

# MFP

MASTER'S FINAL PROJECT

## HYDROGEN EMBRITTLEMENT CHARACTERISATION FOR FCC STEELS FOR H<sub>2</sub> STORAGE AND TRANSPORT

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# HYDROGEN EMBRITTLEMENT CHARACTERISATION FOR FCC STEELS FOR H2 STORAGE AND TRANSPORT

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## ABSTRACT

This document contains the master's degree final project carried out by Mikel Argandoña Sarriegi at Aalto University, in collaboration with Mondragon Unibertsitatea.

The aim of this research focuses on the characterisation of hydrogen embrittlement in steels with FCC structure. For this purpose, two types of stainless steels, 316L and 316L+, have been compared. Samples of both materials have been subjected to electrochemical charging for 72 hours at a temperature of 50° C in an acid solution. Subsequently, the hydrogen concentration has been measured by thermal desorption spectroscopy (TDS).

Both charged and uncharged samples were tested in uni-axial tension to determine their mechanical properties. During the test, the digital image correlation (DIC) method was used to measure the deformation. In addition, specimens with different geometries have been tested to study the effects of stress states.

The results obtained in this study provide a better understanding of hydrogen behaviour in FCC stainless steels and can be useful in the future design of materials with improved hydrogen resistance for various applications. Since a loss of ductility has been observed, it is essential to carefully consider the effect of hydrogen in the selection of materials for hydrogen storage and transport applications.

**Key words:** *hydrogen embrittlement, FCC structure, 316L and 316L+, electrochemical charging, TDS, DIC, stress states, hydrogen storage and transport applications.*

## RESUMEN

Este documento contiene el proyecto de fin de máster realizado por Mikel Argandoña Sarriegi en la Universidad de Aalto, en colaboración con Mondragon Unibertsitatea.

El objetivo de esta investigación se centra en la caracterización de la fragilización por hidrógeno en aceros con estructura FCC. Para ello, se han comparado dos tipos de aceros inoxidables, el 316L y el 316L+. Las muestras de ambos materiales han sido sometidas a una carga electroquímica durante 72 horas a una temperatura de 50° C en una solución ácida, y posteriormente se ha medido la concentración de hidrógeno mediante el método de desorción programada por temperatura (TDS).

Tanto las muestras con carga como las muestras sin ella han sido ensayadas en una máquina de tracción para determinar sus propiedades mecánicas. Durante el ensayo se ha utilizado el método óptico de correlación y seguimiento de imágenes digitales (DIC) para medir la deformación. Además, se han ensayado muestras con diferentes geometrías con el objetivo de comparar los efectos en diferentes estados de tensión.

Los resultados obtenidos en este estudio proporcionan una mejor comprensión del comportamiento del hidrógeno en metales y pueden ser útiles en el diseño futuro de materiales con mejores propiedades de hidrógeno para diversas aplicaciones. Dado que se ha observado una pérdida de ductilidad en los aceros FCC cargados, es fundamental considerar cuidadosamente el efecto del hidrógeno en la selección de materiales para aplicaciones de almacenamiento y transporte de hidrógeno.

**Palabras clave:** *fragilización por hidrógeno, estructura FCC, 316L y 316L+, carga electroquímica, TDS, DIC, estados de tensión, aplicaciones de almacenamiento y transporte de hidrógeno.*

## LABURPENA

Dokumentu honek Mikel Argandoña Sarriegik Aalto Unibertsitatean eta Mondragon Unibertsitatearekin elkarlanean egindako Master Bukaerako Lana jasotzen du.

Ikerketa honen helburua FCC egitura duten altzairuetan hidrogeno bidezko hauskortasuna karakterizatzea da. Horretarako, bi altzairu herdoilezin alderatu dira: 316L eta 316L+. Bi materialen probetei karga elektrokimiko bat ezarri zaie 72 orduz, 50° C graduko tenperaturaren, soluzio azido batean eta ondoren, hidrogeno-kontzentrazioa neurtu da tenperaturaren arabera programatutako desortzio-metodoaren bidez (TDS).

Hidrogeno kargadun eta kargarik gabeko probetak trakzio-makina batean probatu dira, haien propietate mekanikoak zehazteko. Saiakuntzan, irudi digitalen korrelazioko eta jarraipeneko metodo optikoa erabili da deformazioa neurtzeko. Gainera, geometria desberdinetako probetak entseatu dira, tentsio-egoera desberdinetako efektuak alderatzeko.

Ikerketa honetan lortutako emaitzek hidrogenoak metaletan duen portaera hobeto ulertzen laguntzen dute eta hainbat aplikaziotarako hidrogeno-propietate hobeak dituzten materialen etorkizuneko diseinuan erabilgarriak izan daitezke. Kargatutako FCC altzairuetan harikortasuna galdu dela ikusi denez, funtsezkoa da hidrogenoa biltegitratzeko eta garraiatzeko aplikazioetarako materialak hautatzean duen eragina arretaz aztertzea.

**Hitz-gakoak:** *hidrogeno bidezko hauskortasuna, FCC egitura, 316L eta 316L+, karga elektrokimikoa, TDS, DIC, tentsio-egoerak, hidrogenoa biltegitratzeko eta garraiatzeko aplikazioak.*

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## ABBREVIATIONS LIST

<b>Abb.</b>	<b>Full Meaning</b>
FCC	Face Centered Cubic
TDS	Thermal Desorption Spectroscopy
DIC	Digital Image Correlation
HE	Hydrogen Embrittlement
IEA	International Energy Agency
DOE	Department of Energy
C	Carbon
Cr	Chromium
Ni	Nickel
Mo	Molybdenum
N	Nitrogen
PRE	Pitting Resistance Equivalent
CPT	Critical Pitting Temperature
HELP	Hydrogen-Enhanced Localised Plasticity
HEDE	Hydrogen-Enhanced Decohesion
HIC	Hydrogen-Induced Cracking
OES	Optical Emission Spectrometer
SDB	Smooth Dog Bone
NDB	Notched Dog Bone
CH	Centre Hole
SH	Shear
RD	Rolling Direction
DD	Diagonal Direction
TD	Transverse Direction
EDM	Electrical Discharge Machining
WEDM	Wire Electrical Discharge Machining
ROI	Region of Interest
FOV	Field of View
UTS	Ultimate Tensile Strength

YS	Yield Strength
H2	Hydrogen
SDG	Sustainable Development Goals
TE	Total Elongation

## 1. INTRODUCTION

Hydrogen is quickly becoming recognized as a crucial component of a clean energy future, with the potential to become one of the most widely used energy sources. The International Energy Agency (IEA) has noted the importance of hydrogen in supporting clean energy transitions, particularly in industries where the use of renewable energy is more difficult to achieve. Although it is already widely used in some industries, its potential to contribute to a sustainable and low-carbon energy future has not yet been fully realized [1].

Renewable resources, including increased renewable energy usage in the industry, transport, and building sectors, will also need to be utilized to meet the increasing demand for clean energy. While fossil fuel-based energy sources have played a critical role in powering the world's economies over the past century, transitioning towards sustainable, low-carbon energy sources is crucial for combating climate change and ensuring a sustainable future [2][3].

By addressing many of the world's energy challenges, hydrogen has the potential to become a key energy source in the future. Its importance is highlighted by initiatives such as the DOE National Clean Hydrogen Strategy and Roadmap and the Department of Energy Hydrogen Program Plan [4][5]. The importance of hydrogen as a future clean energy source is also being recognized by many governments and organizations around the world, making it an exciting area to explore going forward [6].

### 1.1 HYDROGEN EMBRITTLEMENT

While hydrogen offers numerous advantages as a clean and efficient fuel, the issue of hydrogen embrittlement underscores the need for careful consideration and engineering solutions to ensure the safe and reliable use of hydrogen in various applications.

Hydrogen embrittlement is a phenomenon that occurs when a metal's ductility is reduced due to the absorption of hydrogen [7]. It is mostly observed in steels and higher-strength materials and requires the presence of both atomic hydrogen and mechanical stress to induce crack growth. Hydrogen can be introduced into the metal through sources such as corrosion, electroplating, welding, or exposure to hydrogen gas.

The effects of hydrogen embrittlement have been known since the 19th century [8], and research on the topic continues today to better understand the basic mechanisms. One of the earliest observations of hydrogen embrittlement occurred in the mid-1800s, when it was noted that exposed

iron wires became brittle and eventually shattered after being subjected to hydrogen gas [9]. This early discovery paved the way for further investigation into the phenomenon, and today we have a better understanding of the effects of hydrogen embrittlement on various metals and alloys.

Despite the many advances made in understanding and mitigating hydrogen embrittlement, it continues to be a significant concern in many industries. The aerospace, automotive, and oil and gas industries, for example, are especially susceptible to hydrogen embrittlement and must take measures to prevent it from occurring in their products and equipment [10]. By continuing to study and address the causes of hydrogen embrittlement, researchers hope to find ways to mitigate its effects and ensure the safety and reliability of critical machinery and infrastructure.

## 1.2 STEELS FOR HYDROGEN STORAGE

In the hydrogen sector, the types of steels used for hydrogen storage and transport include mainly austenitic stainless steels such as type 304 and 316 [16], corrosion-resistant low alloy steels, high tensile strength steels such as carbon steel and steels API X70 or higher.

Both AISI 304 (1.4301) and AISI 316 (1.4401) are austenitic stainless steels used in hydrogen storage and transportation. AISI 304 is suitable for moderate-pressure applications, while AISI 316 is used in more demanding environments, including marine and chemical applications. It is suitable for high-pressure hydrogen storage and transportation, such as storage cylinders, pressure vessels, and pipelines.

Hydrogen storage and transport pressure may vary depending on the application and specific requirements. Typical pressures range being 350 to 700 bar [17].

High-pressure hydrogen storage involves compressing the gas to significant pressures and storing it in cylinders or tanks designed to withstand these pressures. Compressors are used to increase the gas pressure. Hydrogen is introduced into the cylinders or tanks, and as it is compressed, its density increases, allowing for larger amounts of hydrogen to be stored in a smaller volume [18].

High-pressure storage is widely used in hydrogen transportation applications, such as fuel cell vehicles and stationary storage systems. It is also used in the distribution and supply of hydrogen for industrial use.

### 1.3 AUSTENITIC STAINLESS STEEL

Austenitic stainless steel is a type of stainless steel that is non-magnetic and has a face-centered cubic (FCC) crystal structure, making it easy to weld and shape [13]. It has excellent corrosion resistance and is frequently used in applications that require good formability, such as kitchen utensils, food processing equipment, and pharmaceuticals.

One of the distinguishing characteristics of austenitic stainless steel is its high concentration of chromium and nickel, which provides its excellent corrosion resistance. It also has lower levels of carbon compared to other types of stainless steel, which enhances its weldability [14].

Applications for austenitic stainless steel include a wide range of industries, such as architecture, automotive, and aerospace. In the automotive industry, it is used in exhaust systems and catalytic converters due to its high-temperature resistance. In aerospace, it is used in structural components, fasteners and couplings due to its excellent strength and corrosion resistance [15].

Some of the most common austenitic stainless steels and their applications include [32][33]:

AISI 304 (1.4301): It is the most used austenitic stainless steel. It is found in general-purpose applications such as kitchen utensils, medical equipment, architectural applications, and industrial components not exposed to aggressive corrosive environments.

AISI 316 (1.4401): It is known for its higher corrosion resistance in marine and chemical environments. It is used in marine applications, chemical processing plants, medical and pharmaceutical equipment, and components exposed to corrosive environments.

AISI 321 (1.4541): It contains titanium as a carbide stabilizer, which provides increased resistance to intergranular corrosion at elevated temperature ranges. It is used in high-temperature applications such as automobile exhaust systems, heat treatment equipment, and gas turbine components.

AISI 347 (1.4550): Similar to AISI 321 but with additional niobium content, providing greater resistance to intergranular corrosion in high-temperature environments. It is found in similar applications as AISI 321, such as exhaust systems, heat exchangers, and chemical reactors.

## 1.4 STUDIED MATERIALS

In this thesis, the materials used are 316L and 316L+ steels, which are part of the high-performance austenitic stainless steel range of the supplier *Outokumpu*. 316L (1.4404) is low-carbon stainless steel, which is used in various process industries and other aggressive environments with higher-than-average corrosion resistance requirements [11]. On the other hand, 316L+ (1.4420) is another type of austenitic stainless steel that has a slightly different chemical composition with improved properties. The materials are used in various applications where increased corrosion resistance and a combination of high strength and good formability are needed [12].

