Mechanical and Aerospace Engineering

Project Report
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A Robust Method to Measure the Fatigue Corrosion of Shape Memory NiTi Wires

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Summary

In the field of NiTi alloys medical implants, it becomes a necessity to have a good understanding of fatigue and corrosion resistance.

In the present study, a reliable method was developed in order to assess experimentally the influence of a corrosive environment on the fatigue resistance of 0.5 mm diameter NiTi wires. For this purpose, a corrosion cell was designed with Solidworks CAD tool and later fabricated by using 3D printing technology. Besides, supports for the setup were fabricated through laser cutting of MDF plates.

Once the corrosion cell was fabricated, it was coupled with a Bending Rotation Fatigue setup and both parts were fixed over their supports. Experiments were carried out using the resulting test rig, to make sure it was a good tool to assess the simultaneous effect of fatigue and corrosion on material integrity.

The tests were performed at slow rotation speed and a certain strain amplitude until the failure of the sample. During their performance, a certain area of the wire was permanently in contact with the electrolyte (3.5% NaCl solution) placed inside the corrosion cell. This experimental procedure allowed getting some preliminary results and validating the functionality and correct design of the test rig.

The sample of the first test, performed with the BRF setup and the corrosion cell, broke prematurely in a wrong site. Observations of the sample at different surface points were done with the optical microscope. Existence of corrosion on the material surface was not detected due to the short exposure time of the wire to the corrosive environment. The second test worked properly but due to a lack of time, the sample was characterized before its failure. Optical images collected with the microscope showed a significant difference compared to the previous observations and what seemed to be the beginning of pitting corrosion was detected.

Based on the results, design modifications were proposed in order to minimize the test rig probability of failure in future investigation.

With this experimental procedure, the main objective was to assure that the designed setup was reliable for its use in further investigation.
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Nomenclature

Symbols:
\( R \) Radius of curvature (mm)
\( d \) Diameter of the wire (mm)
\( \varepsilon_a \) Strain amplitude (%)

Abbreviations:
NiTi Nickel Titanium
3D Three-dimensional
NaCl Sodium Chloride
SMA Shape Memory Alloy
SME Shape Memory Effect
SE Super Elasticity
BRF Bending Rotation Fatigue
MDF Medium-Density Fibreboard
Ms Martensite Start
Mf Martensite Finish
As Austenite Start
Af Austenite Finish
Ph Power of Hydrogen
A Austenite
S Single Variant Martensite
1.0 Introduction

Nickel Titanium shape memory alloys (NiTi SMAs), also known as Nitinol, have become very popular and promising materials for medical purposes, especially in the field of implant applications such as surgical stents and orthodontic implants.

The success of the NiTi as a biomaterial relies on its biocompatibility and two unique functional properties: Shape Memory Effect and Super-Elasticity (1).

Shape Memory Effect (SME) refers to material’s ability of recovering its original shape due to an increase in temperature after being subjected to strong deformations (2).

Super-Elasticity (SE), also referred in the bibliography as Pseudoleasticity, refers to the ability of sustain large strain, up to 8 %, without permanent deformation (1).

Due to the common use of NiTi as an implant, this may be permanently subjected to the conditions inside the body. That means that it can continuously be in contact with corrosion environments such as human blood or saliva and also suffer from static and cyclic mechanical loading (3).

For that reason, it becomes relevant to get a good understanding both of the structural fatigue behaviour and corrosion resistance of the material and the influence that each one produces on the other (3).

Up to now, some experimental methods have been used to address these two fields of study independently, but very few have attempted to connect both structural and chemical studies. On the field of fatigue behaviour, and regarding to literature, Bending Rotation Fatigue tests have been widely used with the purpose of obtaining information about fatigue life of NiTi under different testing conditions.

Miyazaky et al (4) successfully performed several tests with a Bending Rotation Fatigue test rig in order to determine the fatigue behaviour of NiTi under cyclic loading at different temperatures and strain ranges. Also, Wagner et al (5) determined the influence of wire diameter and rotational speed on fatigue rupture behaviour in bending rotation fatigue experiments. As a last example of similar studies, it is worth to mention both Eggeler et al (6) and Takahiro et al (7)
studies, who also tested NiTi wires under cyclic loading in order to assess the fatigue behaviour as well as the crack initiation and propagation applying different parameters.

On the other hand, corrosion resistance of NiTi alloys is mainly related with the formation of a titanium oxide surface film that acts as a protective passive film between the alloy and the corrosive environment (8), and avoids the release of toxic Ni ions into the body (1). This protective layer can locally disappear due to the effect of corrosive ions (chloride mainly) or large strain tensile deformation, and generate pits that can quickly propagate (9). This can be very critic in terms of biocompatibility for implants applications, due to the high toxicity of the Nickel (10). It becomes then very important to study the stability and behaviour of the surface film formed on the NiTi alloys in harsh environments (11,12).

Several studies in the field of corrosion have been developed in order to get information about the corrosion susceptibility of the NiTi alloys under different Ph, solution compositions and temperatures (13). Kanemura et al (14), using physiological saline solution containing hydrogen peroxide, and Figueira et al (1), using Hanks’ solution, are some examples of interesting investigations.

