Fatigue properties of Tungsten Heavy Alloys IT180 and D176

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Abstract
Fatigue properties of the tungsten-based alloys IT180 (W-3.5Ni-1.5wt%Cu) and D176 (W-5Ni-2.5wt%Fe) have been determined using constant amplitude stress-controlled fatigue tests. These tests were performed at room temperature at relatively high cycle fatigue lives. The results indicate that the endurance limit for the alloys is about 210 MPa and 425 MPa for IT180 and D176 respectively. The fatigue strength coefficients and fatigue strength exponents are $S'_f = 1010 \, MPa$ and $b = -0,11$ for IT180 while $S'_f = 3000 \, MPa$ and $b = -0,13$ for D176. Strain-controlled fatigue tests were also performed to observe the response of the materials under cyclic loading. The Multiple Step Test method was used to estimate the cyclic stress-strain curve of the alloys. The tests indicate hardening of both materials when they are subjected to cyclic loading. The cyclic strain hardening exponent of D176 was possible to find and is $n' = 0.08$.

The fatigue response of the material is strongly affected by surface roughness, residual porosity, pore size and its distribution. A finite element analysis was also made to understand the effect of specimen geometry on fatigue data. The results showed that specimen’s geometry affects stress concentrations and stress distribution which might be related with the failure of the specimen. Scanning electron microscopy and energy dispersive spectroscopy have been used to characterize the sample’s microstructures and fracture surfaces.

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Chapter 1 Introduction

1.1 European Spallation Source
European Spallation Source (ESS) is a joint European project, like that of many large-scale research facilities such as CERN in Geneva. The city of Lund in Sweden was chosen to be the host of ESS for being among the most scientifically developed regions worldwide. Currently the ESS is in the pre-construction phase which includes among other things the design of the target station. When it is finished in 2025 it is expected to be the world’s leading research center using beams of slow electrons. ESS can be compared to a large microscope, where neutrons are used to probe various materials. In this sense the field of research will cover a wide range of disciplines such as: chemistry, nano and energy technology, environmental engineering, foodstuff, bioscience, pharmaceuticals, IT, materials and engineering science and archaeology [1-3].

1.2 Problem definition:
The Spallation Source will produce neutron beams for scattering experiments by using an accelerator system to generate high-energy protons, which then knock out neutrons from a target (a so-called spallation process) [1]. The ESS targets have to withstand a large average beam power, therefore submitted to a combined load of high radiation damage, large temperature gradients and stress waves. In this context, the target material has to withstand all the later conditions. ESS on this phase of the project is evaluating tungsten and tungsten heavy alloys as candidates for being target materials. The latter combine the high density and high elastic stiffness provided by the tungsten with the higher mechanical properties of the alloying elements, especially relatively high ductility at room temperature that facilitate among other things the machinability of the tungsten and reduces also the expenses of production. The understanding of the different physical, mechanical and chemical properties of the target material is vital in the design stages of the ESS target station.

1.3 Relevance of the present work
More specifically, the materials to be studied in this work are tungsten heavy alloys Inermet 180 (W-3.5Ni-1.5%wtCu) and Densimet 176 (W-5Ni-2.5%wtFe). Previous
researches have been carried out about the behavior of WHAs under tensile loading, as well as the microstructure and fracture surfaces under this condition and can be found elsewhere in literature. However, little research has been carried out to characterize the fatigue properties of WHAs and no work has been done specifically for Inermet 180 (IT180) and Densimet 176 (D176) in this topic. The study of their fatigue behavior is of interest since high power targets undergo large and changing energy deposition densities, varying in space and time. As a consequence of this, high temperature fluctuations take place thus leading to very short and high cyclic thermal stresses in the target material. The obtaining of fatigue properties of IT180 and D176 is a preliminary research that can also give a baseline for further investigations once the target material is chosen like for example thermo-mechanical fatigue response.

1.4 General Objectives
The scope of the present Master’s Thesis is to characterize the fatigue properties of Inermet 180 and Densimet 176 and to study the behavior of these alloys under cyclic loadings by performing stress-controlled and strain-controlled fatigue tests.

1.4.1 Specific Objectives
- Determine the relationship between the fatigue life and the stress levels at relatively high cycle fatigue regime by estimation of the Stress vs. Cycles to failure curve.

- Description of the cyclic response of IT180 and D176 under cyclic loading by the estimation of the Cyclic Stress-Strain curve.

- Comparison between the cyclic and monotonic response of the alloys.

- Characterize the variables that affect the fatigue behavior of IT180 and D176.

- Description of the fracture surface and fracture mechanisms of these materials.

- Study specimen’s geometry effects and how they may be related to the fatigue response of the materials.

1.5 Limitations
At the beginning of the project samples for fatigue testing of each alloy were provided by ESSS. However, due to budget issues and the interest of just a preliminary study for
the alloys as a baseline for further decisions of the target material, the amount of specimens given was very small making difficult the obtaining of precise and vast information of the fatigue properties of the material. Specifically, 21 specimens IT180 and 12 specimens of D176 were provided. The main difficulty comes with the estimation of an S-N curve that could describe the fatigue behavior of the materials over the entire range of low-cycle and high-cycle fatigue with accuracy. According to literature and fatigue standards at least 6 specimens should be tested to obtain a preliminary estimation of the fatigue life [4] and a minimum of 15 specimens are recommended to estimate the fatigue limit [4-5].

On the other hand, the specimens presented considerable surface roughness in the as-received condition. So, in order to fulfill the requirements about the specimens conditions established in fatigue standards (ASTM), grinding and polishing was carried out under the expense of a reduction in the testing section diameter.

The study of the surface of the Densimet 176 with optical microscopy was performed but no digital images were recorded since the software used for this purpose stopped working during the time length of the thesis.
Chapter 2 Theoretical background

2.1 Tungsten Heavy Alloys
Tungsten heavy alloys (WHAs) are microcomposites of spherical tungsten grains in a continuous ductile matrix of typically Nickel and Iron or Nickel and Copper bonding the grains together. The typical tungsten content varies from 90 to 98%wt [6-8].

2.1.1 Production of Tungsten Heavy alloys
The common procedure for the production of WHAs is liquid phase sintering. This procedure enables achieving of almost theoretical density by the considerable shrinkage during sintering compared to the density obtained only with the pressing of the powders. A brief explanation of how the procedure is carried out will be described:

Elemental powders are blended and then compressed using any common P/M blending and compressing procedure. The finer the W particles the more homogeneity can be achieved thereby W-particles range in size between 2-8μm [6]. In the case of iron and nickel alloys carbonyl powders are used while fine electrolytic powder is used for nickel and copper alloys. The pressed material is sintered in a furnace with an atmosphere of pure hydrogen which is the most general case; nitrogen mixtures, dissociated ammonia or just vacuum can also be used. The sintering of tungsten alloys can take place because of the difference on the melting temperatures of the original composite powders. When heating starts, the alloying elements will melt but thanks to the high melting point of tungsten, this element will remain in a solid state. This melt is called the liquid-phase and flows through the compact between the tungsten particles, wetting them.

W-particles vary in size and since there is a limited solubility of the liquid phase in the solid, the smaller particles will solve in the melt until the final composition of the matrix is set, i.e. the liquid phase reaches saturation. With continued time at temperature, once solved tungsten particles start to precipitate from the liquid-phase and diffuse into the still solid tungsten particles coarsening of these takes place and the grains grow bigger. Final diameter of tungsten grains range between from 50 to 150μm [8]. It is worth mention that even though almost full theoretical density can be achieved with the sintering, there will always be a presence of porosity in the final product making the real density lower.
In general, the mechanical properties of WHA are strongly influenced by the final tungsten grain size, shape and contiguity. That are at the same time are influenced by the tungsten content, sintering temperature and time [8-9].

2.2 The fatigue concept
When a repeated load is applied to a mechanical element it can produce variable tensions, which in the long time run can make the element brake with a lower load than it would have broken if it was exposed to a high constant tension. The repeated loads produce cyclic stresses that are generally described in a sinusoidal waveform using three parameters: the stress range, the stress amplitude and the mean stress as shown in figure 2.1 [10].

Figure 2.1 cyclic stress variations with time and nomenclature for stress parameters which affect fatigue life.

The stress range is defined as the difference between maximum stress and minimum stress while the stress amplitude is a half of stress range. The mean stress would be the algebraic sum of the maximum and minimum stresses.

\[
S_r = S_{\text{max}} - S_{\text{min}} \quad (2.1)
\]

\[
S_a = \frac{S_r}{2} = \frac{S_{\text{max}} - S_{\text{min}}}{2} \quad (2.2)
\]

\[
S_m = \frac{S_{\text{max}} + S_{\text{min}}}{2} \quad (2.3)
\]
In fatigue testing, the stress amplitude or range is the controlled variable, and the number of cycles is the dependant variable. It has to be said that usually the tensile stresses are considered positive and the compressive ones, negative.

The fatigue life \((N)\) is defined as the number of cycles to failure. Each cycle contains two reversals \((2N)\).

One of the most common fatigue routine tests is the uniaxial fatigue test using fully reversed loading, where the maximum and minimum stresses are equal in magnitude, i.e. the mean stress is zero. However a zero mean stress situation is not always representative of many applications so some parameters are introduced to express the mean stress applied to the object; this will be explained further on this chapter. First of all, the stress ratio \(R\) represents the mean stress as the ratio of minimum stress to maximum stress (Eq. 2.4). Then the amplitude ratio is defined as the ratio of stress amplitude to mean stress (Eq. 2.5).

\[
R = \frac{s_{\text{min}}}{s_{\text{max}}} \tag{2.4}
\]

\[
A = \frac{s_a}{s_m} = \frac{1-R}{1+R} \tag{2.5}
\]

Static loads have an \(R=1\) while the fully reversed loading represents \(R=-1\).

\textbf{2.3 Stress-life approach:}

The stress-life approach was firstly introduced by Wöhler between 1852 and 1870. His work gave the idea to obtain the fatigue response of a material from tests where cyclic loads are applied to a specimen of interest. Loads used can be plane bending, rotating bending, multiaxial, or uniaxial tension and or compression among others.

One of the most common tests, and the one used on this thesis consists on the application of uniaxial and equal tension-compression loading to a smooth specimen until failure finally occurs (the failure criteria would be the rupture of the piece). This kind of testing is called constant amplitude, fully reversed fatigue test and data from such an experiment is used to plot the stress amplitude \(S_a\) against the number of cycles to failure \(N\). This plot is called the stress-life or S-N curve. As it can be seen below in figure 2.2, the life of a material increases when decreasing the stress.
amplitude until it reaches a certain point where a plateau is exhibit and stresses below this level do not cause failure of the piece or component. The region above this point is called the finite life region while the plateau level is called in the literature as endurance limit $S_e$ or fatigue limit and is a property that most of materials present typically beyond about $10^6$ cycles. The value of $S_e$ is 35% to 50% of the tensile strength for most steels and copper alloys [11]. However, many high strength steels, aluminum alloys and other non-ferrous materials don’t have a constant endurance limit instead of a continuous increase in life with lowering of the stress. In these cases, the endurance limit is considered the stress amplitude at $10^7$ cycles since from this point the variation of $N$ with the stress level is less compared to that in the finite life region. [11]

![Figure 2.2 Typical S-N curve of a material.](image)

The stress life approach is most used in cases of high cycle fatigue life (HCF, $N$ higher than $10^4$) [12] where low stresses are applied and almost all the deformation taken place is elastic.

When the experimental data is plotted instead on a log-log scale, the curve shows as a straight line, thereby a linear relationship between the stress amplitude and the fatigue life can be determined. This relationship was expressed by Basquin under the equation:

$$S_a = S_f'(2N)^b$$

(2.6)
Where $\sigma_f$ and $b$ are the fatigue strength coefficient and exponent respectively. Is easy to deduce that once these two values are known for a specific material, its fatigue behavior (at least at HCF) can be represented. Then, one of the scopes of this thesis is to determine $\sigma_f$ and $b$ for both tungsten heavy alloys.

2.3.1. The median curve

The fatigue life or behavior of a material cannot be estimated with completely accuracy and a large amount of specimens should be tested in order to obtain a fairly accurate S-N curve. The main reason for this is the inherent scatter in the fatigue properties of a material. Hence, statistical analyses need to be performed and reported along with the data obtained to overcome the scatter problem and be able to report reliable information to design engineers.

In this sense, the median S-N curve is a statistical way of presenting fatigue data and shows the fatigue behavior of 50% of all the population of samples tested. In other words, 50 out of 100 specimens tested are expected to fail at a certain $N$ on the median curve when exposed to a given load.

It is worth to mention that scatter in fatigue properties will always be present while analyzing the test data and they can be due to several factors, for example differences on microstructures in specimens of the same batch (even if they were exactly manufactured) can lead to different results despite testing at equal conditions. [11], [13].

Explanation of how to obtain the median S-N curve will be explained further on this chapter.

