm crack extension and take the intercept with the curve as the ductile initiation point [26]. The initiated stable crack grows with increased \( J \) until a steady state or instability is reached. The slope of the J-R curve after the initiation decreases with increasing constraint [32]. From the slope of the J-R curve the tearing modulus can be defined, which indicates the stability of the growing crack [26]:

\[
T_R = \frac{E}{\sigma_{YS}^2} \frac{dJ_R}{da} \quad [\text{Pa/Pa}]
\] (17)
where $E$ is the Young’s modulus and $\sigma_{YS}$ the yield strength. Instability occurs when the driving force curve, calculated $J$ versus crack extension for the structure (which depends on the geometry of the structure and loading conditions), is tangent to $T_R$.

For measuring the crack extension during testing the compliance or the potential drop method can be used. The compliance method is probably the more common of the two methods. The compliance method utilises the relationship between the specimen compliance (inverted stiffness, $\Delta$CMOD/$\Delta$P) and the crack length. By doing small unloading during the test, the crack length can be calculated, see Figure 11. However, as stated in section 2.2 the symmetry between a non-linear elastic material and an elastic-plastic material is only valid for proportional loading, i.e. unloading violates this condition. However, the error inserted by the unloading during the testing is small compared to other errors if the unloading is around 10-15% of the current load [11, 29].

![Figure 11. The compliance method [11].](image)

The potential drop method uses the fact that the electrical resistance of the specimen depends on the crack length. Both DC and AC can be used. The resulting $J$-$R$ curves for the different methods are slightly different but the evaluated $J_{k}$ is consistent between the methods [11].

The maximum $J$-integral capacity of a specimen for $J_{k}$ evaluation is:

$$J_{max} = \frac{b_0 \sigma_y}{15} \text{ [N/m]}$$

(18)

The crack growth is restricted to the intercept between the $J$-$R$ curve and a 1.5 mm exclusion line. The slope of the exclusion line is $M\sigma_Y$ where $M = 2$ or can be found from the test data [26]. The tentative fracture initiation point, $J_Q$, is qualified as $J_{k}$ if the following restrictions are met:

$$B, b_0 > \frac{25 J_Q}{\sigma_y} \text{ [m]}$$

(19)
The normalization data reduction technique was introduced for developing J-R curves without the need for automatic crack length monitoring during testing [33]. The technique is based on the assumption that J-R curves can be directly constructed from the load-displacement curves if a “key curve function” is available for the material [34]. First, the method was based on a universal key curve for a given material. The method was later developed to use individual normalisation calibration curves for each tested specimens and hence the name for this technique [33].

The assumption is that a functional form with unknown constants can describe the normalized load versus plastic displacement. The unknown constants can be determined at the calibration points which are the points where the load, displacement and crack length are known [33]. The crack length can be found at two points during the test. These positions can be optically measured after the testing. The first one is the crack length at the start of the test (distance of the machined notch plus the fatigue pre-crack). The second one is the crack length at the end of the test when the test is stopped or unstable fracture occurs. If the test was stopped, measuring of the final crack length requires post fatigue cracking, heat tinting or breaking the sample at temperatures were cleavage fracture occurs.

The normalization data reduction technique is described in ASTM E 1820 [26] and this section is a short description of the method. The test method is only applicable on standard test geometries with \(0.45 \leq a_0/W \leq 0.7\) and where the stable crack extension is less than 4 mm or 15 % of the initial uncracked ligament. The normalised load values \(P_i\) up to but not including the maximum load are defined as:

\[
P_{ni} = \frac{P_i}{WB \left(\frac{W-a_{bi}}{W}\right)^{\eta_{pl}}} \quad [N]
\]

where \(W\) is the specimen width, \(B\) is the specimen thickness and \(\eta_{pl}\) is a dimensionless factor that depends on the specimen geometry. \(a_{bi}\) is the blunting corrected crack size which is given by:

\[
a_{bi} = a_0 + \frac{J_i}{2\sigma_Y} \quad [m]
\]

where \(a_0\) is the initial crack length, \(J_i\) is the ith data point of the J-integral found from equation (13) when using the initial crack length \(a_0\). \(\sigma_Y\) is the effective yield strength. The normalised plastic displacement is defined as:
\[ v'_{pli} = \frac{v_{pli}}{W} = \frac{(v_i - P_iC_i)}{W} \quad [\text{m/m}] \]  \hspace{1cm} (23)

where \( C_i \) is the specimen elastic load line compliance based on \( a_{bi} \) and \( v_i \) is the crack-mouth opening displacement at the \( i \)th data point. The final load-displacement point of the test is normalised by equation (21) to (23) except for using the final optical measured crack size, \( a_f \), as \( a_{bi} \). A tangent line from the final normalised data point to the remaining normalised data is drawn and the data points to the right are excluded (except for the final data point). The former normalised data points with \( v_{pli}/W \geq 0.001 \) define the normalised dataset which is fitted to the following function:

\[ P_N = \frac{a + bv'_{pl} + c(v'_{pl})^2}{d + v'_{pl}} \quad [\text{N}] \]  \hspace{1cm} (24)

where \( a, b, c \) and \( d \) are fitting coefficients. The crack extension between the initial crack length and the final crack length during testing can be determined from an iterative procedure. The iteration starts with \( a = a_0 \) and the normalized load, equation (21), is compared with the fitted function, equation (24). The iteration then proceeds with increasing the crack length until the minimum differences between the two equations are found. After this the iteration proceeds to the next data point and repeats this iteration through the dataset. In the thesis a Matlab algorithm was created for the iteration process of finding the crack extension.

With the crack extension determined the J-R curve and \( J_{ik} \) can be evaluated as stated in chapter 2.2. This method has been found to give equivalent J-R results as with the elastic compliance method [26, 33] as long as no pop-ins occur [35].

2.2.4 The master curve method \( T_0 \)

Unstable fracture is usually in the form of cleavage fracture, see section 3.1. The cleavage crack is first initiated as a microcrack from a cracked inclusion, precipitate or other weak constituent or from stress concentrations around mechanical twins. Cleavage failure occurs when this microcrack is able to propagate into and through the surrounding grains [36]. The conditions necessary for the microcrack initiation and subsequent propagation is dependent on the local conditions at the initiation [29]. The fracture toughness depends thereby on the likelihood of finding a critical initiation in the process zone [37] and thus the fracture becomes to an extent random in nature [29]. This give rise to extensive scatter of the measured fracture toughness and that the fracture toughness becomes dependent on the process volume, i.e. the crack length of the specimen [37, 38]. The measured fracture toughness increases with increasing plastic deformation and ductile crack growth prior to the critical cleavage crack initiation [39] as showed in Figure 12. This situation is for example common for ferritic steels in the DBTT region.
Figure 12. Influence of ductile crack growth prior to cleavage crack initiation on the fracture toughness [28].

To characterise the fracture toughness under these conditions, probabilistic models can be used. One approach is to model the fracture event as a Weibull distribution where the failure is governed by the weakest link hypothesis [29, 40]. One such approach is the master curve method which determines the reference temperature $T_0$ as the evaluated fracture toughness. The method is standardised by the American Society for Testing and Material (ASTM) and is designated ASTM E 1921 [40]. The assumption made is that the material volume in front of the crack tip contains a distribution of possible cleavage fracture initiation sites [37]. This material volume can be regarded as infinitesimally small regions and the cleavage fracture will occur when one of these regions fractures [29]. The stress required for fracture (which depends on a complex function of initiator size distribution, grain size, temperature, stress and strain) can vary from region to region and the probability of fracture depends then on both the stress distribution and the probability of finding a critical initiation point [29, 37]. The assumption is however that no global interactions between these regions occur [29, 37]. The probability model for the critical cleavage fracture is illustrated in Figure 13.