As mentioned before, only few studies have referred to the gap between these two fields in relation to this material. Neelakantan (3) designed a setup comprising a BRF test rig and an electrochemical cell with the objective of simultaneously testing corrosion and fatigue behaviour. The design of the electrochemical cell and the interplay between the different components were taken as the main basis for the design criteria in the present study, as it was the strongest reference of a similar study trying to reach similar goals.

In the case of Neelakantan’s study, the performance of the test and consequent results involved the use of three electrodes in order to realise polarization experiments and open circuit potential transient studies. In the present study, however, there is no need to do electrochemical measurements, and this directly affects the design – the integration of the electrodes to the cell is not considered as a design requirement.

The project aim is to design and fabricate a simple corrosion cell that can be integrated with a BRF test rig in order to get a reliable method to assess the combined effects of fatigue and corrosion on NiTi wires. To validate the correct operation of the final setup, the development and performance of an experimental test are also presented in the current study.
2.0 Literature Review

In this section, the main theoretical concepts that are needed to understand the project are widely developed. The objective is to provide the reader a solid basis of knowledge of all the topics that play an important role in the development of the study.

2.1 Functional Properties of NiTi Alloys

Shape memory effect and Super-elasticity are both very important functional properties of NiTi alloys. A good understanding of these properties and their effect on the material behaviour is required in the present study.

2.1.1 Shape memory effect (SME)

The SME of certain materials, being NiTi alloys one of them, refers to their ability of remembering their original shape, even after being subjected to strong deformations. This means that they can recover their original shape due to a phase transformation of their microstructure that occurs when an increase of the temperature is applied (15).

The basis for the memory effect is the ease of these types of materials to transform from and to martensitic state (16). Martensitic transformations take place when cooling the material from a higher temperature phase called Austenite or Parent phase.

The shape memory effect can be described in a macroscopic perspective using the cooling and heating curves represented in Figure 1. In this figure, four relevant temperatures to understand the process are indicated: Mf, Ms, As and Af. They refer to the temperatures at which the transformation to martensitic state stars and finishes, and the temperature at which the reversion to austenite stars and finishes. There is a hysteresis associated with martensitic transformations, which means that transformation temperatures differ upon heating and cooling (16).

When the material is deformed below Mf (martensitic finish) it remains in the deformed shape up to heating induced recovery of the original shape. The shape recovery begins at As (austenite start) and is completed at Af (austenite finish). To deform again the material and keep its deformed shape, it is necessary to cool this below Mf again and deform the martensitic specimen once again (16). A simple outline of this procedure can be also seen in Figure 1.
The shape memory effect is described with reference to a plot of electrical resistance vs. temperature from which the characteristic transformation temperatures $M_s$, $M_f$, $A_s$ and $A_f$ are determined.

The microscopic phase transformation that NiTi alloys undergo during Shape memory effect is represented in Figure 2. When austenite phase material is cooled to martensitic form, its structure can be subjected to several deformations by applying shear stress and moving the twin boundaries that are quite mobile (Figure 2 (b,c)). The material will return to its original austenitic structure and shape through heating (16).

Figure 2, extracted from (16). Austenite (a) is cooled to form twinned martensite (b) without undergoing a shape change, then is deformed by moving twin boundaries (c). Heating either state (b) or (c) will return the originally austenitic structure and shape.
Martensite is a lower symmetry phase than is the austenite. That means there are several ways in which martensite can form from austenite (different distributions of martensitic variants are possible), but there is only one possible route which will return the austenite structure. In other words, there is only one possible reverted structure.

2.1.2 Super-elasticity (SE) or Pseudoelasticity

Super-elasticity refers to the ability of certain metals and alloys to undergo large elastic deformation, during mechanical loading–unloading cycles performed at constant temperatures (1). Unlike shape memory effect, the driving force for the transformation is now mechanical, as opposed to thermal.

Materials that present this characteristic have the capacity of recovering the original shape after large and repetitive loading and unloading, by virtue of stress-induced martensitic transformations (SIMT) (15,17).

When stress is firstly applied to the NiTi alloy, only elastic distortions of the austenitic structure occur, but at some point of critical level of stress, austenite turns unstable and stress induced martensite transformation begin to form (2). When the material is mechanically deformed at temperature greater than Af, the stress induces the transformation from Austenite to Single variant martensite (A → S). Because of martensite is not stable for T > Af, when the load is removed and the stress has reached the new value σSA, the inverse transformation (S → A) takes place and the material recovers the original shape. This process can be seen in Figure 3 (15).

![Figure 3, extracted from (15).](image)

**Figure 3, extracted from (15).** Pseudoelastic effect (T ≥ Af). (1) Elastic deformation of austenite; (2) austenite to single-variant martensite transformation (upper plateau); (3) elastic deformation of single-variant martensite; (4) elastic strain recovery; (5) single-variant martensite to austenite transformation (lower plateau).
In Figure 4 can be seen that NiTi alloys may present recoverable strains as high as 8% (10).

![Figure 4](image_url) **Figure 4, extracted from (10).** The elastic strain range for superelastic Nitinol and 316 stainless steel.

A comparison of the super-elastic behaviour of NiTi alloys regarding other material such as body tissues or others materials used for similar medical purposes appears in Figure 5 (2).

![Figure 5](image_url) **Figure 5, extracted from (10).** The stress vs. strain relationship for superelastic Nitinol, stainless steel, bone and tendon tissue.