The reason for studying austenitic stainless steels is that they are less vulnerable to hydrogen embrittlement due to their chemical composition and microscopic structure. These alloys contain chromium, which forms a protective oxide layer on the surface of the steel. This protective layer helps prevent the diffusion of hydrogen into the steel and therefore reduces hydrogen accumulation in high-stress areas. Regarding the structure, they tend to have a more uniform and homogeneous microscopic structure than other types of steels. This means that there are fewer high-stress zones in the material where hydrogen could accumulate and cause embrittlement. All this makes them a good choice for applications where the material is expected to be exposed to hydrogen.

## 1.3 NOWADAYS PROBLEMS

Technology has been advancing by huge steps in recent decades, with changes affecting industry and everyday life at breathtaking speed. One of the most significant changes on the horizon is the growing importance of hydrogen as the fuel of the future.

However, the arrival of hydrogen as a sustainable renewable energy source faces several challenges, one of which is the effect of hydrogen embrittlement on the materials used in its production and storage. In order to ensure that materials are suitable for use in a hydrogen-powered future, it is necessary to invest in research to better understand this phenomenon.

Fracture in materials due to hydrogen embrittlement is a phenomenon that consists of the weakening of the material structure due to the presence of hydrogen, resulting in premature fracture and failure. To address and prevent this problem, it is necessary to characterise and predict the fracture behaviour under the effect of hydrogen.



When studying the fracture behaviour under the effect of hydrogen, there are several challenges and complexities that can arise.

First is the uneven distribution of hydrogen, this results in localised areas of high stress and increased susceptibility to fracture, making it challenging to predict where and how fractures may occur.

Environmental factors, such as temperature, pressure, and the presence of other gases or corrosive substances, can interact with hydrogen and affect fracture behaviour. Conducting experiments to study hydrogen-induced fracture requires specialized equipment, sample preparation, controlled testing environments and the use of advanced characterisation methods, ensuring the accuracy and reliability of experimental results can be challenging.

Material variability, including microstructure, composition, and processing history, also contributes to the complexity of studying fracture under the effect of hydrogen.

Overall, comprehensive understanding of hydrogen-induced fracture requires careful consideration of these challenges and the implementation of appropriate experimental and analytical approaches.

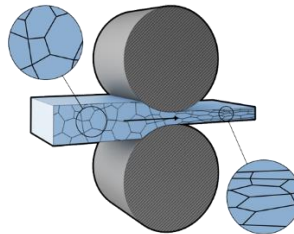
## 2. THEORETICAL BACKGROUND

In this section, the theoretical framework supporting the study on the effect of hydrogen embrittlement on 316L(1.4404) and 316L+(1.4420) metals is presented. The essential concepts related to hydrogen embrittlement, the mechanisms involved in this phenomenon, and the characterised properties of the materials will be addressed. A detailed description of the materials, including the chemical composition, mechanical properties as well as industrial applications, will then be presented. This theoretical analysis will provide the necessary basis for the design and interpretation of the experiments performed.

### 2.1 PROCESSING OF 316L & 316L+

The metal sheets supplied are manufactured in cold rolled form. The cold rolling process is one of the methods used to produce cold-rolled coils and sheets that offer unique benefits compared to hot-rolled steel. The products produced by cold rolling are often used in applications that require a high degree of dimensional accuracy, good surface finish, and improved mechanical properties.

Raw material is first passed through a series of cleaning and pre-treatment steps such as pickling, descaling, or applying a lubricant to the metal surface to prevent damage and reduce friction during rolling. The metal is then fed through a set of rollers that apply pressure to the metal, reducing its thickness and increasing its strength and hardness. The rollers can be arranged in different configurations depending on the desired final product [\[19\]](#). During the process, when the metal is put under mechanical stress, it causes a permanent change to the crystalline structure of the metal. This causes an increase in its strength and often improves corrosion resistance.



**1. Figure** Cold rolling process [\[20\]](#)

*Outokumpu* offers cold rolled steel products in a variety of forms, such as sheets, coils, and strips, to meet the specific needs of their customers in a wide range of industries.

## 2.2 CHEMICAL COMPOSITION AND MECHANICAL PROPERTIES

According to the supplier's specifications, these are the chemical compositions of the materials:

**1. Table** Chemical composition of 316L and 316L+ [\[21\]](#)

Material	C	Cr	Ni	Mo	N
316L	0.02	17.2	10.1	2.1	-
316L+	0.02	20.3	8.6	0.7	0.19

It can be observed that the most important difference in the composition is the presence of nitrogen in the second material. Nitrogen is added to 316L+ stainless steel to help reduce the amount of free hydrogen in the metal, also known as diffusible hydrogen. The nitrogen reacts with hydrogen atoms to form ammonia, which prevents hydrogen from accumulating at defect sites in the metal and causing embrittlement. This results in a material that is more resistant to hydrogen embrittlement, making it a more reliable choice [\[22\]](#).

Regarding the mechanical properties, these are the specifications again provided by the supplier:

**2. Table** Mechanical properties of 316L and 316L+ [\[11\]](#)[\[12\]](#).

316L (1.4404)					
Test Direction	0.2% Yield Strength RP0.2 [MPa]	Tensile Strength Rm min [MPa]	Elongation [%]	Pitting corrosion resistance PRE	Pitting corrosion resistance CPT
Longitudinal	300	625	50-70	24	20 ±2
316L+ (1.4420)					
Test Direction	0.2% Yield Strength RP0.2 [MPa]	Tensile Strength Rm min [MPa]	Elongation [%]	Pitting corrosion resistance PRE	Pitting corrosion resistance CPT
Longitudinal	380	700	50-60	25	35

From the table above it can be seen that 316L+ has better mechanical properties, with yield strength exceeding it by 80 MPa and ultimate tensile strength by 75 MPa. The ductility may vary due to the manufacturing process of the sheet metal. The material processed by cold rolling shows a lower ductility than if it had been processed by hot rolling. It is also visible, as previously mentioned, that the one containing nitrogen shows a higher corrosion resistance.

It is important to consider that the rate at which the tensile test is performed can affect the mechanical properties of the metal. In general, at higher strain rates, the metal tends to exhibit higher strength and lower ductility.

## 2.3 FRACTURE MECHANISMS ASSOCIATED WITH HE

There are several proposed fracture mechanisms associated with hydrogen embrittlement. These are three commonly recognized mechanisms [\[23\]](#) [\[24\]](#) [\[25\]](#):

### Hydrogen-enhanced localised plasticity (HELP)

This mechanism occurs when hydrogen atoms occupy interstitial sites in the material's lattice, causing local softening and an increase in strain localization. The presence of hydrogen promotes localised plastic deformation, leading to the formation and propagation of microvoids, which eventually coalesce to form cracks. The hydrogen reduces the material's ductility and increases its susceptibility to brittle fracture.

### Hydrogen-enhanced decohesion (HEDE)

HEDE involves the hydrogen atoms weakening the atomic bonds in the material, particularly at interfaces such as grain boundaries and phase boundaries. Hydrogen can induce segregation or diffusion to these interfaces, leading to the formation of brittle hydrides or promoting dislocation emission. This leads to reduced cohesion and interfacial strength, making the material more prone to intergranular or transgranular fracture.

### Hydrogen-induced cracking (HIC)

HIC occurs when hydrogen atoms create high local stress concentrations in the material, typically in regions with elevated hydrogen concentrations. The presence of hydrogen lowers the material's fracture toughness and initiates cracks at these localised areas of stress concentration. These cracks can propagate under applied stress, leading to catastrophic failure.

It is important to note that the specific mechanisms of hydrogen embrittlement can vary depending on factors such as material composition, microstructure, loading conditions, and environmental factors. Furthermore, hydrogen embrittlement can manifest as a combination of these mechanisms rather than a single mechanism.

### 3. OBJECTIVES

This section presents a detailed description of the objectives of this project and the plan that has been carried out to achieve them. In addition, the planification, the schedule of conditions and competences and the project budget are defined.

#### 3.1 CHARACTERISATION FOR FCC STEELS

The characterisation of material under the effects of hydrogen embrittlement is of extreme importance in various fields of science and engineering. Hydrogen embrittlement characterisation is relevant in the energy industry, especially in renewable energy production and storage. Materials used in fuel cells, batteries and hydrogen storage systems must be evaluated in terms of their resistance to embrittlement. This is crucial to ensure the efficiency and durability of these devices, as well as to avoid possible failures or ruptures that could compromise the safety of installations and users.

The importance of this research can therefore be summarised in three main points:

- New knowledge on understanding the hydrogen behaviour of metals after complex manufacturing processes.
- Further guide the future design of materials with better hydrogen properties for various applications.
- The safe use and public acceptance of compressed hydrogen gas for the development of hydrogen systems.

#### 3.2 OBJECTIVES

In order to guarantee the correct development of the project, it has been necessary to define more precise objectives. Although it is clear that the main objective is the characterisation of the steels 316L and 316L+, these are some sub-objectives that will allow the project to develop properly:

- Characterisation of the hydrogen diffusion and embrittlement behaviour under different stress states.
- Evaluate the difference in the failure performance among 316L and 316L+.

- Analyse the effect of hydrogen embrittlement in order to predict fracture.
- Follow the plan designed at the beginning.

Through this detailed analysis, it is intended to obtain fundamental information on the mechanisms of hydrogen embrittlement in the metals studied, as well as their relative susceptibility to this phenomenon. The results of this study will contribute to a better understanding of hydrogen embrittlement in 316L and 316L+ metals and will provide valuable information for the design and selection of materials in applications where the presence of hydrogen is a critical factor to be considered.

### 3.3 PLANIFICATION

During eight months, the project has gone through various stages of development and implementation. The initial planning established a clear and detailed structure of the tasks to be carried out, distributed along the established period. However, during the course of the project, some modifications and adjustments were made to adapt to changing circumstances and new findings or challenges encountered. The final planning, with the updated stages and activities workflow, is presented in detail in the attached [ANNEXE A](#).

### 3.4 SCHEDULE OF CONDITIONS AND COMPETENCES

You can find the schedule of conditions document in [ANNEXE B](#). This document has all the necessary information to carry out the project based on the objectives mentioned before. It helps to achieve the defined goals. When creating this document, technical, optional, and legal aspects have been considered to make sure everything is safe and done correctly.

### 3.5 PROJECT BUDGET

Finally, the project budget section details the costs related to the development of the project, as well as the costs associated with the use of warehouses, machine rooms, laboratory and office space. It is important to note that a detailed resource analysis has been carried out and has aimed to maximise efficiency and minimise costs without compromising quality and the achievement of the project's objectives. The breakdown of the economic budget can be found in [ANNEXE C](#).

## 4. EXPERIMENTAL METHODOLOGY

For the experimental methodology, different techniques have been used in order to obtain accurate and reliable results. In this section, the details of the procedures used for data collection and analysis, as well as the description of the research process, will be presented. The experimental design has been adapted to the specific objectives of the thesis, and the variables and possible directions have been carefully considered. The methodological approach has been selected in order to answer the research questions in a clear and rigorous way.

### 4.1 CHARACTERISATION

The characterisation of a material involves evaluating its physical, chemical, mechanical, optical, thermal and electrical properties. For this purpose, different techniques and procedures are used, such as chemical analysis, microscopy, X-ray diffraction, mechanical testing, and spectroscopy, among others. The objective of characterizing a material is to understand its properties and behaviour in different situations and to determine its suitability for various applications.

The experimental methodology used focuses on the characterisation of the mechanical properties of the material. Both tensile and fracture tests will be carried out on the steel samples, with and without hydrogen.

The first step performed upon receiving the 316L and 316L+ sheets was the analysis of their chemical composition using an Optical Emission Spectrometer (OES). This is important as the chemical composition can affect the mechanical properties and behaviour of the material. The *BELEC LAB 3000S* machine has been used, available in Aalto's laboratories. This machine uses the intensity of light emitted from a flame, plasma, arc, or spark at a particular wavelength to determine the quantity of an element in a sample. The wavelength of the atomic spectral line in the emission spectrum gives the identity of the element while the intensity of the emitted light is proportional to the number of atoms of the element [26]. Pure aluminum tape was used to make contact with the machine. The results of the analysis and the comparison with the data of the supplier *Outokumpu* can be found in [ANNEXE D](#).