Figure 13. The probability model for critical cleavage fracture [41].
The cleavage initiation and propagation in the original grain are mainly affected by the size of the initiation and the local stress and strain condition. For propagating into and beyond the neighbouring grains the cleavage fracture is also dependent on the stress gradient in the vicinity of the initiation site [36, 42, 43]. For ferritic steels at the lower shelf temperature (recall Figure 1, the left part) the initiation is guaranteed and the fracture toughness is controlled by the propagation of the crack. With increasing temperature, the microcrack initiation becomes more difficult and the initiation part becomes more and more dominant for the fracture process [37, 44]. The master curve method is based on the assumption that the unstable cleavage fracture is primarily initiation controlled [40, 44] and is thereby only applicable in the transition region, recall Figure 1.

As for the K and J parameter in the previous sections the master curve method requires SSY conditions in the process volume at the crack-tip. When extensive plasticity and/or crack growth occur the outer boundary conditions affects the fracture probability and the model loses its validity [37, 40, 45].

The master curve method is based on the J-integral and the point of unstable fracture (specimen failure or significant pop-in) is defined as \( K_{Jc} \), equation (12) in (14). The \( K_{Jc} \) datum is regarded valid if the measured toughness is less than equation (25) and the crack growth is less than 0.05(W-a0). If equation (25) or the crack growth is exceeded then the measured \( K_{Jc} \) is regarded as invalid and can be used for censoring [40]. If the specimen is unable to terminate in cleavage fracture the test is regarded as a nontest.

\[
K_{Jc(\text{limit})} = \sqrt{\frac{E b_0 \sigma_Y}{M(1-\nu^2)}} \quad [\text{Pa} \sqrt{\text{m}}] \tag{25}
\]

Equation (25) states the measuring capacity of the specimen in regard to the constraint in the specimen, i.e. SSY conditions [46]. \( M \) is a dimensionless constant that states the constraint and is set to 30 in the standard [40].

For adjusting for the influence of the crack length, the \( K_{Jc} \) values can be size adjusted according to the following equation:

\[
K_{Jc(x)} = K_{\text{min}} + (K_{Jc} - K_{\text{min}}) \left( \frac{B_0}{B_{1T}} \right)^{1/4} \quad [\text{Pa} \sqrt{\text{m}}] \tag{26}
\]

where \( K_{Jc} \) is the measured toughness, \( B_0 \) is the specimen thickness, \( B_{1T} \) thickness of prediction and \( K_{\text{min}} \) is the threshold propagation toughness. Without \( K_{\text{min}} \) the model predicts fracture at infinitesimal stresses [37]. According to the standard \( K_{\text{min}} \) is set to 20 MPa \( \sqrt{\text{m}} \) and \( B_0 \) to 25 mm [40]. The exponent \( \frac{1}{4} \) comes from the Weibull shape parameter for SSY conditions [46].

The Weibull scale parameter, \( K_0 \), which corresponds to a 63 % cumulative probability level for cleavage failure can be found from the following expression:
\[ K_0 = \left[ \sum_{i=1}^{N} \frac{(K_{Jc(i)} - K_{\text{min}})^4}{r} \right]^{1/4} + K_{\text{min}} \quad [\text{Pa}\sqrt{\text{m}}] \]  \hspace{1cm} (27)

where \( r \) is the number of valid data points and \( N \) is the sum of valid and invalid data points. The median \( K_{Jc(\text{med})} \) which corresponds to a 50 \% cumulative probability can be found from:

\[ K_{Jc(\text{med})} = K_{\text{min}} + (K_0 - K_{\text{min}}) \left( \ln 2 \right)^{1/4} \quad [\text{Pa}\sqrt{\text{m}}] \]  \hspace{1cm} (28)

The reference temperature, \( T_0 \), is defined as the temperature where the median size adjusted (1T) fracture toughness is 100 MPa\sqrt{m}. For testing where a single testing temperature has been used the \( T_0 \) can be found from the following expression:

\[ T_0 = T - \left( \frac{1}{0.019} \right) \ln \left( \frac{K_{Jc(\text{med})} - 30}{70} \right) \quad [\text{°C}] \]  \hspace{1cm} (29)

If the test data is distributed over a temperature range the \( T_0 \) can be found from iterative solving the following expression:

\[ \sum_{i=1}^{N} \delta_i e^{0.019(T_i - T_0)} - \sum_{i=1}^{N} \frac{(K_{Jc(i)} - K_{\text{min}})^4 e^{0.019(T_i - T_0)}}{(1 + 77e^{0.019(T_i - T_0)})^5} = 0 \quad [-] \]  \hspace{1cm} (30)

where \( \delta \) is 1 for valid datum and 0 for invalid datum.

2.3 The crack-tip opening displacement (CTOD)

Another approach to define the fracture toughness is the crack-tip opening displacement \( \delta \). It was seen during the early years of fracture mechanics that the fatigue crack become blunted during testing, see Figure 14, and that the degree of blunting could be related to the toughness [28].
Several definitions of $\delta$ exist but the two most common is the one in Figure 14 and $\delta$ found from the intersection of a 90° vertex with the crack flanks. For a blunted crack-tip that resembles a semi circle these two definitions are equal [28]. For a three-point bend specimen which rotates about a hinge point the $\delta$ can be defined from a triangular construction:

$$\frac{\delta}{x} = \frac{V}{a + x} \quad [\text{m/m}]$$

(31)

where $V$ is the crack mouth opening displacement, $a$ is the crack length, $W$ is the specimen width and $x$ is the distance from the crack-tip to the hinge point, see Figure 15.

By substituting $x$ with $r(W-a)$ where $r$ (rotational factor) is a dimensionless constant between 0 and 1, equation (31) can be changed to:

$$\frac{\delta}{r(W-a)} = \frac{V}{a + r(W-a)} \quad [\text{m/m}]$$

(32)
By rearranging the terms in (32) the $\delta$ can be found to be:

$$\delta = \frac{r(W-a)V}{r(W-a)+a} \text{ [m]}$$  \hspace{1cm} (33)

However, equation (33) has only satisfactory accuracy when the blunting is plastic and to improve the accuracy one can separate the crack-tip opening into an elastic and plastic part [28]. The elastic part of the blunting can be found from relating the stress intensity factor $K$ to the crack-tip opening. Equation (33) can then be rewritten [26, 28, 47] as:

$$\delta = \delta_{el} + \delta_{pl} = \frac{K^2}{m\sigma_{YS}E'} + \frac{r_p(W-a)V_p}{r_p(W-a)+a} \text{ [m]}$$  \hspace{1cm} (34)

where $K$ is the LEFM SIF $K$ (recall section 2.1), $m$ is a dimensionless function of $a_0/W$ and the strain hardening exponent, $\sigma_{YS}$ is the yield strength, $E'$ the elastic modulus for plane-strain stress condition, $r_p$ is the plastic rotational factor and $V_p$ is the plastic part of the crack mouth opening displacement. Nowadays, $\delta$ can be measured from the J-integral with the following relation [26]:

$$\delta = \frac{J}{m\sigma_y} \text{ [m]}$$  \hspace{1cm} (35)

where $J$ is equation (12) or (13) and $m$ is a dimensionless function of $a_0/W$ and the ratio of tensile strength over yield strength. $\sigma_Y$ is the effective yield strength.

### 2.4 Pop-in

Pop-in is defined as the sudden increase in displacement and decrease in load with subsequent increase in load past the point of the unstable crack extension, recall the type II fracture sequence in Figure 7. The word pop-in refers to the audible “pop-in” sound that occurs when the crack extension occurs. Pop-in is observed to occur during both monotonic and cyclic loading [48]. There are four types of pop-in that can occur during the testing [49]:

- **Type I**: Initiation, propagation and arrest of an unstable crack in the same plane as the fatigue pre-crack.
- **Type II**: Initiation, propagation and arrest of an unstable crack in the planes perpendicular to the plane of the fatigue pre-crack, i.e. delaminations (also referred to as splits and fissures).
- **Type III**: Linking of multiplane fatigue pre-cracks or joining up of flaws (porosity, slag inclusions and lack of fusion).
- **Type IV**: Breaking of ice particles around the rollers during sub-zero temperature testing or electrical interference and clip-gauge malfunction.
The pop-in results from an unstable crack initiated and propagated in a local zone with lower toughness than the surrounding matrix. The crack is then arrested when it propagates into the tougher surrounding matrix [50]. Zones with reduced toughness have been attributed to the presence of grain boundary precipitates, martensite bands and to chemical banding [48]. Local differences in microstructure and chemical composition as in weldments can also cause local zones with reduced toughness.