### 2.2 Nitinol’s Biocompatibility

Biocompatibility described in general terms is the ability that a certain material has to be accepted by the body (18). This refers to non-toxicity and corrosion resistance. NiTi alloy and its application as a biomedical material requires of a well knowledge of its biocompatibility.
Regarding to literature, it is well known that titanium is not a toxic material when in contact with the human body. However, Nickel, the other main component of Nitinol, is extremely toxic. Therefore, toxicity hazards remain a subject of concern. It has been reported that Nickel can produce allergic response from the body tissues and also degeneration of muscle tissue (1).

Body fluids, such as blood, constitute an aggressive environment for a metallic implant. When Nickel and Titanium are coupled together in order to get the NiTi alloy, it becomes a big concern to assure it can provide biocompatibility with the body’s environment. Luckily, it has been studied that Nitinol shows extremely good compatibility with the body due to the protective passive oxide layer that forms on the surface of the material composed mainly by titanium (TiO$_2$) (1,10,18).

This layer, known as passive film, acts as a protection for the metal beneath, preventing it from coming into contact with the aggressive environment of human body fluids (chloride ions and other corrosive substances) (8). The existence of this protective film also prevents the release of the toxic nickel ions into the human body.

### 2.3 Passivity and Localized Corrosion

Passivity refers to the metal state in which it become highly inert due to the formation of a thin oxide film on the surface that acts as a protection against general corrosion. If this protective passive film damages, it usually can re-form rapidly (19).

Localized corrosion is the accelerated attack of a passive metal in a corrosive environment at discrete sites where the otherwise protective passive film has broken down. It is characterized by an intense attack at confined areas on the surface (19).

#### 2.3.1 Crevice corrosion

Crevice corrosion is a localized form of corrosion. It is caused mainly to the existence of gaps, crevices or cavities between material surfaces (metal-metal, metal-nonmetal) where the electrolyte can become stagnant (20).

Factors that affect crevice corrosion may be the alloy composition, the geometry of crevice (depth of the crevice, width of the gap, number of crevices) and the characteristics of the passive film. The onset of crevice corrosion is strongly related with the nature of the passive film. If the passive
film is very stable, the crevice is blocked, but if the protective film breaks down, this may lead to the onset of the crevice corrosion. (8,20).

2.3.2 Pitting Corrosion

Pitting corrosion is a localized form of corrosion, the most insidious one. It consists in the corrosion of small areas in the metal surface, usually leading to the formation of cavities or pits. The presence of a passive film on the metal surface is a condition for this kind of corrosion to happen. The process of pitting destroys the protective film at certain sites resulting in the loss of passivity and initiation of pits on the metal surface (20).

Pitting corrosion can cause the failure of the material by penetration with little weight-loss of the entire structure. This fact makes it more difficult to detect. The pits normally penetrate from the top of a horizontal surface downward in a nearly vertical direction. The most likely environment for pitting is the marine environment as well as environments containing ions such as chloride ions.(19,20)

2.3.3 Hydrogen Embrittlement

Various metal alloys experience a significant reduction in ductility and tensile strength when atomic hydrogen (H) penetrates into the material. Ductile metals experiences brittle fracture when exposed to both tensile stress and corrosive atmosphere. For hydrogen embrittlement to occur, some source of hydrogen must be present (i.e., water vapor, sulfur and arsenic compounds, hydrogen peroxide, etc.) (14,19).

2.5 Fatigue

Fatigue is a form of failure that occurs in structures subjected to dynamic and changing stresses (i.e. aircraft, bridges or medical implants). Under fatigue conditions, failure of materials may occur at a lower stress level than under static loading circumstances. In fact, fatigue is the main cause of failure in metals and it occurs in a suddenly and insidious way (19).

The process of fatigue failure consists in the initiation, propagation of cracks and typically final failure which happens very rapidly once the advancing crack has reached a critical size. Fracture surface is perpendicular to the direction of the applied tensile stress. The fatigue strength is the stress level at which failure will occur for some specified number of cycles.
**Tensile fatigue test:** A specimen is deformed, usually to fracture, with a gradually increasing tensile load that is applied uniaxial along the long axis of a specimen.

**Bending Fatigue test:** In many applications, wires are subjected to various cyclic stresses. The flexure plus the rotation provides a tensile-compression loading of the specimen surface.

In the Bending Rotation Fatigue test, the strain amplitude of the wire in the point of maximum strain (Figure 6) is related with geometrical parameters $d$ (diameter of the wire) and $R$ (bending radius) according to the following equation (6):

$$
\varepsilon_a = \frac{d}{2R}
$$

![Figure 6, extracted from (6). Schematic illustration of the geometry of the wire in the BRF test.](image)

**2.4 Corrosion Fatigue**

Corrosion fatigue is a process in which the failure of a material occurs under the simultaneous action of corrosion and cyclic loading. Material's fracture happens in a premature way, before reaching the normal stress levels that would lead to the material failure in a normal cyclic fatigue test, without the presence of a corrosive environment (8,20).

A corrosive environment promotes crack initiation and shortens the fatigue life. Endurance limit or fatigue limit is defined as the stress below which no failure will occur for a given number of cycles.
3.0 Design of the Corrosion Cell

The design tool used to create the prototype was SolidWorks Student Version 2015 (CAD program). All technical drawings of the different parts of the cell were also made with SolidWorks.