2.3.2. The mean stress effect

The mean stress level is known to play an important role in influencing the engineering materials behavior when fatigue testing [11]. In the high cycle fatigue regime normal mean stresses are responsible for the opening and closing state of microcracks. In this sense tensile mean stresses are detrimental to fatigue strength since they contribute to accelerate the crack growth whereas compressive mean stresses would be beneficial since they retard the propagation of the cracks [13]. However this effect is
not that strong in the low cycle fatigue regime in which the large amounts of plastic deformation erase any effect of a mean stress.

There are some models to calculate the stress amplitude taking into consideration a mean stress different to zero based on the stress amplitude at zero mean stress. Following there are three different models to calculate it: Soderberg relation (Equation 2.7), Modified Goodman relation (Equation 2.8) and Gerber relation (Equation 2.9).

\[
S_a = S_a|_{s_m=0} \left\{ 1 - \frac{s_m}{S_y} \right\} \tag{2.7}
\]

\[
S_a = S_a|_{s_m=0} \left\{ 1 - \frac{s_m}{S_{TS}} \right\} \tag{2.8}
\]

\[
S_a = S_a|_{s_m=0} \left\{ 1 - \left( \frac{s_m}{S_{TS}} \right)^2 \right\} \tag{2.9}
\]

\(S_a\) is the stress amplitude denoting the fatigue stress for a nonzero mean stress, \(S_a|_{s_m=0}\) is the stress amplitude for fully reversed loading, \(S_y\) is the yield strength and \(S_{TS}\) is the material tensile strength.

Equation 2.7 gives a conservative idea of fatigue life for most engineering alloys. Equation 2.8 provides quite close results for brittle metals, and Equation 2.9 is generally good for ductile alloys. Summing up, some calculations will be done with Equation 2.8 to find the difference between making the tests with zero mean stress and using a mean stress value (30 MPa will be used as mean stress according to the purpose of this thesis, as the target material will be submitted to approximately this mean stress while operating).

In 1968 Morrow presented a modification of Basquin equation to take into account the mean stress effect, resulting as:

\[
S_a = \left( S_f - S_m \right) \left( 2N_f \right)^b \tag{2.10}
\]

Where the term added to the original Basquin equation (Eq 2.6) is \(S_m\) which is the mean stress value.
2.3.3. S-N curve vs. service conditions

Even though S-N curves are obtained using fixed test conditions (e.g., fully reversed constant stress amplitude), that in fact are not necessarily the same as the ones the real component is going to experience, they provide a baseline of the fatigue behavior of it that can be used by engineers during the design stages. On the other hand, the S-N curve can be used as a reference condition and there are other methods that combine the data obtained from it with other conditions to adapt it to the service case. During service, parts are subjected to different random levels of stress that are known to eventually cause the fatigue failure of the component. Thereby, the aim of the design engineers is to prevent this eventual failure and it has been proven that the total damage reached when failure occurs is an accumulation of the damage caused during each level of cyclic stress which can be calculated from the S-N curve (Cumulative theory). Explanation of this theory can be found elsewhere in the literature [11], [13].

2.3.5. Estimation of the S-N curve:

2.3.5.1 The median curve within the finite life region

In this work statistical analysis of fatigue data will be performed using the procedure proposed by ASTM on their practice E739 [4] where the stress vs cycle curve will be computed following the methodology suggested in this standard. Yet this practice is also applicable for the estimation of strain vs. cycle curves. Note that the results presented in the following chapters and the terminology used tries to go in accordance with ASTM standards references in E739 [14-15].

The tests are carried out in the high cycle fatigue regime but at stresses above the suspected endurance limit so they will fail at relatively shorter lives, i.e. number of cycles higher than $10^4$ but less than $10^6$, to ensure the endurance limit is not reached. The exact localization of the endurance limit will not be found in this thesis since a lot of specimens are needed for this purpose and they were simply not available. There are especial methods for the estimation of the endurance limit among which the Staircase Method is the most popular but it is recommended to test at least 15 specimens [5], [16].
Due to the unavoidable scatter present on fatigue data, the final stress-life fatigue curve of the IT180 and D176 will be presented in terms of a median curve and its associated statistics. According to ASTM, statistical analysis applies when the given data can be reasonably assumed to be a representation of some specific defined population or universe of material of interest (under specific test conditions), and it is desired either to characterize the material or to predict the performance of future random samples of the material. This goes on accordance to the principal objective of the present work which is to characterize the fatigue behavior of the alloys in order to predict the performance of future samples of the same material during service on the spallation device.

Fatigue life data obtained from the stress controlled fatigue tests, this is number of cycles $N_i$ at an specific stress amplitude $S_i$ during fully reversed axial loading until failure occurs, will be plotted on a log-log scale where the relationship between the life of the specimen and the stresses experienced is expected to follow a straight line as stated before. For the notation of this work, the subscript “$i$” denotes each sample tested whereas the total amount of samples is $n$.

The intention is then finding a line that better describes the fatigue response of the material, i.e. the line of best fit that will be determined by the maximum likelihood method as recommended by ASTM in [4].

The linear S-N relationship is represented by:

$$Log(N) = A + BLog(S_a)$$  \hspace{1cm} (2.11)

in which $S_a$ is the stress amplitude and $N$ is the fatigue life of the specimen in number of cycles.

The fatigue life $N$ is the dependent (random) variable in S-N tests, whereas $S$ is the independent (controlled) variable.

Even though the real distribution of the S-N curve is unknown it is assumed that the logarithms of the fatigue lives are normally distributed, that is, the fatigue life is log-normally distributed. This implies that the scatter in $Log(N)$ is assumed to be the same at low and high stresses (the variance of the log life is constant over the entire
range of the stresses used in testing). However this is just an assumption because it is known that the scatter in the logarithm of the life increases when decreasing the stress. Nevertheless, the use of a log-normal distribution for the S-N curve in metals has been used in countless of studies and it has also been proved to provide a good fit or description of the fatigue curve of materials [12].

Accordingly, the independent variable is denoted with \( X \) and the random variable is denoted with \( Y \). In this case \( X = \log(S_a) \) and \( Y = \log(N) \), however this practice can be applied to analyze other data and \( X \) and \( Y \) can represent some other variables. Then, equation (2.11) can be written as:

\[
Y = A + B \cdot X
\]  

(2.12)

The maximum likelihood estimators are as follows:

\[
\hat{A} = \bar{Y} + \bar{B} \cdot \bar{X}
\]  

(2.13)

\[
\hat{B} = \frac{\sum_{i=1}^{n}(X_i-\bar{X})(Y_i-\bar{Y})}{(X_i-\bar{X})^2}
\]  

(2.14)

Where \( \bar{X} \) and \( \bar{Y} \) are the average values of the independent and dependent variables and calculated as follows:

\[
\bar{X} = \frac{\sum_{i=1}^{n}X_i}{n}
\]  

(2.15)

\[
\bar{Y} = \frac{\sum_{i=1}^{n}Y_i}{n}
\]  

(2.16)

\( n \) is the total number of specimens (the total sample size). Then the variance of the normal distribution for Log \( N \) is

\[
\sigma^2 = \frac{\sum_{i=1}^{n}(Y_i-\bar{Y})^2}{(n-2)}
\]  

(2.17)

in which \( \bar{Y}_i \) is \( Y_i = \hat{A} + \hat{B} \cdot X_i \) and represents the estimated value of \( Y \) using the calculated estimators \( \hat{A} \) and \( \hat{B} \). The (n-2) term in the denominator is used instead of \( n \) to make \( \sigma^2 \) an unbiased estimator of the normal population variance \( \sigma^2 \).
On the other hand, if one takes logarithms of both sides of Basquin’s equation (2.6) and rearranges the terms, the following equation can be obtained:

\[
\log(N) = -\frac{1}{b} \log(S_f) + \frac{1}{b} \log(S_a)
\]  

(2.18)

Then, by comparing the latter equation with the MLH equation 2.12 it can be determined that if \( Y = \log(N) \) and \( X = \log(S_a) \) then, the estimators would be \( \hat{A} = -\frac{1}{b} \log(S_f) \) and \( \hat{B} = \frac{1}{b} \).

Thereby, the values of the strength coefficient and the strength constant can be deduced as:

\[
S_f = \exp(-\hat{A}b)
\]  

(2.19)

\[
b = \frac{1}{\hat{B}}
\]  

(2.20)

2.3.5.2 Design curve (lower safety band):
A design curve is a line that characterizes the minimum fatigue life at a given fatigue stress level so that the majority of the fatigue data fall above this minimum or lower bound value. This line can be obtained by taking the median S-N curve and shifting it to the left by a constant value times the standard deviation. Hence the design curve can be estimated as:

\[
Y_l = Y - K \cdot \sigma
\]  

(2.21)

where \( Y_l \) is defined as the lower limit of \( Y \) at a given \( X_m \), \( \sigma \) is the standard deviation and \( K \) is a multiplier whose value varies depending on the design curve methodology chosen. The ASTM methodology suggests the use of a hyperbolic double sided confidence band as a design curve. However, they agree on the use of confidence and tolerance bands parallel to the fitted median S-N line. On the other hand, the choice of a lower tolerance limit is arbitrary and dependent on materials cost, safety policy and industry standards [13]. Based on all these and for the sake of the simplicity of the calculations the design curve will be estimated using the Owen one-sided tolerance limit found in reference [13] where the constant value is approximated by \( K_{owen} \).
The design S–N curve then can be expressed as follows:

\[ Y_{R95C90} = Y - K_{OWEN} \cdot \sigma \]  \hspace{1cm} (2.22)

The K value is derived with the following expression:

\[ K_{OWEN} = K_D \cdot R_{OWEN} \]  \hspace{1cm} (2.23)

Where:

\[ K_D = c_1 \cdot K_R + K_C \sqrt{c_3 K_R^2 + c_2 a} \]  \hspace{1cm} (2.24)

\[ R_{OWEN} = b_1 + \frac{b_2}{f b_3} + b_4 \exp(-f) \]  \hspace{1cm} (2.25)

In which

\[ K_R = \varphi^{-1}(R) \]  \hspace{1cm} (2.26)

\[ K_C = \varphi^{-1}(C) \]  \hspace{1cm} (2.27)

\[ f = n - 2 \]  \hspace{1cm} (2.28)

\[ a = \frac{1.85}{n} \]  \hspace{1cm} (2.29)

Where \( \varphi \) is the error function.

The values of C and R are the confidence level and the reliability respectively. For example when R95C90 is used for design purposes, that value ensures that there is a 95% chance of survival with a 90% of confidence level for fatigue life at a specified fatigue level [9]. In this thesis values of C=90% and R=95% will be used.

It was decided to use \( K_{owen} \) because it takes into account the sample size and also allows to use any reliability value higher than that of E739 (R50%).

2.3.5.3 Sampling for strain-controlled testing

Standard practice E739 recommends a minimum of specimens to be tested depending on the purpose of the research. They are summarized as follows:
Table 2.1 Minimum of specimens required depending on the type of test program to be carried out

<table>
<thead>
<tr>
<th>Type of Test</th>
<th>Minimum of specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preliminary and exploratory (exploratory research and development tests)</td>
<td>6 to 12</td>
</tr>
<tr>
<td>Research and development testing of components and specimens</td>
<td>6 to 12</td>
</tr>
<tr>
<td>Design allowable data</td>
<td>12 to 24</td>
</tr>
<tr>
<td>Reliability data</td>
<td>12 to 24</td>
</tr>
</tbody>
</table>

On the other hand, when performing this kind of tests where statistics are involved the more specimens tested the better the reproducibility of the results. In this sense ASTM proposes replication guidelines which depend in the number of stress levels and the number of specimens tested at each level. Table 2.2 shows ASTM replication recommendations:

Table 2. Percentage of replication depending on the type of test program to be carried out

<table>
<thead>
<tr>
<th>Type of Test</th>
<th>Percentage of replication</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preliminary and exploratory (exploratory research and development tests)</td>
<td>17 to 33</td>
</tr>
<tr>
<td>Research and development testing of components and specimens</td>
<td>33 to 59</td>
</tr>
<tr>
<td>Design allowable data</td>
<td>50 to 75</td>
</tr>
<tr>
<td>Reliability data</td>
<td>75 to 88</td>
</tr>
</tbody>
</table>

They percentage of replication is calculated as:

\[
\%\text{Replication} = 100 \left[ 1 - \frac{\text{Total number of different stress levels used in testing}}{\text{Total number of specimens tested}} \right]
\]

The percent replication indicates the portion of the total number of specimens tested that may be used for obtaining an estimate of the variability of replicate tests.
2.4 Strain-Life approach

In contrast with the stress-life approach, this methodology for describing the fatigue behavior of a component uses the local strain as the governing fatigue life parameter. As it was mentioned before, the stress-life approach is suitable in those cases of low stress loading where the majority of the deformation is elastic. However, there are cases of high stresses where plasticity is present. Furthermore, there are also situations where even when the component appears to have nominally cyclic elastic stresses local plastic deformation can be present (stresses can locally exceed the yield stress) in areas of stress concentrations such as welds or notches in the component. In this context, the use of a strain-life approach is recommended and has been shown to be more effective in the prediction of the fatigue life [13].