The problem with pop-in is the transferability to real structural components. Would the crack arrest also occur in a structural component or is the crack arrest due to the testing setup (specimen size and geometry and the stiffness of the testing machine and the loading conditions)? Even though the stress field around the crack-tip in the test specimen is in SSY condition which can be transferred to a real structural component, recall Figure 6, there are still some size differences that have to be considered. When a crack extension occurs, the material behind the crack-tip will be unloaded and a stress wave will travel outwards. When this wave reaches the boundaries of the specimen, the wave will be reflected and may thereby interfere with the propagating crack. The time (i.e. amount of crack extension that can occur) until the stress wave returns then depends on the size of the loaded body. Willoughby estimated on how large the crack extension during a pop-in had to be until the stress waves returned in a three-point bending specimen [51]. It was concluded that if the crack extension was less than 4 % of the original ligament (W-a₀) the pop-in could be regarded as insignificant, i.e. structurally irrelevant.

Another issue is the difference in loading condition. Real structural components are loaded in something between load and displacement controlled condition. Fracture toughness testing is most often done in displacement control. The reduction in load during the pop-in is due to the constant displacement condition and depends on the stiffness of the testing machine and the length of the crack extension [52, 53]. Due to this load reduction, it is possible for the SIF at the propagating crack-tip to decrease and fall below K_{IC} and the resulting crack arrest causes the pop-in behaviour in the test specimen [50, 54].

The assessment in the test standards for the significance of one or multiple pop-ins is based on the change of the specimen compliance after a pop-in event. The ASTM 1820 standard specifies that if the compliance change is larger than 5 %, then the pop-in is regarded to be significant while the British Standard BS 7448 standard specifies a 1 % change [26, 47]. The change in compliance corresponds to a certain crack extension which seems to be continuity from the K_{IC} standard [50]. It is also stated that a metallographic examination should be conducted for verifying that the crack arrest occurred in similar microstructure as the crack initiation.

### 2.4.1 The type II pop-in

The type II pop-in occurs in a specimen subjected to high through-thickness stress (e.g. a high triaxial stress state under plane-strain stress condition) and the existence of “weak” planes perpendicular to the fatigue pre-crack plane [49]. Under this condition, delaminations can occur which changes the specimen compliance which results in a measured load drop and increased displacement. Because the delaminations do not lead to specimen failure, the load
starts to increase after the new compliance have been settled. The “weak” planes have been
classified as inclusion-type or structure-type [49].

The structural significance of type II pop-in has gain little attention in the literature. In the
fracture mechanical testing standards, it is specially noted that delamination can result in pop-
ins but no further information is given [26, 47]. In BS 7448 part II [55] it is stated that: “The
fracture toughness at pop-in caused by a split shall be reported. However, the assessment of
the structural significance of the split is outside the requirements of this standard”. Pisarski et
al. [56] state that: “The structural significance of the split/pop-in is not considered in testing
standards”. Wiesner et al. [49] and Pisarski et al. [56] conclude that the type II pop-in is
structural insignificant as long as there is no significant loading in the through-thickness of
the material. If so is the case the through-thickness fracture toughness should be determined.
3 Fracture mechanisms

3.1 Transcrystalline fracture (cleavage fracture)

Cleavage fracture occurs by the breaking of the atomic bonds between atoms in certain crystallographic planes in the grains. The plane for the separation is usually the plane with largest distance between adjacent atoms (i.e. the least dense plane) [10, 57]. For phases with bcc structure (ferrite) the cleavage fracture occurs in the (100) plane [10]. Estimated stresses at the crack-tip from finite element analyses are ~50 times less than the theoretical fracture stress for crystalline solids [28]. For overcoming this difference in stress, the concept of sharp microcracks is used. The microcracks provide enough local stress concentrations for exceeding the atomic bond strength [28]. The microcracks can originate from dislocation interactions, mechanical twinning and perhaps the most common way from the cracking of inclusions and precipitates [28]. A cleavage crack is initiated when the local stress at the tip of the microcrack exceeds the atomic bond strength and crack-tip blunting from dislocation motion is restricted [29, 58]. Experiments indicate that the yield strength has to be reached before cleavage fracture can occur thus indicating that some dislocation motion (plasticity) is needed [59, 60, 61].

Cleavage fracture is rarely observed in fcc materials which is due to the properties given by this type of crystal structure. The fcc structure have higher planar densities and can also have sustained activation of glide systems for most circumstances [62]. Cleavage fractures have been observed in austenitic steels when the steel is highly alloyed with nitrogen [63].

When observing the fracture surface by naked eye, the surface usually has visible facets and no or little plastic deformation. Using scanning electron microscopy (SEM) reveals flat facets with no or little sign of plastic deformation. White branching streaks are a usual characteristic of cleavage fractures and are due to the propagation of several parallel fracture planes [11, 28, 64], see Figure 16. These streaks are called “river patterns”. Due to the higher energy “cost” of having multiple fracture planes the river lines eventually converges which then makes it possible to determine the fracture direction and the point of cleavage initiation.
Figure 16. Cleavage facet showing river pattern (right side) and tongues (upper centre to the right) [65].

The river pattern emerges when the cleavage fracture crosses over to another grain which has a different crystallographic orientation. Due to the misalignment between the current cleavage plane and the cleavage plane in the adjacent grain, several parallel fracture planes appear, see Figure 17. River lines can also result from local defects in the lattice, e.g. dislocations, inclusions and subgrain boundaries [62]. Other distinctive characteristics are feather markings (fan-shaped arrays of very fine river lines) and tongues (due to the formation of mechanical twins which has the cleavage plane locally changed), see Figure 17 [64].

Figure 17. Cleavage fracture crossing a grain boundary (from left to right) [65]. River lines (“crack paths”) and tongues (“twins”) are shown.

Ideal cleavage fracture occurs only under well-defined conditions, e.g. for single-crystals with limited number of active slip systems. Most engineering alloys are polycrystalline which thereby contains varying fraction of cleavage fracture and deformation by slip. Depending on the grain orientation with respect to the axis of loading the grains can be favourably oriented for slip. When both cleavage fracture and slip operate together which results in dimple rupture and/or tear ridges that accompany the cleavage morphology the fracture process is
termed quasi-cleavage. The dividing line between cleavage and quasi-cleavage is somewhat arbitrary [62, 64].

The phenomenological models for cleavage fracture are built upon the assumption of critical tensile stress (the cleavage fracture stress) over a characteristic length for propagating the microcrack out from the grain and into the surrounding grains [28, 42, 61]. The arrest of the microcrack is most likely to occur at the grain boundary where twisted orientations offer higher resistance to crack propagation than tilted orientations [66, 67]. The susceptibility to cleavage fracture is increased by triaxiality and high strain rates [29] and by mechanisms that increase the yield strength without increasing the cleavage fracture stress [28].

3.2 Ductile fracture

Ductile fracture results from the nucleation, growth and coalescence of voids. In most engineering metals there exist inclusions and second phase particles. The initiation of the voids has been largely explained by either the interfacial failure between inclusions or second phase particles and the metal matrix or cracking of the inclusions or second phase particles during plastic deformation [11, 28, 68].

As the tensile stresses open up the crack-tip, plastic deformation causes crack-tip blunting. Due to the blunting the stresses in the vicinity of the crack-tip are reduced [28]. However, at some distance from the crack-tip the local stresses and strains will be sufficient to nucleate voids. As the crack-tip blunting continues the voids grow and eventually the blunted crack-tip (the stretch width zone) reaches a critical value where the growing voids coalesce with the crack-tip and a ductile fracture initiation ($J_{lc}$) has occurred [69]. Further crack extension occurs by repeating these steps of void nucleation, growth and coalescence.