3.1 Design Basis

According to Neelakantan et al (3), specific design criteria must be accomplished in order to succeed in subsequent tests:

- The electrolyte should be confined into a specific volume without any or minimum leakage out of its container. This way, the area of the wire in contact with the electrolyte can be always controlled and known by those who perform the tests. It is important then, to isolate the electrolyte as much as possible (leak-free).
- The cell should not interfere with free rotation of the wire.
- Inert material to avoid chemical interaction has to be used.
- Avoid stagnation of the electrolyte in order to prevent the appearance of crevice or pitting corrosion.

3.2 First Prototype

The first prototype of the cell had some differences regarding the model (3) that had been taken as the design basis, although it fulfilled all the design criteria. Efforts were made to simplify the original model so it was possible to avoid the use of many screws and fabricate fewer parts to get a more compact cell. In the performed tests, there was no need of electrodes, but the design permits the incorporation of them in future investigation, just keeping in mind that some little modifications in the design should be done.

The design of the prototype that is shown in Figure 7 supports different amplitude strains of the wire. In other words, it works properly with a wide variety of bending radius of the wire.
Figure 7. Isometric view of the first prototype

Description of cell’s first prototype

The main dimensions of the prototype cell were 150 mm length x 20 mm width x 70 mm height. A description of the different components of the cell is presented below. In addition to this, all drawings with their specified dimensions can be found in Appendix B.

Cell parts:

1) Front Plate, with a rectangular shape and 10 screw holes all over its edge is shown in Figure 8.

Figure 8. Front plate

2) Base plate can be seen in Figure 9: with double ‘U’ shape and 10 threaded screw holes all over the edge. A rectangular cavity in the middle, which purpose is to contain the electrolyte. Also two thin slots in each side of the electrolyte container to allow free rotation of the NiTi wire inside the cell.
3) Base plate details are shown in Figure 10: reservoir for the electrolyte and 2mm thickness slots. The bended wire rotates inside the electrolyte container and the two slots. A wide range of bending radius is possible due to the depth and length of the slots.

4) Plastic tapes: two fluoro rubber or silicone thin tapes that act as a division for both the electrolyte container and the slots, shown below in Figure 11. Their use avoids the leakage of electrolyte from the container to the slots. The wire pierces the soft plastic tape easily, at the desired height depending on the required bending radius of the wire.
5) Chemically inert fluoro rubber, placed between the base plate and the front plate, can be seen in Figure 12. It basically acts as a sealant and prevents leakage (3).

3.3 Modifications

At this point, the prototype was validated and ready to be fabricated.

However, due to a problem in times and terms of manufacturing, it became impossible to have the cell ready on-time to implement the tests. The process of getting all the necessary materials and machining the different parts of the cell was too long for the available term. It became necessary then to reinvent the design from zero and try to get the most simplified model that accomplished the main design criteria (avoid leakage of electrolyte, confined volume for the electrolyte, free rotation of the wire) so it could be 3D printed. This technique allowed to get the model in a fast and accurately way.
The design was modified to one single piece formed by a rectangular container for the electrolyte with one small hole in each one of its lateral sides. The electrolyte volume should never exceed the height of the holes to avoid the leakage of it out of its container. At the bottom of the electrolyte container there were two flanges with two holes that allowed fixing the cell to a bigger base plate using screws in order to provide enough stability.

The rendering of the isometric view of the cell with can be seen on Figure 13. Engineering drawings for this design can be found in Appendix B. The main dimensions for the corrosion cell were 60 mm length x 20 mm width x 35 mm height and a 2 mm thickness for all the cell walls for the electrolyte container. For the cell base they were 80 mm length x 20 mm width x 2 mm height.

![Figure 13. Cell’s final design Isometric view](image)

This final model allowed to perform the corrosion fatigue test and interfaced properly with the BRF setup. However, the simplification of the model brought some restrictions to the tests. The fact that the electrolyte container only had one hole in each side for the wire to pass through them meant that there was only a possible diameter for the bending wire (the chosen diameter was exactly 85 mm). That meant it was not possible to vary the diameter of the wire in the setup so the surface area of the sample inside the electrolyte container was always the same.

One solution that was raised to perform tests with different areas in contact with the electrolyte was to vary the volume of the electrolyte so it covered more or less of the wire surface area inside the container.
3.4 Cell and Setup supports Design and Construction

The last step on the design process was to design a solid support for the BRF setup in order to keep it raised the necessary distance from the base so it fitted perfectly with the cell and allowed the free rotation of the wire.

Different options were discussed and finally the one showed in Figure 15 was chosen as the most suitable. This option consisted of two rectangular blocks that should be placed underneath the BRF setup in order to hold it properly at the desired height. In this case, it was 73 mm.

In accordance to the technicians, the material chosen to build this support was MDF, as it turned out to be the easiest and fastest way to obtain the blocks. It was machined by laser cut using MDF sheets of 12 mm each (maximum plate thickness available at Queen’s University Workshop). A total of 6 equal cuts were necessary to obtain each block. Once all the pieces were laser cut, they were put one above the other and glued together to avoid instability of the whole set.

The final appearance of the support blocks can be seen in Figure 14.