The results of a strain-controlled test generally are used to plot the strain-life relationship where the number of cycles to failure is plotted against the correspondent strain levels. A statistical analysis can be performed as the one explained before for the stress-life curve to obtain the entire curve. However, the estimation of the strain-life curve is out of the scope of this thesis since just the relationship between the applied stress and the fatigue life is of interest specifically at relatively low stresses which can be fairly estimated by performing stress-controlled tests. Still, strain-controlled testing also allows estimating the cyclic stress-strain curve of the material by applying fully reversed constant amplitude strains. According to A. Jones [17] it is necessary to derive the cyclic stress-strain curve of the material since the information provided by a monotonic curve is just an approximation, especially when significant hardening or softening takes place.

2.4.1 Material response under cyclic loading:

Under constant amplitude, strain-controlled fatigue loading the stresses in the material at the initial cycles varies with each reversal. This initial stage of change in stress amplitudes is known as the transient cyclic response of the material and describes how the resistance to deformation changes due to cyclic loading. In this respect, the material can behave in one of the following manners: cyclic hardening,
cyclic softening, remain stable or a combination of both. In the case where there is transient cyclic hardening, the flow stress will increase with increasing the number of cycles. In transient cyclic softening the flow stress is lower at each successive cycle. However, the rate of change in stress in both scenarios decreases and after a certain number of cycles the material reaches a steady-state where the stress remains almost constant despite continuous cycling, i.e. the material stabilizes and it is said that it has reached saturation. Once the steady state condition is reached it can be observed by plotting the stress versus the strain amplitude. Generally the stabilization is taken at half of the fatigue life. The resulting plot (shown in fig. 2.3) is known as the hysteresis loop of the material which represents the elastic plus plastic work on it after being subjected to loading and unloading. The stress at which the hysteresis loop stabilizes at a given strain amplitude is called peak stress. In this sense, if a group of stabilized hysteresis loops at different strain levels is plotted, a cyclic stress-strain curve can be obtained by joining the tips of the peak stresses of each loop as shown in figure 2.3.

![hysteresis loops](image)

**Fig. 2.3** Hysteresis loops at different strain levels and the cyclic stress-strain curve formed by joining the locus of the loop tips. The tensile curve is superimposed in the figure to show the relationship between the cyclic and monotonic behavior of the material. (Figure extracted from reference [13])
2.4.2 Cyclic vs. Monotonic behavior:
The cyclic stress-strain curve (CSS) reflects the resistance of a material to cyclic
deformation and can be different from the monotonic stress–strain curve. Commonly
these two curves are compared by plotting them in a same graph and observing if the
material under cyclic conditions hardens, softens, remains stable or has a mixed
behavior compared to that under a monotonic tensile situation.

When the true stress vs. the true strain from a tensile test is plotted, the curve is
approximately described by Ramberg-Osgood relationship as [11] [13]:

\[ \varepsilon = \frac{\sigma}{E} + \left( \frac{\sigma}{K} \right)^{1/n_m} \] (2.30)

Where \( \sigma \) is the true stress, \( E \) is the Young’s modulus, \( \varepsilon \) the total true
strain, \( K \) is a constant called the monotonic strength coefficient and \( n_m \) is known as the monotonic
strain hardening exponent.

The total strain experienced by the material can be composed in the elastic and plastic
part, then:

\[ \varepsilon_e = \frac{\sigma}{E} \] (2.31)

\[ \varepsilon_p = \left( \frac{\sigma}{K} \right)^{1/n_m} \] (2.32)

Where \( \varepsilon_e \) and \( \varepsilon_p \) are the elastic and plastic strains.

Similarly, the resultant CSS from the strain controlled test can be described by:

\[ \varepsilon_a = \frac{\sigma_a}{E'} + \left( \frac{\sigma_a}{K'} \right)^{1/n'} \] (2.33)

The latter expression is based on Masing’s assumption (Masing, 1926) found in
reference [13], [18]. Here \( \sigma_a \) is the stress amplitude, \( E' \) is the Young’s modulus, \( \varepsilon_a \) the
strain amplitude, \( K' \) is the cyclic strength coefficient and \( n' \) is known as the cyclic strain
hardening exponent. The CSS is plotted using the values of the stress and strain
amplitudes of the stabilized hysteresis loops, which commonly are taken at half of the
fatigue life.
The material constants $K'$ and $n'$ can be estimated as the intercept and slope of the line that best fit the plastic strain vs. stress amplitudes in a log-log scale which has the form:

$$
\sigma_a = K'(\varepsilon_p)^{n'}
$$

(2.34)

Or in an equivalent form would be:

$$
\log(\sigma_a) = \log(K') + n'\log(\varepsilon_p)
$$

(2.35)

The former has a similar fashion to equation 2.12 and the statistical analysis presented in ASTM standard practice E739 explained in section 2.2 allows to estimate $K'$ and $n'$ as in reference [9]. In this case, the statistically independent variable is the plastic strain amplitude and the statistically dependent variable is the stress amplitude. In other words: $Y = \sigma_a$ and $X = \log(\varepsilon_p)$. The equivalent procedure applies for the estimation of the monotonic parameters $K$ and $n_m$. The typical range of $n_m$ for alloys is 0-0.5 and for $n'$ is 0.1-0.2 [13].

The plastic strain amplitudes are calculated as:

$$
\varepsilon_p = \varepsilon_a - \frac{\sigma_a}{E_t}
$$

(2.36)

Where $\varepsilon_a$ is obtained from the strain-controlled fatigue test.

As a rule of thumb (Bannantine et al., 1990) [13] the material will harden if $Su/Sy$ bigger than 1.4, it will soften if $Su/Sy$ is smaller than 1.2 and if $Su/Sy$ is between 1.2 and 1.4 the material can exhibit hardening, softening or a combination of both.

It has been mentioned that the materials during the spallation process undergo low cyclic loadings and are expected to have a long life; hence information at high cycle fatigue is of main interest. However, since the aim of this research is to characterize as much as possible the behavior of both WHAs under fatigue conditions it was decided to perform strain controlled testing as well.

2.4.3 Estimation of the Cyclic Stress Strain Curve

Strain controlled tests have gained increasing use in the determination of CSS curves for engineering alloys [11]. There are three common strain-controlled methods to
obtain the CSS curve of material which differ in the amount of specimens needed for testing, hence the time expended in the procedure. These methods are listed below and shown figure 2.4:

-Constant Strain Amplitude Method or Companion Specimen Test (CST): the specimen is cycled within a constant strain limit, until failure occurs. Multiple specimens are needed each tested at a different strain level in order to obtain the CSS curve from their resultant stabilized hysteresis loops.

-Multiple Step Test (MST): a specimen is cycled at a constant strain level until a stable hysteresis loop results. Then the strain level is incremented until stabilization again takes place. The strain level is increased until the entire CSS curve is obtained by using just one specimen.

-Incremental Step Test (IST): the specimen is subjected to blocks of increasing and decreasing strains until the stress at each level of strain becomes constant. The resulting stable hysteresis loop provides the CSS plot.

<table>
<thead>
<tr>
<th>Method</th>
<th>Strain waveform</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant Strain Amplitude</td>
<td><img src="image" alt="Waveform" /></td>
</tr>
<tr>
<td>Multiple Step Test</td>
<td><img src="image" alt="Waveform" /></td>
</tr>
<tr>
<td>Incremental Step Test</td>
<td><img src="image" alt="Waveform" /></td>
</tr>
</tbody>
</table>

Figure 2.4 Test methods for obtaining the cyclic stress-strain curve. The multiple step test method is the one used in this work.
These methods that require just one specimen are often used by investigators and it after doing literature research these authors found reports were they have been used successfully such as in reference [17, 19-21]. Researches with different materials have observed differences in CSS curves obtained by each method [17] where the differences could be explained by the dislocation substructures generated depending on the actual strain history. The cyclic strain rates might also influence since the tests can be performed using either constant strain rate or constant frequency in which case the strain rate increases with increasing the strain amplitude. However, the results from the MST and the CST should be similar and this was found successfully in the study of low carbon steel done by Polak et. al [20]. The latter giving gives a very good approximation of the cyclic stress-strain curve of the material with a long saturation stage. Considering this and for facilitate the testing procedure MST was the method used in this work for the estimation if the CSS curve of IT180 and D176.

2.5 Loading situation of the specimens

2.5.1 Stress concentrations
When an axial force is applied to a member, the stress distribution created is not uniform, but a complex stress distribution appears along the character. In the figure below (Fig 2.5) it can be seen that at the top of the member, where the force is applied (1), some distorts lines are present. Then, at the middle of the character (2) the lines are straight because they are away from the load and support, and finally at the bottom (3) distort lines also appear caused by the support [22].

![Fig 2.5](image)

The stress concentrations not only appear in this situation, but they are also present where the cross-sectional area changes. The maximum normal stress will occur at the smallest cross-sectional area, and this is what the engineering practice takes into...
account instead of determining the stress distribution when designing. On the other hand, if the material is brittle or if it will be subjected to fatigue loadings, the stress distribution becomes important. In IT180 and D176 samples this maximum stress could be placed in any cross-sectional plane of the sample neck (in the gage area), which is the part of the sample with the smallest cross-sectional area. Moreover, a deeper study will be carried out to find out where the stress concentrations appear.

When calculating stress concentration, is commonly used the stress-concentration factor $K_t$, which is defined as:

$$K_t = \frac{\sigma_{\text{max}}}{\sigma_{\text{avg}}} \tag{2.37}$$

Where $\sigma_{\text{max}}$ the maximum stress and $\sigma_{\text{avg}}$ is the average stress acting at the smallest cross section. $\sigma_{\text{avg}}$ is calculated as the load $P$ divided by the smallest cross-sectional area. $K_t$ value depends only on specimen’s geometry and the type of discontinuity.

Regarding the specimens geometry, the sharp corners and close radios should be avoided, because in those cases high stress concentrations appear. For the brittle materials, the proportional limit may be at the rupture stress, so for these materials failure will start when the proportional limit is reached. The crack will start and then propagate along the cross section, resulting in sudden fracture of the member.

### 2.5.2 Residual stresses

When compressive and tensile stresses are applied to a member, residual stresses can rely on it when the stresses are removed. When unloading, there is an elastic recovery in the material which can cause permanent deformation while the material tries to recover plastic strain. These residual stresses caused by fatigue, and also when machining and treating the materials, affect the material fatigue life.
Chapter 3 Experimental work

3.1. Specimens for testing

3.1.1 Specifications
Tungsten Heavy Alloys samples were received from Plansee, the supplier. Following is shown 3D image of the sample modeled with SolidWorks and a plan with the sample dimensions (Fig. 3.1).

![3D model and plan of the sample](image)

**Figure 3.1** Sample modeled with SolidWorks; (a) 3D view, (b) sample dimensions in mm.

The specimen’s geometry was designed so it could adapt to the testing machine available in LTH laboratory. However, reviewing ASTM standard practices to perform stress and strain controlled fatigue tests and tensile tests [4], [18], [23-24]; some differences were noted between their recommendations for the specimen’s geometry, preparation and storage and the working specimen’s conditions. These remarks are worth mention since they might affect the fatigue data obtained and the reproducibility of the results.

**Geometry:**

The specimen design should ensure that failure occur within the test section area. There is several possible specimens’ geometry but only that similar to our samples
shall be discussed. In this sense, specifications for specimens with tangentially blended fillets between the test section and the ends described in [23] are of interest. The uniform and circular gage section (testing section) should preferably have a diameter between 25.4mm and 5.08mm; to ensure the test section failure, the grip area should be at least 1.5 times the test section area, but preferably 4 times bigger. Standard practice E606 for strain controlled fatigue testing [18] suggests solid circular cross sections with a minimum diameter of 6.35mm. However, specific cross-sectional dimensions are listed there only because they have been dominant in the generation of the low-cycle fatigue database that exists in the open literature. Specimens possessing other diameters or tubular cross sections may be tested successfully within the scope of that practice; however, crack growth rate, specimen grain size, and other considerations might preclude direct comparison with test results from the recommended specimens. It is observed in figure 3.1 (b) that the nominal testing section diameter of the specimens is 5mm which already is lower than that suggested in both practices. Also the specimens were ground and polished to fulfill recommendations regarding surface conditions. As a consequence, the real diameter of the test section in the samples was lowered down to 4.99±0.01mm. It was decided to carry the surface machining under the consideration that a poor treated surface could be more detrimental to fatigue crack initiation than the effect of reducing the gage section diameter.

The blending radius should be at least eight times the test section diameter to minimize the theoretical concentration factor $K_t$ and to minimize the stress concentration. The test section length should be at least 2 times the test section diameter, especially to minimize buckling in compression. Samples with a continuous radius between ends should have a curvature radius at least 8 times the test section diameter to minimize the stress concentration [23]. In this case, the samples received didn’t accomplish this statement, since the diameter in the test section was 5mm and the blend radius was 10mm, so the results may have been affected by this issue. As the specimen geometry may affect the fatigue and fracture behavior, a FEM analysis on specimen geometry explained in following sections has been performed to observe how the stresses are distributed and where are they concentrated. In general, special
care has to be taken in low ductility materials which will be exposed to high stresses since this has been shown to be a factor in variability of test results.