The coalescence of voids is the main crack growth mechanism for ductile fracture and hence the name microvoid coalescence (MVC). The resulting fracture surface from the coalesced voids is a characteristic dimple structure as seen in Figure 18. The shape of the dimples depends on the shape of the inclusion/second phase particle but also on the resulting loading mode [65]. Fourteen different dimple shapes have been postulated to exist due to the possible mixtures of loading modes [70].
In the centre of the dimple, particles can sometimes be found that lead to the void formation. Depending on the dimple size and resolution of the SEM the intersecting slip-band formation can be seen on the inner dimple surface [65].

The MVC fracture process is strongly governed by the strain state along the crack front and for the crack to macroscopically extend MVC must occur along the whole crack front. MVC is therefore governed by the mean toughness properties of the material [29, 37, 71]. The ductile crack growth is thus of a more deterministic nature than the cleavage fracture and shows a small amount of scatter [29, 37]. After void nucleation, void growth commences and it has been seen that fracture toughness increases with increasing dimple depth and width [11, 65, 72].

The void coalescence is the final stage of the ductile fracture. It consists of the localization of plastic deformation between neighbouring voids. Two modes of coalescence are usually described, the first one is internal instability of the ligament between adjacent primary voids, see Figure 19a, the other one is a shear localisation at 45° from the main loading direction between primary voids, see Figure 19b [11, 29, 73, 74]. It has been observed for metals with bimodal size distribution of particles that inside these localized plastic deformation ligaments there is nucleation and rapid growth of second population of smaller voids. If these smaller voids cause failure before the coalescence of the primary voids the fracture mechanism is called void sheet mode of failure [74, 75].

Figure 18. Microvoid and coalescence fracture in LDX 2101® weld metal.
Figure 19. Micrograph showing the different void coalescence mechanism in metals. 100 μm thick aluminium alloy sheet with laser-drilled cylindrical holes, loading axis is vertical [73]. a) Internal instability between adjacent voids, b) shear localization between adjacent voids.

For metals where the void coalescence occurs after negligible void growth, the fracture mechanism is said to be nucleation controlled [73].

The void nucleation occurs more readily in higher triaxial stress state which results in a faster crack growth in the centre of the fracture toughness specimen. This is usually seen as a thumbnail shape for the crack growth that occurred during the fracture toughness testing [28].
4 Mechanical properties of duplex stainless steels at sub-zero temperatures

The two phases ferrite and austenite in DSS have different mechanical and physical properties, e.g., elastic modulus, yield strength, hardness and thermal expansion coefficient. During loading the macroscopically strains have to be accommodated locally by the phases. The different physical and mechanical properties cause micro deformation heterogeneity and stress partitioning between the phases and grains [76]. Finite element modelling of a duplex stainless steel with elastic-plastic properties taking measured crystallographic texture into account showed that stress concentrations occur at the phase boundaries between austenite and ferrite due to the elastic-plastic incompatibility between the two phases [77]. Another example of the micro heterogeneity is the observed cleavage crack initiation in aged DSS (see chapter 4.5) where certain austenite grains (depending on the crystallographic orientation) are constrained by the harder ferrite. This produces local stress concentrations in the austenite which are relieved by the formation of cleavage cracks in the ferrite [78].

The hardness (resistance to deformation) of the phases depends on the grain size and chemical content. Due to the different crystal structure of the two phases, the concentration of alloying element is favoured in one phase over the other. Chromium and molybdenum are enriched in ferrite while nickel and nitrogen are enriched in austenite. Microhardness measurement on 2205 showed that the ferrite was slightly harder than the austenite [79] and microhardness measurement on the SAF 2906 super duplex steel showed the opposite behaviour [80]. The increased hardness in the austenite has mainly been assigned to increased nitrogen content [2]. It has been observed that the austenite has higher work hardening than the ferrite which results in a transition point where the austenite becomes harder than the ferrite even if the austenite was initially softer [80]. Due to the differences in hardness the softer phase will exhibit higher plastic deformation during loading than the harder phase. It has been observed that evolution of the dislocation structures in ferrite during low cycle fatigue depend on the initial hardness difference between the two phases [80].

The difference in thermal expansion coefficient between the ferrite and austenite is causing residual stresses in the austenite when the metal is quenched from the solution annealing temperature. The residual stresses have been observed to be tensile in the austenite and compressive stresses in the ferrite [77, 81].

Rolled DSS exhibit strong anisotropy for the mechanical properties yield and tensile strength and toughness. The yield and tensile strengths are found to be higher in the transverse direction compared to the longitudinal direction. The cause for the anisotropy between transversal and longitudinal direction has been explained by the crystallographic texture [77, 82, 83]. However, for loading where the loading axis is 45° oriented to the elongated microstructure, the anisotropy cannot be explained by the crystallographic texture alone. In this case the elongated microstructure is parallel to maximum shear stress and the observed slip lines in the ferrite have longer distance to the phase boundaries compared to the other two loading directions. The phase boundaries act as obstacles to slip which causes dislocation pileups which in turn results in reduced dislocation mobility in the grain. The
grain size in the 45° orientation therefore becomes effectively larger and thus the yield strength and work hardening are reduced compared to the longitudinal and transversal direction [84].

No major influence of the crystallographic texture on the impact toughness has been observed [82].

4.1 Yield and tensile strength

The DSS have around twice the yield strength comparable to the most common grades of austenitic stainless steels at room temperature. The ultimate tensile strength is also high and the elongation greater than 25 % [2]. High mechanical strength is the result of several mechanisms in the alloy [2]:

- Interstitial solid solution hardening. Modern DSS have low carbon content so nitrogen is usually the main interstitial element.
- Substitutional solution hardening, e.g. from Cr, Mo and Ni.
- Strengthening from grain size refinement due to the presence of two phases that restrict mutual grain growth during heat treatment.
- High phase content of ferrite. For similar chemical composition the ferrite is harder than the austenite.
- Residual stress state after cooling from the solid solution treatment. This is due to the different thermal expansion coefficients for the two phases.

In Figure 20 the influence of low temperature on the yield and ultimate tensile strength is shown. Both the yield and tensile strength increase rapidly with decreasing temperature both for base and weld metals [85]. This large increase in strength is due to the increased resistance to dislocation movement by the increased Peierls stress in the ferrite at lower temperatures [11].
From comparison with austenitic stainless steels the yield strength of the austenite seems to be relative insensitive to sub-zero temperatures [86]. However, the tensile strength of austenitic stainless steels can increase rapidly with decreasing temperature. This has been explained by strain induced martensitic transformation of the austenite [86]. Strain induced martensitic transformation of the austenite has been observed for the 2205 grade during low cycle fatigue at room temperature [87]. Ferrite measurements by a digital ferritometer on impact toughness specimens of 2205 tested at -196 °C showed no evidence of martensitic transformation [88]. No temperature induced martensite was observed for a duplex Fe-24Cr-5Ni alloy with 17.2 % austenite content when held at -208 °C. However, during tensile testing strain induced martensite was observed at and below -52 °C [8]. Strain induced martensite has been found to not only increase the static strength but also improve high cycle fatigue properties [89], ductility and toughness [90]. Strain induced martensitic transformation is also the basis of transformation induced plasticity (TRIP). The TRIP effect has been observed in low nickel DSS [91, 92, 93]. It should be noted however that the TRIP effect is only likely to give positive toughness contribution when the temperature is above the thermal martensitic transformation temperature ($M_s$) [90].

### 4.2 Fracture toughness – Base metal

In Table 1 some published work on fracture toughness testing of base metal below room temperature is summarised. In the listed papers, the fracture toughness has been evaluated according to the crack-tip opening displacement (CTOD) method, recall chapter 2.3. The failure events have been the attainment of maximum force plateau, pop-in or unstable

![Figure 20](image-url)  
**Figure 20.** Influence of temperature on the yield and ultimate tensile strength for duplex stainless steels. Specimen extracted from a 30 mm plate of 2205, transversal direction [85].
fracture. The attainment of maximum force plateau and unstable fracture are labelled as m and c respectively [47].