![Figure 14. Rectangular block supports](image)

A support for the corrosion cell was also fabricated using MDF plates.
4.0 Experimental Details

4.1 Materials

NiTi Wires
The material used in the tests was commercial Ti-55.91 wt% Ni wire of 0.5 mm in diameter (9). The samples were cleaned up with propanol before their use in the laboratory in order to remove impurities from the surface. The cleaning process was carried out by laboratory technicians. All the samples were obtained from a long roll of wire and the longitude of each sample was approximately 225 mm (134 mm for the test region and extra 60 mm and 30 mm on each side for the correct fixation with the setup).

It is also important to comment that the NiTi wires used in all the tests were all in their austenitic phase because the test temperature (room temperature) was above Af temperature (9).

Electrolyte
The electrolyte used in the corrosion tests was a saline solution of 3.5% NaCl. This high saline concentration, similar to the sea water composition, was used because of its strong corrosive effect. The idea was to reproduce human body fluid corrosive environment, highly rich in chloride ions.

Fabrication materials
The material used to fabricate the corrosion cell was VERO GREY, an opaque rigid plastic usually used in 3D printing. For the supports, MDF plates were used.

4.2 Methods

BRF test rig
A Bending Rotation Fatigue test rig was used to determine the mechanical resistance of the NiTi wire. When the bending wire rotates, its surface is subjected to tensile strain in the first half cycle and compressive strain in the second half cycle (17). The BRF test rig can be seen in Figures 15 and 16.
In the BRF test rig, a NiTi wire is bent into a semi-circular shape with a specific radius of curvature and forced to rotate by a motor attached to one end and running at a constant rotational speed. The speed value can be modified by manipulating the variable frequency drive tool (arrow 1) (3). A proximity sensor mounted over the setup plate, next to the wire, acted as a counter for the number of cycles to fatigue failure of the wire (arrow 2). The number of cycles appeared in the screen attached on the basis of the setup (arrow 3). Also, the diameter of the wire could be modified making larger the distance between the wire fasteners that is, increasing the bending radius (arrows 4 and 5). The maximum distance between fasteners was around 85 mm (3).

**Corrosion cell**

Corrosion effect on NiTi was determined using an electrochemical cell designed and built in the present study. The entire design process, materials, and the final outcome are described in detail.

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*Figure 15. Bending Rotation Fatigue test*

*Figure 16. Components of the BRF setup detail (speed controller and screen for cycle counting)*
in section 3.0 Design of the Cell. A picture of the final cell that was used in the experimental studies appears in Figure 17.

![Figure 17. Final 3D printed cell](image)

**Final test rig**

The BRF test rig and the cell were fixed on their respective supports in order to avoid instability of the equipment. Design process and materials of the supports are also described in section 3.4 Cell and Setup supports Design and Construction.

The corrosion cell was coupled with the BRF test rig as showed in Figure 18 in a way that when rotating, a certain area of the wire surface was permanently in contact with the electrolyte contained inside the corrosion cell.

![Figure 18. Final BRF test rig setup](image)
**Setup parameter: Wire radius of curvature**

Due to simplifications applied in the cell design, the final prototype only was compatible with one specific radius of curvature (R) of the wire (only one perforation machined in each side of the cell). It was assessed that the best radius of curvature for the purpose of the experiments was the maximum that was possible to get with the BRF test rig (R = 42.5 mm) in order to minimize the surface strain (this corresponds to a surface strain of 0.6 %). The corrosion process requires plenty of time so the longer the fatigue life of the sample, the best for obtaining some corrosion influence results.

It is also worth to comment that it is possible to vary the contact area of the wire surface with the electrolyte decreasing the level of the electrolyte inside the cell.

### 4.3 Experimental procedure

**Mechanical and Corrosion Measurements**

Experimental procedures that have been carried out are explained below:

1) In the first test only the bending rotation fatigue setup was used to determine the fatigue life of NiTi samples and to validate the proper operation at slow rotation (155 rpm). This was a preliminary test in order to detect problems that may appear while the test rig was running and it served as a base for necessary future design modifications.

2) Second test was performed with the corrosion cell coupled to the BRF test rig with the aim to study corrosion fatigue and detect possible corrosion effect on the broken sample surface. It was also performed at slow rotation, 155 rpm, and the volume of NaCl solution used was 10 ml. Slow rotation was used in order to avoid a fast failure of the wire and have enough time to observe the influence of the corrosion in the wire surface. This experimental procedure was repeated once more keeping the same experimental parameters except for the volume of electrolyte, which was increased up to 15 ml. The surface area of the sample submerged under the NaCl solution was bigger than it was in the previous test.
Microstructural Analysis

The cross-sectional microstructure in the fracture point of the samples and the surface of the wire in the test area (under the influence or not of corrosion) were characterized using an optical microscope (shown in Figure 19) and NIS-Elements F 3.0 software.

Figure 19. Nikon SMZ800 microscope
5.0 Results and Discussion

Results obtained from the tests, possible design improvements and procedural enhancements are presented in this section. All the performed tests had as the main objective to validate the operation of the method and find errors that require design and procedure improvements.

5.1 Preliminary test with the BRF test rig

The preliminary test was accomplished using only the BRF test rig. The objective of this preliminary test was to assure the good working order of the mechanical setup and detect procedure problems that could appear while the test was running. Also, it could give information about the number of cycles until failure under the chosen conditions (explained in section 4.3: Experimental procedure). This could serve as a basis to compare the fatigue life with and without corrosion influence and see if there were any differences.