Surface preparation:

About the surface preparation, the specimens have a “surface preparation history” as consequence of all the machining processes, the heat treatments performed and the environmental conditions when storing. That amount of facts can affect the fatigue behavior and the fracture behavior as well, so they should be minimized as much as possible, or in fact, they should be done to minimize their influence on specimen behavior. A smooth and uniform surface should be obtained, and machining or finishing operation should be done to ensure the minimal surface distortion. A better explanation of this is given later on this chapter.

Storage:

Oxidation and corrosion should be avoided by using protective atmospheres. The exact procedure of specimen should be clearly documented. The samples have to be stored in a suitable protective environment; in the present case the specimens were stored in deshumidificator to prevent surface attacks. In the case of tungsten special care has to be taken in this aspect, since this material has a high tendency to oxidize.

3.1.2 Inspection and preparation of the specimens:

Before performing all the tests, the specimen’s surface was inspected in the microscope at x20 magnification. According to ASTM standard practice [23], no roughness should be able to be seen in a specimen at this magnification. However all the samples in their “as received” form presented clearly visible stripes and in order to prevent this defect from being a mechanism of micro-crack growth, each specimen was grinded and polished to obtain a smoother surface. After machining, great improvement in surface roughness was observed but thin radial lines resulting from the manufacturing procedure were still visible with x20 magnification. However these lines were clearly thinner in width. The decrease in the diameter of the samples is between 0.02-0.03 mm, thereby the fact that there was still presence of stripes provides an estimate in the depth of the roughness. Below there are two images which
show the difference of the surface observed before and after the grinding and polishing.

During the length of the project the optical microscope that allowed us to record digital images of the samples as well as measure the size of the porosity on the specimens was damaged. Even though the smoothness of the Densimet 176 was checked after the grinding and polishing and every sample was inspected qualitatively no digital images of the pores or surface of this alloy could be recorded.

On the other hand, another defect observed in the specimens was the presence of pores, which is a typical characteristic of materials made of powder metallurgy process. It was of interest to see if once grinded and polished the pores size could be decreased or even eliminated.

3.1.3 Density calculations
The density of both alloys was theoretically and experimentally calculated using Archimedes’ principles and the percentage of porosity have been calculated. Moreover, the experimental value can be compared with the values given by Plansee, the specimens’ provider. Following tables show the results obtained by calculating the theoretical density and measuring the density for 3 specimens of each alloy by Archimedes’ principle.
Table 3.1 Results obtained by calculating the theoretical density for IT180

<table>
<thead>
<tr>
<th>Elements</th>
<th>%weight</th>
<th>Theoretical Density [g/cm$^3$]</th>
<th>Density [g/cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>95</td>
<td>19,25</td>
<td>17,29</td>
</tr>
<tr>
<td>Ni</td>
<td>3,5</td>
<td>8,908</td>
<td>0,64</td>
</tr>
<tr>
<td>Cu</td>
<td>1,5</td>
<td>8,96</td>
<td>0,28</td>
</tr>
<tr>
<td>TOTAL</td>
<td></td>
<td></td>
<td>18,2</td>
</tr>
</tbody>
</table>

Table 3.2 Results obtained by calculating the theoretical density for D176

<table>
<thead>
<tr>
<th>Elements</th>
<th>%weight</th>
<th>Theoretical Density [g/cm$^3$]</th>
<th>Density [g/cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>92,5</td>
<td>19,25</td>
<td>16,27</td>
</tr>
<tr>
<td>Ni</td>
<td>5</td>
<td>8,908</td>
<td>0,88</td>
</tr>
<tr>
<td>Fe</td>
<td>2,5</td>
<td>7,874</td>
<td>0,44</td>
</tr>
<tr>
<td>TOTAL</td>
<td></td>
<td></td>
<td>17,59</td>
</tr>
</tbody>
</table>

Table 3.3 Results obtained by calculating the experimental density for IT180

<table>
<thead>
<tr>
<th>SPECIMEN</th>
<th>Mass(air) [g]</th>
<th>Mass(water) [g]</th>
<th>Density [g/cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>41,4884</td>
<td>39,1891</td>
<td>18,0439264</td>
</tr>
<tr>
<td>9</td>
<td>41,471</td>
<td>39,1746</td>
<td>18,059136</td>
</tr>
<tr>
<td>8</td>
<td>41,7085</td>
<td>39,3992</td>
<td>18,0611008</td>
</tr>
</tbody>
</table>

Table 3.4 Results obtained by calculating the experimental density for D176

<table>
<thead>
<tr>
<th>SPECIMEN</th>
<th>Mass(air) [g]</th>
<th>Mass(water) [g]</th>
<th>Density [g/cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>40,7474</td>
<td>38,4369</td>
<td>17,6357498</td>
</tr>
<tr>
<td>9</td>
<td>40,455</td>
<td>38,1493</td>
<td>17,5456477</td>
</tr>
<tr>
<td>10</td>
<td>40,5266</td>
<td>38,2107</td>
<td>17,4992875</td>
</tr>
</tbody>
</table>
The mean value for the experimental density of IT180 is 18,05 g/cm\(^3\) while for D176 is 17,56 g/cm\(^3\). The values got from Plansee were 18,0 g/cm\(^3\) and 17,6 respectively, so the results obtained are correct. Then, the percentage of porosity can be calculated as:

Table 3.5 Percentage of porosity for each alloy

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Porosity [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>IT180</td>
<td>0,782430608</td>
</tr>
<tr>
<td>D176</td>
<td>0,187861074</td>
</tr>
</tbody>
</table>

As observed with the optical microscope, the IT180 has more porosity than D176.

3.1.2.1 Grinding and polishing procedure:
The selection of grinding and polishing procedure was limited to the available facilities. First, the grinding was done using a sequence of grinding paper of 320, 500, 1000 and 4000 particles per square inch. Then, the sample was polished using 3µm diameter diamond (DP suspension). The specimen was observed in the microscope at x20 magnification and since some stripes were still visible, it was proceed to polish again but using a 1µm diameter diamond so a smoother surface was finally obtained.

3.2 Microstructural Examination

3.2.1 Preparation of the samples
Extraction of two small and perpendicular sections on the grip area of one specimen of each alloy that had been used during tensile testing was performed. This particular specimen was used under the belief that since it had been subjected to tensile testing no remarkable difference on the microstructure should exist compared to that of the as-received samples. The sectioning was done with an aluminium oxide cut-off wheel (Φ=125mm) for Accutom for cutting of hard ferrous materials (HV>500) (Fig 3.4). Coolant was used to ensure an almost plane surface and avoid mechanical or thermal damage.
Encapsulation of the sample was done in two layers of Struers compression molding resins. The bottom layer consisted of a bakelite hot mounting resin and the top consisted of a diallyphtalate hot mounting resin that facilitates the subsequent machining of the sample. The complete mounting of the sample was done in a Predopress Struers machine that preheats, heats, press and cools the capsule so the resins can melt and the solidify. (Fig 3.5)

Grinding with a sequence of 320, 500, 1000 and 4000 of SiC paper followed by polishing with 3μm and 1μm Diamond suspension was performed using an abrasive rotating wheel (Fig 3.4). Cleaning of the specimen was finally done and it was then ready for inspection on the microscope.
3.2.2 Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS)
The SE microscope uses a high-energy beam of electrons to scan the samples surface in a raster scan pattern that can be of a rectangular or a square form. The electrons scan the surface until they escape from the material or are absorbed by it. A volume of the part that is analyzed with the incident beam is irradiated and different signals are generated, i.e. secondary electrons, back-scattered electrons and X-rays. Each of these signals varies in intensity depending on the properties of the material and the topography of the surface. Thus these intensities can be measured and allow to reconstruct the image and extract information of the surface and its properties. Back scatter electrons were used in our work to analyze both the microstructures and the fractures surfaces of the materials of interest. X-rays were used for mapping of the microstructure by energy dispersive microscopy since these kinds of beams are characteristic of the atoms being irradiated. Hence information of the chemical composition of the different phases of the material could be obtained [25].
Figure 3.7 XL 30 ESEM used to study the simple surface

The prepared sample was put into the SEM and vacuuming of the chamber was proceeded. The analysis used a 1.75 A filament current and 20 kV beam voltage.

3.3. Equipment needed for mechanical testing.
A machine for tensile and fatigue testing have been used to perform both type of tests at the M building Laboratories in LTH. This machine is shown in the following pictures. The machine used consists in two arms, the lower one which is fixed, and the upper one is the responsible for the load application. When the test starts the lower arm holds the specimen in a fix position while the second arm applies the force. In the case of the tensile test it slowly increases the force until the specimen brakes. In the case of the fatigue tests it applies a sinusoidal load with a frequency of 30 Hz until the specimen brakes as well and the machine stops. The machine has a hydraulic system that provides the ideal pressure to hold the specimen with the grips (10 bar).

Below, there are some images of the machine used to perform the tests.
3.3.2. Preparation of the test
Firstly, the strain and load are calibrated and set to zero. It’s necessary to align the specimens; otherwise multiaxial stresses could appear inside it and consequently falsify the results. To do so, a metal piece with 3 sensors (Fig 3.9) is put between the arms, where the specimen will be put afterwards, and it is held by the clamps. After alignment is ready, the metallic piece with sensors is changed for the sample. In the case of the tensile test, an extensometer is placed in order to measure the elongation of the specimen. After this last step, everything is ready to run the test.

Figure 3.8 Equipment used for tensile testing and fatigue testing

Figure 3.9 Metal piece with sensors used to align the specimen
Figure 3.10 Sample ready for the test
Figure 3.11 Extensometer used to measure the deformation in the tensile test.
3.4. Tensile test
First of all, in order to know the material behavior and some important properties necessary to do the fatigue tests, a tensile test was performed. In this case, 2 samples of each heavy tungsten alloy have been tested.

The tensile test consists in applying an increasing load in the specimen axial direction, until final failure occurs, i.e. rupture of the specimen. With this kind of test, one can measure the material resistance to the application of a static load (or a slowly increasing one). In this case, the rate of elongation was set to 3mm/min (0,05mm/s).

Remark in 1st tensile test of IT180: When the first test was started there was a sudden stop of the equipment due to a mistaken configuration during its preparation. There was a load control system on and it was activated because the pressure of the grips was less than the recommended (10 bar). However the test was run again after setting the right pressure level (the sample was not removed and the machine was not calibrated again).

Remark in 1st tensile test of D176: a default setting of the program was to stop the test after a length of 10 mm. Since D176 is very ductile, the machine stopped before the rupture of the piece. The test was run again with another sample and the results presented here are based on this.

Due to the inconvenient of the first tensile tests, a second specimen of each alloy was tested and the results from these tests are the ones used for the calculations.

3.5 Fatigue Tests

3.5.1 Stress-Controlled testing:
As it was mentioned before, stress controlled fatigue testing have been performed. However, the entry parameter for the machine is the applied load and not the stress amplitude. Thus the equivalent load for each desired stress level has to be calculated. Being an axial load case the applied load \( P \) is calculated according to equation 3.1 below, where \( \sigma \) is the stress amplitude and \( d \) the sample testing section diameter. It has to be said that the diameter is not the same value in all the samples because as mentioned before, all the samples were equally prepared and grinding and polishing
was done, so since it’s a hand-made process the same diameter for all the samples couldn’t be achieved after the surface treatment.

\[
\sigma = \frac{P}{A} \rightarrow P = \sigma \cdot A \rightarrow \sigma = \sigma \cdot \pi \cdot \frac{d^2}{4} \quad (3.1)
\]

The failure criterion is the complete separation of the piece and the number of cycles to run-out is \(2 \cdot 10^6\).

IT180 was the first WHA to be tested. The strategy was to test at different stresses trying at the same time to come as close as possible to the fatigue limit. In this sense, after the first test, if failure occurred then the next stress level would be lowered while it would be higher in case of no failure.

Keeping in mind that the UTS for this alloy is 631 Mpa and that the endurance limit for powder metallurgy materials is usually found between 30-40% of this value [16], the first test for this alloy was run at a stress amplitude of 300 Mpa (~50%) expecting that the sample will fail but not far from the fatigue limit. Failure occurred at 33,831 cycles. In order to get reproducibility on the results a second experiment was run also at 300 MPa, and 40,177 cycles of life were obtained. Afterwards, the stress amplitude was decreased to 200 MPa, obtaining for the 3\textsuperscript{rd} specimen tested an infinite life, in this case the specimen didn’t brake until a life up to \(6 \cdot 10^6\) cycles so it was decided to stop it and consider it a run-out. This is why due to the length of the testing (this last run for more than two days) and to the fact that for most materials\(^1\) the fatigue limit is reached beyond about \(10^6\) cycles the fatigue life for a run-out was considered at \(2 \cdot 10^6\) cycles. Then, one more specimen was tested at 200 MPa stress amplitude, and it didn’t brake either, so the test was stopped at \(2 \cdot 10^6\) cycles. At this point, the endurance limit was situated clearly between 200 and 300 MPa, so 5\textsuperscript{th} specimen was decided to be run at 250 MPa, obtaining 139,854 cycles as a result. Next test was run at 225 MPa, and the specimen failed after 336,042 of fatigue. Following 3 tests were run at 210 MPa in a row, having as a result 312,378, \(2 \cdot 10^6\) and 1,626,921 cycles respectively. At this point, the endurance limit was set around 210 MPa for the IT180. Finally two more

\(^1\) Value typical for mild steels and other materials which harden by strain-ageing [11].
specimens were tested at 250 and 225 MPa breaking at 329748 and 424545 cycles respectively.