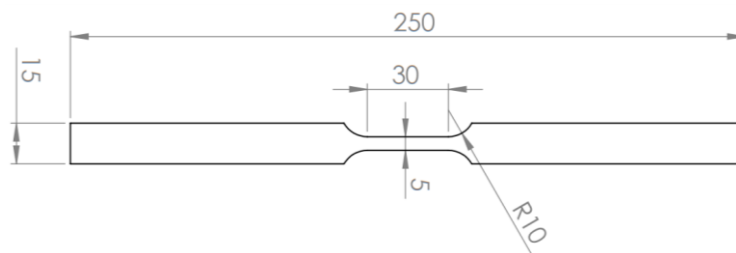
**2. Figure** BELEC LAB 3000S machine

After chemical composition verification, the following are the tests and the geometries used.

#### 4.1.1 Tensile tests

Firstly, tensile tests were performed to determine the elastic and plastic properties of the material. These tests involve the application of a gradually increasing uniaxial force to the specimens and the simultaneous measurement of the applied load and the resulting deformation. This provides data on the tensile strength, yield strength and ductility of the material.

For the tensile tests, the SDB (Smooth Dog Bone) geometry was employed. The SDB geometry features a standard shape with a narrowed neck section and enlarged ends. The dimensions of the sample can be observed in the next Figure 3.



**3. Figure** SDB geometry dimensions in mm

SDB geometry is widely accepted and recognized in the field of materials testing. It is a standard specimen shape recommended by various international standards organizations, ensuring comparability and reproducibility of results.

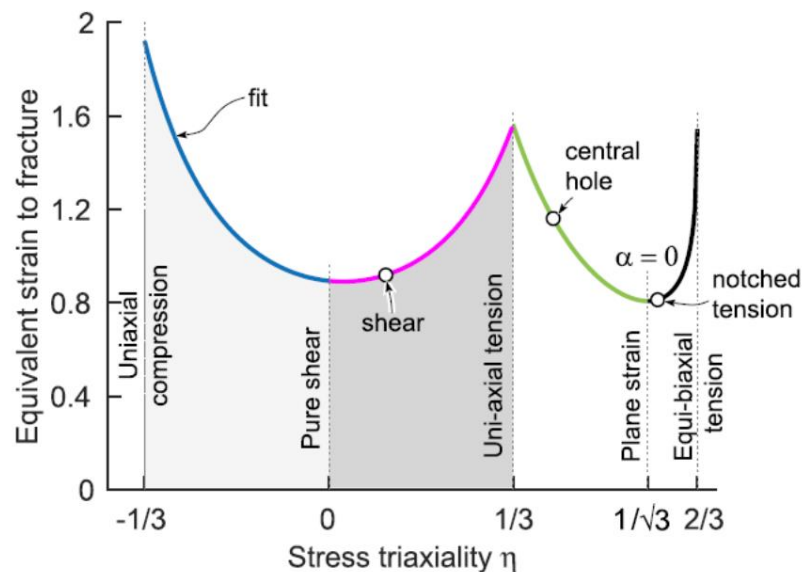
The dimensions (250 mm x 15 mm) of the specimens are conditioned to fit properly within the charging cell, which will be introduced later.

#### 4.1.2 Fracture tests

Fracture tests have been carried out to evaluate the resistance of the material to crack propagation at different stress states. To cover a wide range of stress triaxialities, different specimen geometries are used in the tests.

The choice of specific specimen geometries allows different stress states to be simulated and provides a more complete understanding of the material's behaviour in terms of toughness and fracture.

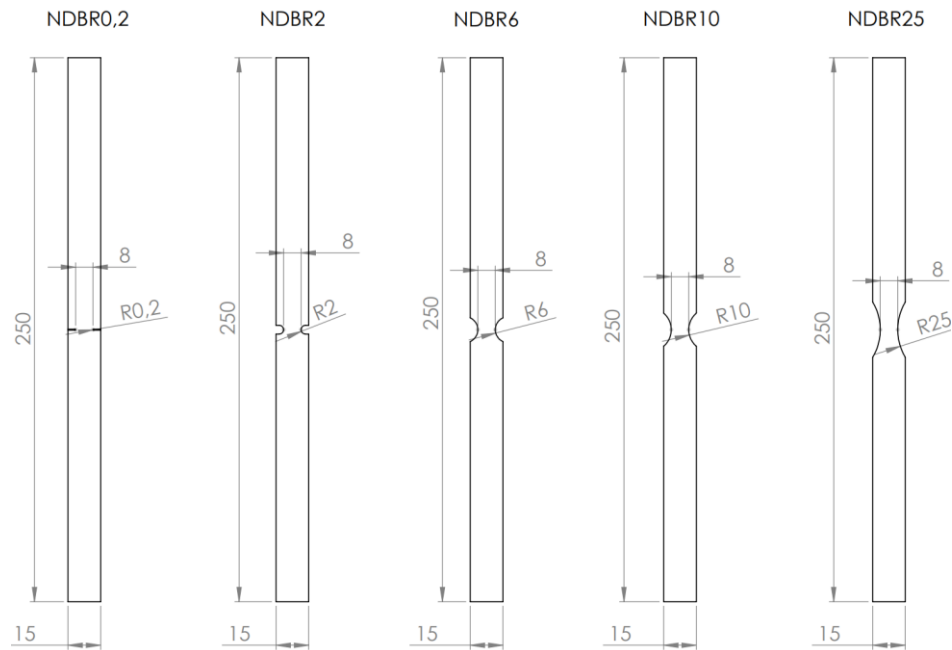
Therefore, geometries such as NDB (Notched Dog Bone), CH (Centre Hole) and SH (Shear) have been used to address different loading modes and stresses in the tests. This is shown in the following Figure 4.



**4. Figure** Stress triaxiality vs Equivalent strain to fracture graph for fracture tests [34].

#### NDB (Notched Dog Bone)

The NDB geometries are used to evaluate fracture toughness under conditions of uniaxial stresses and stress concentration at the notch tip. The dimensions of the samples can be observed in the next Figure 5.

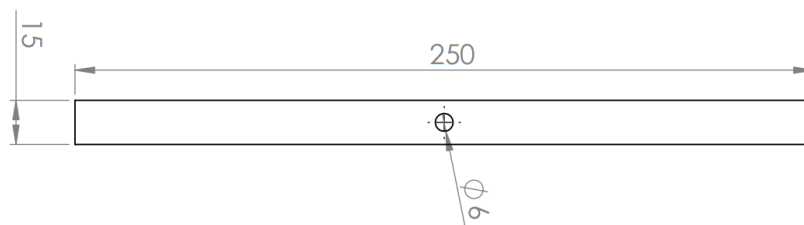


**5. Figure NDB geometries dimensions in mm**

Those geometries represent the transitional stress states from uniaxial to plane strain at the critical point. The sample with a large notch radius (NDBR25) tends more towards uniaxial tension, while the one with a smaller notch radius (NDBR0.2) is closer to plane strain.

### CH (Centre Hole)

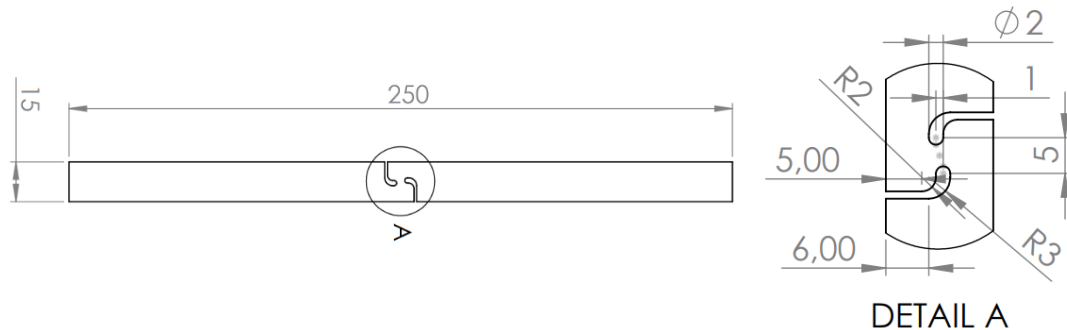
CH geometry is used to investigate the resistance to crack propagation in biaxial or radial stress situations, through the opening of a crack from the centre of the specimen. The dimensions of the sample can be observed in the next Figure 6.



**6. Figure CH geometry dimensions in mm**

## SH (Shear)

Shear geometry is used to study creep resistance and crack propagation under pure shear loading conditions. The dimensions of the sample can be observed in the next Figure 7.



**7. Figure SH geometry dimensions in mm**

This geometry involves the use of samples with a parallelogram cross-section or a specific shape that promotes the shearing of the material.

### 4.1.3 Anisotropy

The anisotropy of the material due to its manufacturing process, cold rolling, has been analysed. During the cold rolling process, the grains of the material experience deformation, which can have a significant impact on its mechanical properties.

To understand how this anisotropy affects the material, tensile tests have been carried out using SDB (Standard Dog Bone) geometry in three different directions: 0 degrees, 45 degrees, and 90 degrees.

In the 0° direction, the tensile force is applied parallel to the rolling direction (RD). This gives an assessment of the mechanical properties of the material in the main rolling direction.

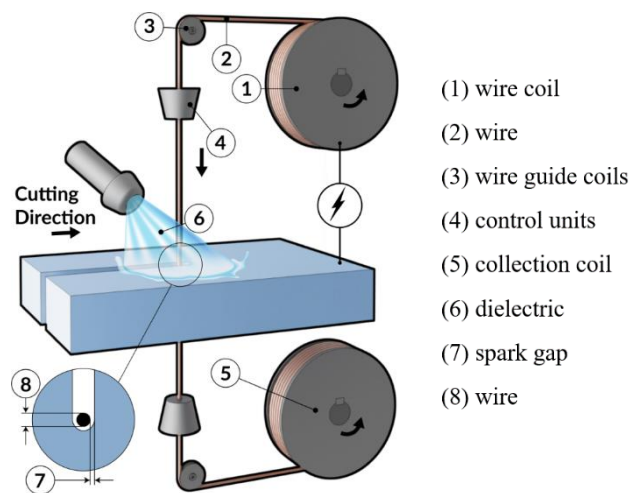
In the 90° direction, the tensile force is applied perpendicular to RD as the transverse direction (TD).

The 45° direction is therefore the diagonal direction (DD).

#### 4.1.4 Specimen manufacturing

The samples tested for this project have been manufactured by EDM. The wire electrical discharge machining (EDM) process is a relatively simple but precise method of cutting and shaping workpieces. In this process, an electrical discharge is created between the tool and the workpiece, generating a controlled spark with extremely high temperatures ranging from 8000°C to 12000°C. This intense heat vaporizes the conductive materials, enabling precise material removal [27].

The spark is produced within a small gap between the tool and the workpiece, which is immersed in a dielectric fluid, usually deionized water. The dielectric fluid serves multiple functions: it acts as a non-conductive medium for the electrical discharge, provides cooling during the machining process and helps to remove eroded metal particles [27]. Figure 8 shows what it looks like and its components.



8. Figure EDM machine and components [27]

Unlike traditional machining methods, WEDM uses a thin electrode wire to follow a pre-programmed path, cutting or shaping the workpiece with high precision. The diameter of the electrode wire typically ranges from 10 mm to 30 mm, depending on the specific requirements of the machining project.

One of the main advantages of WEDM is its ability to control and precisely locate the spark, limiting its effects on the surface of the material. This allows intricate and complex shapes to be achieved without affecting the structural integrity of the part.

## 4.2 ELECTROCHEMICAL CHARGING

The objective of this study is to investigate and compare the behaviour of 316L(1.4404) and 316L+(1.4420) metals against hydrogen embrittlement. For this purpose, electrochemical loading has been used as a method of charging the samples, which allows the simulation of realistic and controlled hydrogen exposure conditions.

In this process, the sample is placed as the cathode in an electrochemical cell, and electricity is used to carry out a redox reaction that involves the production of hydrogen gas at the cathode. During the process, the hydrogen gas diffuses into the metal matrix, charging it with hydrogen and causing changes in its mechanical properties.[\[28\]](#) [\[29\]](#).

Another method is gas charging. Gas charging and electrochemical charging are two commonly used methods for introducing hydrogen into metal samples for research purposes.