Table 1. Published work on the sub-zero temperature fracture toughness testing of the base metal of duplex stainless steels

<table>
<thead>
<tr>
<th>DSS grades</th>
<th>Plate thickness</th>
<th>Specimen thickness</th>
<th>Ferrite content</th>
<th>Failure event</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNS S31803</td>
<td>13.5 mm</td>
<td>-, SE(B)</td>
<td>-</td>
<td>m/pop-in</td>
<td>E. Erauzkin et al. [21]</td>
</tr>
<tr>
<td>UNS S31803</td>
<td>40 mm</td>
<td>-, SE(B)</td>
<td>-</td>
<td>m/ pop-in</td>
<td>A. Dhooge et al. [18]</td>
</tr>
<tr>
<td>UNS S32760</td>
<td>35 mm</td>
<td>-, SE(B)</td>
<td>-</td>
<td>m/ pop-in</td>
<td>A. Dhooge et al. [18]</td>
</tr>
<tr>
<td>UNS S32760</td>
<td>25 mm</td>
<td>10 mm, SE(B)</td>
<td>50%</td>
<td>m/ pop-in or</td>
<td>T. J. Marrow et al. [22]</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>c &lt; -90 °C</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>80 mm pipe</td>
<td>-, CT</td>
<td>-</td>
<td>-</td>
<td>V. Kozak et al. [94]</td>
</tr>
</tbody>
</table>

The results from this work can be seen in Figure 21. The reduced fracture toughness with decreasing temperature is clearly shown. The fracture process was stable with increasing susceptibility for pop-in with decreasing temperature [18]. Marrow et al. reported unstable fracture below -90 °C.

The reason for the large discrepancy between different papers is unknown but may be due to different specimen sizes (which were mostly not stated in the articles) and/or different microstructure. It should also be stated that the attainment of maximum force plateau occurs when the rate of strain hardening of the material is balanced by the rate of decrease of the remaining cross section [28]. This point is not directly associated with the crack extension in the specimen and likely to depend on specimen geometry (size and crack depth) and displacement rate and has therefore been removed from the ASTM E 1290-08 standard [95]. For specimens that only show stable crack growth, the CTOD at the end-of-test can be used for quality control and specifications of acceptance. The discrepancy between the results may therefore have been less if the ductile initiation fracture toughness ($J_{Ic}$ or $\delta_{Ic}$) had instead been measured.
Kozak et al. [94] performed fracture toughness testing on CT-specimens from a 700 mm pipe (wall thickness 80 mm). The results can be seen in Figure 22. Valid $K_{lc}$ values were obtained at the lower shelf temperatures (-100 °C and lower).

Room temperature fracture toughness of a hot extruded and cold rolled tube of 2205 resulted in ductile initiation fracture toughness ($J_{lc}$) of 230-500 kN/m depending on specimen orientation [96]. The fracture process was fully ductile and it was also reported that the microvoids nucleated preferentially in the ferritic phase or at the phase boundaries.
4.3 Fracture toughness - Weld metal

Compiled results from published works on sub-zero temperature fracture toughness testing of DSS weld metals can be found in Table 2 and Figure 23. The summary contains DSS grades from super duplex to lean duplex.

Table 2. Published work on the sub-zero temperature fracture toughness testing of the weld metal of duplex stainless steels

<table>
<thead>
<tr>
<th>DSS grades/welding method</th>
<th>Joint type</th>
<th>Plate thickness</th>
<th>Specimen thickness</th>
<th>Ferrite content</th>
<th>Failure event</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNS S31803, SMAW, SAW</td>
<td>V, β2 = 0</td>
<td>40 mm</td>
<td>-</td>
<td>-</td>
<td>m/c</td>
<td>A. Dhooge et al. [18]</td>
</tr>
<tr>
<td>UNS S32760, SMAW, SAW</td>
<td>V, β2 = 0</td>
<td>35 mm</td>
<td>-</td>
<td>-</td>
<td>m/c</td>
<td>A. Dhooge et al. [18]</td>
</tr>
<tr>
<td>LDX 2101®, SMAW+FCAW</td>
<td>X</td>
<td>30 mm</td>
<td>30 mm</td>
<td>45 %</td>
<td>c</td>
<td>H. Sieurin et al. [19]</td>
</tr>
<tr>
<td>2304, SMAW+FCAW</td>
<td>X</td>
<td>30 mm</td>
<td>30 mm</td>
<td>44 %</td>
<td>c</td>
<td>H. Sieurin et al. [19]</td>
</tr>
<tr>
<td>2205, SAW</td>
<td>K</td>
<td>30 mm</td>
<td>30 mm</td>
<td>64 %</td>
<td>c</td>
<td>H. Sieurin et al. [16]</td>
</tr>
<tr>
<td>LDX 2101®, SMAW+FCAW</td>
<td>X</td>
<td>30 mm</td>
<td>30 mm</td>
<td>59 %</td>
<td>m'/c</td>
<td>Paper D</td>
</tr>
</tbody>
</table>

*The maximum force plateau was reached but the ductile initiation fracture toughness was measured (δ_{lc})
The fracture toughness for the super duplex Zeron 100 (UNS S32760) was evaluated at the attainment of maximum force plateau except for two specimens that had unstable fracture (one at -40 °C for the SMAW weldment and the other at -60 °C for the SAW weldment). The 2205 weldments tested by Dhooge et al. [18] failed by unstable fracture at -10 °C (one specimen of the SMAW weldment), at -40 °C (two specimens of the SMAW weldment) and at -60 °C (all of the three specimens of the SAW weldment), all other specimens were evaluated at the attainment of maximum force plateau. The specimens shown in Figure 23 from the testing conducted by Sieurin et al. [16, 19] all failed by unstable fracture. The result from Paper D is shown for comparison. The data points at room temperature and at 0 °C are ductile initiation fracture toughness. At -20 °C and at -40 °C unstable fracture occurred.

![Figure 23. Crack-tip opening displacement for the weld metal of duplex stainless steels. See table Table 2 for more information.](image)

4.4 The heat-affected zone (HAZ)

For the modern DSS with improved austenite reformation the heat-affected zone (HAZ) in duplex stainless steels has been found to be quite narrow, which makes it difficult to test the toughness with ordinary methods [1, 97]. For experimental testing of the mechanical properties of the HAZ, Gleeble thermo-mechanical simulator can be used [98].
Room temperature impact toughness testing of a submerged arc welded (SAW) 2205 showed that the toughness increased stepwise from the weld metal, fusion line and into different locations in the HAZ [85].

4.5 Influence of ageing and hydrogen

When exposed to elevated temperatures numerous structural changes can occur in the duplex stainless steel. This structural change with time is called aging. Most often the changes occur in the ferrite because of the much higher diffusion rates in this phase compared to the austenite [2]. The DSS are alloyed with chromium and molybdenum which are known for promoting the precipitation of intermetallic phases, e.g. sigma, chi, R, tau and pi. The interstitial elements nitrogen and carbon can also precipitate as nitrides and carbides. In Figure 24 a schematic time-temperature transformation diagram (TTT) over the possible precipitations in duplex stainless steels is shown. With increasing alloying content the boundary for precipitation expands to shorter time and higher/lower temperatures thus making the higher alloyed grades of DSS more susceptible to aging than lower alloyed DSS grades.

![Figure 24. Schematic TTT diagram for the possible precipitations in duplex stainless steels](image)

It should be noted that the time axis in Figure 24 is logarithmic so the time for precipitation can be from minutes to thousands of hours depending on the temperature. The precipitations that seems to have caused most concerns for DSS are the sigma phase (σ), chromium nitrides and the decomposition of ferrite (α'). However, as seen in Figure 24 there is no clear boundary between the different types of precipitation which makes it difficult to ascribe the changed mechanical properties to a certain type of precipitation. The chi phase (χ) is for example often observed together with the σ phase [99, 100].