Fatigue testing of D176 followed next. Starting with run-outs of specimens 3 and 4 at 300 Mpa and 400 respectively. Thereby specimen 5 was tested at a higher stress equal to 500 MPa, braking at 958.423 cycles. Next test was run at 450 MPa, obtaining 885.444 cycles as a result. Then, 550 MPa was decided as the stress amplitude for the next test, and the specimen failed at 213.838 cycles. Specimen 8 was run again at 450 MPa, obtaining 1.468.220 cycles as a result, and Specimen 9 was run at 550 MPa, braking at 258.788 cycles.

The following tables summarize the specifications for every specimen, as well as the load at which it was subjected to.

**Table 3.1.** Contains the stress amplitude at which every specimen was exposed to, its diameter, the load according to the amplitude mentioned and the cycles before breaking for IT180.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Stress [MPa]</th>
<th>Diameter [mm]</th>
<th>Load [N]</th>
<th>Life [cycles]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>300</td>
<td>4,99</td>
<td>5866,94784</td>
<td>33831</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>4,99</td>
<td>5866,94784</td>
<td>40177</td>
</tr>
<tr>
<td>5</td>
<td>200</td>
<td>4,98</td>
<td>3895,63772</td>
<td>6,00E+06</td>
</tr>
<tr>
<td>6</td>
<td>200</td>
<td>4,98</td>
<td>3895,63772</td>
<td>2,00E+06</td>
</tr>
<tr>
<td>7</td>
<td>250</td>
<td>4,98</td>
<td>4869,54715</td>
<td>139854</td>
</tr>
<tr>
<td>8</td>
<td>225</td>
<td>4,98</td>
<td>4382,59244</td>
<td>336042</td>
</tr>
<tr>
<td>9</td>
<td>210</td>
<td>4,99</td>
<td>4106,86349</td>
<td>312378</td>
</tr>
<tr>
<td>10</td>
<td>210</td>
<td>4,99</td>
<td>4106,86349</td>
<td>2,00E+06</td>
</tr>
<tr>
<td>11</td>
<td>210</td>
<td>4,99</td>
<td>4106,86349</td>
<td>1626921</td>
</tr>
<tr>
<td>12</td>
<td>250</td>
<td>5,00</td>
<td>4908,73852</td>
<td>329748</td>
</tr>
<tr>
<td>13</td>
<td>225</td>
<td>5,00</td>
<td>4417,86467</td>
<td>424545</td>
</tr>
</tbody>
</table>

**Table 3.2.** Contains the stress amplitude at which every specimen was exposed to, its diameter, the load according to the amplitude mentioned and the cycles before breaking for D176.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Stress [MPa]</th>
<th>Diameter [mm]</th>
<th>Load [N]</th>
<th>Life [cycles]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>300</td>
<td>5</td>
<td>5890,48623</td>
<td>2,00E+06</td>
</tr>
</tbody>
</table>
3.5.2. Strain-Controlled Testing:
Constant strain amplitude axial fatigue tests were carried out in three specimens of IT180 and D176 in order to obtain the cyclic stress-strain curve of the alloys. All the tests were fully reversed where the mean strain was zero. One specimen of IT180 was tested at a strain ratio R of . In all cases the control variable was the total strain range even when some literature reports suggest the plastic strain as the measured variable because this one was not able to measure by the equipment used. The servo-hydraulic machine is controlled by a computer where a wave-maker program allows applying the desired loading during the tests. During the length of the test data of the applied load together with the longitudinal deformation (in mm) was stored in a file in the computer. Every specimen was aligned as it was done for the stress-controlled and tensile tests. The contacting extensometer had a gauge length of 12.5 mm.

Creation of the strain program:

Since the amount of specimens available was small it was decided to use and for the sake of the simplicity the method chosen for the estimation of CSS curve was the multiple step strain test. A set of blocks was created each of them having a triangular loading waveform with constant strain amplitude which was increased at every new block. The initial straining direction was tensile in all tests.

Firstly, two specimens of IT180 were tested at strain amplitudes of 0.0042, 0.0046, 0.0050, 0.0054, 0.0058, 0.0062, 0.0066, 0.0070, 0.0074, and 0.0078 mm. One specimen was tested at fully reversed loading and the other with R=-0.5 with a constant frequency of 0.2Hz and 5 cycles per block. It was observed that these strain
sequence gave strain in pct that ranged only within the elastic part of the alloy. Also it was noticed that using a constant strain rate was more convenient for the desired purpose than using a constant frequency since similar experiments found in literature reports used a constant strain rate [17, 19-20]. Because of it the information obtained with these two tests was not considered much useful and no further discussion of the data was done.

Thereby, for subsequent tests the frequency at each block was changed in order to maintain a constant strain rate of 0.05mm/s equal to that used in tensile testing. In the case of IT180 the strain amplitudes programmed were 0.1, 0.2, 0.3, 0.4, 0.5 pct. Since the entry parameter for the program was the gage elongation in [mm], the correspondent elongation amplitude was calculated based on the extensometer length to provide the pct desired. For example an elongation of 0.0125 mm is equivalent to 0.1 pct.

The first fully reversed test consisted in 200 cycles per block. However the specimen broke during the second block so the number of cycles for the next specimen tested was lowered to 30 cycles. Yet the specimen broke again at the second block. In the case of D176 the strain amplitude sequence was of 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, and 1 pct. Even when complete separation of the sample didn’t occur, a crack on the surface was visible when the test finished.

According to ASTM standard E606 [18] there are several failure criterions depending on the final use of the fatigue life information. In this sense, it was not of interest to cycle the specimen until complete separation occurred so the existence of a surface microcrack was enough to consider the specimen as failed. Table 3.3 below summarizes the strain amplitudes together with their correspondent frequencies for the last three specimens.

Table 3.3 Cyclic frequencies at each strain amplitude. Note that IT180 was tested at strains between 0.1 and 0.5pct. D176 was tested at all strains shown starting from 0.2pct.

<table>
<thead>
<tr>
<th>Strain amplitude [pct]</th>
<th>Strain amplitude [mm]</th>
<th>Frequency [Hz]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.0125</td>
<td>1</td>
</tr>
<tr>
<td>0.2</td>
<td>0.025</td>
<td>0.5</td>
</tr>
<tr>
<td>0.3</td>
<td>0.0375</td>
<td>0.333</td>
</tr>
<tr>
<td>0.4</td>
<td>0.05</td>
<td>0.25</td>
</tr>
<tr>
<td>0.5</td>
<td>0.0625</td>
<td>0.2</td>
</tr>
<tr>
<td>0.6</td>
<td>0.075</td>
<td>0.167</td>
</tr>
<tr>
<td>0.7</td>
<td>0.0875</td>
<td>0.143</td>
</tr>
<tr>
<td>0.8</td>
<td>0.1000</td>
<td>0.125</td>
</tr>
<tr>
<td>0.9</td>
<td>0.1125</td>
<td>0.111</td>
</tr>
<tr>
<td>1</td>
<td>0.125</td>
<td>0.100</td>
</tr>
</tbody>
</table>
Chapter 4: Results and Discussion

4.1. Surface inspection
The valleys on a rough surface serve as stress concentrations and a possible place for the nucleation of a dominant crack. In order to reduce this effect as much as possible, the samples were polished and grinded as explained in chapter 3. However, residual stresses arising from the mechanical treatments when superimposed with the applied fatigue loads alter the mean level cycle as the mechanical stresses superimposed on cyclic stress amplitude [11]. In this sense, residual stresses would be detrimental if tensile and favourable if compressive as explained in section 2. Yet it was decided to carry out the surface treatment with the thought that the effect on fatigue strength of a rough surface could be more detrimental that the possible residual stress induced by the surface treatment. However it could be recommended a heat treatment after the wanted surface is achieved to release these residual stresses.

Porosity is a typical defect of materials produced by powder metallurgy process, since the complete theoretical density is almost impossible to achieve after sintering. However, the inspection of the IT180 with the optical microscope showed that every sample presented a considerable amount of pores and cracks of different size lengths that could actually affect the fatigue response of the material. The largest pore registered had a length of 280 μm found on specimen number 4. Pores larger than 100 μm diameter are considered dangerous and potential causes of future crack growth, thereby a register of the approximate localization of them is relevant to later compare if the failure of the specimens is related to these pores. Following there are two pictures of the same area of specimen 2, before and after grinding and polishing, taken at x100 magnification. Note also the diminishing of the roughness of the surface.
During the final inspection on the microscope it could be observed that pores and cracks of significant magnitude (more than 100 µm length) kept more or less the same dimensions and shape. However, there are some areas where the pores were eliminated while appearance of new pores was seen in other areas. In some places, small pores joined together to form a bigger one. Since there is no specific pattern in the presence, absence or shape of the porosity it is thought that this defect is present throughout all the material and is a defect that cannot be eliminated. Furthermore, it is known that pores on the free surfaces are likely to start a crack, however they are not necessarily the only cause for crack initiation since defects present within the material can also induce failure, and these ones cannot be registered or inspected prior testing.

N. Chawla on his work with powder metallurgy steels [26,27] used a pore shape form factor established by DeHoff to characterize the porosity of the material. Following the Shape Factor formula is shown:

\[ F = \frac{4 \pi A}{P^2} \]  

(4.1)

Where A is the area of the pore, and P its perimeter.

As some samples were exhaustively observed with an optical microscope at x100 magnification, all the pores bigger than 100µm were registered with their position and size.
It was decided to use the same $F$ to characterize pore shape as N. Chawla in this work so the area and perimeter of the pores were measured and the irregularity of pores was estimated.

![Shape Factor](image)

**Figure 4.3** Shape factor calculated for all the pores of 3 specimens

Values of $F$ near to one represent a nearly spherical pore shape while values tending to zero are pores completely irregular. As shown in graph 4.3 the bigger pores presented a lower shape factor whereas the smaller ones had a higher shape factor what indicates a more regular and near to round shape. On the other hand, while crack initiation in powder metallurgy materials is associated with the size and shape of the pores, the mean pore spacing is the principal factor that affects crack propagation rate. When pores are near each other the crack will propagate faster because it needs to travel a shorter distance before finding another pore which will increase instantly the crack length and continue propagating. Due to the higher volume of porosity of IT180 the material had more zones with clusters of pores instead of isolated pores like in D176. However, in materials with a high density but yet a certain amount of porosity, the presence of pores act as a delay or deviation for cracks, reducing the speed of the crack propagation because in this case pores represent a discontinuity in the material, making the path for the crack more tortuous.
On the other hand, it has been observed that the porosity on the specimen was not uniformly distributed, i.e., there is more concentration of pores near the ends of the samples neck, where all the fractures have taken place indicating that failure might have been related to porosity localized in that zone.

A study on this field has been done to see where the pores were concentrated as it can be a potential cause of fracture initiation in the samples. The next picture (Fig 4.4) shows where the pores were observed in a population sample of 8 specimens. It’s clearly visible that near the blend radius, where the neck starts, there is a high amount of pores not present in other parts of the specimen. The rest of superficial porosity is uniformly distributed along the testing area.

Figure 4.4 Pore distribution along the specimen neck
4.2 FEM Analysis
A Finite Element Analysis (FEM) analysis has been carried out in order to find out how the specimen geometry affects to the rupture pattern. The specimens used in this project have the same geometry for both alloys; the study was carried out with IT180 properties.

First of all, the sample geometry was modeled with the 3D CAD software SolidWorks, and afterwards it was analyzed and simulated using ABAQUS. The simulation consisted in applying pressure in two opposite sides at the top of the specimen, perpendicular to the faces, simulating the pressure made by the grips, and then a distributed shear force in the same faces, simulating the force applied by the testing machine. The specimen is fixed by the other side. By this simple way, the stress distribution will be obtained, so it can be a good clue when designing new specimens for future studies.

The first analysis was done to a model with exactly the specimen size and dimensions, applying a 10kN force on the top of the specimen. This force was chosen because it’s not enough to make the specimen fail but enough to create considerable stresses on it. As the purpose is to see where the stresses are concentrated, the force value chosen is good for this goal. Most part of the meshing was done using hexahedral elements, while the two neck ends were meshed using tetrahedral elements because of the complicated geometry. Once the meshing was ready, the simulation was obtained (Fig 4.4)

![Figure 4.4](image)

**Figure 4.4** First analysis of specimen stress distribution to the real size model
The scope of the simulation was to find out the maximum principal stress value and location, as it is the responsible of the sample failure, to be able to study its variation when modifying softly the specimen geometry. After obtaining the real sample size simulation, the radii from the grips to the neck (blend radii) were modified, making them bigger and shorter, to see how this affect to the stress distribution. Three more analysis were done to samples with different radios, showed below:

**Figure 4.5** Second FEM analysis, with 8 mm radius model

**Figure 4.6** Second FEM analysis, with 20 mm radius model
4.2.4 FEM Results
The FEM showed interesting results about stress distribution in the specimen geometry. The first test, as said before done with the real sample dimensions, showed a value of 526.9 MPa of maximum principal stress on the sample. It’s important to know the value to compare it with the following analysis with different radius, to see how much better or worse is to modify the critical parameters. Another field of study is where the stress concentrations take place, because the other goal of the FEM study is to try to distribute the principal maximum stresses along the neck sample, and not concentrate them in the neck ends. Because of this fact, in the other analysis the neck ends radiiuses were modified, making them both bigger and smaller, to observe how the specimen behaves with these soft changes. As the sharp corners and sudden cross-section area changes should be avoid not to concentrate stresses, the radius should be bigger, but the contrary case was also studied to prove this argument.