5.1.1 Results

The test was running for 24 hours but the setup was turned off before the failure of the wire. This decision was taken because the rotating sample shifted a little respect its initial position. The results that could be extracted from the test were then consequently unreliable. With the movement of the sample, the bending radius changed, so the section subjected to the maximum surface strain also changed. That meant the number of cycles until failure could not be taken as a valid fact.

5.1.2 Causes of failure and Improvements

The movement of the NiTi wire was caused by a bad fastening of itself. Usually, when performing a BRF test, the wire is subjected with a plastic tape that prevents its movement. The plastic tape used to avoid wire’s escape in this test was too thin and it was not tightly subjected to the wire, so it slipped from its starting position.

It was determined that for further studies it was necessary to use a thicker plastic tape and was crucial to assure it was well attached to the sample at the correct point.
5.2. First test: BRF test rig coupled with corrosion cell

These tests were carried out with the interplay of the corrosion cell and the BRF test rig.

5.2.1 Results

The sample in this experiment broke down in an unexpected point. As shown in Figure 20, the failure happened in one side of the sample instead of breaking in the middle section of the bending wire, the section that suffers the maximum surface strain and that is expected to fail first. The wire made 239.857 cycles before it failed, which corresponds to approximately 26 hours rotating at 155 rpm, but this number is not a representative fact of its fatigue life because the sample have been subjected to additional stresses. As mentioned before, the point of fracture can be seen in Figure 20, just at the bottom of the rectangular metallic plate that act as a guiding path for the wire to get the bending shape.

![Figure 20. ... Fracture of the sample circled in red](image)

The green paper attached to the top of the cell with plastic tape was a preventive cover in order to avoid the entrance of atmospheric impurities inside the cell.

It was also observed that around the cell holes through which the wire passed, there was a concentration of solid salt as showed in Figure 21. One possible explanation for this fact is that the wire acted as a capillary while the test was running, displacing the saline solution upwards. The solution got out of the cell and when evaporating, only the salt remained on the surface.
5.2.2 Possible causes of failure

The most likely cause for the failure of the wire just at the bottom of the rectangular metallic guiding plate was a wrong positioning of the cell with a consequent imperfect bending radius that caused little deformations.

The wire passed through the corrosion cell holes and also through the metal plate’s conduits at each side of the setup. That meant that the cell should be perfectly positioned in the middle, between the metal plates, to get a perfect radius. This is hard to get, although it was as much accurate as possible. But any little displacement made the hole of the cell to press onto the wire, diverting more the wire path.

The sample then, pressed also onto the metal guiding plate’s conduit and combining the friction and the rotation, it was to expect that it would break through this point.

Microscope images

As it can be seen in Figure 22, an image from a microscope observation (x40 magnifications) shows the rupture zone. This image corroborates the idea that the breakdown was caused by friction of the wire against the path guidance metal plate.
In addition, observations of the area in contact with the electrolyte were made, and no influence of corrosion was observed, any pit was observed. Compared with images taken from free electrolyte areas, there was no significance difference between them (see Figures 23 and 24 below). Little striations on the surface of the samples can be seen in both figures caused by the manufacturing process of the NiTi wire. There is no relation between these striations and the corrosion effect.

This fact reinforce the idea that corrosion takes long time to show any influence

### 5.2.3 Design improvements and Actions to be taken

Regarding the problem with precision when positioning the cell to get the specific bending radius that is required for the test, a design modification was developed in order to avoid the need of such precision without being affected by external tensions that could influence in the modification of the break down point.
The modification can be seen in the SolidWorks rendering that appears in Figure 25. Instead of little holes in each side, a thin groove of the same width as the holes diameter has been machined in the electrolyte container sides. This groove allows free rotation of the wire. A cover for the cell was also designed to replace the provisional paper lid. This cover should be also fabricated by using 3D printing.

![Figure 25. Improved cell design](image)

5.3. Second test: BRF test rig coupled with corrosion cell

5.3.1 Results and Observations

This test was performed using the same procedure as in the previous one. The positioning of the cell in the middle of the bending radius was made as accurate as possible trying to avoid a premature fracture of the wire due to unwanted stresses.

Some regular grease was smeared on the bottom of the metal plate, where the sample had broken in the previous test. The objective was trying to avoid as maximum as possible the friction between the NiTi wire and the thin metallic conduit of the wire.

During the course of the experiment, it was necessary to refill twice the electrolyte container with NaCl solution due to the decrease of the electrolyte volume. This decreasing was caused by the capillarity effect of the sample, (its effect already explained in the previous section 5.2.1).

This time, the test presented a great performance and after 746.351 cycles, the equivalent to 80 hours, the sample was still rotating. However, due to a lack of time, it was not possible to wait until failure of the sample. It is unpredictable to know when a specimen will fail under fatigue conditions.
The decision of turning off the setup was taken in order to realize some observations with the microscope and check whether the sample showed signs of corrosion influence.