Gas charging involves exposing the metal samples to a controlled hydrogen gas environment. The samples are placed in a chamber or vessel filled with hydrogen gas, and the pressure, temperature, and exposure time are carefully regulated. It requires specialized equipment to handle the hydrogen gas safely.

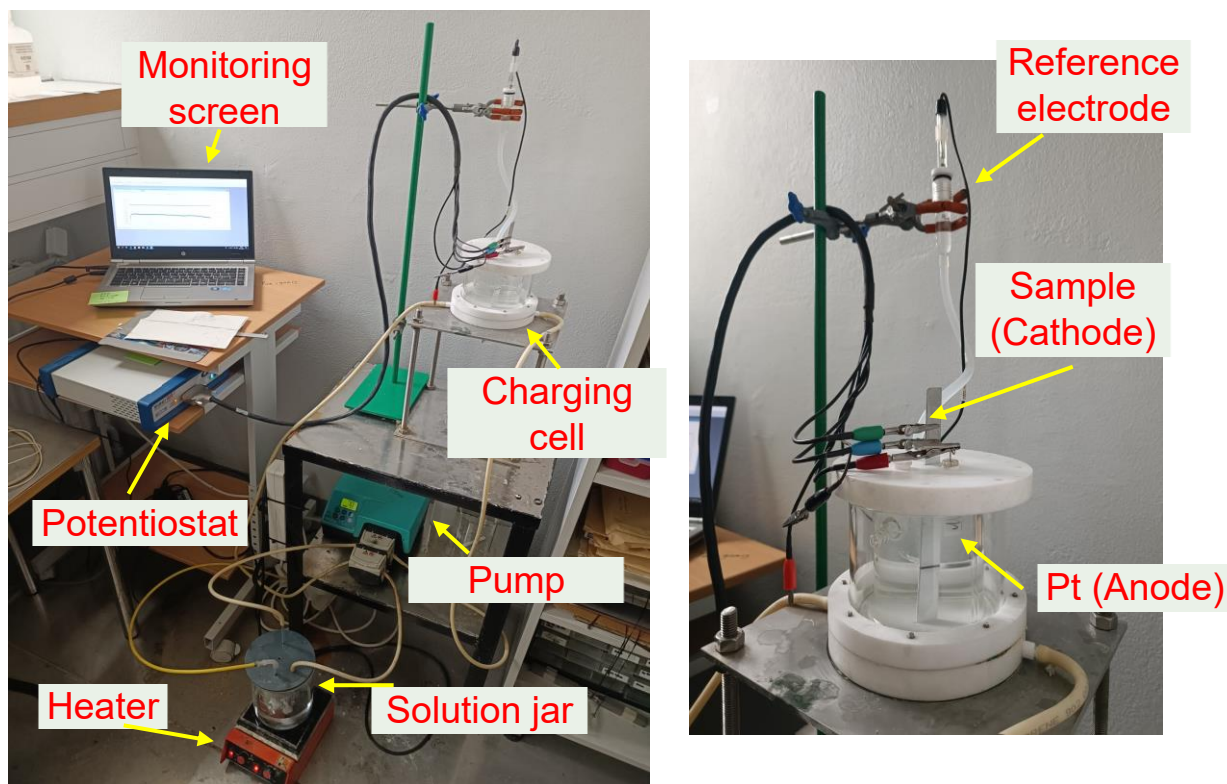
Gas charging generally allows for faster hydrogen uptake compared to electrochemical charging. The exposure time required for gas charging can be shorter, leading to accelerated hydrogen diffusion into the material. In contrast, electrochemical charging can take longer because the hydrogen diffusion rate depends on the charging current density and the material's electrochemical behaviour, it can be a relatively time-consuming process. However, electrochemical charging provides better control over the hydrogen distribution and allows for studying the effects of specific electrochemical conditions.

Safety considerations also differ between the two methods. Gas charging requires careful handling of hydrogen gas due to its flammable nature, and appropriate safety measures must be in place. Electrochemical charging, on the other hand, is considered a safer alternative as it does not involve direct handling of hydrogen gas.

The choice of electrochemical charging as the charging method was mainly based on the availability of resources and equipment in the laboratory where the research was carried out. Within the laboratory, there were the necessary instruments and infrastructure to perform electrochemical charging in a precise and controlled manner.

### 4.2.1 Experimental Setup

The design of the experimental setup used is detailed below, as well as the different components used in the electrochemical charging process of the samples. This setup was carefully selected to guarantee the precision and repeatability of the results obtained.



**9. Figure** Hydrogen charging setup

Figure 9 shows the setup that has been used at Aalto University's facilities to charge the metal samples. In the charging cell is where the sample is placed and the electrolysis takes place. The solution is stored in the jar above the heater in case the temperature of the solution needs to be changed. The pump is responsible for recirculating the solution from the jar to the charging cell. During loading, the pump is constantly recirculating the solution to ensure a homogeneous distribution of ions and to prevent the products of the chemical reaction from accumulating in any one place. The electricity is supplied by the potentiostat, which is connected to the sample and to a platinum piece that works as the anode in the electrolysis. Finally, there is the reference electrode, which is used to monitor the electrochemical conditions during charging.



## 4.2.2 Electrolysis

During electrolysis, water is split into its component parts by applying an electric current through two electrodes placed in the water. The anode (positive electrode) forms oxygen gas while the cathode (negative electrode) forms hydrogen gas. This process can be carried out in either an acidic or basic solution.

The electrical conductivity of water is increased by adding electrolytes, such as salts, acids, or bases. These electrolytes dissociated in the water provide ions that facilitate electrical conduction and allow electrochemical reactions at the electrodes.

Using acidic solutions in electrochemical charging for introducing hydrogen into stainless steel samples offers advantages over saline solutions. Acidic solutions provide better pH control, allowing adjustment for optimized electrochemical reactions. They also offer selectivity, promoting specific electrochemical reactions. Additionally, acidic solutions have a controlled corrosive potential, minimizing corrosion and defects in the metal samples during charging.

In this project, the solution used is an acidic aqueous solution with a concentration of 1N H<sub>2</sub>SO<sub>4</sub>. To this solution, 20 mg/L thiourea (CH<sub>4</sub>N<sub>2</sub>S) is added as “hydrogen poison”, and acts as a hydrogen evolution inhibitor, meaning it suppresses the formation of hydrogen gas during the electrochemical process. By inhibiting the hydrogen evolution reaction, the concentration of hydrogen in the metal sample increases, facilitating the hydrogen charging process.

On the other hand, in the reference electrode, there is a curved Luggin-type salt bridge filled with potassium sulfate salt (K<sub>2</sub>SO<sub>4</sub>). Works as a stable medium to maintain a constant reference potential during electrochemical charging by remaining inert and not participating in the reactions. Due to its high ionic conductivity, the reference electrode ensures a reliable and consistent electrical response, allowing accurate monitoring of the electrochemical charging process. It was kept at a 2 mm distance from the sample during charging.

It is recommended to change the acid solution after each electrochemical charge, as the acid can become contaminated with impurities and affect the consistency of the results. Additionally, the acidity of the solution can change during the electrochemical charge, which can also affect the results of subsequent charges if the same solution is reused. So, it is best to use a fresh acid solution for each electrochemical charge.



### 4.2.3 Electrodynamic Scan

Electrochemical charging is carried out at constant potential, which implies maintaining a fixed potential value during the charging process. As a first step, a potentiodynamic scan has been performed for the two materials to obtain information about the electrochemical behaviour of the system.

The potentiodynamic scan is a technique that gradually varies the potential in a specific range and records the current generated as a function of the applied potential. This scan makes it possible to identify and characterise the electrochemical processes occurring at the electrode interface and to determine the appropriate potential to carry out electrochemical charging at a constant potential.

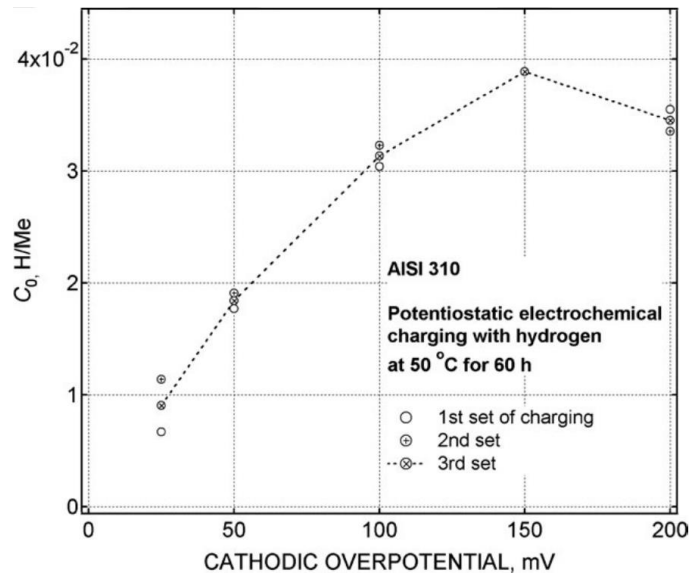
In this case, considering the specific electrochemical properties of the sample's material, the potential range is from -1.3V to -0.6V. The potentiodynamic scan was performed at a rate of 1mV per second and the curves can be seen in [ANNEXE E](#).

After these measurements, the corrosion potentials are known to be -802mV and -816mV, for 316L and 316L+ respectively.

### 4.2.4 Charging Conditions

The charging conditions have been defined at the beginning and are maintained for all samples to guarantee the same hydrogen absorption and good repeatability of the results.

To determine the charging potential, an overpotential of 150 mV above the corrosion potential has been chosen with the solution at 50 ° Celsius. These conditions have been chosen based on a previous study carried out at Aalto University, where hydrogen solubility and diffusion in austenitic stainless steels were studied to find the optimum loading conditions for AISI310.



**10. Figure** [Hydrogen solubility and diffusion in austenitic stainless steels studied with thermal desorption spectroscopy][30]

In the graph,  $C_0$  refers to the solubility of hydrogen and can be observed as it reaches the peak at an overpotential of 150 mV. Hydrogen solubility refers to the ability of a material to dissolve and retain hydrogen in its structure. The solubility of hydrogen varies according to the type of material and its crystalline structure, it is worth mentioning that AISI310 is used as a reference due to the limitation of resources and time to study the solubility for 316L and 316L+.

On the other hand, the reason for heating the solution to 50 degrees Celsius is that as the temperature increases, the diffusivity of the ions in the solution and the hydrogen atoms in the material tends to increase. This can accelerate the charge transfer and diffusion processes, facilitating the incorporation of hydrogen into the material.

The higher temperature can also help promote a more uniform distribution of hydrogen in the material during electrochemical charging. This is especially important in hydrogen embrittlement studies, where the aim is to analyse and understand the effects of hydrogen on the structure and properties of the material.

Finally, the charging time of 72 hours has been defined due to the characteristics of the samples under study. These samples are relatively wide, with a thickness of 1.5 mm. Due to their thickness and the low diffusivity of stainless steels, a prolonged loading time is required to allow sufficient hydrogen absorption in the material and to observe the effects of hydrogen embrittlement. It was important to consider the balance between the loading time needed to obtain meaningful results and the practical duration of the experiment.

#### 4.2.5 Sample Preparation

Proper sample preparation is necessary to ensure accurate and reliable results.

Metal samples undergo a polishing process to remove any surface layers, imperfections, or contaminants that may affect the results of the experiment. The samples studied are finished with P1200 sandpaper.

After polishing, the samples are cleaned using ethanol. This helps to remove any residue or particles that may be present on the sample surface and ensures a clean base for the electrochemical charging process.

Finally, to ensure that only the region of interest (gauge) is charged with hydrogen, the parts of the sample that are not to be charged are covered with insulating tape. The insulating tape acts as a barrier that prevents direct exposure of these areas to the electrochemical charging solution, ensuring that only the area of interest is affected by the hydrogen.

In Figure 11 below, a sample of SDB is shown prepared for introduction into the charging cell with the described preparation.