The sigma phase precipitates at temperatures between 650 and 1000 °C and the time for transformation is between minutes to hours depending on the temperature and steel composition [2]. The sigma phase precipitates at the phase boundaries and then grows into
the ferrite [100]. The sigma phase increases the hardness, yield and tensile strength of the steel but at cost of reduced ductility, toughness and corrosion resistance [2, 101]. It has been observed that 1 vol% of intermetallic phases (χ+σ) is enough to significantly reduce the impact toughness for both base metal and weldments of DSS [100, 102]. It has also been observed that decreasing the test temperature has little influence on the impact toughness of DSS embrittled by intermetallic precipitation [103]. This should be interpreted as that the embrittlement shifts the transition temperature to higher temperature.

In modern DSS the carbon content is kept to very low levels so the carbide precipitation does not seem to be an issue [2]. The nitrogen content is on the other hand a very important alloying element for DSS and the nitrogen content is between 0.1 to 0.5 wt% depending on the grade. Nitride precipitation has often been observed after cooling of the metal from the solid solution treatment or welding. The nitrogen has higher solubility in the ferrite phase at higher temperatures and with cooling, depending on the cooling rate and chemical composition, the ferrite can be supersaturated with nitrogen which precipitate as chromium nitrides when the excess nitrogen no longer can be dissolved in the ferrite. The preferential sites are the α/α and α/γ phase boundaries due to the higher diffusion rates there. In aged lean duplex stainless steels where no χ and σ phase precipitation is observed, the chromium nitrides can cause significant toughness reduction when precipitated at the α/α and α/γ phase boundaries. However, no toughness degradation was observed when the precipitation occurred inside the austenite grains (at secondary austenite phase boundaries) [104].

The α' precipitation is the decomposition of the ferritic phase into a chromium depleted phase and a chromium enriched phase [99]. This ageing phenomenon is also known as the 475 °C embrittlement. It is common in ferrite with high amount of chromium and it is found in both ferritic and duplex stainless steels. This decomposition occurs due to a miscibility gap in the iron-chromium system [2]. The temperature interval for this decomposition is 300 to 525 °C [2] and is most rapid around 475 °C. The time scale for the decomposition is from minutes (at around 475 °C) to thousands of hours at the boundary temperatures [105]. The decomposition can occur by spinodal decomposition or nucleation and growth outside the spinodal but within the miscibility gap [2]. The result of the decomposition of the ferrite is increased resistance for dislocation movement in the ferrite [105, 106]. Due to the reduced dislocation mobility slip planarity is increased, which favours mechanical twinning [107]. The change from slip to twinning dominant failure in aged specimens has been observed in impact and tensile testing [106, 108, 109]. The austenite seems however to be unaffected by the ageing [106, 110, 111, 112]. The resulting mechanical properties after ageing depend thereby directly on the amount and morphology of the ferritic phase [113]. The aging results in macroscopically increased hardness, yield and tensile strength but with reduced ductility and toughness [106, 114]. The fracture toughness of a forged thick wall pipe of DSS aged at 5 h at 475 °C can be seen in Figure 25. Below -100 °C the aged samples have the same fracture toughness as the as-received samples indicating a propagation controlled fracture. At the higher temperatures, influence of embrittlement is more evident as cleavage crack initiation is facilitated by the aging.
Room temperature testing, in particular impact toughness, seems to be the most common approach for evaluating embrittlement by aging. However, it is recommended to compare the transition temperature for the aged material with the transition temperature of the non-aged material when evaluating embrittlement due to aging \cite{115}. The reason for this is that many metals are fully ductile at room temperature and the embrittlement from the aging shifts the transition curve, recall Figure 1, to higher temperatures. The material can be embrittled by the aging but at room temperature the aged material may still be on the upper shelf and low toughness degradation is measured. If the transition temperatures are instead compared, the embrittlement is more evident. The transition temperature can for example be defined as the temperature for the average value between the upper and the lower shelf, however no strict definition exist.

Hydrogen embrittlement is a phenomenon that can occur when hydrogen enters the metal and reduces the ductility and toughness. It can occur by the presence of hydrogen from chemical or electrochemical reactions. Once the hydrogen is in the steel the transport through the metal can occur by diffusion which can be greatly enhanced by the dislocation motions \cite{11}. The hydrogen tends to accumulate at grain boundaries, inclusions, voids and dislocations arrays \cite{11}. Several mechanisms for the hydrogen embrittlement have been proposed, e.g. the formation of hydrogen gas inside the metal, interaction with the metal lattice to lower its cohesive strength and interaction with the dislocation mobility \cite{11}. The hydrogen embrittlement can involve cleavage, microvoid and coalescence and intergranular fracture so there is no single fracture mechanism that is characteristic for hydrogen.
embrittlement [11]. The general trend is that single-phase ferritic steels are most susceptible to hydrogen embrittlement while single-phase austenitic steels have the least susceptibility [116]. The duplex stainless steels have an intermediate susceptibility [116] where the austenite phase inhibits cleavage crack growth in the ferritic phase [117].

The solubility and diffusion rate for hydrogen differs between the two phases in duplex stainless steels. The solubility is highest in austenite while the diffusion rate is highest in ferrite [2]. This results in uneven hydrogen distribution between the phases where the austenite contains the major part of the dissolved hydrogen [116]. Microhardness measurements of hydrogen charged duplex stainless steels showed that the hardness increases significantly in the austenite with increasing charging time while the increase in microhardness of the ferrite was marginal compared to the austenite [31].

The major embrittlement effect of hydrogen on duplex stainless steels seems to be the change in fracture behaviour where the ferrite becomes increasing susceptible to cleavage fracture [116, 117, 118]. The change in fracture behaviour for the ferrite may be explained by the induced stress concentrations in the ferrite from the localized plasticity that occurs in hydrogen affected austenite [116].
5 Experimental details and methods in the thesis

5.1 Fracture mechanical testing and specimen orientation

The fracture mechanical testing was performed in a test rig with a 100 kN servo-hydraulic cylinder. The cylinder can be load or displacement controlled with a stroke distance of 150 mm. A clip-gauge mounted on the top of the specimen by side notches was used for measuring the crack mouth opening displacement (CMOD).

For controlling the temperature a double-sided cooling box with mounted stirrer and thermostat was used. The thermostat included a thermometer (placed closed to the crack-tip) and an electric heater. The specimens were submerged in alcohol which was cooled by pouring liquid nitrogen into the chambers located between the sides of the box. The lowest temperature that can be reached is approximately -110 °C (the freezing point for the alcohol used). The testing temperature could be held constant with ±1 °C during the entire testing.

The definitions of different specimen orientations that exist for plate material can be seen in Figure 26. In this thesis the T-L orientation has been used for the plate material. For the bar material the X-Y(Z) orientation has been used.

![Figure 26](image)

**Figure 26.** Specimen orientations, a) plate, b) bar, the grain flow in X-direction [47].

The welded specimens were made from two plates where one side of each plate was bevelled. The two plates were assembled in X-joint configuration and welded, see Figure 27. The notch was placed in the centreline of the weld and across the thickness.
Figure 27. X-joint weld configuration. B = plate/specimen thickness, W = specimen width. L is the rolling/flow direction of the base metal.

In Table 3 the specimen dimensions for the singe-edged notched bend bars, SE(B), used in the thesis can be found.