A significant difference was detected comparing the solution exposed surface and the non-exposed surface. In Figure 26 early pits seemed to begin. What seem small round holes can be seen in these images. On the contrary, in Figure 27 the non-exposed surface doesn’t show any peculiarity on it. However, it would be very risky to affirm that these small rounded shapes correspond to pitting corrosion. The maximum magnification that the microscope can get is not enough to clearly distinguish if they are a directly consequence of corrosion or not.

![Figure 26. Early pits on the wire’s surface (exposure x50)](image1)

![Figure 27. No-exposure surface (x50)](image2)
6.0 Conclusions

- A corrosion cell was successfully designed and fabricated using 3D printing technology. The designed allowed its coupling with a Bending Rotation test rig. To get a good interrelationship of the components in the final setup, supports for the BRF test rig and the corrosion cell were also fabricated.

- The designed corrosion cell that was fabricated and used to perform the corrosion fatigue test was a simplification of the first prototype, but it equally demonstrated to meet all the required design criteria for reaching its purpose. The final result turned to be a useful tool to assess the corrosion fatigue of NiTi wires. The capability and functionality of combined operation of BRF test rig and corrosion cell were demonstrated by performing experimental tests.

- Some procedural issues were detected during the performance of the first test. Consequently, some design modifications were proposed in order to improve these limitations. The initial holes of the cell were replaced by thin slots that made easier the positioning of the wire through the cell and allowed, at the same time, the free rotation of the wire. A cover to avoid the entrance of atmospheric impurities was also added.

- The second test using the final setup was successful (some grease was used as lubricant to avoid the friction between the sample and the setup components). The sample was subjected to cyclic loading for about 80 hours. Due to a lack of time, it was no possible to wait for the sample to break. However the test was a proven fact that and it can be used as a reliable method to measure the fatigue corrosion in future investigations.

- Optical microscope observations showed a clear difference between the surface of the sample exposed to the electrolyte and the non-exposed surface. In the exposure area small round holes seemed to begin its formation. Due to the fact that corrosion requires a long time to produce any influence over the material surface, it is risky to assure that the pits observed are related with localized corrosion.
References


# Appendix A Project Management

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**Table A1** Project Work Plan
# Appendix B Engineering Drawings

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### Review of actions from previous meeting

*FIRST MEETING.*

### Discussion, decisions, assignments

- Brief introduction to the key points of the project.
- Discuss the main objectives.
- Delivery of one paper that discusses a similar test to which we need to perform.
- Paper Title: Design and fabrication of a bending rotation fatigue test rig for in situ electrochemical analysis during fatigue testing of NiTi shape memory alloy wires.
- To focus on attention on the electrochemical cell (design and construction).

### Agreed actions and completion dates

- To read and understand the paper.
- To understand the purpose of the test, the design criteria, and the results obtained.
- To get a deeper idea of the different stages of the test design.

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Review of actions from previous meeting:
Discuss some doubts about the paper.

Discussion, decisions, assignments:
Decide what are the main issues to be adresssed theoretical.
Creating a list of items to study in depth:
- CORROSION / Type of Connexion
- FATIGUE / Traction and Bending Fatigue
- FATIGUE Corrosion
- SHAPE MEMORY ALLOY

To find and read literature about these topics.

Agreed actions and completion dates:
Elaborate on these concepts and write down any question that may arise.
Get a good understanding of the theoretical basis.

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### Review of actions from previous meeting

Review all the information about the main topics of research and talk about some doubts about them related to these.

### Discussion, decisions, assignments

Discuss about the theoretical concepts. Resolve all the doubts that have arisen.
Explanation by the supervisor of more complex and specific concepts.
Decide which issues need further research.

### Agreed actions and completion dates

Finish deepening with the research of theoretical issues and ensure a good understanding of the subject.
Read two more papers related with this topic.

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### Review of actions from previous meeting:

- Solve past theoretical questions.

### Discussion, decisions, assignments:

- Start thinking about the design of the cell.
- Elaborate a table with different Design Criteria and the possible consequence of each issue and how to tackle them.
- Think of a design overview.

### Agreed actions and completion dates:

- Design a first prototype of the electrochemical cell using CAD tools (SolidWorks).

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**Review of actions from previous meeting**

- First prototype of the cell, designed using SolidWorks.
- Design based on the general cell description of the paper “Design and fabrication of a bending notched fatigue test rig for in situ electrochemical analysis during fatigue testing of NiTi shape memory alloy wires” (2013, PeerJ).

**Discussion, decisions, assignments**

- Discuss and decide which modifications have to be done regarding the first design.
- Send the modifications to the supervisor via email, and once I get an OK from him, start doing the technical drawings of the CAD model.

**Agreed actions and completion dates**

- Contact the technicians and in order to decide if the cell design is feasible to build and talk about the best materials to be used.
- Decide dates of construction.

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### Review of actions from previous meeting

Discuss about the problems that have been considered in terms of time and materials to fabricate the SolidWorks prototype. Talk about possible solutions proposed by technicians.

### Discussion, decisions, assignments

- Validate the modifications that have been introduced to the design.
- Validate if the simplifications meet the design criteria.
- Validate the whole design for future construction.

### Agreed actions and completion dates

To have the connection cell constructed (1 day using 3D printing) and review the final result.

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**Review of actions from previous meeting**

The connexion cell is already built. Also the support for the cell has been fabricated.