In the second analysis, a model with 8mm of radius was studied (the real one radius is 10mm), and a higher maximum principal stress appeared in the same region as the first simulation. The high stress is still present in the ends of the neck, as was in the first simulation. Then, the third analysis was performed, in this case with a specimen with 20mm of radius, and occurred that the maximum principal stress was lower than the first and second analysis and the stresses were more uniformly distributed along the neck, not that concentrated in both sides. Finally, a fourth simulation was
performed, modeling the specimen with 30mm radius, and in this case the maximum principal stress was lower, and the tensions were more distributed along the neck. The following table shows the numerical results obtained with the simulations:

Table 4.1: Contains the radius of the different models created and the maximum principal stress appeared in the simulation.

<table>
<thead>
<tr>
<th>Radius [mm]</th>
<th>8</th>
<th>10</th>
<th>20</th>
<th>30</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. Principal Stress [Mpa]</td>
<td>560,5</td>
<td>526,9</td>
<td>426,9</td>
<td>363,3</td>
</tr>
</tbody>
</table>

The concentration factor $K$ (Eq x) can be calculated for last cases, calculating first $\sigma_{avg}$.

\[
\sigma_{avg} = \frac{F}{A_{neck}} = \frac{F}{\pi \left(\frac{d^2}{4}\right)} = \frac{4 \cdot 10000}{\pi 5^2} = 509,31 \text{ MPa}
\]

Table 4.2: Contains the radius of the different models created, the maximum principal stress appeared in the simulation and the concentration factor $K$ in each case.

<table>
<thead>
<tr>
<th>Radius [mm]</th>
<th>8</th>
<th>10</th>
<th>20</th>
<th>30</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. Principal Stress [Mpa]</td>
<td>560,5</td>
<td>526,9</td>
<td>426,9</td>
<td>363,3</td>
</tr>
<tr>
<td>$K$ [-]</td>
<td>1,10</td>
<td>1,03</td>
<td>0,84</td>
<td>0,71</td>
</tr>
</tbody>
</table>

It’s clear that when increasing the radius, the maximum principal stress is decreasing. The fact of increasing the radius makes the cross-sectional area change softly so the stresses are distributed along the specimen’s neck, making its geometry not that responsible of the break.

Observing the $K$ value, in the models with a blend radius of 20 and 30 mm it becomes lower than 1. It could be due to the stress distribution, so maybe the shear stresses were bigger in those cases, so the shear stresses were also analyzed for the first model. The 20 and 30 mm blend radius models show lower shear stresses and maximum as well, so it seems that all the stresses get a lower value when the blend radius is increased. Following, figures 4.8, 4.9 and 4.10 show the shear stresses in the 3 planes. This concentration also affects to the fracture behavior.
Figure 4.8 FEM analysis of the first model (10 mm blend radius) showing shear stresses $S_{12}$

Figure 4.9 FEM analysis of the first model (10 mm blend radius) showing shear stresses $S_{13}$

Figure 4.10 FEM analysis of the first model (10 mm blend radius) showing shear stresses $S_{23}$
4.3 Microstructural Examination
According to the literature, both chemical composition and microstructural features are the responsible variables influencing the mechanical properties of tungsten heavy alloys. This is why in order to provide a better comprehension as possible of the fatigue behaviour of the Inermet 180 and Densimet 176 microstructural and fracture surface analyses are performed. The objective is then to try to relate these analyses with the results obtained from the fatigue tests.

![Image of Inermet 180 microstructure](image)

**Figure 4.8** Microstructure of Inermet 180 alloy (SEM magn. 500x)

Figure 4.8 shows the microstructure of IT180 (W-3.5Ni-1.5%wtCu) specimen (º2) taken by scanning electron microscopy. It can be distinguished in the picture the presence of tungsten grains with an average diameter of 62µm and a bonding phase between these grains. Jiten Das and G. Appa Rao on their study of the microstructure and mechanical properties of tungsten heavy alloys [9] worked with an alloy of the same composition as IT180 that presented tungsten grains of ~60µm diameter.

In general W-grains are rounded in shape but flattening of the grains is observed due to rearrangement during sintering. In the picture clear coarsening of tungsten grains is
observed and a considerable presence of W-W interface areas along with porosity trapped between grains. This porosity is also related to the sintering process.

Figure 4.2 shows the microstructure of the Densimet 176 alloy. The average tungsten grain size of this alloy is 34µm. This alloy presents more rounded pores and less coarsening of the tungsten grains. In general, WNF alloys present less W-W interface areas and smaller W grains than WNC alloys.

The tungsten content is one of the main responsible for microstructural differences and subsequent change in mechanical properties of tungsten heavy alloys. Higher tungsten content requires higher sintering temperature that at the same time implies bigger grains and further coarsening of them [9]. On the other hand, increasing the amount of tungsten decreases the amount of matrix and consequently the W-matrix interface area [8]. There will be then less matrix phase located between W grains in which case a higher amount of W-W interface areas will be present. This last feature also know as contiguity characterizes the amount of W-W interface area in proportion
to the total interface area and is another important feature that affects most of all the ductility of this alloys.

X-ray mapping of the microstructure of IT180 showed the presence of the alloying elements nickel and copper mainly in the ductile bonding phase, see figure 4.3 below. The small presence of Ni and Cu within the grains areas could be particles removed by polishing during the preparation of the specimens and not completely taken away during cleaning. Whereas the X-ray mapping of D176 microstructure show a weak presence of the alloying elements nickel and iron in the bonding phase (See figure 4.4). From a quantitative point of view, electron probe micro analysis (EPMA) of the composition of both alloys helps noticing that the matrix of D176 is richer in tungsten (~50 wt%) as compared to that of IT180 (~30%wt). Furthermore, in the case of D176, the amount of nickel is very low compared to the amount of iron, what is not correspondent as it was expected since theoretically the nickel contribution should be higher than that of the other two composites.

![X-ray mapping by electron dispersive microscopy of IT180](image)

*Figure 4.9 X-ray mapping by electron dispersive microscopy of IT180*
Figure 4.10 X-ray mapping by electron dispersive microscopy of D176

Figure 4.11 Composition analysis in a spot within the ductile phase of each alloy showing (a) Amount of nickel, copper and tungsten in IT180 matrix and (b) Amount of nickel, iron and tungsten in D176 matrix. Note the high presence of W in the ductile phase of this alloy

Table 4.3 EPMA analysis (%wt) of the matrix phase of the alloys.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>W</th>
<th>Ni</th>
<th>Cu</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>IT180 spot 1</td>
<td>33.34</td>
<td>47.12</td>
<td>19.54</td>
<td></td>
</tr>
<tr>
<td>IT180 spot 2</td>
<td>30.66</td>
<td>50.77</td>
<td>18.57</td>
<td></td>
</tr>
<tr>
<td>D176 spot 1</td>
<td>51.38</td>
<td>16.39</td>
<td></td>
<td>32.23</td>
</tr>
<tr>
<td>D176 spot 2</td>
<td>50.77</td>
<td>17.32</td>
<td></td>
<td>31.91</td>
</tr>
</tbody>
</table>
4.4 Tensile testing

Two specimens of each alloy were tested under tensile loading. Information about the mechanical properties of both IT180 and D176 was provided by Plansee. However it was considered necessary to characterize the same parameters at the laboratory at LTH using the equipment that would be further used for the fatigue testing. Also, the samples to be tested did not have the same condition when as received since surface grinding and polishing was performed. Table 1 summarizes all the results obtained along with the data from Plansee.

Unfortunately, the first test run for the IT180 doesn’t provide reliable information because of the stop and later continuation of the test. The percentage of elongation to failure was 0.3% which differs from the theoretical value (3%). Strengthen of the material is supposed to have occurred during the first try of the test. The gage length used was 12.5mm.

As it can be seen Inermet180 shows a significant reduction on ultimate tensile strength (UTS), Young’s modulus and especially in percentage of elongation to fracture compared to the as-received data. The variability between the two data is sufficiently great to be ascribed to experimental error or different testing conditions. Furthermore the decrease on these properties can be due to the significant amount of porosity present on the alloys received at LTH, that may not have been present in those tested by the company. In the case of the Densimet176 an enhancement on the mechanical properties is observed that may have been induced by cold working while grinding and polishing of the specimens.

Table 4.4 Tensile properties of IT180 and D176 provided by Plansee and obtained at LTH laboratory after surface machining.

<table>
<thead>
<tr>
<th></th>
<th>IT180</th>
<th></th>
<th>D176</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>LTH</td>
<td>Plansee</td>
<td>LTH</td>
<td>Plansee</td>
</tr>
<tr>
<td>Yield strength (offset = 0.2%) [MPa]</td>
<td>612</td>
<td>610</td>
<td>654</td>
<td>620</td>
</tr>
<tr>
<td>Ultimate Tensile strength [MPa]</td>
<td>631</td>
<td>685</td>
<td>945</td>
<td>880</td>
</tr>
<tr>
<td>Fracture elongation [%]</td>
<td>0.6</td>
<td>3</td>
<td>42</td>
<td>20</td>
</tr>
<tr>
<td>Young’s Modulus [GPa]</td>
<td>308</td>
<td>360</td>
<td>309</td>
<td>360</td>
</tr>
</tbody>
</table>
Porosity produced during densification can reduce the mechanical properties of this alloys. Under monotonic tensile loading, porosity reduces the effective load bearing cross-sectional area and acts as a stress concentration site for strain localization and damage, decreasing both strength and ductility [22]. Small and spherical pores seem to have no effect on properties whereas larger pores formed during coalescence or gas evolution in over sintered specimens and porosity higher than 0.5% sharply reduce the properties specially ductility [12].

This would be confirmed with the results for D176 that had just small, rounded and almost isolated porosity within the material. However, the enhancement gained with the cold work would be overcome by the porosity effect on IT180.

The strength and ductility are controlled by the amount of “load-bearing” cross-sectional area, i.e., the cross-sectional area of material that contains no pores [22]. Thus, a high density material will have a larger deformed volume, which results in much higher ductility than a material with interconnected porosity, where deformation is more localized and the macroscopic ductility is diminished.

Comparing the properties between the two alloys, it can be noticed that there is none or almost no difference in the elastic behaviour of them, having similar yield strengths of 615 Mpa for the IT180 and 650 Mpa for the D176. However, a comparison between the ultimate tensile strengths and percentage of elongation shows a significant variation. This is explained by the higher amount of tungsten contained in the IT180. The ductility of WHAs is very sensitive to the tungsten content of the material decreasing with an increase of it. However an increase in tungsten content has no relevant effect on the elastic behaviour of these alloys.
Figure 4.12 Stress vs Strain diagrams of Inermet 180.

Figure 4.13 Stress vs Strain diagrams of Densimet 176.
4.5. Fatigue results

4.5.1 Stress-Controlled Tests:
The results obtained for IT180 and D176 is plotted in a log-log scale according to E739 Standard practice for statistical Analysis of Linear or Linearized Stress-Life ( S-N ) of fatigue data. This practice intends to generate a statistical S-N curve in the finite fatigue life region therefore those samples tested at stress levels below the fatigue limit (and had infinite life) are not taken into account to do the computations.

In the case of IT180, four stress levels were used: 300, 250, 225 and 210 MPa and a total amount of specimens to perform the calculation equal to 9, being then the percentage of replication equal to 55.6%.

The data for these stress levels was plotted in log-log scale. Noting that the distribution of the points seemed to follow a straight line the computations to obtain the median curve were performed. In this sense, the estimators $A$ and $B$ and the variance of the curve are respectively:

$$
\hat{A} = 26,783 \quad \hat{B} = -8,967 \quad \sigma = 0,268
$$

Then, substituting these values in equation (2.11) the median curve would be:

$$
Log(N) = 26,783 - 8,967Log(S_a)
$$

and expressing it in the form of the Basquin equation (2.6):

$$
S_a = 1048(2N)^{-0,112}
$$

Where the fatigue coefficient and exponent are $S'_f = 1048 \text{ MPa}$ and $b = -0,112$ respectively.

Finally the design curve with a reliability of 90% and a confidence of 95% was estimated. For this: $K_{OWEN} = 2,727$ and the design curve is:

$$
Y = \hat{A} + \hat{B} \cdot X - K_{OWEN} \cdot \sigma \rightarrow Y = 26,783 - 8,967LogX - 2,727 \cdot 0,268
$$

or its equivalent version in a logarithmic scale:
For the case of the D176, six specimens were tested at 3 different stress levels that were 550, 500 and 450 MPa (2 specimens at each level) then the percentage of replication is equal to 50%.