**11. Figure** Sample prepared before electrochemical charging

#### 4.3 THERMAL DESORPTION SPECTROSCOPY (TDS)

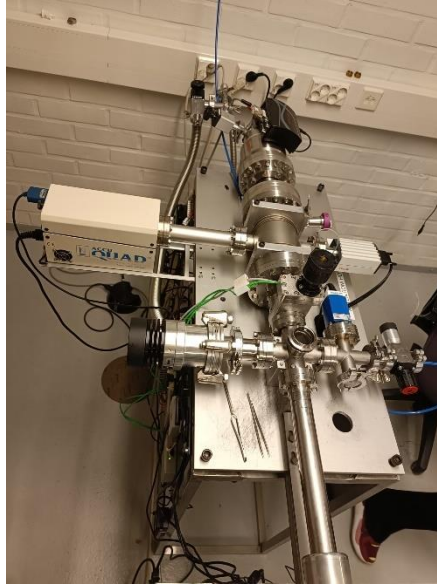
After the electrochemical charging process, it is crucial to determine the hydrogen concentration within the two materials, 316L (1.4404) and 316L+ (1.4420). To achieve this, Thermal Desorption Spectroscopy (TDS) has been employed as an analytical technique.

TDS is a technique used for the measurement of hydrogen concentration in metallic materials. It involves heating a sample in a vacuum or a controlled environment to desorb trapped hydrogen and analyzing the released gases [\[31\]](#).

Allows the determination of desorption temperatures and activation energies associated with hydrogen desorption, revealing the stability and strength of hydrogen-metal bonds. In

addition, provides quantitative information about the amount of hydrogen adsorbed on the metal surface at different temperatures, aiding in understanding surface coverage and saturation levels.

The following Figure 12 shows the TDS machine available in Aalto, which was used to determine the hydrogen concentration in 316L and 316L+ materials by replicating the charging conditions defined above.



**12. Figure** TDS machine

## 4.4 MECHANICAL TESTING

This section presents the methodology of the mechanical tests that have been carried out to determine the mechanical properties of the samples with and without hydrogen.

### 4.4.1 Zick Rowell Tensile Machine

The tests have been conducted at room temperature with a *Zwick/Roell Z020* screw-driven tensile testing machine, which has a maximum load capacity of 20kN. To start the tensile testing, the specimen was gripped at each end and stretched along its length direction at a constant crosshead velocity until it fractures.

#### 4.4.2 Strain Rate

The chosen constant crosshead velocity was 0.9 mm/min for tensile tests, and for the specimen gauge length of 30 mm, it corresponded to the quasi-static strain rate of  $5 \times 10^{-3} \text{ s}^{-1}$  before necking. For the fracture tests, a strain rate of 0.45 mm/min was used.

It is important to find an appropriate balance in the strain rate used in the tests, allowing sufficient time for the redistribution of hydrogen in the material, but without causing excessive loss of hydrogen concentration.

Very fast deformation would result in the hydrogen concentration remaining localised in certain regions and not diffusing uniformly. As a result, the effects of hydrogen embrittlement may not be fully evident in tests, which may underestimate the real effects of hydrogen on the mechanical properties of the material.

In the opposite case, hydrogen may desorb from the material and diffuse into the atmosphere as a gas. This may result in a decrease in the hydrogen concentration in the material and affect the test results. This happens because the sample is taken out of the charging cell for testing, a solution to this problem would be to test in situ.

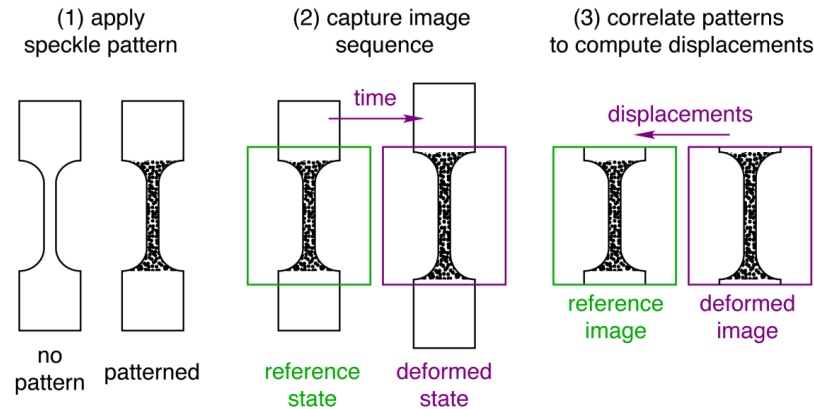
With the deformation velocities of 0.9mm/min for the tensile test and 0.45 mm/min for the fracture test a balance is achieved, and the time to failure is between 20-25 minutes.

#### 4.4.3 Digital Image Correlation (DIC)

The applied load and full-field displacement at the uniform deformation zone were measured using a load cell and an optical DIC system.

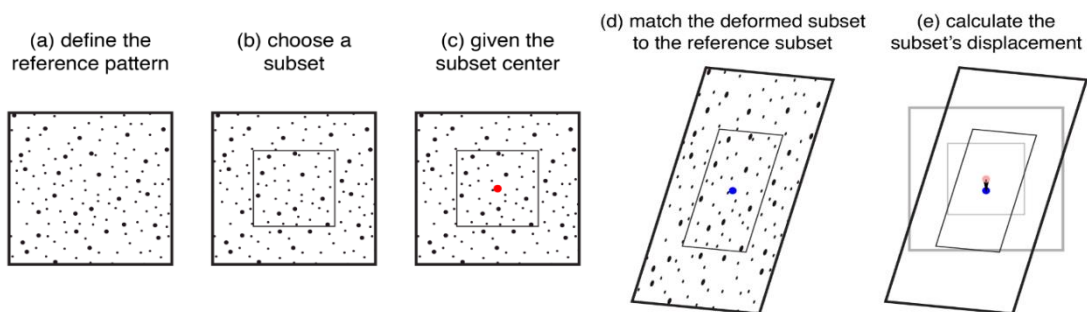
DIC is an optical-based technique to measure the evolving full-field 2D or 3D displacements on the surface of a specimen throughout a mechanical test. The measured displacement fields can be used to calculate derived field quantities such as strains, strain rates, velocities, and curvatures. As DIC is a non-contact technique that is independent of the material being tested, it can be used in a wide variety of applications to investigate and characterise the deformation of solids. To perform DIC measurements, the specimen surface to be measured is typically randomly patterned with features of the desired speckle size that is chosen based in part on the intended Image Scale (Figure 13.1). This surface pattern is then imaged using calibrated digital cameras and lenses system (Figure 13.2). These images are taken both before and during

specimen motion and/or deformation (strain) all within the Field of View (FOV) of the cameras (Figure 13.3). The process is shown in Figure 8.



13. Figure DIC measurements process [35].

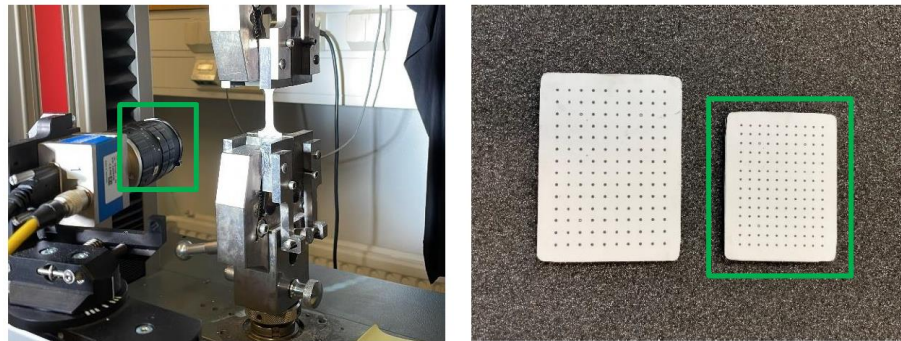
2D displacements can be measured using a single camera system, whereas 3D shapes and displacement measurements require a pair of cameras oriented at a Stereo-Angle to perform 3D photogrammetry in addition to the image correlation. In this method, the first image of the sequence is set as the reference image (Figure 14.a), so that all other images obtained are compared with it. The resulting images are then interrogated in a Region-of-Interest (ROI) within the FOV, which is sub-divided into Subsets (Figure 14.b) at some spacing (Step Size) (Figure 14.c). The subsets are numerically correlated from the reference image (before motion/deformation) to each subsequent image (during motion/deformation) (Figure 14.d). This correlation is performed by approximating the pattern in each subset using an interpolant function, and then allowing that function to deform from the reference image based on a Subset Shape Function that is then compared (Figure 14.e). The result of the correlation is the measured displacement of the centre of each subset that is related to physical units based on the calibration of the used digital cameras and lenses system used.



14. Figure DIC 3D acquisition method [35].

In this study the 3D system was used. The observed specimen surface is lacquered with a white paint coating layer as a background to ensure the image quality. Besides, to improve the spatial resolution and displacement sensitivity of the DIC measurement, the black pattern was obtained by using a black paint spray.

DIC system has been used as an optical extensometer to measure the deformation in the gauge of specimens during mechanical testing. the DIC provides detailed information on localised strain fields, allowing for a more complete analysis of the mechanical behaviour of the material. For the SDB samples, the extensometer used for the y-axis has been 30 mm and for the x-axis 9.3 mm. For the NDB it has been 25mm only in the y-axis. Finally, for the calibration, the smallest calibration target (Figure 15) of 4mm from Correlated solutions was used.



**15. Figure** Hikvision camera and Calibration targets for DIC measurements



## 5. RESULTS AND DISCUSSION

This section presents the results of the tests performed, showing the outcomes obtained from the tests. The focus is on examining the effects of hydrogen embrittlement on the mechanical properties, particularly on the ductility of 316L (1.4404) and 316L+ (1.4420) stainless steel materials. The analysis of the data allows a better understanding of the phenomenon of hydrogen embrittlement. The results are presented by means of figures, tables and graphs, accompanied by explanations to facilitate their understanding.

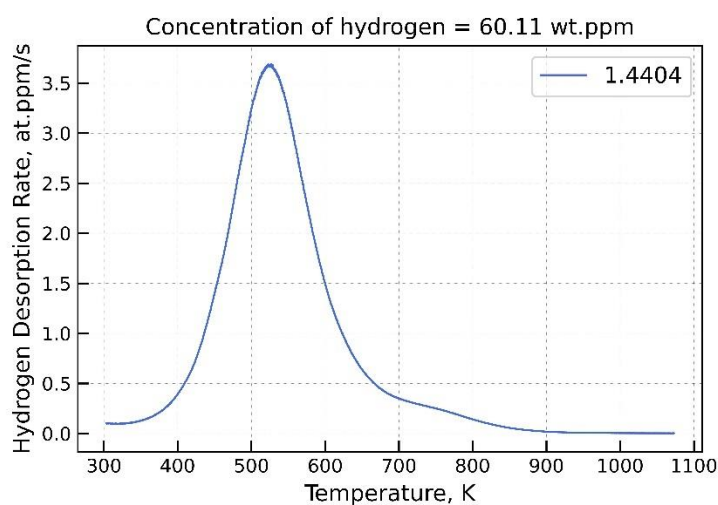
Due to time constraints, not all planned geometries could be tested. So, the experimental focus is initially on the SDB and NDB specimens with both large and small radii to cover different points of triaxiality.

This results section is divided into four parts, hydrogen concentration results, the results for the anisotropy tests, the tensile tests and finally the fracture tests.

### 5.1 HYDROGEN CONCENTRATION RESULTS

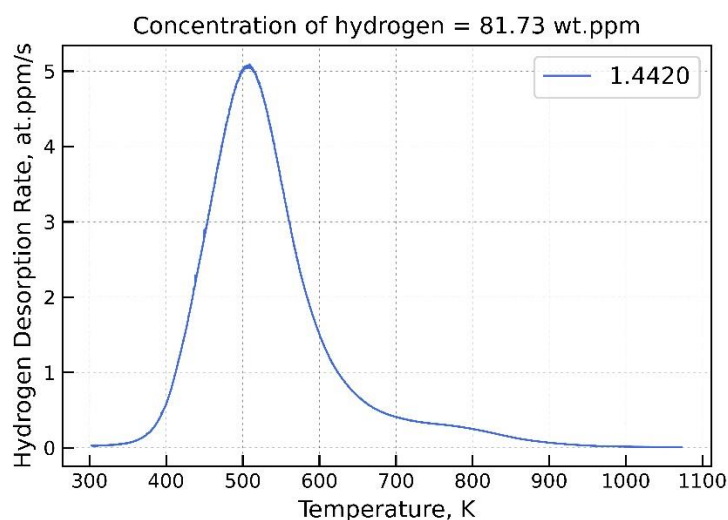
The results of the hydrogen measurement by thermal desorption spectroscopy are presented below.