**Discussion, decisions, assignments**

Check that the connexion cell is well fabricated and discuss which tests should be performed in the lab, in order to evaluate the connexion influence on the fatigue life of the specimen.

**Agreed actions and completion dates**

- To design a feasible support for the BRF setup that allows to maintain the setup at a certain height over the connexion cell, so the tests can be performed properly.

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#### Review of actions from previous meeting

- Review that we have all the setup parts ready: the BRF, the connection cell and the supports.
- Check that everything for the tests is already prepared and ready to use: Wire chow, Saline Solution and Lab equipment.
- Validate the support for the BRF (that has been fabricated).

#### Discussion, decisions, assignments

- Assemble all the parts of the whole setup. Find a method of fixing and securing the BRF setup to avoid it from falling down. Finally, wide plastic tape is used on this purpose.
- Make some variations on the rotational speed with using our sample, and decide which a speed is the best for the performance of the tests.
- Check the chosen rpm with a stopwatch/timer.

#### Agreed actions and completion dates

- Perform a test in the lab only with the BRF setup (without the connection cell) to assess the fatigue life of the sample at few rotational speeds.

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### Review of actions from previous meeting

The results of the preliminary BRF test were not reliable because the sample has moved slightly from its original position. The bending radius has changed.

### Discussion, decisions, assignments

- Validate the assembly of the whole test jig (conjunction cell + BRF setup + supports) in the Lab.
- Decide notation speed (155 upm) for the performance of the test.
- Begin the test, and leave the set-up running all weekend.

### Agreed actions and completion dates

- Check if the wire has broken during the weekend. Decide next steps (more tests, microscope observations).
- Check the feasibility of the experimental procedure and the reliability of the results obtained.

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**Review of actions from previous meeting**

Check if the wire is broken.

**Discussion, decisions, assignments**

The wire has broken, but in the wrong place. Design modifications are required to make the setup work properly.

Decide which is the initial point in the cell design and try to understand why the wire has broken in the wrong place.

Discuss possible modifications.

**Agreed actions and completion dates**

Discuss and validate the changes proposed for the design.

<table>
<thead>
<tr>
<th>Date and time of next meeting</th>
<th>15/05/2015</th>
<th>Location of next meeting</th>
<th>Ashby Building, 6th Floor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supervisor signature</td>
<td></td>
<td>Student signature</td>
<td></td>
</tr>
</tbody>
</table>
## Individual Project Meeting Record

<table>
<thead>
<tr>
<th>Project Title</th>
<th>A Robust Method to Measure the Fatigue Cycles of Shape Memory NiTi Wire</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supervisor</td>
<td>Dr. Chi Wai Chan</td>
</tr>
<tr>
<td>Student</td>
<td>JANA LLORART PRIETO</td>
</tr>
<tr>
<td>Date and time</td>
<td>15/05/2015</td>
</tr>
<tr>
<td>Location</td>
<td>Ashby Building, 6th Floor</td>
</tr>
</tbody>
</table>

### Review of actions from previous meeting

Evaluate the performance of the first test (second with BRF+ connection card)

### Discussion, decisions, assignments

- Discuss the test details about the results obtained from the tests.
- Talk about next conclusions.
- Resolve past doubts related to the project.

### Agreed actions and completion dates

Complete and submit the project.

<table>
<thead>
<tr>
<th>Date and time of next meeting</th>
<th>LAST MEETING</th>
<th>Location of next meeting</th>
<th>LAST MEETING</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supervisor signature</td>
<td></td>
<td>Student signature</td>
<td></td>
</tr>
</tbody>
</table>
# Project Submission Checklist

A copy of the checklist can be found on the Student SharePoint site and must be included at the very end of your report.

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Tick if Met</th>
</tr>
</thead>
<tbody>
<tr>
<td>Does the report meet the formatting stipulated in the Module handbook and template provided?</td>
<td>X</td>
</tr>
<tr>
<td>Line Spacing (1.5)</td>
<td>X</td>
</tr>
<tr>
<td>Font (Calibri 11Pt)</td>
<td>X</td>
</tr>
<tr>
<td>Margins: Top &amp; Bottom (25mm), Left (30mm), Right (25mm)</td>
<td>X</td>
</tr>
<tr>
<td>Paragraphs are fully justified</td>
<td>X</td>
</tr>
<tr>
<td>Does the main body of the report meet the strict 30 Page Limit (40 Page limit for MEE7012)? (excluding Title Page, Table of Contents, Turn-It-In Summary, References and Submission Checklist)</td>
<td>X</td>
</tr>
<tr>
<td>Do the Appendices meet the strict 10 Page Limit (15 Page Limit for MEE7012)?</td>
<td>X</td>
</tr>
<tr>
<td>Are all tables and figures numbered correctly, captioned and referenced if required?</td>
<td>X</td>
</tr>
<tr>
<td>Has the report been checked using Turn-It-In?</td>
<td>X</td>
</tr>
<tr>
<td>Is the Turn-it-in summary report included in the report?</td>
<td>X</td>
</tr>
<tr>
<td>Pages are numbered.</td>
<td>X</td>
</tr>
</tbody>
</table>

**Statement of originality**

I hereby declare that this project is my own work and that it has not been submitted for another degree, either at Queen’s University Belfast or elsewhere. Where other sources of information have been used, they have been acknowledged.

Signature: 

Date: 16/11/15