In both cases for IT180 and D176 the minimum number of specimens and the percentage of replication used are in accordance with the requirements suggested on E739-ASTM standard practice for conducting a preliminary and exploratory research and development fatigue testing.

Following the same procedure as used with IT180, the estimators for the statistical median S-N curve for the D176 are:

\[
\hat{A} = 26,79015 \quad \hat{B} = -7,79133 \quad \sigma = 0,15325
\]

Therefore the median S-N curve can be expressed as:

\[
Log(N) = 26,79015 - 7,79133Log(S)
\]

And its equivalent version as the Basquin equation for the D176 would be:

\[
S_a = 3000 \cdot N^{-0,12835}
\]

Where the fatigue exponent and coefficient are \( S_f = 2744,47 \text{ MPa} \) and \( b = -0,12835 \), respectively.

Finally the design curve with a reliability of 90% and a confidence of 95% was estimated. For this \( K_{OWEN} = 3,55962 \) and the design curve is:

\[
Y = \hat{A} + \hat{B} \cdot X - K_{OWEN} \cdot \sigma \rightarrow Y = 26,79015 - 7,79133 \cdot X - 3,55962 \cdot 0,15325
\]

Or its equivalent version in a logarithmic scale:

\[
S_{R90C95} = 2553 \cdot (2N)^{-0,12835}
\]

The computed curves together with the experimental data for both alloys are shown in figures below:
Figure 4.14. Experimental data of the IT180 in the finite life region along with the statistical median curve and the confidence band.

Figure 4.15. Experimental data of Densimet 176 in the finite life region along with the statistical median curve and the confidence band.
Comparing the results between IT180 and D176, the latter has higher fatigue strength having higher lives when subjected to the same stresses. On the other hand, the endurance limit for D176 is about 45% of its ultimate tensile strength whereas the endurance limit of IT180 is about 33% of its ultimate tensile strength which is in the lower limit of the typical endurance limit values for most metals.

Since the sample size is usually limited and in our case is very small, the real distribution of the fatigue behaviour of the material is hard to predict exactly. Then 95% of confidence bands with 90% of reliability are drawn in the plots together with the median curves because if a series of analyses is performed to this material and design curves are computed for the data, so the 95% of the samples tested will have a 90% chance of survival at that stress.

Note that according to ASTM standard practice E739, the confidence band suggested for them is double-sided hyperbolic confidence bands. However, this practice also agrees on the use of a linear one-sided lower bound as a confidence band. This is then the procedure followed in this thesis for sake of the simplicity of the calculations.

All specimens endured more than $10^4$ cycles before failing, proving that we are definitely in a zone of relatively high cycle fatigue life and thereby the study of the data was accurately obtained from a load controlled constant amplitude test and described using the stress-life approach.

On the other hand, for the specific interval of stress tested, the behaviour of the fatigue life can reasonably be approximated to a straight line so the mean S-N curve by ASTM standards can be obtained.

The values of the stress exponent for most metals range between -0.05 and -0.12 [11] what indicates that both our alloys are in the top limit of this range. On the other hand, $S'_{f}$ can be approximated to the true fracture strength $\sigma_f$, corrected for necking, in a monotonic tension test for most metals [11]. This value would correspond to the intercept of the stress-life curve with the ordinate at one-quarter of the first cycle. In the present case the true fracture strength for the IT180 and D176 are 633 MPa and 1180 MPa respectively. In contrast, the fatigue strength coefficients for each alloy are
3000 MPa and 1010 MPa respectively. Keeping in mind that because the actual S-N relationship is approximated by a straight line only within a specific interval of stress, and because the actual fatigue life distribution is unknown, it is not recommended that the S-N curve be extrapolated outside the interval of testing. Then the difference between the values of the true strength and the computed values of $S'_f$ (especially for D176) indicate that it is not recommended to use the median curve obtained for extrapolation to lower fatigue lives. Even when the comparison between $S'_f$ and $\sigma_f$ is just an approximate idea and doesn’t necessarily mean that these stresses have to be equal.

It is important to mention that one of the conditions for the statistical model proposed by E739 is that there are neither run-outs nor suspended tests for the entire interval of lives used in testing. However, the specimen #10 of IT180 alloy didn’t fail even up to $2 \times 10^6$ cycles, thereby it is considered as a run-out. Despite this fact we decided to still use this value for the calculations since there are other two values for the life at the same stress level (210 MPa) that actually did fail.

Finally, Morrows equation (eq. 2.10) was used to see the influence of a mean stress of 30 MPa in the fatigue curve and is shown in pictures 4.16 and 4.17. It is easy to see how the median S-N curve for the constant stress amplitude almost overlaps with the S-N modified by the mean stress. This shows that if would have run the tests using a mean stress, the results wouldn’t have varied considerably (at least for the mean stress of interest).
Figure 4.16 Mean stress effect in the S-N curve of IT180

Figure 4.17 Mean stress effect in the S-N curve of D176
Looking at the fatigue data plotted in figures 4.14 and 4.15 it is noticeable how the scatter between the lives obtained at each of the stress level increases the lower the stress levels are which is in accordance to the literature.

Despite the fact that there is a large scatter in the fatigue data of the D176 alloy it is easily observed the higher fatigue strength that this material presents compared to the IT180 alloy. These can lead to the assumption that fatigue strength and specially the endurance limit of a material can be related or determined by the ductility of it since the most noticeable difference between them is on this aspect.

The fracture surface was observed by scanning electron microscope shows the different fracture modes of both tungsten alloys that in fact are related to their ductility and therefore their fatigue strength.

In this sense the fracture mechanisms or modes of tungsten heavy alloys are a combination of intergranular W fractures, interfacial matrix-W decohesion, W transgranular cleavage and ductile rupture of the matrix. A graphical description of these mechanisms is shown below in figure 4.16.

![Figure 4.16](image-url)  
*Figure 4.16 Schematic illustration of major failure modes. A1= ductile fracture of matrix-phase; A2= intergranular fracture of matrix-phase; A3= transgranular cleavage; A4= intergranular fracture of W-grains network; A5= tungsten side of W-matrix interface fracture; A6= matrix side of W-matrix interface fracture. (Picture taken from reference [8])*
In general, low ductility alloys (lower elongation to fracture) have a fracture surface where matrix-W debonding and intergranular decohesion of contiguous W grains predominate. Whereas an alloy with higher ductility have less presence of interfacial decohesion and a high proportion of transgranular cleavage of W grains.

W-W interfaces are a weak link in the microstructure and the easiest path for crack growth since these interfaces represent the lowest strength fracture mechanism because the critical energy release rate is lowest for the decohesion of these interfaces [28,29]. This is why alloys with a high contiguity have lower ductility because they have a large amount of low strength intergranular bondings that separates very easy reducing the load which can be carried by the grains decreasing their ductile properties.

On the other hand, K. M Ostoloza Zamora [28] points out that cracking of the W grains by cleavage needs good transmission of stresses from the matrix to the spherical or ellipsoidal grains through the interfaces so that, rather paradoxically, good interfacial bonding produces ductile alloys whose fracture surfaces show a maximum of cleavage.

From a microstructural point of view the difference between these two alloys can be explained as follows. As it was mentioned before, higher tungsten content, increases the final grain size, more coarsening of the W-grains is created and larger areas of W-W interface are presented. This fact is also helped by the lesser amount of bonding ductile phase to provide more W-matrix interfaces.

So, IT180 alloy that presents a lower ductility is expected to have a majority of W-matrix interface failure together with decohesion of tungsten grains. Figure 4.17 shows the fracture surface of this alloy, where tungsten grains have flattened areas due to the separation between them and other tungsten grains. Also it is noticeable a crack growing through the matrix phase and continuing between this one and the W grain boundary.
In the case of Densimet 176 alloy a combination of W-matrix failure and transgranular cleavage of tungsten grains can be observed in figure 4.18. Here it can be seen how cracks propagate between the grains boundaries and the ductile matrix and then go through the grains giving them these radial striations that continue growing. Note that there is also the presence of smooth tungsten grain areas associated with an intergranular fracture mode but this mechanism is overcome by the two latter ones.
Cyclic stresses induce deformation in the material due to back and forth sliding on closely spaced parallel planes in a narrow band known as permanent slip band. This deformation causes striations on the surface and accumulates progressively concentrating the stress until a crack is formed. Once the crack is formed two scenarios can occur. If the crack is long enough then it will propagate and cause the part failure. Or the crack form but do not propagate giving the chance to the formation of several other cracks that can also cause failure. The stresses at the tip of the crack are very high and break the atomic bonds at that area and the crack can increase in length.

4.5.2 Strain controlled tests
The specimen of the IT180 tested with blocks containing 200 cycles broke at the second block of strain amplitude 0.2 pct at the 29th cycle. The fact of giving the wave program a strain in [mm] which was equivalent to the desired strain in [%] was performed properly since the real strain amplitudes obtained are almost exact to those programmed. For example at 0.2% strain amplitude the peak strain is 0.193%.
Figure 4.19 shows the stress vs. the strain amplitude for this specimen. At 0.1% strain the behavior of the material is completely elastic and the specimen failed before yielding of the material could take place. The reason why this material has failed so early could be due to alignment of the specimen. In this sense, ASTM standard practice E606 recommends that the bending strains should not exceed 5% of the minimum strain amplitude, especially in testing of low ductility material such as IT180. The minimum strain amplitude was equivalent to 0.0125 mm whereas the lateral displacement measured by the strain gages was in the magnitude order of $10^{-2}$ mm which is much higher than 5%.

At a strain rate of 0.1% the specimen stabilizes at about 40 cycles, while at 0.2% stabilization takes place at 15 cycles where the peak tensile stress is 519 MPa. Comparing the results from the specimens tested with blocks of 200 or 30 cycles it was noticed that the stabilized peak stresses in tension are almost the same (with an approximate difference of 4 MPa) whereas the difference in compression is more considerable (about 40 MPa). In this respect, the specimen that was subjected to the initial 200 cycles reached a higher stress amplitude at the next strain level which can be because the material was able to harden more with the higher amount of cycles.

Figure 4.19 Hysteresis loops for IT180 until 0.2% of strain amplitude.
In the case of D176 the specimen was tested until a deformation of 1%. The resulting hysteresis loops are shown in figure 4.20. Figure 4.21 shows the variation of the stress amplitudes with cycling in time for the entire test length. For initial strain amplitudes between 0.2-0.5pct cyclic hardening rate is more noticeable while the stress stabilizes at about the twentieth cycle of each block. However at higher strain amplitudes up to 1pct, the stress amplitudes variation at each cycle is very small and the obtained hysteresis loops are stabilized.

Figure 4.20 Hysteresis loops for D176 until 1% of strain amplitude.
Cyclic vs. Monotonic response:

In order to compare the cyclic and monotonic behavior of the alloys, the tensile curve of IT180 and D176 is shown in figure 4.22 and 4.23 (red line) superimposed to the tensile side of the hysteresis loops (first quadrant). The cyclic parameters $K'$ and $n'$ were not estimated since just a small part of the curve could be drawn. However even when the fatigue test reached only until the elastic region it can be observed that the cyclic curve lies above the monotonic curve and it is expected to would have followed this behavior if cycling would have continued. It this sense it may be said that IT180 hardens under cyclic loading.
The cyclic yield stress ($S_{y'}$) is the stress at 0.2% plastic strain on a cyclic stress–strain curve. This value was determined as proposed in reference [13] by constructing a line parallel to the modulus of elasticity through the 0.2% strain offset at the zero stress point. The stress at which the line intersects the cyclic stress–strain curve is taken as the cyclic yield stress. The cyclic yield strength of the D176 is about 800 MPa, which is higher than the monotonic yield strength (654 MPa). This indicates that D176 hardens under cyclic loading.

The cyclic parameters $K'$ and $n'$ were estimated and compared to the monotonic parameters $K$ and $n$. The procedure was done as follows:

The values of the stabilized stress amplitudes at the tip of the hysteresis loop for each strain amplitude were read from the cyclic stress-strain curve shown in figure 4.23. Then, the elastic and plastic strain was calculated by equations 2.31 and 2.36. These data is presented in table 4.5.

Figure 4.23 Tensile side of the hysteresis loops for IT180 together with the tensile stress-strain curve.
Table 4.5 Stress and strain amplitudes extracted from the CSS curve used to estimate the cyclic strength coefficient and the cyclic strain hardening exponent. The calculated elastic and plastic components of the total strain amplitudes are also presented.