The dimensions of the loaded sample are limited to 4.5 mm x 15 mm with a thickness of less than 0.9 mm by the machine. The measurements can be seen in the following charts. The heating rate is 10 K/min.



**16. Figure** Hydrogen desorption spectra of 316L





**17. Figure** Hydrogen desorption spectra of 316L+

The results show a 21.62 wt. ppm. the difference in hydrogen concentration after electrochemical charging between the two materials. Material 1.4404 exhibits a concentration of 60.11 wt. ppm., while material 1.4420 shows a concentration of 81.73 wt. ppm. “wt. ppm” refers to “weight parts per million,” which is a unit of measurement used to express the concentration of a substance in a solution or material.

Both materials, 316L (1.4404) and 316L+ (1.4420) exhibit a desorption activation temperature range of 510-525 Kelvin associated with hydrogen desorption. This indicates that the release of adsorbed or dissolved hydrogen in these materials predominantly occurs within this temperature window.

The presence of this activation temperature suggests the existence of specific trapping sites or bonds in the materials that require a certain amount of energy to release hydrogen. The observed desorption peaks at this temperature indicate the contribution of these sites in the hydrogen release during TDS analysis.

The secondary peak around 780 Kelvin could be attributed to the desorption of hydrogen from more strongly trapped sites or bonds within the material. These sites may require higher temperatures or a longer heating time to release the trapped hydrogen.

Both materials were subjected to the same electrochemical charging conditions, indicating that the difference in hydrogen concentration is not due to the experimental conditions.

One possible reason is that material 1.4420 has a higher affinity for hydrogen due to the presence of nitrogen in its chemical composition. This can result in greater hydrogen absorption and retention in the material, leading to a higher concentration after charging. [22]

Additionally, the structure and microstructure of the materials could also influence their ability to absorb and diffuse hydrogen. It is possible that material 1.4420 has a structure or microstructure that facilitates the absorption and retention of hydrogen compared to material 1.4404.

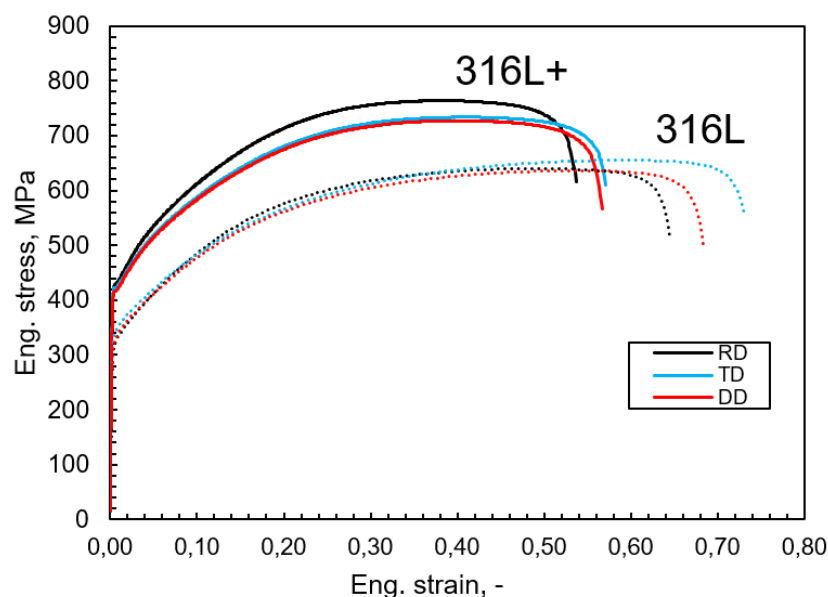
Furthermore, the difference in hydrogen concentration could be attributed to the corrosion of the material 1.4420 during the charging process, as it was visibly corroded to the naked eye. Corrosion can lead to the formation of localised areas with increased hydrogen uptake, resulting in a higher concentration in the material.

Another factor to consider is the sample preparation. From removal from the charging cell to analysis in the TDS can have an impact on the hydrogen concentration results. During this preparation process, changes in the distribution and release of hydrogen in the material are possible. It is essential to take into account all the steps involved in sample preparation and handling to minimise any possible loss or alteration of hydrogen.

In conclusion, the observed difference in hydrogen concentration can be attributed to various factors. However, it is important to note that the concentrations of both materials (1.4404 and 1.4420) are relatively similar. Therefore, we can assume that the mechanical tests are initiated under comparable charging conditions. To ensure the reliability and repeatability of the measurements, it is recommended to perform the hydrogen concentration measurements multiple times in the future.

## 5.2 ANISOTROPY TESTS

First, the results of the anisotropy tests in the three directions (RD, TD and DD) are studied to see how cold rolling affects the mechanical properties of the materials. The following graph shows the results of the Eng. stress and Eng. strain graphs.



**18. Figure** Eng. Stress vs Eng. Strain in RD, DD and TD directions

It can be observed how the 316L material shows a quite isotropic behaviour with a variation in ductility of 8.53% of total elongation. However, the 316L+ material shows considerable anisotropy in the RD direction showing an increased yield strength and ultimate strength compared to the other directions, showing up to a difference of up to 30MPa between the RD and the other two directions in the UTS.

It is also curious to mention that the RD in both cases is when the material presents the lowest ductility. This is possible because, during the cold rolling process, the grains of the material are aligned in the rolling direction. This means that the grains are preferentially oriented in the rolling direction and elongate in that direction.

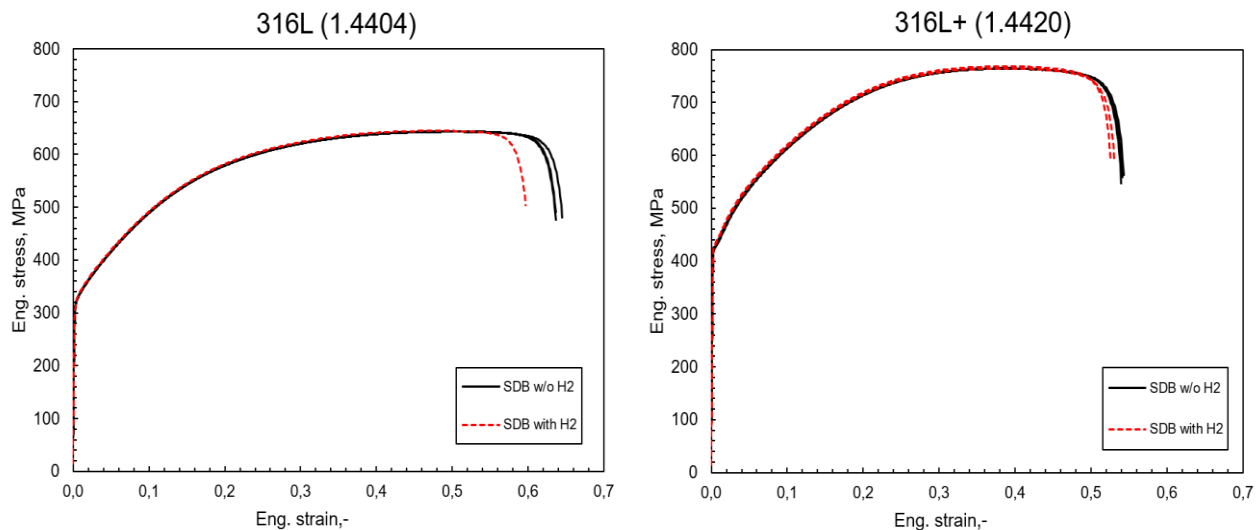
This alignment of the grains in the rolling direction can result in a higher mechanical strength in that direction due to a higher blocking of dislocations. However, it can also cause sliding of the crystalline planes to be more difficult and thus reduce the ductility in that direction.

In contrast, in the transverse direction, the grains are not aligned in a preferential direction and their orientation is more random. This allows for greater movement of dislocations and easier sliding of the crystalline planes, which can lead to higher ductility in this direction.

This difference in grain orientation and its impact on ductility is based on the crystalline structure of the material and the associated deformation mechanisms.

### 5.3 TENSILE TESTS

The following Figure 19 shows the results of the tensile tests for both materials, with and without hydrogen.



**19. Figure Eng. Stress vs Eng. Strain for SDB**

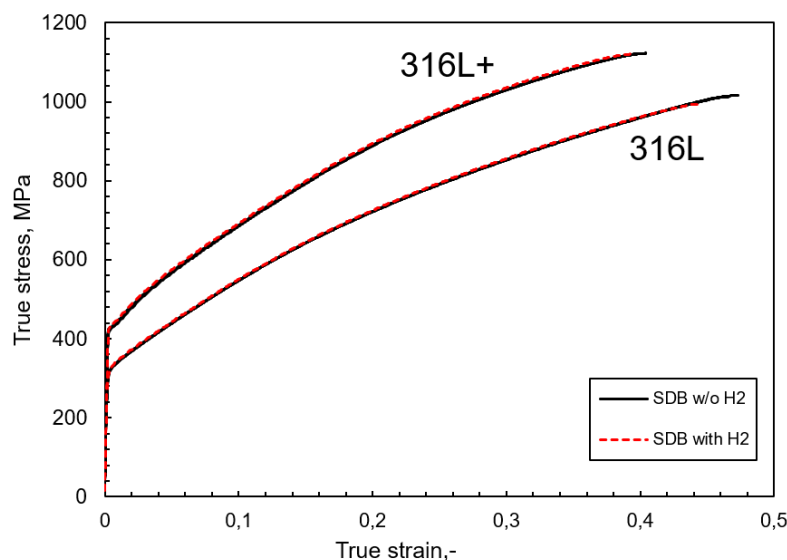
The test results clearly show that the presence of hydrogen has an impact mainly on the ductility of the material, while the rest of the stress values remain almost unchanged. This can be seen in the engineering stress-strain graphs, where a 4.33% ductility reduction in the plastic deformation capacity of the 316L material and 1.79% for 316L+ when exposed to hydrogen can be seen.

The mechanical properties are summarised in the Table 3 below, and it can be seen that even in the samples with hydrogen a minimally higher UTS is measured. Hydrogen atoms could interact with defects in the crystalline structure of the material and strengthen the bonds between metal atoms, increasing the resistance to deformation.

**3. Table Mechanical properties of 316L and 316L+ for SDB tests**

316L (1.4404)		
	Tensile Strength [MPa]	Elongation [%]
w/o H2	644	64.4
with H2	645	60.0
316L+ (1.4420)		
	Tensile Strength [MPa]	Elongation [%]
w/o H2	765	54.7
with H2	767	52.8

The results of the true stress-strain are shown below and it is even difficult to see with the human eye the difference between the lines with and without hydrogen, which confirms that HE does not affect the UTS and YS.



**20. Figure** True stress vs True strain for SDB 316L and 316L+

The YS for 316L material is 358 MPa for both cases and the UTS 1014Mpa w/o H2, while 994 MPa for with H2. In the case of 316L+, the YS is 416 MPa and 415 MPa, and the UTS 1123 MPa and 1119 MPa, for w/o H2 and with H2 respectively. The results have been compiled in the following Table 4 for easier viewing.

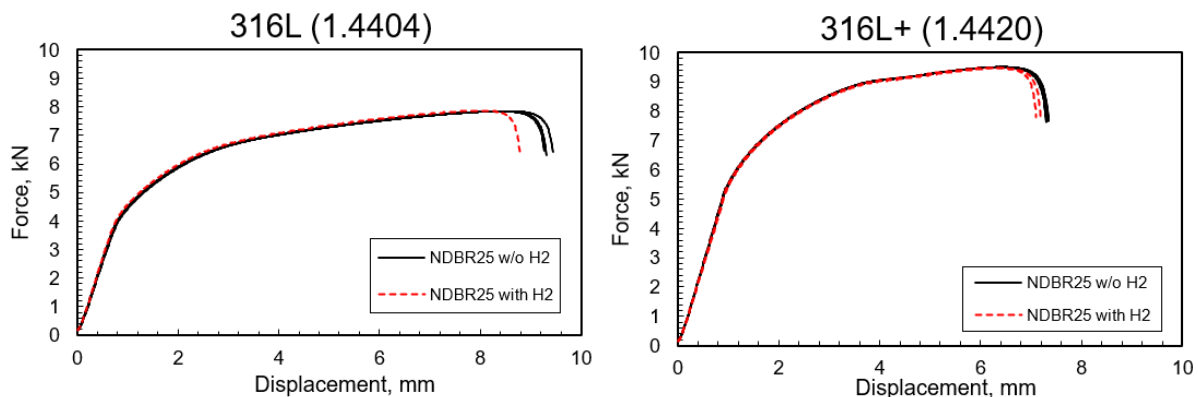
**4. Table** Mechanical properties of true stress vs strain

316L (1.4404)		
	YS [MPa]	UTS [MPa]
w/o H2	358	1014
with H2	358	994
316L+ (1.4420)		
	YS [MPa]	UTS [MPa]
w/o H2	416	1123
with H2	415	1119

All these results support the hypothesis that material 1.4420 (316L+), with its modified chemical composition and higher corrosion resistance, has a higher resistance to hydrogen embrittlement compared to material 1.4404 (316L).