<table>
<thead>
<tr>
<th>Grade</th>
<th>BxWxL</th>
<th>Average a/W</th>
<th>Temperature interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>2205</td>
<td>10x20x200 mm³</td>
<td>0.58</td>
<td>-18 ≤ T ≤ -94 °C</td>
</tr>
<tr>
<td>2205</td>
<td>30x60x400 mm³</td>
<td>0.55</td>
<td>-55 ≤ T ≤ -94 °C</td>
</tr>
<tr>
<td>2205</td>
<td>50x50x400 mm³</td>
<td>0.57</td>
<td>-50 ≤ T ≤ -94 °C</td>
</tr>
<tr>
<td>LDX 2101® WM</td>
<td>30x60x400 mm³</td>
<td>0.51</td>
<td>+20 ≤ T ≤ -60 °C</td>
</tr>
<tr>
<td>SAF 2906 BM</td>
<td>30x60x400 mm³</td>
<td>0.54</td>
<td>-60 ≤ T ≤ -103 °C</td>
</tr>
<tr>
<td>SAF 2906 WM</td>
<td>28x60x290 mm³</td>
<td>0.49</td>
<td>-72 °C</td>
</tr>
</tbody>
</table>

The test standards and methods for evaluating the fracture toughness that were used in the thesis can be found in Table 4. The J<sub>k</sub> was evaluated from J-R curves found from the normalisation data reduction technique, see chapter 2.2.3.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Method</th>
<th>Standard</th>
<th>Chapter in the thesis</th>
</tr>
</thead>
<tbody>
<tr>
<td>2205 BM</td>
<td>CTOD</td>
<td>ASTM E 1290-02</td>
<td>2.3</td>
</tr>
<tr>
<td></td>
<td>J&lt;sub&gt;c&lt;/sub&gt;, J&lt;sub&gt;k&lt;/sub&gt;</td>
<td>ASTM E 1820-06</td>
<td>2.2.1, 2.2.2</td>
</tr>
<tr>
<td>LDX2101® WM</td>
<td>J&lt;sub&gt;c&lt;/sub&gt;, J&lt;sub&gt;k&lt;/sub&gt;</td>
<td>ASTM E 1820-06</td>
<td>2.2.1, 2.2.2</td>
</tr>
<tr>
<td></td>
<td>K&lt;sub&gt;c&lt;/sub&gt;</td>
<td>ASTM E 1921-05</td>
<td>2.2.4</td>
</tr>
<tr>
<td></td>
<td>K&lt;sub&gt;k&lt;/sub&gt;</td>
<td>ASTM E 1820-06</td>
<td>2.1.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>BS7448 Part 2&lt;sup&gt;a&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>SAF 2906 BM</td>
<td>K&lt;sub&gt;k&lt;/sub&gt;</td>
<td>ASTM E 1921-05</td>
<td>2.3</td>
</tr>
<tr>
<td>SAF 2906 WM</td>
<td>K&lt;sub&gt;k&lt;/sub&gt;</td>
<td>ASTM E 1921-05</td>
<td>2.2.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>BS7448 Part 2&lt;sup&gt;a&lt;/sup&gt;</td>
<td></td>
</tr>
</tbody>
</table>

<sup>a</sup> Used for verifying specimen geometry and weld quality. Not used for fracture toughness evaluation

5.2 Tensile testing
The tensile testing was conducted with an electromechanical tensile machine equipped with a 50 kN load cell. The tests were performed with round specimens 5 mm in diameter and with 60 mm length including threads (36 mm gauge length). The specimens were extracted along the centre of the weld metal 2 mm below the surface. The specimens were submerged in ethanol, and liquid nitrogen was used to cool the ethanol down to the desired testing temperature. With a thermometer and a stirrer the temperature was manually controlled within ±2 °C during the entire tensile testing. The temperature was held constant at 10 min prior to testing. The strain rate was 0.00028 s⁻¹.

Due to the test setup the extensometer could not be reset after the testing had begun so only the initial strain (ε < 5%) was measured by the specimen extensometer. Larger strains after were found by assuming that the specimen strain could be found from the actuator displacement adjusted for the machine compliance [119]. The total displacement is given by:

\[ \delta = \delta_{\text{specimen}} + \delta_{\text{machine}} = \delta_{\text{ext}} + C_m P \]  \hspace{1cm} (36)

where \( \delta \) is the total displacement of the actuator, \( \delta_{\text{ext}} \) is the displacement of the extensometer applied on the specimen, \( C_m \) is the machine compliance and \( P \) is the applied load. Rearranging the terms in equation (36) gives the machine compliance:

\[ C_m = \frac{\Delta \delta - \Delta \delta_{\text{ext}}}{\Delta P} \]  \hspace{1cm} [m/N]  \hspace{1cm} (37)

Equation (36) and consequently equation (37) are only valid when the displacements are elastic. The machine compliance is thereby found from the elastic part of the tensile curve prior to the yield strength is reached. The specimen strain can then be found from equation (36) by dividing the specimen displacement by the specimen gauge length:

\[ \varepsilon_{\text{specimen}} = \frac{\delta_{\text{ext}}}{L} = \frac{\delta - C_m P}{L} \]  \hspace{1cm} [m/m]  \hspace{1cm} (38)

where \( L \) is the specimen gauge length. Due to contact issues between the actuator and specimen no general machine compliance could be found. The machine compliance was then determined for each individual tested specimen. After the testing the total elongation of the specimen was measured by measuring the length of both halves.
Summary of appended papers

This thesis is about the fracture mechanics of duplex stainless steels at sub-zero temperatures and can be divided into two parts, base metal and weld metal. In Paper A to C the fracture toughness and fracture mechanisms for the base metal are investigated. In Paper D and E the fracture toughness and fracture mechanisms for the weld metal are studied.

6.1 Paper A

Sub-zero temperature (°C) fracture toughness testing of 10, 30 and 50 mm plates of hot-rolled 2205 duplex stainless steel (DSS) was performed. The study was concentrated to the phenomenon of splits which are delaminations of the fracture surface, see Figure 28a. They are commonly observed after fracture and impact toughness testing of hot-rolled DSSs. The results indicated that the pop-in phenomenon becomes more common and more severe with thinner plate thickness. The pop-in was also observed to be due to the delaminations. Except for the pop-in phenomenon all specimens exhibited stable fracture where increasing actuator displacement was needed for increasing crack extension. Fractographic investigation with scanning electron microscope reveals that the fracture surface between the delaminations consists of a mix with microvoid and coalescence and locally arrested cleavage facets, see Figure 28b. It was also observed that the amount of crack growth depends on the distance between two delaminations. This indicates loss of constraint along the crack front. The ductile initiation fracture toughness, $J_{IC}$, evaluated from the normalization method was found to be independent of the specimen thickness.

![Figure 28](image_url) SEM photos of delaminated fracture surface, fatigue pre-crack at the upper side of the photos. a) The fracture surface of 10 mm plate, b) showing delamination, arrested cleavage crack and the surrounding ductile fracture.
Despite the presence of cleavage fracture, the specimens exhibited a stable fracture process. This was interfering with the master curve method. For evaluating the fracture toughness at sub-zero temperatures an assessment of the fracture resistance curve was instead suggested. For assessing the brittle crack behaviour at sub-zero temperatures it was proposed to use the split initiation as “failure” criteria.

6.2 Paper B

Standard Charpy V impact toughness testing on three different specimen orientations (T-L, L-T and T-S) where conducted for further investigating of the delamination phenomenon in Paper A. The material was a 30 mm plate of LDX 2101® and 10 and 30 mm plates of 2205. The testing temperatures were between room temperature and -120 °C. From fractographic examination together with the measured energy absorption, it can be concluded that the impact toughness starts to fall when the delaminations occur. Fractographic examination of delamination surfaces revealed low deformation fracture surfaces with structures similar to river lines. Together with the observed unstable propagation of the delaminations this indicated that the delaminations were cleavage fractures. Until the lower shelf is reached the cleavage cracks are constrained to follow the direction of the austenite lamellas. This gives a predefined crack path that causes the delamination phenomenon. The toughness anisotropy in hot-rolled DSS can therefore be explained by the cleavage fracture and the appearance of the microstructure. Interrupted fracture toughness tests of the 10 mm plate and a 50 mm plate of 2205 were also performed. The result from the interrupted fracture toughness test reveals that the delaminations initiate prior to the maximum force plateau and propagates ahead of the stable crack growth during testing, see Figure 29.