<table>
<thead>
<tr>
<th>$\varepsilon$ [-]</th>
<th>$\sigma_a$ [MPa]</th>
<th>$\varepsilon_e$ [-]</th>
<th>$\varepsilon_p$ [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,0019</td>
<td>654,2</td>
<td>0,0020</td>
<td>-0,0001</td>
</tr>
<tr>
<td>0,0030</td>
<td>740,9</td>
<td>0,0023</td>
<td>0,0007</td>
</tr>
<tr>
<td>0,0040</td>
<td>779,7</td>
<td>0,0024</td>
<td>0,0017</td>
</tr>
<tr>
<td>0,0051</td>
<td>813,9</td>
<td>0,0025</td>
<td>0,0026</td>
</tr>
<tr>
<td>0,0061</td>
<td>836,6</td>
<td>0,0026</td>
<td>0,0035</td>
</tr>
<tr>
<td>0,0071</td>
<td>852,5</td>
<td>0,0026</td>
<td>0,0044</td>
</tr>
<tr>
<td>0,0081</td>
<td>869,8</td>
<td>0,0027</td>
<td>0,0054</td>
</tr>
<tr>
<td>0,0090</td>
<td>876</td>
<td>0,0027</td>
<td>0,0064</td>
</tr>
<tr>
<td>0,0101</td>
<td>882,5</td>
<td>0,0027</td>
<td>0,0074</td>
</tr>
</tbody>
</table>

The data at 0.2% of strain amplitude was not used for the computations according to [2][13] where it is suggested that strains lower than 0.0005 [mm/mm] should be neglected to avoid calculation errors. On the other hand, the Young’s modulus for the CSS was estimated as the slope of the line between the origin and the peak point at 0.2 strain amplitude in the graph. To facilitate the calculations it was assumed that $E$ was constant for each strain loop which in essence seems to be true and just some small variations are seen at higher strains.

As it was mentioned in section 2.3.1, the stress vs. plastic strain amplitude follows a straight line when plotted in a logarithmic scale as seen in figure 4.23.

The estimators of the curve A and B and the variance are respectively:

$A = 3,11 \quad B = 0,08 \quad \sigma = 0,002$

Hence, the cyclic strength coefficient and the cyclic strain hardening exponent are respectively:

$K' = 1314 \text{ MPa} \quad n' = 0,08$

In the case of the estimation of the tensile parameters some assumptions had to be made. Since the instantaneous test section area was not able to measure, the engineering stresses and strains were used for the computations. Even when this is not
correspondent with the literature, it was thought to be a considerable assumption since the final area at fracture was measured and seen that it didn’t vary so much. Again, those strains lower than 0.0005 [mm/mm] were not taken into account to do the computations. The estimators of the best fit line are:

\[ A = 3.00 \quad B = 0.07 \quad \sigma = 0.007 \]

Then,

\[ K = 1008 \, \text{MPa} \quad \text{and} \quad n_m = 0.07 \]

Despite there is not a big difference between the cyclic and monotonic strain hardening exponents, it may be said that the D176 will harden when undergoing cyclic loading. This is also seen in figure 4.22 where the monotonic curve lays below the cyclic strain-stress curve. This explains that this material yields at a higher stress under cyclic loading than under monotonic loading. The parameters are obtained statistically if more strain levels would have been tested; the value of \( n' \) could have been higher.
Chapter 5: Conclusions

The S-N curves of the alloys show that Densimet 176 has higher fatigue strength than Inermet 180. Even when the exact endurance limit for the alloys was not obtained it is suspected to be about 45% and 33% of the UTS for the latter and former respectively.

Tungsten content affects the fatigue behavior of these alloys. In this sense, the higher the tungsten content, the lower the fatigue properties which explains why the D176 having 92.5%wt of tungsten has higher fatigue strength than IT180 (95%wt of tungsten).

Porosity was a common defect present in both alloys. However its effect has been more detrimental in the case of IT180 who presented bigger pores, with a very irregular shape and in a high amount that acted as stress concentrations and zones of crack initiation. The scatter in the fatigue data for this alloy is higher than that of D176 and might be related to this fact.

The use of stress-controlled testing for the estimation of the S-N curve is fairly valid since all the specimens endured more than $10^4$ cycles before failing proving that the experiments were in the high cycle fatigue regime as where this type of test is suitable.

Since the S-N relationship was approximated by a straight line within the interval of stresses tested and because the actual fatigue distribution is unknown, the results obtained should not be extrapolated to other stress levels and are only valid within this interval.

Surface roughness has a detrimental effect on crack initiation in a material under cyclic loading. Grinding and polishing to reduce radial lines on the surface of the specimens avoided this effect helping in the reproducibility of the fatigue results.

The stress concentration associated to the blend radius of the tested specimens may have influenced that most of the specimens broke near the grips. For more accurate results, the best results would be obtained by testing a sample with the same shape that the target material will have when operating in the spallation device.

The principal fracture mechanism under fatigue loading observed in D176 was tungsten grain cleavage as it had been observed for ductile WHAs under tensile
loading conditions. In the case of IT180, failure by separation of W-W interfaces was predominant which during failure reduces the load that can be carried by the grains and thereby its fatigue strength.

The estimation of the cyclic stress-strain curve showed that both alloys harden under cyclic loading compared to their response under tensile loads. The cyclic strain hardening exponent and the cyclic yield strength of D176 were able find and are higher than the monotonic strain hardening exponent and yield strength.

The IT180 for being a highly brittle material is very sensitive to misalignment during the strain-controlled tests and broke at low strain amplitude levels causing that it was not possible to find the cyclic parameters n’ and $\sigma'$, for this alloy.
References


Appendix A  Statistical analysis of linearized Stress-Life curve fatigue data

Fatigue Data from the tests:

<table>
<thead>
<tr>
<th>Stress levels:</th>
<th>Cycles to failure:</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>33831</td>
</tr>
<tr>
<td>300</td>
<td>40177</td>
</tr>
<tr>
<td>250</td>
<td>329748</td>
</tr>
<tr>
<td>250</td>
<td>139854</td>
</tr>
<tr>
<td>Inermet 180</td>
<td></td>
</tr>
<tr>
<td>225</td>
<td>424545</td>
</tr>
<tr>
<td>225</td>
<td>336042</td>
</tr>
<tr>
<td>210</td>
<td>312378</td>
</tr>
<tr>
<td>210</td>
<td>2.00E+06</td>
</tr>
<tr>
<td>210</td>
<td>1626921</td>
</tr>
<tr>
<td>Densimet 176</td>
<td></td>
</tr>
<tr>
<td>550</td>
<td>258788</td>
</tr>
<tr>
<td>550</td>
<td>213838</td>
</tr>
<tr>
<td>500</td>
<td>629575</td>
</tr>
<tr>
<td>500</td>
<td>958423</td>
</tr>
<tr>
<td>450</td>
<td>885444</td>
</tr>
<tr>
<td>450</td>
<td>1468220</td>
</tr>
</tbody>
</table>

*The subscripts "I" and "D" refer to Inermet 180 and Densimet 176 respectively.

Now, expressing the data to logarithmic scale:

\[
Y(N) := \text{for } i \in 0..\text{rows}(N) - 1 \quad Y_i \leftarrow \log(N_i) \\
X(S) := \text{for } i \in 0..\text{rows}(S) - 1 \quad X_i \leftarrow \log(S_i)
\]

New variables definition:

Life of IT180: \( Y_I := Y(N_I) \)

Stress of IT180: \( X_I := X(S_I) \)

Life of D176: \( Y_D := Y(N_D) \)

Stress of D176: \( X_D := X(S_D) \)
The average of the stress and the fatigue life would be from equations 2.15 and 2.16 respectively:

\[ Y_1 = \frac{\sum_{rows(Y_1)} Y_1}{X_1} = 5.44 \quad \text{and} \quad X_1 = \frac{\sum_{rows(X_1)} X_1}{rows(X_1)} = 2.38 \]

\[ Y_D = \frac{\sum_{rows(Y_D)} Y_D}{X_D} = 5.773 \quad \text{and} \quad X_D = \frac{\sum_{rows(X_D)} X_D}{rows(X_D)} = 2.698 \]

The maximum likelihood estimators are calculated from as follows:

The numerator of equation 2.14 is:

\[ \text{Bup}(X, Y, X, Y) := \text{for } i \in 0..\text{rows}(X) - 1 \]
\[ \text{Bup}_i \leftarrow (X_i - X)(Y_i - Y) \]
\[ \text{Bup} \]

The denominator from equation 2.14 is:

\[ \text{Bdown}(X, Y, X) := \text{for } i \in 0..\text{rows}(X) - 1 \]
\[ \text{Bdown}_i \leftarrow (X_i - X)^2 \]
\[ \text{Bdown} \]

\[ \text{Bup}_D := \text{Bup}(X_D, Y_D, X_D, Y_D) \quad \text{Bdown}_D := \text{Bdown}(X_D, Y_D, X_D) \]

\[ \text{Bup}_I := \text{Bup}(X_I, Y_I, X_I, Y_I) \quad \text{Bdown}_I := \text{Bdown}(X_I, Y_I, X_I) \]

The estimators of IT180 are:

\[ B_I := \frac{\sum_{Bup} Bup}{\sum_{Bdown} Bdown} = -8.967 \quad \text{and} \quad A_I := Y_1 - B_I X_1 = 26.783 \]

\[ B_D := \frac{\sum_{BupD} BupD}{\sum_{BdownD} BdownD} = -7.791 \quad \text{and} \quad A_D := Y_D - B_D X_D = 26.7902 \]
Expected fatigue life for each stress level according to the estimators

\[ y_I := A_I + B_I X_I \]
\[ y_D := A_D + B_D X_D \]

The variance is:

\[ \text{IT180: } \text{Square}\sigma_I := \frac{\sum(y_I - \bar{y}_I)^2}{\text{rows}(y_I) - 2} = 0.072 \quad \sigma_I := \sqrt{\text{Square}\sigma_I} = 0.268 \]
\[ \text{D176: } \text{Square}\sigma_D := \frac{\sum(y_D - \bar{y}_D)^2}{\text{rows}(y_D) - 2} = 0.023 \quad \sigma_D := \sqrt{\text{Square}\sigma_D} = 0.153 \]

The fatigue strength exponents are:

\[
\begin{align*}
\frac{1}{b_I} &= \frac{1}{B_I} = -0.112 \\
\frac{1}{b_D} &= \frac{1}{B_D} = -0.128
\end{align*}
\]

The fatigue strength coefficients are:

\[
\begin{align*}
S_{f_I} &= 10 \left( \frac{1}{2} \right)^{b_I} = 1.048 \times 10^3 \\
S_{f_D} &= 10 \left( \frac{1}{2} \right)^{b_D} = 2.999818 \times 10^3
\end{align*}
\]

The term "1/2" is added because Basquin’s equation is expressed in number of reversals to failure instead of cycles to failure which is the measured variable.

The median curves in a Basquin’s equation fashion for each alloy are expressed as:

\[ S_{a_I}(x) := S_{f_I}(2x)^{b_I} \]
\[ S_{a_D}(x) := S_{f_D}(2x)^{b_D} \]
**Estimation of the design curve:**

Variables definition:

\[ K_R := \text{qnorm}(0.90, 0, 1) = 1.282 \quad K_C := \text{qnorm}(0.95, 0, 1) = 1.645 \]

\[ f_I := \text{rows}(Y_I) - 2 \quad f_D := \text{rows}(Y_D) - 2 \]

\[ a_I := \frac{1.85}{\text{rows}(Y_I)} \quad a_D := \frac{1.85}{\text{rows}(Y_D)} = 0.308 \]

Empirical coefficients for Kowen with 95% confidence level

\[ b_1 := 0.9968 \quad b_2 := 0.1596 \quad b_3 := 0.60 \quad b_4 := -2.636 \]

Note that \( b_1 \), \( b_2 \), \( b_3 \) and \( b_4 \) are the same for both alloys. The rest of the constants are:

\[ c_1 := 1 + \frac{3}{4(f_I - 1.042)} = 1.126 \quad c_1 := 1 + \frac{3}{4(f_D - 1.042)} = 1.254 \]

\[ c_2 := \frac{f_I}{f_I - 2} = 1.4 \quad c_2 := \frac{f_D}{f_D - 2} = 2 \]

\[ c_3 := c_2 - c_1^2 = 0.132 \quad c_3 := c_2 - c_1^2 = 0.429 \]

\[ K_I := c_1 K_R + K_C \sqrt{c_3 K_R^2 + c_2 a_I} = 2.612 \]

\[ K_D := c_1 K_R + K_C \sqrt{c_3 K_R^2 + c_2 a_D} = 3.497 \]

\[ \text{Rowen}_I := b_1 + \frac{b_2}{f_I} + b_4 e^{-f_I} \quad \text{Rowen}_D := b_1 + \frac{b_2}{f_D} + b_4 e^{-f_D} \]

\[ \text{Kowen}_I := K_I \cdot \text{Rowen}_I = 2.727 \quad \text{Kowen}_D := K_D \cdot \text{Rowen}_D = 3.56 \]
The fatigue coefficient for the design curve of each alloy is:

\[
S'_I = 10 \frac{A_{I-Kown} \sigma_I}{-B_I \left(\frac{1}{2}\right)^{b_I}} \quad S'_D = 10 \frac{A_{D-Kown} \sigma_D}{-B_D \left(\frac{1}{2}\right)^{b_D}}
\]

\[
S'_I = 868.318 \\ S'_D = 2.553 \times 10^3
\]
Annex 2

Sample pictures after fracture
In this chapter, pictures of all the samples after testing are shown, and it’s clearly visible that almost all of them broke, but mainly at the ends of testing section, as mentioned in the report.