## 5.4 FRACTURE TESTS

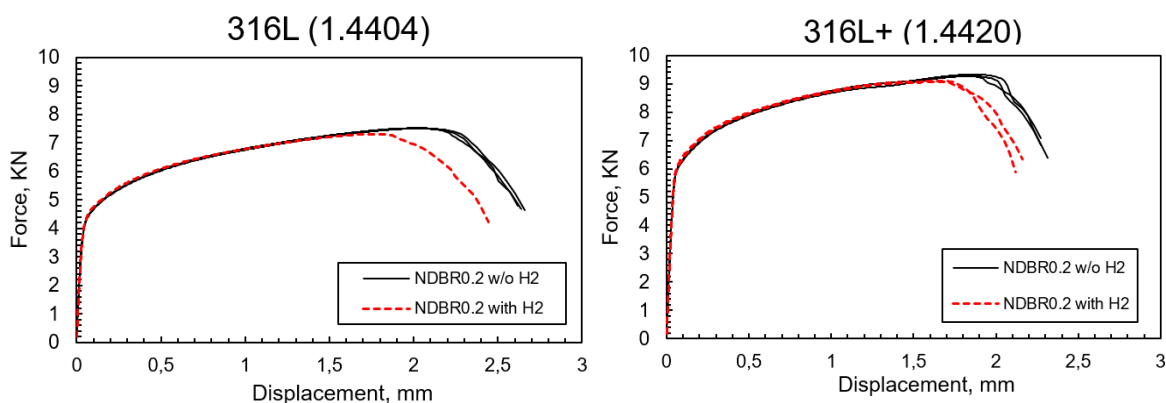
This section shows the results for the fracture tests for both NDBR0.2 and NDBR25 geometry. The force-displacement graphs of the R25 mm notches are shown in the following Figure 21.



**21. Figure** Force vs Displacement for NDBR25

We can see how the effect of HE has the same effect by advancing the fracture of the sample. The NDBR25 samples go from having an elongation of 7.22 mm to 6.78 mm at the gauge, assuming a 6.2% loss in elongation capacity in the 316L material. With respect to 316L+, the samples go from 6.20 mm to 5.95 mm due to the effect of hydrogen, resulting in a loss of 4%.

In the next Figure 22 the force-displacement graphs if the R0.2 mm notches are shown.



**22. Figure** Force vs Displacement for NDBR0.2

As in the previous tests, it is visible how the hydrogen charging affects the premature fracture of the sample. In this case, there has been a loss of 11% for 316L and 6.4% for 316L+.

In the following Table 5, the results of the fracture tests have been compiled for easier viewing and understanding.

### 5. Table Fracture tests results summary

NDBR25			
	Condition	Elongation [mm]	Loss [%]
316L	w/o H2	7.22	6.17
	with H2	6.77	
316L+	w/o H2	6.20	4.04
	with H2	5.95	
NDBR0.2			
	Condition	Elongation [mm]	Loss [%]
316L	w/o H2	2.75	10.98
	with H2	2.44	
316L+	w/o H2	2.29	6.41
	with H2	2.14	

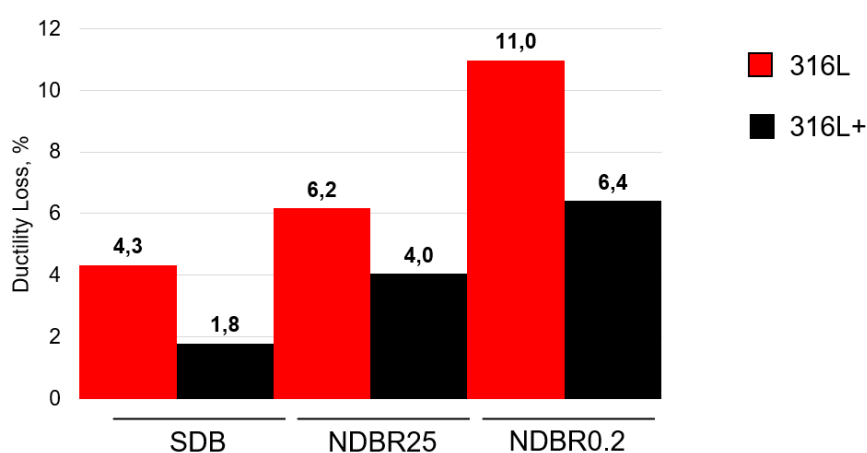
Analysing the results, it is clear that the 316L+ material shows a higher resistance to hydrogen embrittlement. But it can also be seen that the effect of this phenomenon is more pronounced when the notch of the sample is smaller.

## 5.5 DUCTILITY LOSS COMPARISON

In this section, the loss of ductility among different geometries and materials is compared. The aim is to highlight the significant differences in ductility.

For this purpose, the total elongation (TE) of the samples with h2 with respect to the samples w/o h2 has been calculated. The following equation has been used.

$$\text{Ductility Loss [\%]} = \frac{L_{TE \text{ w/o } h2} - L_{TE \text{ with } h2}}{L_{TE \text{ w/o } h2}} \times 100$$



**23. Figure** Ductility loss comparison

The analysis of the results reveals a clear trend regarding the effect of hydrogen on ductility loss. It is evident that as the size of the sample notch decreases, the impact of hydrogen becomes more pronounced. This can be attributed to the higher concentration of hydrogen in smaller notched samples, leading to a greater susceptibility to hydrogen embrittlement.

Furthermore, it is worth noting that the material 316L+ demonstrates superior resistance to hydrogen embrittlement (HE) compared to the other tested material. This can be attributed to its composition, which includes nitrogen that exhibits an affinity for hydrogen, reducing its diffusivity and mitigating the effects of hydrogen embrittlement.



## 5.6 GENERAL DISCUSSION

The research presented in this study focused on the characterisation of hydrogen embrittlement (HE) in austenitic stainless steels. By comparing two different stainless steel alloys, 316L and 316L+, the effects of hydrogen on their mechanical properties were investigated. Through electrochemical charging and subsequent thermal desorption spectroscopy (TDS) analysis, the hydrogen concentration in the materials was measured. Additionally, mechanical testing and digital image correlation (DIC) were employed to evaluate the deformation and fracture behaviour of the samples.

Although time constraints prevented the completion of all initially planned tests, a clear trend has emerged from the conducted experiments. It has been observed that hydrogen embrittlement (HE) is more pronounced in the 316L material compared to 316L+. Additionally, preliminary results indicate that samples with smaller notches exhibit a higher susceptibility to HE.

The susceptibility to embrittlement varies depending on the microstructure and composition of the material. Different materials exhibit varying levels of resistance to hydrogen embrittlement due to factors such as grain size, alloying elements and heat treatment.

Another important aspect to take into account is that the solubility and diffusivity of hydrogen can vary among different stainless steels and under different conditions. Factors such as alloy composition, microstructure, and temperature can influence the solubility and diffusivity of hydrogen within the material. Therefore, when choosing potentials for electrochemical charging, it would be appropriate to carry out material-specific solubility studies.

It is crucial to characterise materials under the effects of hydrogen embrittlement (HE) to predict, prevent fractures and to ensure safe usage.

## 6. CONCLUSIONS

The general conclusions drawn during the research on the characterisation of hydrogen embrittlement for FCC steels are listed below.

- The main mechanical property affected by hydrogen embrittlement is ductility. The presence of hydrogen leads to a significant reduction in ductility, with a maximum loss of 10.98% observed in the most severe case of this study.
- The effect of HE is more pronounced in geometries with small radii. This is due to higher hydrogen concentration in these areas. The reduced radius results in localised stress concentrations, which facilitate hydrogen diffusion and accumulation. It is therefore essential to consider the geometry of the component when considering susceptibility to hydrogen embrittlement.
- The presence of nitrogen in the material reduces the effect of hydrogen embrittlement and enhances its resistance. Nitrogen forms interstitial compounds with hydrogen, limiting its mobility and diffusion within the material's structure [22]. This prevents hydrogen from accumulating in high stress areas and reduces the risk of embrittlement. In addition, the increased corrosion resistance of the 1.4420 (316L+) material also provides additional protection against hydrogen penetration.
- Stainless steel has low diffusivity, which allows for testing to be conducted in ambient air. Samples can be taken directly from the charging cell without the need for specialized testing environments. This enables more efficient and cost-effective testing procedures for evaluating the susceptibility of stainless steel to hydrogen embrittlement, although it is important to be aware that there is a loss of hydrogen during the preparation of the tests.
- The thickness of the sample plays a significant role in the charging time required to observe the effects of hydrogen. In this study, the sample thickness was relatively large (1.5 mm) compared to the surface area being loaded with hydrogen. As a result, a charging time of 72 hours was necessary to ensure sufficient hydrogen absorption and diffusion within the material. A reduction in sample thickness would lead to a shorter charging time and consequently more tests could be done in the same time.
- The TDS measurements reveal a higher concentration of hydrogen in the 316L+ material. This could be attributed to the affinity between nitrogen and hydrogen, as

well as corrosion found in the sample. However, it is worth noting that despite the higher hydrogen concentration, the 316L+ material exhibits greater resistance to hydrogen embrittlement.

- The cold rolling does have an impact on the mechanical properties. In the case of 316L, it primarily affects ductility but the effect is relatively isotropic. However, in the case of 316L+, it exhibits improved ultimate tensile strength (UTS) and yield strength (YS) specifically in the rolling direction, indicating a higher level of anisotropy. In the transverse direction, the grains are not aligned in a preferential direction and their orientation is more random. This allows for greater movement of dislocations and easier sliding of the crystalline planes, which leads to higher ductility in this direction.

## 7. FUTURE LINES

The following steps and recommendations are provided for the continuation of the main project, taking into account its ongoing state.

- Conduct tests on the rest of the geometries designed in order to cover different points of stress triaxiality.
- EBSD to measure grain size, determine crystallographic orientations, identify grain boundaries, and quantify the percentage of each phase present in the material.
- EBSD to identify the mechanisms responsible for hydrogen-induced cracking and embrittlement.
- Calculate the hydrogen concentration profile. The hydrogen concentration is known but not the distribution. By mapping the distribution of hydrogen within the material, insights into how hydrogen diffuses and accumulates, particularly in regions susceptible to embrittlement are gained.
- Perform in-situ tests to measure hydrogen loss during test preparation.
- Repeat the TDS measurements to check the reliability of the results.
- Perform chemical composition analysis to check the nitrogen reduction in the 316L+ material after testing.

## 8. IMPACT ON THE SUSTAINABLE DEVELOPMENT GOALS (SDG)

The research conducted in this study has potential implications for several Sustainable Development Goals.

Firstly, SDG 9: Industry, Innovation, and Infrastructure, as the investigation focuses on improving the understanding of hydrogen embrittlement in stainless steels. This knowledge can contribute to the development of more durable and reliable materials for various industries, promoting innovation and supporting sustainable infrastructure.

Secondly, SDG 7: Affordable and Clean Energy, as hydrogen is a promising alternative energy carrier. By addressing the challenges associated with hydrogen embrittlement, such as the loss of ductility in materials, this research can help advance the use of hydrogen as a clean energy source and support the transition to a more sustainable energy system.

Furthermore, SDG 12: Responsible Consumption and Production, is relevant as the study explores the behaviour of materials under specific conditions, aiming to optimize their performance and lifespan. By understanding the effects of hydrogen embrittlement, industries can make informed decisions about material selection, reducing waste, and promoting responsible consumption and production practices.