![Figure 29](image.png)

Figure 29. Schematic drawing over the crack growth during fracture toughness testing. The straight black lines are delaminations.

With the data from the interrupted specimens together with the data from Paper A, an estimate of the upper limit for the fracture delamination initiation toughness at sub-zero temperatures for the 2205 base metal was made. The fracture delamination initiation toughness was defined as the fracture toughness at the point of delamination. Measured by the crack-tip opening displacement method the fracture delamination initiation toughness was
28-61 μm for the 10 mm plate, 70-106 μm for the 30 mm plate and below 100 μm for the 50 mm plate.

6.3 Paper C

In this paper the role of crystallographic orientation of hot-rolled plates of 2205 has been examined to determine possible influence on the delamination phenomenon. The crystallographic orientation was measured by electron backscatter diffraction (EBSD) on electropolished metal samples of 10 and 50 mm plates of 2205 DSS. The results showed that 10 mm plates had a strong cube-on-edge texture in the ferritic phase while the 50 mm plate had a weak texture. The more uniform lattice orientation for the ferritic phase in the 10 mm plate can explain why thinner plates of 2205 are more prone to exhibit pop-in behaviour during fracture toughness testing.

6.4 Paper D

The aim was to evaluate the susceptibility for brittle failure in the weld metal at sub-zero temperatures (°C). Three weldments with the nickel contents 1.3, 4.9 and 6.0 wt% were made from 30 mm LDX 2101® plates. The weldments were subjected to tensile, impact and fracture toughness testing. The amount of ferrite was higher for the 1.3 wt% nickel weldment compared to the other two which had similar phase composition and mean free ferrite distance. For the two weldments with higher nickel content the tensile specimens failed after necking at all temperatures while the 1.3 wt% nickel weldment failed in a brittle way at all temperatures except at room temperature. The ductility remained unchanged for the weldment with highest nickel content with decreasing temperature while the other two weldments became less ductile with decreasing temperature. The 1.3 wt % nickel weldment was very brittle according to the impact toughness testing at all test temperatures while the other two were ductile at room temperature. With decreasing temperature the 4.9 and 6 wt% nickel weldments became increasingly more brittle with a \( T_{40J} \) temperature at -13 and -43 °C respectively. J-integral based fracture toughness testing showed a significant difference in the susceptibility for brittle failure at sub-zero temperatures between the weldment with 6 wt% nickel and the two with lower nickel content, see Figure 30. Published fracture toughness data on other duplex stainless steel weldments together with the results of the present investigation showed that the fracture toughness at sub-zero temperatures increases with increasing nickel content in the range from 1 to 9 wt% nickel.
6.5 Paper E

The aim was to evaluate the susceptibility for brittle failure in the base and weld metal at sub-zero temperatures (°C) for the super duplex stainless steel SAF 2906. Fracture toughness testing was conducted on standard single-edge notched bend bar specimens. The base metal was tested between -103 and -60 °C. The fracture sequence at and below -80 °C can be described as ductile until critical cleavage initiation occurs, which causes unstable failure of the specimen. No delamination was observed. At -60 °C the test was stopped shortly after maximum load had been reached without any unstable failure. Below -80 °C the master curve method can be used to determine the fracture toughness. The welding method used was submerged arc welding (SAW) with a 7 wt% nickel filler metal. The welded specimens were post weld heat treated (PWHT) at 1100 °C for 20 min and then quenched. Energy-dispersive X-ray spectroscopy analysis showed that during PWHT substitutional element partitioning occurred which resulted in decreased nickel content in the ferrite. The PWHT weld metal specimens were tested at -72 °C. The fracture sequence was critical cleavage fracture initiation after minor crack-tip blunting and ductile fracture.
7 Conclusions

This thesis includes fracture toughness testing of hot-rolled 2205 base metal, LDX 2101® weld metal and SAF 2906 bar material (base and weld metal). The testing temperature were between –103 to +20 °C. Impact toughness testing for the 2205 base metal and LDX 2101® base and weld metal between –120 to +20 °C has also been performed.

The conclusions are:

- The observed pop-ins in the 10 and 30 mm plate of 2205 are due to the sudden delamination of the material, see Figure 31a.
- Fractographic study on these delaminations indicated that the delaminations are cleavage cracks constrained by the microstructure to propagate along the direction of the austenite lamellas.

![Figure 31. Unstable fracture during pop-in. 10 mm plate of 2205 at -68 °C. a) The resulting delamination, b) higher magnification of the arrest of the delamination in a).](image)

- Delaminations occur for all tested plate thicknesses of 2205 while the pop-in phenomenon was absent for the 50 mm plate. The proposed explanation for this is that the cleavage fracture (the delamination) is not able to propagate far enough to cause a noticeable load drop during testing. One explanation for this is the ferrite grain size. Higher thickness reduction during hot-rolling gives a more elongated microstructure with longer and wider ferrite grains. Another explanation is the difference between textures. Electron backscatter diffraction measurement on the crystallographic orientation showed that the ferrite in the 10 mm plate had a stronger texture compared to the weaker texture found in the 50 mm plate. A stronger texture results in overall lower misorientation between the cleavage fracture planes which gives a lower cleavage crack propagation resistance.
- The delaminations can occur during the crack-tip blunting process (as in Figure 31a) or after a stable crack has formed. As the new specimen compliance sets in and the load decreases on the specimen and the crack-tip is far away from the main crack-tip,
the delamination arrests as seen in Figure 31b. The arrested delamination is likely to be stopped until the main crack eventually approaches.

- The crack growth in the main fracture plane is a mixture between high plastic deformation resulting in microvoid and coalescence and tearing with arrested cleavage cracks.
- The overall fracture process was stable for all specimens regardless of thickness. For the 10 and 30 mm plates the stable fracture process was continuously interrupted with unstable crack extension that was arrested quickly after (defined as pop-ins prior to the maximum force plateau).
- The consequence of delaminations was that the constraint along the crack front was reduced due to the existence of free surfaces. This hampers cleavage initiation and propagation. Each part between two delaminations acts as an individual subunit and the specimen thickness has less impact on the crack front constraint. The fracture toughness becomes therefore independent on the specimen thickness, see Figure 32.

![Figure 32](image-url)

**Figure 32.** Ductile initiation fracture toughness of 50 mm plate of 2205. B in the figure is the specimen thickness.

- No delaminations were observed for specimens extracted from a SAF 2906 bar. The fracture sequence for these specimens can be described as ductile until critical cleavage initiation occurred which caused an unstable fracture and thereby failure of the specimen. Below -80 °C the master curve method can therefore be used for characterise the bar materials fracture toughness.
The cause of the different fracture behaviour between the hot-rolled plates and the bar material is not known but it may be due to the coarser microstructure of the bar material.

The conclusion from this thesis regarding the fracture toughness testing of hot-rolled plates of DSS is that the evaluation of the fracture toughness is more complex than previously thought. The delamination phenomenon seems not to be included in the various existing fracture toughness standards. It is recommended that further work is done on e.g. the delaminations influence on the compliance method (and how to define the crack extension from highly irregular crack growth) and on the assumption of a J-dominating zone at the crack-tip.

The fracture toughness testing on the welded specimens in this thesis was on the weld metal. The conclusions are:

- Investigation with energy-dispersive X-ray spectroscopy showed that the substitutional element partitioning between the austenite and ferrite in the weld metal was small. However, during post weld heat treatment substitutional element partitioning occurred which resulted in reduced nickel content in the ferrite.
- The only found weld defect in the specimen was gas pores. However, from examination with scanning electron microscope ductile fracture was found around the gas pores in the fracture toughness specimens indicating that the gas pores did not contribute to the cleavage fracture.
- When considering the phase composition, microstructure and chemical content of various duplex stainless steel weld metals, the increase in fracture toughness at sub-zero temperatures correlated well with the increase in nickel content, see Figure 33.
Figure 33. Sub-zero temperature fracture toughness of DSS weld metals (SAW).
8 References


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