Lastly, SDG 13: Climate Action, can also be related to this research. By improving the understanding of hydrogen embrittlement and contributing to the development of more resistant materials, it supports efforts to mitigate climate change by advancing sustainable and clean technologies.

## PERSONAL EVALUATION

My experience at Aalto University in Finland has definitely been a very enriching academic and personal experience. During my time at Aalto, I had the wonderful opportunity to immerse myself in the world of research and to get a first-hand experience of how researchers work.

One of the most valuable aspects of my stay was being able to interact directly with the researchers and be part of their working teams. I witnessed the dedication and enthusiasm with which they approached each project, sharing their knowledge and experience generously. Through collaborations and discussions, I was able to draw from their vast academic background and learn valuable lessons on research methodology, data analysis and how to develop innovative approaches. Lectures, seminars and meetings with experts in different fields allowed me to broaden my horizons and expand my knowledge in areas previously unknown to me.

Throughout my experience at Aalto University, I have faced a variety of challenges, from lack of resources to unexpected results and situations that required better planification. These setbacks, while frustrating at the time, have been valuable learnings. They have taught me the importance of resilience, adaptability and the ability to find solutions to difficulties. In addition, I have learned to appreciate even more the value of careful planning, efficient resource management and the importance of setting realistic objectives in any research project. In addition, I am deeply grateful for the constant support of my supervisors, who have always been willing to help.

Being surrounded by people from different cultural and academic backgrounds was exciting. Meeting new people and hearing their perspectives and experiences allowed me to see the world from different points of view. The diversity present at Aalto University provided me with a more complete global view and helped me develop valuable intercultural skills.

In addition, immersing myself in a challenging academic environment allowed me to expand my knowledge and skills. Although I was initially insecure because I did not know much about the specific subjects being covered, this situation motivated me to push myself even harder. I was pleasantly surprised to discover how much I could learn and grow when I faced new challenges and embraced the opportunity to acquire new knowledge.

Throughout my time at Aalto University, I have experienced moments of self-improvement, where I have realised my ability to adapt to unfamiliar situations and face obstacles with determination. This experience has taught me that stepping out of one's comfort zone is not only enriching, but also essential for personal and academic growth.

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## ANNEXES

The following section includes a number of documents that have been referenced throughout the report. These annexes provide additional and detailed information that supports the findings and conclusions presented in the main document. However, it is important to note that there are additional documents, such as test and post-processed data files, which are not presented in the annexes due to their nature and size. These files are available upon request for those interested in further technical details of the research.

## ANNEXE A

MBL Hydrogen embrittlement characterization for FCC steels for H<sub>2</sub> storage and transport.



24. Figure Project planification

## ANNEXE B

This document refers to the master's thesis "hydrogen embrittlement characterisation for FCC steels for h2 storage and transport" developed at Aalto University as accreditation for the title of the Master's Degree in Industrial Engineering by Mondragon Goi Eskola Politeknikoa (MGEP).

Aalto University (Mechanical engineering K2 department) is the research centre that requested the collaboration of the student from Mondragon Unibertsitatea to carry out the characterisation at microscopic and macroscopic level of a high-strength steel for the automotive industry.

During the execution of the project, the requirements present in the agreement established between the two companies, as well as those agreement established between both companies, as well as the agreement signed with the author of the project. the project.

### Objective and scope of the specification

Since the main objective of the project is to characterise 316L and 316L+ under HE phenomenon, the tests, and the analysis that the project must follow for its correct development are visible in the Section: EXPERIMENTAL METHODOLOGY. The experimental work necessary to achieve the objectives will be carried out in Aalto's workshop and laboratory, guided by the necessary experts.

Various activities have been developed related to the management of all the test and analysis that where needed, from the initial state of art around the topic, from the development of all those tests, through the post-analysis of the obtained data.

### Technical requirements

In order to carry out the project, a series of material and software elements must be available to carry out the necessary actions.

- Personal desktop computer:
  - CPU: Intel Core i7-8665U, 1.90 GHz 2.11 GHz.
  - RAM 16 GB. o 64-bit operating system.

- 256 GB solid-state hard drive.
- Windows 10 operating system
- Software licenses:
  - Matlab R2021.
  - SolidWorks 2021.
- Others:
  - Steel cutting machine.
  - Grinding and polishing machine.
  - Machine for the creation of microscope specimens.
  - Chemical products.
  - Zwick-Rowell universal tensile testing machine.
  - Software for testing and post-processing of results.
  - DIC System o Data acquisition system o Manual for performing DIC tests.
  - Equipment for analysis of tensile machine test results. o DELL external memory disk.

### **Facultative conditions**

The MGEP student has been assigned to do an external academic internship under the supervision of Aalto University. The aim is for the student to apply the knowledge acquired so far in their academic training. And so that they are able to acquire new skills and knowledge that will prepare them for the development of professional activity, improving their employability and entrepreneurship. As this is a 42 ECTS project, the competences to be acquired will be the three that can be seen below:

- M2H203: Capacity for the design and testing of machines.
- M2H216: Capacity for the management of Research, Development and Technological Innovation.
- M2H115: To have integrated project management skills.

For a correct development of the practices, the rights, and obligations of all the parties involved in this project are defined.

The author, Mikel Argandoña Sarriegi, will be the one developing the project with his own resources as well as the materials mentioned in the previous section. The student must comply with the agreement signed with MGEP and Aalto as a student and internship in the second entity. The following obligations must be fulfilled:

- Know and comply with the regulations regarding external internships at MGEP. • To carry out the FMT following the instructions of the tutors.
- Comply with the timetable established by the company. • Comply with the company's occupational risk plan.
- Notifying the tutor of trips outside the workplace using the form on Mudle. On the other hand, the tutors' obligations are as follows:
- Provide the student with the necessary resources for the development of the project. • To monitor both the internship and the project.
- To carry out the evaluation of the student's internship.

### **Legal conditions**

The workplace encompasses a number of laws and regulations that must be complied with in order to perform the work safely. The laws shown below summarise the safety and necessary PPE to be used in the workplace, manufacturing, assembly or testing:

- Law 31/1995, on the Prevention of Occupational Risks.
- Law 54/2003, on the reform of the regulatory framework for the prevention of occupational hazards.
- Royal Decree 485/1997 on minimum requirements for health and safety signs at work.

## ANNEXE C

This section of the document specifies the costs related to the development of the project, as well as the costs associated with the usage of the warehouse, machine rooms, laboratory, and office. The costs, therefore, are divided into the following groups:

- Labour
- Material
- Software licenses

The value of the costs associated with the purchase of the material necessary for the development of the project has been calculated according to the prices offers given by the material suppliers. In the same way, to be able to calculate the cost associated to the use of the machines and microscopes, an approximation of the economic value of the machines and the time of usage has been done. The value of the software license costs specified in this budget matches the value of the retail price offered to individual users.

### 6. Table Project budget

LABOUR HOURS	Cost / hour [€/h]	Time [h]	Total [€]
Traineeship student	0	1064	0
Project tutor	15	50	750
<b>Subtotal [€]</b>			<b>750</b>
USED MATERIAL	Unitary cost [€/u]	Quantity [u]	Total [€]
Consumables			
Office material	20	5	100
EPIs	5	3	15
Spaces & Machinery			
EDM	100	1	100
Laboratory machines	50	3	150
Laboratory products	30	10	300
Testing machines	150	2	300
DIC	100	1	100
Charging set up	100	1	100
Metal sheets	280	2	560
<b>Subtotal [€]</b>			<b>1.725</b>

SOFTWARES	Unitary cost [€/u]	Quantity [u]	Total [€]
Desk computer	195	1	195
SolidWorks license	86	1	86
Office			
MatLab license	35	1	35
<b><i>Subtotal [€]</i></b>			<b>316</b>
<b>TOTAL COST [€]</b>			<b>2.791</b>



## ANEXXE D

This annex shows the results of the chemical composition analysis carried out at Aalto using the *BELEC LAB 3000S* machine.

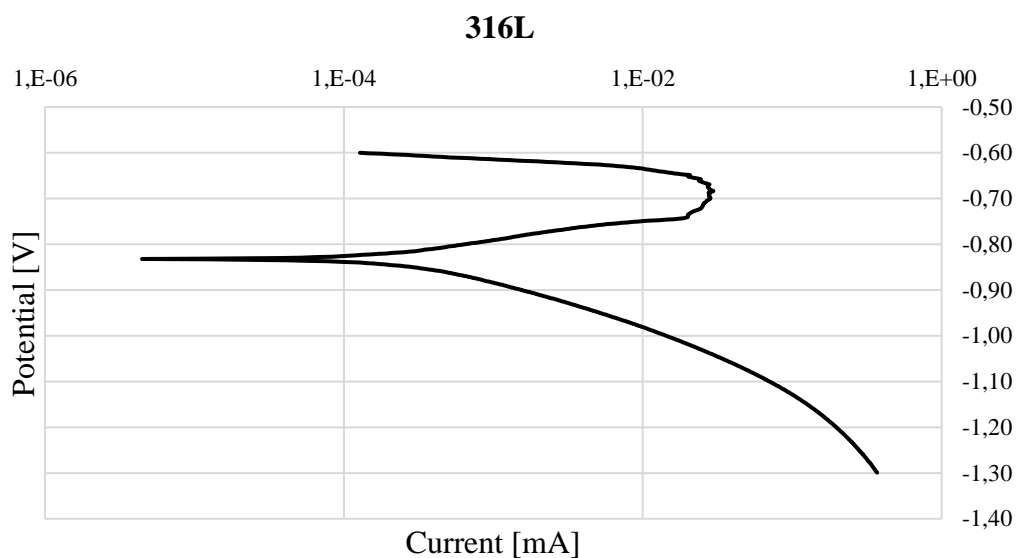
**7. Table** Chemical composition analysis at Aalto

Source	Material	C	Cr	Ni	Mo	N
Outokumpu	316L	0.02	17.2	10.1	2.1	-
Aalto	316L	0.012	17.18	9.43	1.994	-
Outokumpu	316L+	0.02	20.3	8.6	0.7	0.19
Aalto	316L+	0.01	20.15	8.35	0.567	-

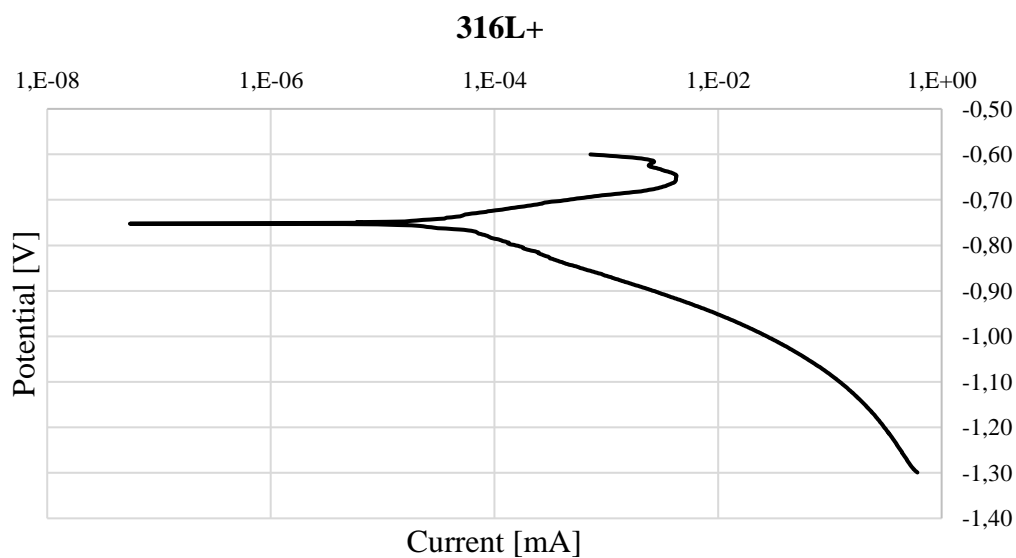
As can be seen in the table, the results are satisfactory. However, it is important to mention the inability of the machine to measure nitrogen, which is the reason for the absence of nitrogen in the 316L+ measurement at Aalto.

## ANEXXE E

This annex shows the potentiodynamic scans for 316L and 316L+.



**25. Figure** Potentiodynamic scan for 316L at 50° C



**26. Figure** Potentiodynamic scan for 316L+ at 50° C

